

RiO5 METHOD (6)

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^{234}Th — method — seawater samples

^{234}Th in 4 Liter method in seawater

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.

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1 SCOPE

A method is described for the analysis of ^{234}Th in 4 L seawater samples. ^{234}Th is pre-concentrated with MnO precipitates and filtered in 25mm QMA filters. Dried filters are measured at sea or onshore using beta counting system. Chemical recoveries are determined measuring ^{230}Th by ICP-MS, and are usually >90%.

2 EQUIPMENT and CHEMICAL REAGENTS

2.1 Equipment and consumables

- 4 liter bottles
- Filter holders
- Aspirator pump: Cole-Parmer
- Vacuum pump
- pH meter
- 25 mm QMA filters
- Mylar
- Foil
- Beta mounts

2.2 Tracers

- ^{230}Th tracer: 50 dpm/g
- Eckert & Ziegler Analytics: Atlanta, Georgia USA

2.3 Chemical reagents

- Nitric Acid: Fisher Reagent grade
- Ammonium Hydroxide: Fisher Reagent grade
- KMnO_4 : Fisher solid
- MnCl_2 : Fisher solid
- H_2O_2 : Fisher 30% solution

2.4 Solutions

- KMnO_4 : 0.45 g KMnO_4 /60 mL H_2O
- Mn Cl_2 : 1.2 g MnCl_2 /60 mL H_2O
- Rinse Solution= 1.0N HNO_3 / H_2O_2 (990 mls 1N HNO_3 + 10mls 30% H_2O_2)

3 PROCEDURE

1. Rinse container 3X with seawater, dump rinses.
2. Fill labeled 4 liter bottle with seawater to graduated mark on neck.
3. Adjust ph to ~1.5 with conc HNO₃ (around 7ml) and shake well.
4. Add 1ml of 230-Th (10 dpm/g) yield monitor and shake well. Let sit >8 hours.
Bring ph to 8 +/- 0.15 using NH₄OH.
5. Check ph with OAKTON electronic pH meter just to dial in amount.
6. Add 100ul of diluted 1:20 (0.45 g/60 mL H₂O for KMnO₄) solution cap and mix.
7. Add 100ul of diluted 1:20 (1.2 g/60 mL H₂O for MnCl₂) solution cap and mix. Note time.
8. Let sample sit capped for at least 8 hours to form MnO₂ ppt.
9. Filter sample using 25mm QMA filter, in labeled gray pvc filter rig. Open valve as you invert bottle into rack, note start and stop time of filtering.
10. When sample is done close valve, remove pvc filter head and rinse using bench top filtration.
11. Rinse filter funnel 3X with ph 9 water.
12. Carefully remove filter and place in a labeled petri dish.
13. Dry at ~ 60 C for 2 hours.
14. Mount with 1 mylar and 2 foil layers, trim, label and beta count.
15. Rinse container, filtration rig with RS (rinse solution) and 3X with distilled H₂O.

Check Th recovery by measuring ²³⁰Th with ICP-MS. Average recovery of 230-Th from this 4-liter precipitation procedure is 92%. See method #45 *Determination of 234Th in Seawater samples* for specific instructions on this procedure.

4 REFERENCES

Pike, S.M., Buesseler, K.O., Andrews, J., and Savoye, N., 2005, *Quantification of ²³⁴Th recovery in small volume sea water samples by inductively coupled plasma-mass spectrometry*, Journal of Radioanalytical and Nuclear Chemistry, Vol. 263, No. 2, pp. 355-360.

5 FLOW CHART

Simplified Flow Diagram

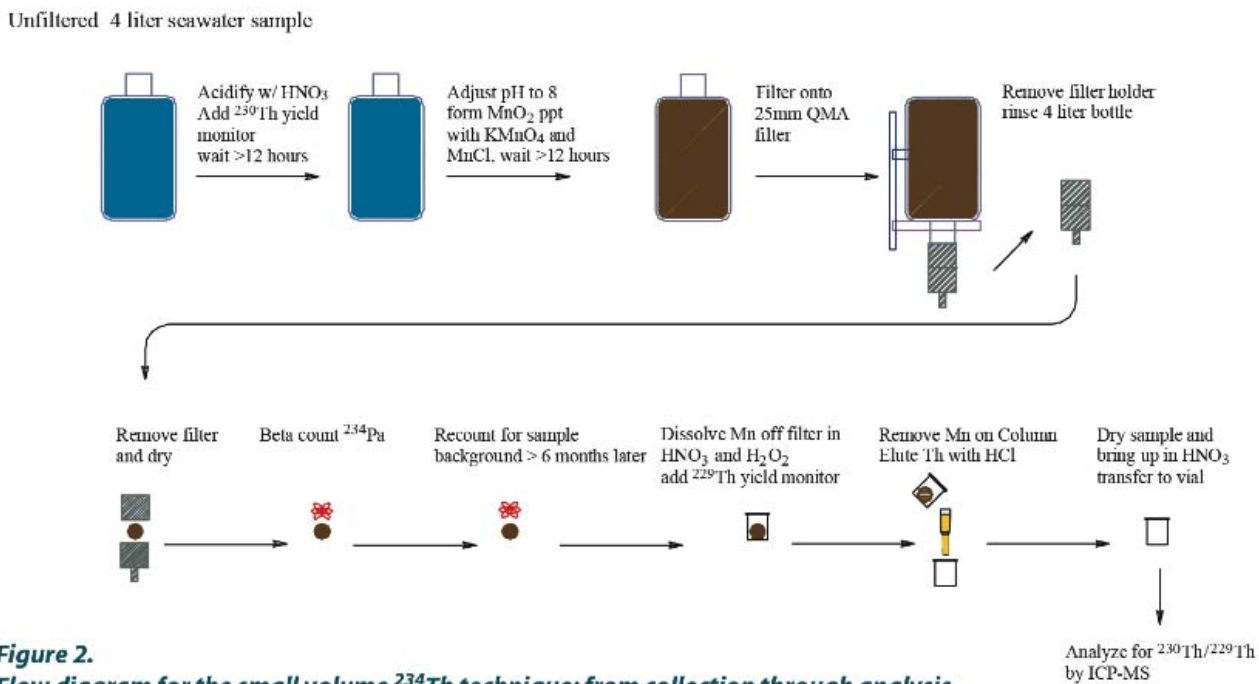


Figure 2.
Flow diagram for the small volume ²³⁴Th technique: from collection through analysis.

Figure 1. Flow diagram for the small volume ²³⁴Th technique: from collection through analysis.

6 IMAGES



Picture 1. 4 liter filter head assembly.



Picture 2. Filter head assembly with vacuum manifold attached.



Picture 3. At sea filtration set up for small volume ^{234}Th sampling.