RiO5 METHOD (35)

Ken Buesseler
Steve Pike

Woods Hole Oceanographic Institution

kbuesseler@whoi.edu

239Pu, 240Pu — Mass Spectrometry — Sediments

Sediment leaching of Pu and analysis by ICP-MS

Table of Contents

1 SCOPE

2 EQUIPMENT CHEMICAL REAGENTS

2.1 Equipment

- 500 ml beaker (acid clean)
- Watch glasses (for beaker)
- Heat plate
- Stir rods
- GFF Filter
- AGI-x8 column with 50-100 mesh Bio-Rad resin
- Glass wood
- 5 ml pipette with acid clean tips
- AGMP-1- 100-200 mesh Bio-Rad Resin column
- 1cc disposable syringe
- Acrodisc 0.2 um filter
- PE snap vials (acid clean)

2.2 Tracers

- ^{242}Pu Tracer solution

2.3 Chemical reagents

- NaNO2 (solid)

2.4 Solutions

- 8N HNO_3
- Concentrated HCl
- 1N NH4
- Aqua regia
- SEASTAR HCL/H2O2 solution (10 ml conc. HCL with one drop 30% H2O2)
- 2N HCl

3 PROCEDURE

Sediment Preparation:

1. 1-10 grams of dry sediment.

2. Place aliquot of dried sediment into an acid cleaned 500 ml beaker and add 100 ml of 8N HNO3.

3. Add 1 ml of 242Pu tracer to solution. Volume may vary depending on tracer concentration. Tracer is weighed in separate acid cleaned Teflon vial. Vial is rinsed x5 with dilute HNO3 to ensure quantitative transfer.

4. Cover beaker with watch glass and heat gently to near boiling (80o C) overnight stirring occasionally.

5. Filter sediment slurry through acid cleaned GFF. Rinse beaker x3 with 8N HNO3 and pass through filter.

6. Collect filtrate in clean 500 ml beaker and slowly evaporate.

7. Return sediment to original beaker and re-leach in hot HNO3 for 6-8 hours. Repeat Step #5 and combine filtrates.

8. Evaporate to 50 ml. Add 50 ml Q-H2O to form 8N HNO3.

9. Add 1 gram NaNO2 and heat at 40o C for 10-15 minutes. Sample is cooled and ready for first ion exchange column.

Column #1: (30 cm goose neck column w/ 250 ml reservoir)

1. Build a 20 ml column using AG1-x8, 50-100 mesh Bio-Rad resin. Place glass wool plugs at top and bottom of column.

2. Place aliquot of dried sediment into an acid cleaned 500 ml beaker and add 100 ml of 8N HNO_3.

Preconditioning (use TM acids):

3. 80 ml conc. HNO_3. Adjust flow rate to 2 ml/min.

4. 100 ml 8N HNO_3 with 1 g $NaNO_2$.

Sample Loading:

Waste:

5. Add sample to reservoir. Adjust flow rate to 1 ml/min.

6. Rinse beaker x3 with 8N HNO_3 and add to column.

7. Further wash column with 250 ml 8N HNO_3. (Fe and U removed)

8. Pass 150 ml of conc. HCl through column. Adjust flow rate to 2-4 ml/min. (Th removed)

Collect in clean 250 ml beaker:

9. Pass 150 ml of conc. HCl with 7.5 ml 1N NH_4I through column. Adjust flow rate to 1 ml/min.
10. Evaporate to dryness.
11. Add 1ml aqua regia and evaporate to dryness. Repeat x1.
12. Add 2 ml conc. HNO_3 evaporate to dryness. Repeat x2

Column #2: (2 cm column)

1. Build a 2 ml column using AG1-x8, 100-200 mesh Bio-Rad resin. Place glass wool plugs at top and bottom of column.
2. Bring up dried eluted Pu sample from column #1 in 5 ml of 8N $HNO_3$with 0.02 g $NaNO_2$. Heat slightly.

Preconditioning (use TM grade acids):

3. 6 ml conc. HNO_3. Adjust flow rate to 1 ml/min.
4. 7 ml 8N HNO_3 with 0.07 g $NaNO_2$.

Sample Loading:

Waste:

5. Load sample using acid cleaned pipette tips. (flow rate: 1 ml/min)
6. Rinse beaker x2 with 5 ml 8N HNO_3 and add to column.
7. Further wash column with 20 ml 8N HNO_3.
8. Pass 25 ml of conc. HCl through column.

Collect in clean 50 ml beaker:

9. Pass 30 ml of conc. HCl with 1.5 ml 1N NH_4I through column. (flow rate: 1 ml/min)
10. Evaporate to dryness.
11. Add 1ml aqua regia and evaporate to dryness. Repeat x1.
12. Add 1 ml conc. HCl evaporate to dryness. Repeat x1.

Column #3: (In clean room)

1. Build a 1 ml column using AGMP-1, 100-200 mesh Bio-Rad resin. Place glass wool plugs at top and bottom of column.
2. Bring up dried eluted Pu sample from column #2 in 1 ml of SEASTAR HCl/H_2O_2 solution. (10 ml conc. HCl with 1 drop 30% H_2O_2). Heat at 40^o C for 60 min.

Preconditioning (use high purity acids):

3. 3 ml of HCl/H_2O_2 solution.

Sample Loading:

Waste:

4. Load sample using acid cleaned pipette tips.
5. Rinse beaker x1 with 2 ml HCl/H_2O_2 solution and add to column.
6. Further wash column with 2 ml 8N HNO_3.

Collect in clean 5 ml Teflon vial:

7. Pass 3 ml of conc. HBr.
8. Evaporate to 1 drop under heat lamp.
9. Add 8 drops conc. HNO_3 and evaporate to dryness to remove trace HBr.
10. Bring up in 1ml of 10% HNO_3 /1% HF for ICPMS analysis.

Filtration

Prepare 1cc disposable syringe and Acrodisc 0.2 μm filter by taking up 2N HCl with syringe and washing through the filter. Repeat and let HCl stand in syringe and filter over night. Prior to use, rinse x3 with Q-H_2O.

1. Draw sample into syringe, replace filter and filter into acid cleaned PE snap vials. Repeat until all of sample has been filtered.
2. Store vials for ICP-MS analysis.

4 REFERENCES

Modified from Buesseler 1986