

RiO5 METHOD (33)

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Procedure for the separation/purification of Th-234 on EQPAC Filter samples (53 or 0.7 micron)

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.

PROCEDURE FOR THE SEPARATION/PURIFICATION
OF TH-234 ON EQPAC 53-MICRON SAMPLES
or 0.7-MICRON SAMPLES

PRE WEIGH THE DRIED GF/F FILTER!

Cut approximately 10% from the filter for C-H-N analysis.

RE WEIGH THE REMAINING ~ 90% OF THE GF/F FILTER.
+ 10%

Date Started : _____

1. Place the (90%) GF/F filter in a 250 ml beaker and add approximately 50 ml 8N HNO₃. Heat to solubilize the particles.
2. Add 6 dpm (by weight) Th-230. Continue heating.
3. If the solution is cloudy, cool and add a few drops of 30% H₂O₂. Heat for 5 hours. ✓ ok
4. Filter through a glass fiber filter, ~~saving the filter for possible future analysis.~~
5. Condition a gooseneck column containing 10 ml AG1-X8 50 - 100 mesh resin with 50 ml 8N HNO₃ (1 - 2 ml/min). 100-200?
6. Pass the solution through the column (1 - 2 ml/min), collecting in a waste beaker. This fraction contains Ra and should be discarded.
7. Rinse the filter flask 3x with 5 ml 8N HNO₃, then rinse the column with 75 ml 8N HNO₃, collecting in a waste beaker.
8. Change from the waste beaker to a 150 ml beaker and elute Th with 50 ml 8N HCl (open stopcock).
9. Evaporate the Th fraction to dryness and resolubilize in 40 ml 8N HCl.
10. Condition a column containing 10 ml of AG1-X8 100 - 200 mesh resin with 50 ml 8N HCl (1-2 ml/min).
11. Pass the 40 ml Th fraction through the column (1 - 2 ml/min), collecting in a 100 ml beaker.

Date and time of Th separation from U : _____ (should be ~ 4 U)

12. Rinse the 250 ml beaker 3x with 2 ml 8N HCl, then rinse the column with 20 ml 8N HCl, collecting in

the same 100 ml beaker as step 11.

13. Evaporate the Th fraction to dryness and resolubilize ^{x2} in 5 ml 8N HNO₃. Evaporate and resol. in 5 ml 8N HNO₃.
14. Condition a column containing 2 ml of AG1-X8 100 - 200 mesh resin with 10 ml 8N HNO₃ (1-2 ml/min).
15. Pass the 5 ml Th fraction through the column (1-2 ml/min), collecting in a waste beaker.
16. Change from the waste beaker to a 50 ml beaker and elute the Th with 15 ml 8N HCl. → HNO₃ waste?
17. Add 5 drops of concentrated H₂SO₄ to the Th fraction and evaporate to dryness. Cool. Hold for electroplating.

ELECTROPLATING PROCEDURE

1. Clean and label the plating disks with N#. Assemble the plating cells, allowing time for each to sit with 5 ml of DI water, to check for leaking. Toss the DI.
2. Add exactly 5 ml of DI to the dried Th fraction from step 17, washing down the walls of the beaker with a disposable pipette.
3. Add 4 drops of 0.02% thymol blue indicator and 10 drops of 14M NH₄OH (in the hood). The indicator should turn blue (yellow is OK), indicating a basic pH. Mix the solution using a disposable pipette. *** It is important that the same amount of NH₄OH be added to each cell per plating run, since the (NH₄)₂SO₄ conc. establishes the electrical resistance of the solution and thus affects the current/cell.
4. Neutralize the solution with 18M H₂SO₄ and add one drop in excess of the neutral point (soln ~ pink).
5. Transfer the solution to the plating cell, rinsing the beaker 2x with 2 ml of 0.02 M H₂SO₄ (3 drops of 18 M H₂SO₄ per 100 ml DI).
6. Adjust the pH to 2 - 3, using 2 M NH₄OH (mix with a disposable pipette). The end point should be orange. Check the pH and if > 3, then adjust with an extremely dilute H₂SO₄ solution (1 drop conc. in 50 ml DI).

7. Plate for 2 hours at 1 amp/cell (ie. 4 cells = 4 amps). Check periodically, adjusting the voltage.
8. At the end of the plating period, add 1 ml of 14 M NH_4OH to each sample, wait 1 minute, then switch off.
9. Disassemble the cells as quickly as possible. Wash the plating disk with DI and acetone. Dry and store for counting in a labelled envelope.
** Save the plating solution until the chemical efficiency has been established**

Place the plating apparatus in a beaker with warm soapy water and warm on a hot plate. Cool overnight, rinse with tap water and put everything but the copper bottoms into a beaker with 2N HCl, warming on a hot plate. Put the copper bottoms into a cool acid bath. Rinse the plating equipment with distilled water, dry and store in plastic bags. ✓