

RiO5 METHOD (22)

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^{228}Th , ^{230}Th , ^{232}Th —anion-exchange column—Seawater sample

Coastal seawater sample preparation for thorium isotopes

(^{228}Th , ^{230}Th , ^{232}Th)

Disclaimer

It is the responsibility of each analyst to follow established practices when handling and examining the samples referenced in this Rio5 Cookbook. Although the methods may have been tested by each laboratory identified as the source, each user must perform a validation procedure to ensure the validity of their results. Woods Hole Oceanographic Institution, its officers, directors and employees are not responsible for any of the data or the results that may be achieved from using the information in the Rio5 Cookbook and disclaim all liability for the same.

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1 SCOPE

This method specifies the minimum requirements and laboratory methods for measuring ^{228}Th , ^{230}Th and ^{232}Th in river water and coastal seawater with high thorium contents.

River- and sea-water samples are collected in acid-cleaned containers and pre-treated on board, by filtrating, acidifying and co-precipitating. Post processing is conducted in the land laboratory by purifying Th isotopes via anion-exchange column. Thorium isotopes are finally measured by alpha spectrometry. Influence of Ra on Th measurement is corrected (Zhang et al., 2005).

2 EQUIPMENT CHEMICAL REAGENTS

2.1 Equipment

- Plastic container
- Plastic columns (BioRad)
- Glass beaker
- Analytical balance with an accuracy of ± 0.1 mg
- Hot plate
- Alpha-spectrometry

2.2 Tracers

- ^{229}Th (11 dpm g^{-1})

2.3 Chemical reagents

- Minimum info required: name and purity (if relevant)
- Hydrochloric acid (HCl)
- Ammonium hydroxide (NH_4OH)

2.4 Solutions

- 30 mg Fe mL^{-1} , use $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ to prepare
- TTA-benzene solution, use TTA and benzene to prepare

3 PROCEDURE

3.1 Separation of dissolved and particulate Th

1. Filtrating 30 L seawater through polycarbonate membrane with 0.4 or 0.8 μm pore size, particulate matter, after washing with Milli-Q water, retained on the membrane is operationally defined as particulate sample for measuring thorium isotopes, the filtration is defined as the dissolved sample for measuring Th.

2. Add the dissolved sample with concentrated HCl until the pH value is less than 2.0.
3. Kept particulate sample at 18°C.

3.2 Pre-concentration of dissolved Th

1. Add pre-weighed ^{229}Th as spike.
2. Add Fe^{3+} carrier (i.e. FeCl_3), usually 5 mg Fe L⁻¹.
3. Mix the sample and equilibrate for 24 hours with regular mixing.
4. Add ammonium hydroxide to a pH of 8.5-9.0 to form $\text{Fe}(\text{OH})_3$, and the sample is left for no less than 10 hours.
5. Separate the precipitate from solution by siphoning and successive centrifugation.
6. Dissolve the precipitate with concentrated HCl and evaporate to nearly dryness.

3.3 Digestion of particulate Th

1. Add a known amount of ^{229}Th spike to particulate sample.
2. Digest particulate sample with mixed HF/ HNO_3 / HClO_4 to clean solution, and evaporate to nearly dryness.
3. Add 5 ml concentrated HCl and evaporate to nearly dryness.

3.4 Separation and purification of Th

1. Dissolve the pre-concentrated dissolved and digested particulate samples with 9 M HCl.
2. Solution flows through a pre-treated anion-exchange column (AG1-X8) using 9 M HCl, collecting effluent.
3. Use 80 ml 9 M HCl to wash off Th isotopes retained in the column, and combine all the effluent.
4. Evaporate the effluent to dryness, and dissolve it using 8 M HNO_3 .
5. Solution flow through a pre-treated anion-exchange column (AG1-X8) using 8 M HNO_3 , collecting effluent.
6. Use 100 ml 9 M HCl to wash off Th isotopes retained in the column, and collect the effluent.
7. Evaporate the effluent to nearly dryness, and extract Th isotopes into TTA-benzene solution.
8. Drop the Th-contained TTA-benzene phase onto a heated stainless steel disc and evaporate to dryness.

3.5 Measurement of Th isotopes

Th isotopes (i.e. ^{228}Th , ^{229}Th , ^{230}Th and ^{232}Th) in the stainless steel disc are counted using alpha-spectrometry.

4 REFERENCES

Zhang, L., Chen, M., Yang, W., Xing, N., Li, Y., Qiu, Y., Huang, Y.(2005). Size-fractionated thorium isotopes (^{228}Th , ^{230}Th , ^{232}Th) in surface waters in the Jiulong River estuary, China. *Journal of Environmental Radioactivity*, 78(2): 199-216.

5 FLOW CHART

