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RiO5 METHOD (44)

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^{90}Sr — beta counting — 20L seawater samples

Determination of ^{90}Sr activity in seawater via its daughter ^{90}Y

Adapted from Tazoe et al., 2016
by Jennifer Kenyon, MIT-WHOI Joint Program

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

This method describes the radiochemistry of seawater to analyze ^{90}Sr through the detection of its daughter ^{90}Y using beta counting. 20 L of seawater sample are pre-concentrated with iron hydroxide precipitates. This method needs to be done relatively quickly not to let 90Y ($T_{1/2}$=64 hours) decay once it is separated from ^{90}Sr. Chemical recovery is monitored with stable Y throughout the process.

2 EQUIPMENT and CHEMICAL REAGENTS

2.1 Equipment and consumables

- Filter set-up: Filter stands, peristaltic pump, tubing, plastic tweezers
- Transfer pipettes, glass beakers, plastic beakers, 2 mL vials
- Pipette and tips
- Hot plate
- Large filters: GE Healthcare Whatman, ME24 Membrane Filters, Mixed cellulose ester, 0.2 µm, 143 mm diameter, 25 per pack. Cat No. 10401-731.
- Eichrom DGA Resin: 1 mL cartridge
- Petri-dish: USA Scientific, 150 x 15 mm Disposable polystyrene petri dish
- QMA filters

2.2 Tracers

- Stable Yttrium spike: Y set in HNO_3
    - Inorganic Ventures (CGY1) Yttrium
        - Product name: 1000 ppm (µg/mL) Yttrium for ICP - CGY1-125ML
        - 998 +/- 4 µg/mL Yttrium
        - 2% v/v HNO_3

2.3 Chemical reagents

- FeSO4*7H2O: Iron (II) sulphate heptahydrate, CAS #7782-63-0, Merck KGaA (Germany), 500 g
- HCl (trace metal grade)
- HNO3 (trace metal grade)
- Ammonia (trace metal grade)

2.4 Solutions

- Iron precipitate solution (Fe carrier)
- 0.1 M HCl

- 8M HCl
- 8M HNO3
- 3M HNO3 + 0.3M Hf
- 0.02M HNO3
- 13.3M HNO3

  Note:

  - Aliquot samples will be used to run recoveries via ICP-MS

3 PROCEDURE

1. Sample preconditioning

1.1. Record sample weight (kg)
1.2. Acidify to ~2 pH (roughly ~1 mL HNO_3 per kg seawater)
1.3. Add Y spike
- Shake to mix the sample
- **Must be completed >12 hours before next step**

1.4. Shake sample again after waiting period
1.5. Take aliquot sample (~ 1 mL)
1.6. Add Fe carrier (~1 mL)
1.7. Bring to pH ~8.8 – 9 by adding NH_4OH
- Record time added
- Sample should turn yellow
- **Must be completed >30 minutes before filtration**
- Shake sample again after waiting period

2. Filtration

2.1. Prepare filtration system with clean gloves
- Filtration stand → large mesh screen → small mesh screen → filter → O-Ring
- Use 0.2 µm, 1433 mm ME24 Membrane Filter

2.2. Wet filter with MilliQ water mixed with ammonium (pH 9)
2.3. Filter the sample using peristaltic pump and record time
- To begin, use low velocity and increase speed after it is clear there are no leaks

2.4. Upon completion, remove filter and place in petri dish—immediately cover to avoid contamination
2.5. Add 13.3M HNO_3 to sample move solution to a clean glass beaker until the filter is completely rinsed of any orange precipitate
2.6. Evaporate solution out of beaker on hot plate until liquid is completely evaporated
- Avoid bringing the solution to a boil
- Be careful not to let the sample dry out and burn

2.7. Add 5 mL 13.3 HNO_3 into sample beaker
2.8. Prepare to add solution to column

3. Column chemistry

3.1. Prepare column analysis: peristaltic pumps, tubing, column (DGA Resin), waste beaker
3.2. Rinse tubing (not column) with 10% HCl (~ 15 mL) and Milli Q water (~ 20 mL)
3.3. Attach column to tubing and pre-treat DGA resin by adding solutions, flow rate at **2 mL/min**
- 20 mL – 0.1M HCl
- 10 mL – 8M HCl
- 10 mL – 8M HNO_3

3.4. Load sample to column at **1 mL/min**
3.5. Add 8M HNO_3 (15 mL) to sample beaker and load to column at **2 mL/min**
3.6. Add 8M HCl (10 mL) direct to column at **2 mL/min**
3.7. Add 3M HNO_3 + 0.3M HF (20 mL) direct to column at at **2 mL/min**
3.8. Add 0.02 M HNO_3 (20 mL) direct to column at at **2 mL/min**
3.9. Remove waste beaker, replace with clean beaker
- Record clean beaker mass before and after adding final solution

3.10. Add 0.1M HCl (20 mL0 direct to column at at **1 mL/min**
- **Be careful**, as this is the final sample
- Upon completion, remove beaker from column chemistry set-up

