

GEOCHEMISTRY PREPARATION PROCEDURE

SAFETY: See associated COSSH form. Always wear PPE (lab coat, safety glasses and nitrile gloves). Inform safety officer of any spills.

HCl – mild burns, respiratory irritant

HNO³ – severe burns, highly flammable, respiratory irritant

Indium – irritant

First aid – run with water for ~20 minutes, seek medical help for eye incidents.

CHEMICALS REQUIRED:

MilliQ water

7% Aristar/Analar HCl

10% Aristar/Analar Nitric Acid (HNO³)

Teepol detergent

Neutracon

Indium ICP-MS Aristar Quality

EQUIPMENT REQUIRED:

50ml centrifuge tubes with lids

50ml test tube racks

Centrifuge

Drying oven (105°C)

Hot plate

Ultrasonic water bath

Fume cupboard

15ml centrifuge tubes with lids

15ml graduated glass cylinder

Scales

Porcelain crucibles and pen

Teflon pots

Large glass beaker

Hot plate

Metal blocks to hold Teflon pots

15ml test tube racks

NOTES: It is of fundamental importance that in order to obtain geochemical data from your sediment samples that is accurate and able to stand up to rigorous scrutiny by your peers that during every step in the sample preparation process cleanliness of all equipment used is paramount and the chance of any cross contamination from any other source if minimized.

PROCEDURE:

1. Before sub-sampling all tubes, porcelain crucibles etc. must be scrubbed in Teepol (added to MilliQ), then rinsed in MilliQ and then left over-night in Neutracon (in MilliQ). On removing from the Neutracon the tubes etc. must be acid rinsed 7% HCL (Aristar quality diluted in MilliQ) at least three times and then rinsed in MilliQ (five times); note: do not place tubes in dryer, as they do not need to be dry (reducing chance of contamination in dryer). All metal spatulas also need to be acid rinsed and MilliQ rinsed, do not place on aluminium foil or paper towels (both will contaminate samples).
2. Sub-sample 1cm³ of sediment into 15ml graduated glass cylinder, (note: weigh sample before subsample is removed and after). 1cm³ of sediment is obtained by adding 5ml of MilliQ to tube and then adding sediment until the level of MilliQ in the

tube due to displacement increases to 6ml. (Remember re-use of this tube for another sample requires rinsing in MilliQ-Acid-MilliQ thoroughly and after one batch of samples is prepared cleaned as outlined above (Teepol-Neutracon-Acid-MilliQ). The sample can then be transferred to a Centrifuge tube (with a screw cap) labelled as required e.g. sample 1, placed in a rack.

3. Centrifuge the samples for 5 minutes at 3500rpm, having topped all tubes up with MilliQ to the same level.
4. Drain off the MilliQ retaining the sediment in the tube, then transfer the sediment to dry porcelain crucible that has been weighed and labelled (ensure pen ink is capable of heating to 105°C for a 12 hours). Weigh wet sample in crucible. Dry the samples at 105°C until sediments are dry (6-12 hours). Weigh dried sample in porcelain crucible.
5. Before the sample can be transferred to the Teflon pot the pots must be extensively cleaned as Teflon is porous and will have absorbed the chemical elements from its last use, simply rinsing in 7% HCL or performing the Teepol-Neutracon-Acid rinse-MilliQ process is not adequate. Place the Teflon pots in 5-7% HCL in MilliQ and heat to 100°C for 24 hours ensuring that the pots remain submerged. Remove pots from acid and rinse in MilliQ 5 times.
6. Transfer 0.25 g of dried sample from the porcelain crucible to the Teflon pot (labelled).
7. Add 5ml of Aristar Nitric Acid (HNO₃) diluted to 10% in MilliQ to the sample in the Teflon pot. From this stage onwards prepare the required number of blank regents (Earth Sciences suggest 4-5 blanks per 30 samples) and perform the same processes as for the samples.
8. Ultrasonicate the samples in the Teflon pots for 20 minutes, note: Due to buoyancy of the Teflon pots ensure less than 1cm of water is in the sonic bath or the Teflon pot will tip over and your sample will be lost.
9. Place the machined metal blocks on to the hot plate (these balance the Teflon pots and reduce the uneven heating across the hotplate surface). Pre-heat the hotplate to 105°C, once the temperature has been reached then place the samples in the Teflon pots on the hotplate, ensure that the extraction fan is functioning and that no other chemical processes or samples are in the fume cupboard at the same time. Samples remain on the hotplate until all fluid has evaporated this will takes several hours.
10. Transfer sample residue to a 15 ml graduated (labeled) tube with screw top and add 1% nitric acid (HNO₃) diluted in MilliQ; continue to add acid until there is 6 ml of solution in the tube (debatable what pipettes to use to reduce contamination, disposable? If so a new pipette must be used for each sample) Note: Nitric Acid needs to be of a minimum Aristar quality, use distilled Analar nitric acid if it can be obtained this contains less contaminants. The tube you use should not be used again for geochem!

11. Centrifuge the samples at 3000rpm for 15-20 minutes to separate the acid from the sediment (most of the sediment will remain at the bottom of the tube, but a film of light material may remain at the surface).
12. Place a new 15 ml tube (labelled), the tube should be clear plastic not opaque (so ICPMS operator can see if any sediment exists in acid) and have a screw top, place on balance; tare to 0.00mg.
13. Using a autopipette/dispenser and a disposable tip, transfer 0.1ml of supernatant into the 15 ml graduated tube from the middle of the sediment tube avoiding take up of the sediment in the bottom of the tube or the floating material.
14. To the 0.1 ml supernatant add 1% nitric acid solution containing 50 ppb (parts per billion) of Indium ICP-MS Aristar Quality until the sample weight is 10 grams. (again the question arises as to the pipette to use, whatever you chose a new pipette is required per sample). Place lid on to tube and place in tube rack.