

ICP-MS method for soft sediment extracts

Materials

- 13 ml plastic test tubes (ICP-MS tube; 2 per sample).
- 50 ml plastic test tubes (ICP-MS tube; 1 per blank and standard).
- High-purity HNO₃.
- Kimwipes.
- Chlorox bleach.
- DI water.
- Teflon spatula.
- Various pipettes.

Dissolution and preliminary work

1. Collect your samples and dry them.
2. On a semi-micro balance, weigh one 13 ml test tube (cap on) 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.
4. Carefully add 2 ml of Chlorox bleach and replace the cap. Agitate to suspend sediment but try not to get material onto the cap. Allow to soak overnight.
5. Dilute with DI water. Centrifuge the test tube. Decant the Chlorox solution. Repeat this step 3 more times.
6. Add 0.5 ml of high-purity HNO₃ and 9.5 ml of DI water. Let react until fizzing stops.
7. Shake to mix.
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. Allow particles to settle at least one day.

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 (ppm) 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.

Example trace element standard, concentrated solution

Element	Stock bottle, ppm	ml of stock solutions diluted to 100 ml	Solution 1, ppm	ml of solution 1, diluted to 50 ml to make solution 2	Solution 2, ppb
Al	1000	1	10		100
Mn	1000	1	10		100
Fe	1000	5	50		500
Co	1000	0.05	0.5		5
Zn	1000	0.2	2	0.5	20
Cu	1000	0.1	1		10
Sr	1000	1	10		100
Ba	1000	1	10		100
Pb	1000	0.1	1		10

Solution 1 made in a 100 ml volumetric flask, solution 2 made in the 50 ml autosampler tube.

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 the volumes of 1000 µg/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 standard solution 1.

Diluting solution

Half-fill a clean one-liter bottle with DI water. Add 14 ml of high purity HNO₃, and 10 ml of the internal standard solution, and mix. This is the diluting solution. You don't need to use a volumetric flask. The important thing is that all samples are the same, not that the concentrations are exactly known.

Calibrate the pipettes

1. Take the 0.5 ml pipette and the 10 ml pipette set to 9 ml. Use one sample and determine the weight of 1 ml of this sample. Use the 10 ml pipette to determine the weight of 9.5 ml of the diluting solution. They will not be exactly 0.5 and 9.5 g, respectively; do not worry.
2. Diluting solution weight + sample solution weight / sample solution weight = **dilution factor #2**.
3. Use these pipettes exclusively in doing the dilutions below.

Dilute the samples

1. Take 0.5 ml from the particle-free top of a sample solution with the 0.5 ml pipette and put it in a new, labeled 13 ml test tube.
2. Add 9.5 ml of the diluting solution, and shake.

Dilute the standards

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

Diluting solution weight + standard solution 1 weight + Fe solution weight / standard solution 1 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. The total dilution factors should be put into the Elan software, samples window, prior to analysis.

Standard concentrations

The nominal standard concentrations in the table above are not quite correct.

1. A = the actual standard concentration labeled on the stock standard bottle (e.g., 1002 µg/ml; ppm).
2. B = volume of stock standard diluted to 100 ml in standard solution 1.
3. Actual concentration for all elements except Fe = $A * B/100 / \text{dilution factor \#3}$.
4. Actual Fe concentration = $A / \text{Dilution factor \#3}$.
5. These calculated standard concentrations should be entered into the Elan software, Method window, calibration tab, prior to analysis.