4. Final Filtration and Mounting

4.1. Take aliquot sample (1 mL) from final sample – make sure to weigh vial before and after aliquot
- Make sure to take this into account in final calculation

4.2. Add Fe carrier (0.3 mL) to final sample
4.3. Add NH_4OH (0.3 mL) to final sample
- Precipitate should form
  - Gently stir the solution (ideally by swirling the beaker and not adding a stir rod or another potential source for contamination)
- **Must be completed >5 minutes before filtration**

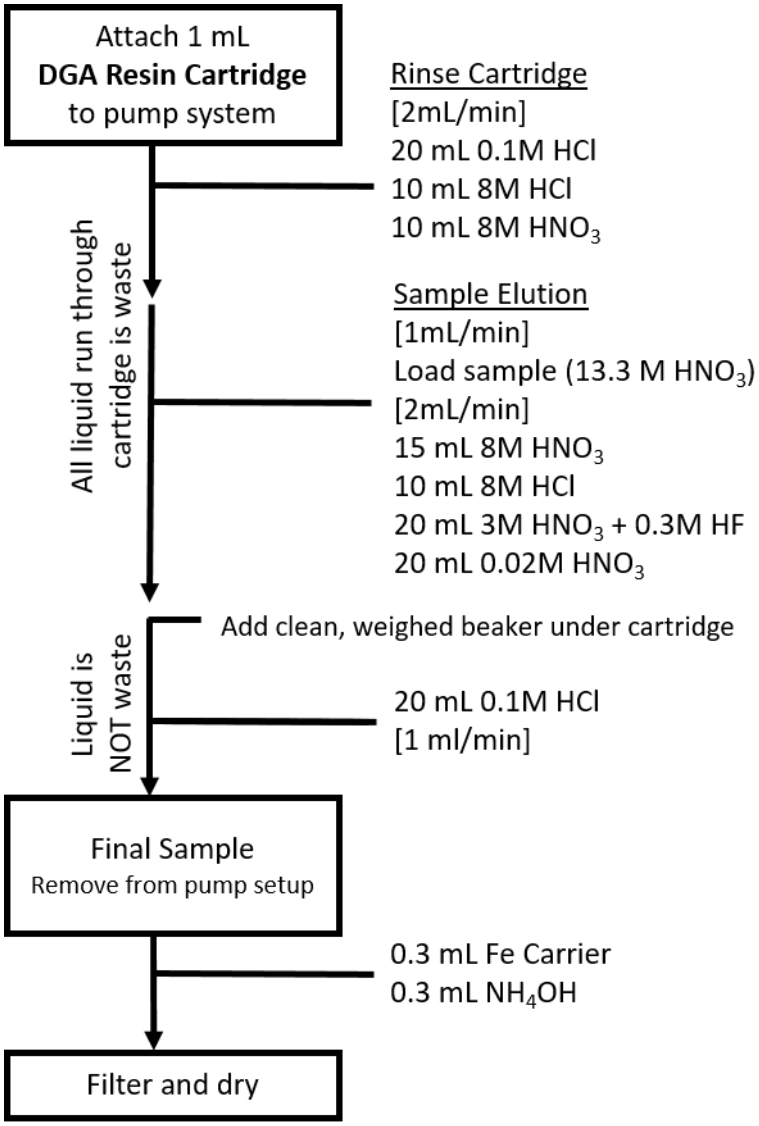
4.4. Filter final sample through QMA filter
- Pour precipitate solution slowly into filter holder with pump on
- Rinse sides of filter holder using milli Q + ammonia solution (pH 9) to remove Fe build-up
- Fe sticks to sides of filter holder, do not let the filter run dry but rather turn off pump just as the last drop of liquid is sucked through

4.5. Remove filter from pump and place on labeled, clean watch-glass and heat (low) to dry
4.6. Transfer filter to beta mount by placing filter precipitate side up onto the mount, covering with Mylar film
4.7. Samples are counted on a RISO beta counter for at least 70 hours

