ICP-MS method extracts from silicate soft-sediment cores

Materials

- 13 ml plastic test tubes (1 per sample, with tops).
- 50 ml plastic test tubes (1 per each standard and blank made).
- High-purity HNO_3.
- Kimwipes.
- Chlorox bleach.
- DI water.
- Teflon spatula.
- Various pipettes.

Dissolution and preliminary work

1. Collect your samples and dry them. For best homogeneity, they should be broken up to dust.
2. On a semi-micro balance, weigh one ICP-MS tube (cap off) and *record* its weight.
3. Tare the test tube and add ~100 mg of sample using a Teflon spatula. *Record* the actual sample weight to five decimals. Replace the cap and label tube with sample name and depth. Do this for all samples.
4. Add 2 ml of Chlorox bleach and replace the cap. Agitate to suspend the sediment, but try not to get any sediment onto the cap . Allow to soak overnight.
5. Dilute with ultra-pure DI water. Centrifuge the tube. Decant the Chlorox solution. Repeat this step 3 more times to remove the Chlorox.
6. Add 0.5 ml of high-purity HNO_3 and 9.5 ml of DI water. Let react until fizzing stops.
7. Mix vigorously.
8. Weight the test tube with the solution in it. A) Total weight - tube weight = solution weight. B) Solution weight / sample weight = **dilution factor #1**.
9. Let sit for at least a day to allow particles to settle.

Internal standard solution

1. Fill a clean 100 ml volumetric flask half full with DI water.
2. Add 7 ml of high-purity HNO3.
3. Add to the flask 0.1 ml each of the 1000 μg/g stock solutions of Rh, In, Re, and Bi.
4. Fill the flask to volume and transfer to a clean storage bottle. Internal standard concentrations are 1 ppm in 5% HNO3.

Example Trace Element Standard

Element	Single element solutions, µg/g	ml of single element solutions added to 100 ml flask	Concentrated standard elements, ppm	ml of stock standard used, diluted to 50 ml to make dilute standard	Dilute standard elements, ppb
Al	1000	3	30	0.5	300
V	1000	0.1	1		10
Cr	1000	0.1	1		10
Mn	1000	2	20		200
Co	1000	0.05	0.5		5
Ni	1000	0.1	1		10
Zn	1000	0.2	2		20
Cu	1000	0.1	1		10
Sr	1000	0.5	5		50
Mo	1000	0.05	0.5		5
Cd	1000	0.05	0.5		5
Ba	1000	0.5	5		50
Pb	1000	0.2	2		20
U	1000	0.05	0.5		5
Fe	1000	None	None	0.1*	2000

The concentrated standard solution (green) is made in a 100 ml volumetric flask; the dilute standard solution is made in the 50 ml autosampler tube.

*Added directly from the 1000 ppm stock bottle to the 50 ml autosampler test tube.

1. Fill a clean 100 ml volumetric flask half full with DI water.
2. Add 7 ml of high-purity HNO_3.
3. Add to the flask the volumes of 1000 g/ml (ppm) stock solutions indicated in the green region of the table above.
4. Fill the flask to volume and transfer it to a clean storage bottle. This is the concentrated standard solution.

Diluting solution

Half fill a clean one-liter liter bottle with DI water. Add 14 ml of high purity HNO_3, and 10 ml of the internals standard solution. This is the diluting solution. Note that it does not have to be mixed in a volumetric flask. The important thing is that all samples are the same, not that the concentrations are exactly known. Shake well.

Calibrate the pipettes

Take the 0.1 ml pipette and set the 10 ml pipette to 10 ml. Use *one sample* and determine the weight of 0.1 ml of this sample. Use the 10 ml pipette to determine the weight of 10 ml of the *diluting solution*. They will not be exactly 0.1 and 10 g, respectively, but that's OK.

(10 ml diluting solution weight + 0.1 ml sample solution weight) / (0.1 ml sample solution weight) = **dilution factor #2**.

Use these pipettes exclusively in doing the dilutions below.

Dilute the samples

1. Take 0.1 ml from the particle-free top of the sample solution with the 0.1 ml pipette and put it in a new, labeled, 13 ml plastic test tube.
2. Add 10 ml of the diluting solution, and shake.

Dilute the standards

1. Transfer 0.5 ml of the concentrated standard solution to a 50 ml autosampler tube.
2. Add 50 ml of the diluting solution.
3. Add 0.1 ml of the 1000 μg/g (ppm) stock Fe solution, and shake.

(Diluting solution weight + concentrated standard solution weight + Fe solution weight) / concentrated standard solution weight = **dilution factor #3**.

Blank

Transfer 50 ml of the diluting solution to a 50 ml autosampler tube.

Sample calculations

1. For each sample, calculate: Total dilution factor = dilution factor #1 * dilution factor #2.
2. These dilution factors should be put into the Elan software sample file prior to analysis.

Standard concentrations

1. The nominal standard concentrations in the table above are not quite correct.
2. A = the actual standard concentration labeled on the stock standard bottle (e.g., 1002 μg/ml; ~ppm).
3. B = volume of stock standard diluted to 100 ml in the concentrated standard solution.
4. Actual concentration for all elements except Fe = ((A * B)/100) / dilution factor #3.
5. Actual Fe concentration = A / Dilution factor #3.
6. These calculated standard concentrations should be entered into the Elan software method file prior to analysis.