RiO5 METHOD (34)

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Procedure for the separation/purification of Th-234 on EQPAC Mn Cartridge samples

PROCEDURE FOR THE SEPARATION/PURIFICATION
OF TH-234 ON EQPAC MN CARTRIDGE SAMPLES

ASH THE CARTRIDGES **SEPARATELY** IN 600 ML BEAKERS AT 500C.

Date Started : _____

1. Add approximately 50 ml 8N HNO3 to the ashed sample in the 600ml beaker and heat to solubilize.

2. Add 6 dpm (by weight) Th-230. Continue heating.

3. Cool and add 30% H2O2 dropwise until fizzing stops and the solution clears. Heat for 5 hours.

4. Warm and add 8N HNO3 to solubilize all salts (if necessary), then filter through a glass fiber filter. Save the filter in a labelled petri dish for possible future analysis.

5. Condition a gooseneck column containing 17 ml AG1-X8 50 - 100 mesh resin with 100 ml 8N HNO3 (1 - 2 ml/min).

dilute to 4N ?

6. Pass the solution through the column (1 - 2 ml/min), **collecting in a 500 ml bottle.** This fraction contains Ra and should be dated and held for purification.

order ?
500 ml bottles

Date and time of Ra separation from Th : _____

7. Rinse the filter flask 3x with 5 ml 8N HNO3, then rinse the column with 150 ml 8N HNO3, collecting in the same bottle containing the Ra fraction.

8. Change from the 500 ml bottle to a 250 ml beaker and elute Th with 100 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 : _____

not necessary
should type of collector

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 in 5 ml 8N HNO3. Evaporate and resol. in 5 ml 8N HNO3. x2

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. → HNO_3 wash + beaker rinse

16. Change from the waste beaker to a 50 ml beaker and elute the Th with 15 ml 8N HCl.

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 resistence 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 H_2SO_4 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.