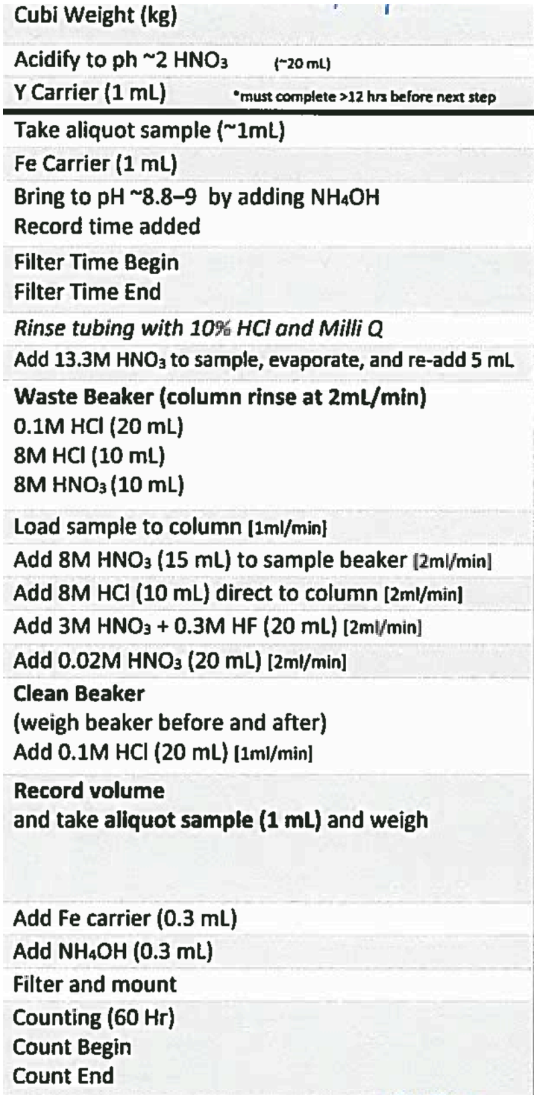
4 REFERENCES

Tazoe, H., Obata, H., Yamagata, T., Karube, Z., Nagai, H., Yamada, M., 2016. Determination of strontium-90 from direct separation of yttrium-90 by solid phase extraction using DGA Resin for seawater monitoring. Talanta. 152, 219-227.

5 FLOW CHART



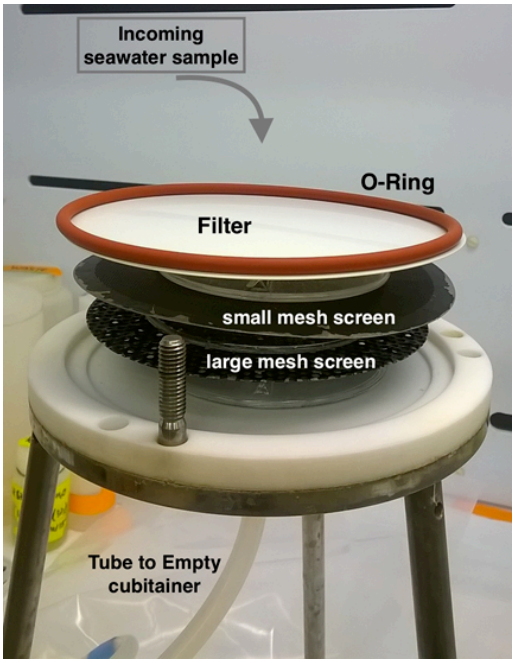
Flowchart of elution process through DGA Resin.



Cubi Weight (kg)
Acidify to ph ~2 HNO_3 (~20 mL)
Y Carrier (1 mL) *must complete >12 hrs before next step
Take aliquot sample (~1mL)
Fe Carrier (1 mL)
Bring to pH ~8.8–9 by adding NH_4OH Record time added
Filter Time Begin Filter Time End
Rinse tubing with 10% HCl and Milli Q
Add 13.3M HNO_3 to sample, evaporate, and re-add 5 mL
Waste Beaker (column rinse at 2mL/min) 0.1M HCl (20 mL) 8M HCl (10 mL) 8M HNO_3 (10 mL)
Load sample to column [1ml/min]
Add 8M HNO_3 (15 mL) to sample beaker [2ml/min]
Add 8M HCl (10 mL) direct to column [2ml/min]
Add 3M HNO_3 + 0.3M HF (20 mL) [2ml/min]
Add 0.02M HNO_3 (20 mL) [2ml/min]
Clean Beaker (weigh beaker before and after) Add 0.1M HCl (20 mL) [1ml/min]
Record volume and take **aliquot sample (1 mL)** and weigh
Add Fe carrier (0.3 mL)
Add NH_4OH (0.3 mL)
Filter and mount
Counting (60 Hr) Count Begin Count End

Step-by-step “checklist” for the author’s experiment setup.
Note: this list was added as a source of reference, but should not substitute thoroughly reading the step-by-step instructions and may not be applicable to every lab setup.

6 IMAGES



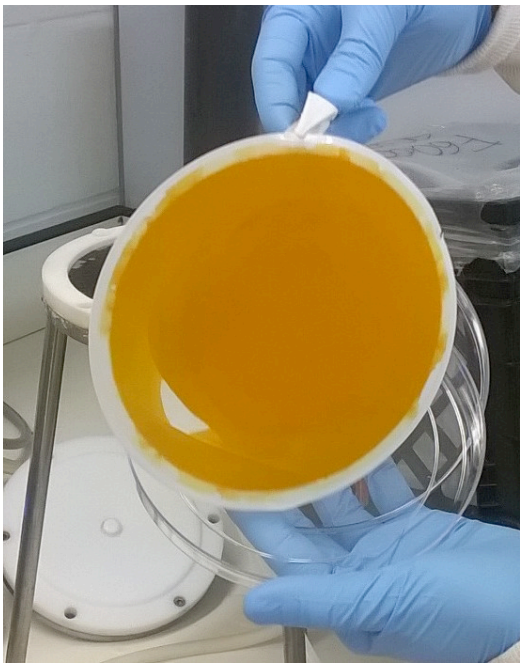


Image of step 2.1: initial filtration on a 0.2 µm, 1433 mm ME24 Membrane Filter and the completed filtered sample.