RiO5 METHOD (8)

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^{129}I — resin — seawater

IODINE-129 RADIOCHEMISTRY IN SEAWATER SAMPLES AND TARGET PREPARATION.

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 ^{129}I in seawater samples. It is based on the method described in Szidat et al (2000) and Michel et al (2012), modified during 2015 with own tests at EAWAG and ETH (Casacuberta et al., 2015). The method covers the radiochemistry of a seawater sample right after its collection to the final preparation of the cathode (target) before its measurement with Accelerator Mass Spectrometry (AMS). One should note that every AMS system has its own targets and thus final steps of this method should be adapted

2 EQUIPMENT and CHEMICAL REAGENTS

2.1 Equipment and consumables

- Glass or plastic bottles 500 mL where to process the sample.
- Stirring plates
- Glass or plastic funnel (500 – 1000 mL) with a stopper (see Figure)
- 10 mL column ((from BioRad). It is important that the columns fit to the gummy-stoppers of the funnel above
- A setup that holds both the funnel and column (see Figure).
- Beakers of different sizes (for wastes and final collection of the sample)

2.2 Tracers

- Stable ^{127}I (Wood Ward Iodine).

2.3 Chemical reagents

- Sodium metabisulfite: $Na_2O_5S_2$
- Calcium hypoclorite: $Ca(ClO)_2$
- Hydroxylamine hydrochloride: $[NH_2OH]\cdot HCl$
- Sodium hydroxide NaOH
- Potassium nitrate: KNO_3
- Resin DOWEX 1x8
- Nitric Acid (suprapure): HNO_3
- Silver nitrate: $AgNO_3$

2.4 Solutions

- Tracer solution ^{127}I WWI of 30 mg/mL (see step 0 of procedure to prepare it)
- Calcium hypoclorite: $Ca(ClO)_2$, concentration 2%.
- Hydroxylamine hydrochloride: $[NH_2OH]\cdot HCl$, concentration 1M.

- Potassium nitrate: KNO3, one solution 0.5 M, another solution 2.25 M.
- Silver nitrate: AgNO3, concentration 0.1 M.
- Sodium hydroxide NaOH, concentration 7 M and another solution of 0.5 M.

3 PROCEDURE

0. Prepare a solution of WWI (Woodward Iodine) – Stable I isotope (carrier)[1]
    a. Weight about 0.1 g of WWI.
    b. Prepare 10 mL $NaHSO_3$ (1 g $Na_2O_5S_2$ per 10 mL milliQ water).
    c. Take small dark crystal bottle. Tare it.
    d. Add 3 mL of $NaHSO_3$ solution (a). Note the weight.
    e. Add 0.2 mL NaOH 7M. Note the weight.
    f. Add pre-weighted 0.1 g of WWI. Note the weight.
    g. Mix it until WWI is completely dissolved.
    h. Add 0.2 mL NaOH 7M. Note the weight.
    i. Label the bottle with the number of the solution (e.g. WWI sol. 3).

For a batch of 4 samples (use the spread sheet of the radiochemistry steps to mark every step):

1. Weight samples. Note the weight.[2]
2. Check pH of samples. Should be around 6-7. When lower, add NaOH 0.5M.
3. Add WWI[3], normally 50 uL of the solution prepared at (0). Note the code of the solution used (e.g. 2016_1). Shake vigorously to ensure total mixing of the sample.
4. Put the samples on the stirring plates. Add a stirrer in each sample and start stirring them.
5. Add 5 mL $Ca(ClO)_2$ 2% (30g $Ca(ClO)_2$ in 100mL milliQ water). This step is to convert all iodine species into iodide.
6. Stir for 5 minutes.

[1]This solution should be freshly prepared. Maximum Keep it for 2 or 3 weeks.

[2]The volume used will depend on the expected amount of ^{129}I. See report by Casacuberta et al, for clarifications.

[3] The amount of WWI will depend on the expected amount of 129I and the volume of sample. See report by Casacuberta et al, for clarifications.

7. Add 20 mL $[NH_2OH]\cdot HCl$ 1 M together with 1 g. $Na_2O_5S_2$. This oxidation step was described as a useful method to destroy organic iodine compounds so that not only inorganic iodine species are measured. Shake vigorously to ensure total mixing of the sample.
8. Stir for 45 minutes.
9. Prepare anion exchange columns (Figure 1).
    a. Weight 5 g. of resin DOWEX 1x8 (100-200 mesh). 5g = 5-6 mL of resin.
    b. Add water and let it go through the column.
    c. Add 25 mL KNO_3 0.5 M and let it go through the column. You should not add the solution before all the water has gone through.
10. After 45 minutes stirring, raise sample pH to 5-6 using 7M NaOH. Add NaOH in 0.2 mL steps and check the pH. Be careful, this solution is corrosive.
11. Put samples into the decantation funnels (Figure 1). Make sure the funnels are closed before poring the sample into them. Connect the funnels to the columns and open the funnel so the sample starts going through the column. Make sure that connection is properly sealed not to loose any sample.
12. Let the sample go trough the column. This might take long (1-4 hours, depending on the sample volume).
13. Once the sample has gone through the column you can do the Iodide extraction:
    a. Rinse the resin:
        i. Add 20 mL milliQ water.
        ii. Add 50 mL KNO_3 0.5 M.
        iii. Discard fractions i, ii.
    b. Prepare beakers for Iodide collection:
        i. Prepare 3 beakers for each sample. In total, 12 beakers.
        ii. Add 0.2 mL $AgNO_3$ in each beaker.
        iii. Add 0.1 mL HNO_3 conc. In each beaker.
    c. Extract Iodide (Figure 2).
        i. Add 15 mL KNO_3 2.25 M. Collect it in a small beaker.
        ii. Add 20 mL KNO_3 2.25 M. Collect it in a second small beaker.
        iii. Repeat step ii and collect it in a third beaker.
14. Combine fractions in 13c that have more precipitate. Normally should be beakers 2 and 3.
15. Let precipitates settle overnight in a dark place.

