

RiO5 METHOD (2)

K. Buesseler
Woods Hole Oceanographic Institution
kbuesseler@whoi.edu

Contributor:
Steven Pike
Spike@whoi.edu

¹³⁷Cesium — AMP Chemistry — 20 liter Seawater Samples

AMP-PAN

¹³⁷Cesium AMP Chemistry for 20 liter Seawater Samples

Disclaimer

It is the responsibility of the analyst to follow established safety and health practices. Although each laboratory identified as the source has tested the methods, each user should perform an individual validation procedure.

Table of Contents

1	SCOPE	1
2	EQUIPMENT AND CHEMICAL REAGENTS	1
2.1	EQUIPMENT AND CONSUMABLES	1
2.2	TRACERS	1
2.3	CHEMICAL REAGENTS	1
3	PROCEDURE	2
4	REFERENCES	3
5	IMAGES	ERROR! BOOKMARK NOT DEFINED.

1 SCOPE

This method requires approximately 20 L seawater sample (filter or unfiltered) where water acidified and followed by the addition of AMP-PAN resin. Samples are then concentrated and AMP is dissolved for gamma counting.

2 EQUIPMENT and CHEMICAL REAGENTS

2.1 Equipment and consumables

- Pipette (1 mL)
- Plastic graduated cylinder
- 2 plastic squeeze bottles
- pH meter
- Polybottles-500ml
- Acid bottle top dispenser
- Cubitainers with caps and spigots
- Waterproof marker
- Acrylic snap vials (large counting vials)
- 3 ml ICP snap vials (cryo vials)
- Tygon tubing, 3/8" I.D.
- AMP-PAN resin (Czech Technical University, Prague)
- Safety gloves and glasses
- 50 ml plastic beaker

2.2 Tracers

- Stable Cesium carrier (0.5-0.7 mg/ml), J.T. Baker

2.3 Chemical reagents

- Concentrated hydrochloric Acid

3 PROCEDURE

FIELD SAMPLING

1. **Cs-1** Fill a labeled twenty-liter cubitainer with sample water. Either tare cubitainer or gross weight at sea or measure volume. For volume, it is important to fill each cubitainer equally and save one full cubitainer to weigh ashore to establish a good sample weight. Pour (or siphon) off exactly 1 liter to both know the sample volume and to make room for reagents.
2. **Cs-2** Spike with 1ml of Cs carrier (25 mg/ml) using calibrated pipette, cap cubitainer and agitate to mix. Equilibrate for at least 1 hour.
3. **Cs-3** Adjust pH of sample to 1.5-2 with concentrated HCl (approx.. 30 ml for 19L sample). Use NaOH if necessary to raise the pH back if it goes too low.
4. **Cs-4** Take 1 ml aliquot of sample w/ carrier and store in labeled 3 ml ICP vial for recovery analysis.
5. **Cs-5** Add 4 g of AMP (ammonium molybdophosphate) by dumping APM into a 50 ml beaker and mixing with ~20 ml of D.I. water to form a smooth slurry. Add this to the cubitainer, cap and agitate well.
6. **Cs-6** Let the sample settle overnight or as long as possible
7. **Cs-7** Pour off as much of the supernatant as possible by using smaller clear tubing to siphon off water. This can be done in steps if the sample needs to resettle. Pour off precipitate (<500ml) into labeled polybottle. Rinse the cubitainer with DI water. *since a known amount of carrier has been added do not worry about getting every bit of precipitate out of the cubi. Let it settle in the polybottle then pour off any more supernatant if possible.
8. **Cs-8** Wrap the cap of the polybottle with black tape and/or parafilm for shipping. Do not ship back the cubitainers
9. **Cs-9** do one blank sample using your DI water and reagents.

LAB PROCESSING

10. Record all information found on the sample bottles. Label all vialing vials and mark calibration level before filling.
11. Decant remaining supernate carefully using a vacuum aspirator (found in sink hood). Remove as much liquid as possible. Small losses can be accounted for by subsequent recovery corrections.
12. Add 1.5-2 ml of concentrated NaOH (10M) and 4 ml of Q-H₂O. Shake vigorously to dissolve the AMP.
13. If necessary add NaOH drop by drop until AMP is completely dissolved.

14. Pour off into a counting vial using a plastic funnel (about 5-8 ml)
15. Rinse sample bottle x2 with 3 ml of Q-H₂O. Pour off into counting vial after each rinse.
BE CAREFUL – DO NOT over fill the vial beyond calibration mark. Sample can be brought up to mark using Q-H₂O.
16. Cap samples and count on gamma detector.

4 REFERENCES

Includes AMP dissolution for gamma counting, range of the pH is adapted from Aoyama et al 2000. The total amount of Cs carrier the same as Wong et al 1994, Radiochemical procedures for analysis of Pu, Am, Cs, and Sr in water, soil, sediments and biota samples. Lawrence Livermore National Lab.

Aoyama, M., Hirose, K., Miyao, T., Igarashi, Y., 2000. Low level ¹³⁷Cs measurements in deep seawater samples. Applied Radiation and Isotopes, 53, 159-162.