16. Filtration of precipitates (Figure 3):
    a. Prepare filtration setup.
    b. Filtrate each sample in a 0.8 um pore size filter.
    c. Put filters into a stove at 50ºC to dry them.
17. Store dried filter in labeled boxes.
18. Prepare targets.
    a. Tare the Achat stone mortar.
    b. Scratch the filter into a Achat stone mortar.
    c. Weight the amount of sample that has been scratched. Optimal is to get 1 mg of precipitate.
    d. Add Ag powder in a proportion 1/4 [4], so around 4 mg of Ag, that all together sum up to 5 mg.
    e. Mix it all together until it gets very homogeneous.
    f. Add this into the Ti target[5].
    g. Label the target and store it until wheel preparation.

4 REFERENCES

Casacuberta, N., M. Christl, and C. Vockenhuber (2014), Chemistry of 129I in seawater samples: setting up the 129I radiochemistry at LIP/EAWAG*Rep.*, 140 pp, ETH Zürich.

Michel, R., et al. (2012), Iodine-129 and iodine-127 in European seawaters and in precipitation from Northern Germany, *Science of The Total Environment*, *419*(0), 151-169, doi:http://dx.doi.org/10.1016/j.scitotenv.2012.01.009.

[4] If proportion is not ¼ does not really make a bit difference for the AMS measurements. So do not worry if you add a bit more or a bit less Ag to the sample.

[5] Always use Ti targets, not Al targets. Very important!!

5 IMAGES

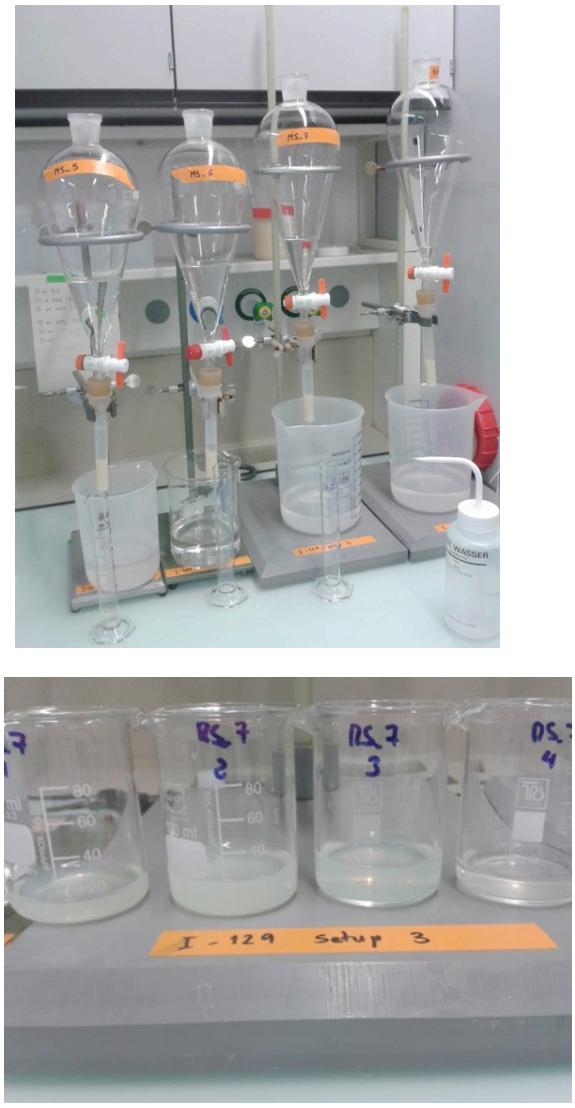

Figure 2: Collection of the different fractions of iodine from seawater. Solutions contain AgI precipitates.

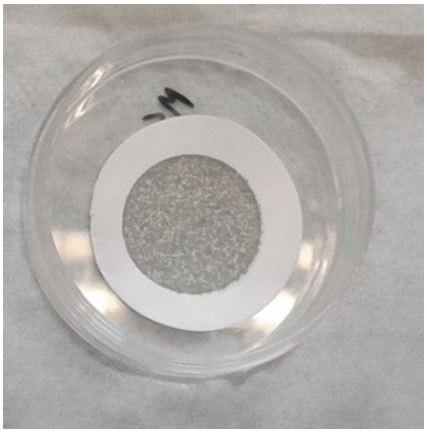

Figure 3: Filter containing the final AgI precipitates.