RiO5 METHOD (28)

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^{210}**Pb**—Auto-plating —Sediment sample

Sediment sample preparation for lead-210

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.

Table of Contents

1 SCOPE

This method specifies the minimum requirements and laboratory methods for the analysis of ^{210}Pb with alpha-spectrometry in sediment samples.

Samples are collected using crab sampler or gravity sampler, sealed on board and finally kept at -18°C. Post processing will take place in the land laboratory by freeze-dry. ^{210}Pb is determined through its grandparent ^{210}Po. Isotope decay correction is applied if the duration between sampling and measurement introduce discernible variation in ^{210}Pb activity. (see Yang et al., 2016)

2 EQUIPMENT CHEMICAL REAGENTS

2.1 Equipment

- Sediment sampler
- Analytical balance with an accuracy of ± 0.1 mg
- Teflon beakers
- Teflon-coated magnetic rotor assembling silver disk holder
- Hot plate with magnetic stirrer
- Alpha spectrometry

2.2 Tracers

- ^{209}Po (4.54 dpm g^{-1})

2.3 Chemical reagents

- Hydrochloric acid (HCl)
- Ammonium hydroxide (NH_4OH)
- Nitric acid (HNO_3)
- Ascorbic acid ($C_6H_8O_6$, powder form)
- Hydroxylamine hydrochloride
- Sodium citrate

3 PROCEDURE

Lead-210 in sediment is usually expected to be in equilibrium with its granddaughter ^{210}Po although ^{210}Po is excessive with respect to ^{210}Pb in some areas with very high sedimentation rates. Thus, determining ^{210}Pb in marine sediments via ^{210}Po is widely used. To grantee

measured ^{210}Po equal to ^{210}Pb, we suggest that the sediment is left for half a year after sampling.

Preparation of sediment sample

1. Put 0.5-1.0 g dried sediment into a Teflon beaker.
2. Add a known amount of ^{209}Po (~2 dpm) to the sample as a yield tracer of ^{210}Po.
3. Add 20 ml concentrated HNO_3 and 2 ml HF, and digesting the sample at 200°C for 2 hours, centrifuge and decant the solution into the other Teflon beaker.
4. Repeat the above step two times and combing the leached solution.
5. Evaporate the solution to nearly dryness,
6. Add 5 ml concentrated HCl and evaporating to nearly dryness.

Deposition of Po on silver disc

1. Dissolved the prepared sample with 6 M HCl, and adjuste to a pH range of 0-2.0 with ammonium hydroxide.
2. Add ascorbic acid to a colorless solution, then add 1 mL 20% hydroxylamine hydrochloride and adjust to a pH ~1.5.
3. Place the silver disc in the magnetic rotor with one side free of touching the solution.
4. Auto-plate Po isotopes (i.e. ^{209}Po and ^{210}Po) onto a silver disc at 95°C for 4 hours with stirring.

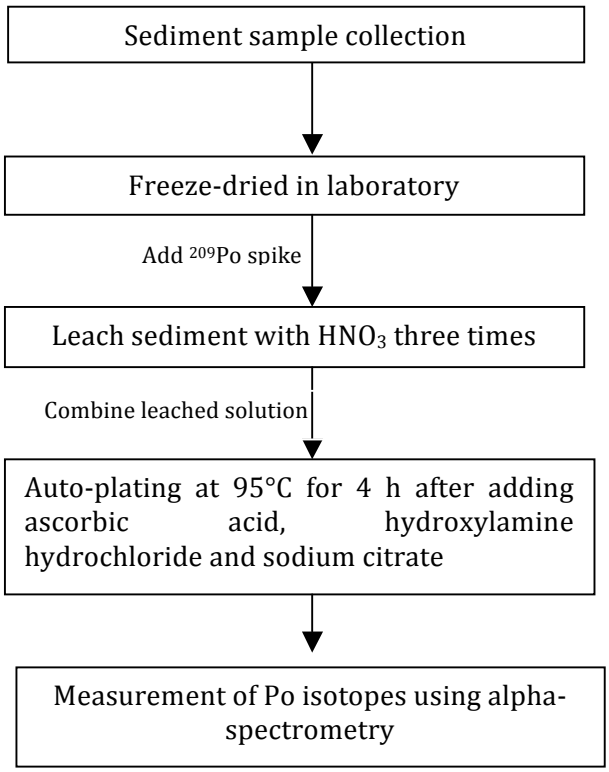
Measurement of Po

Po isotopes (i.e. ^{209}Po and ^{210}Po) are counted using alpha-spectrometry, for ^{209}Po and ^{210}Po the energy are 4.881 MeV and 5.115 MeV respectively.

4 REFERENCES

Yang W., Chen M., Zhang F., Zhao X., Fang Z., Ma H. (2016), Anthropogenic impacts on sedimentation in Jiaozhou Bay, China. Journal of Coastal Conservation, 20, 501-506.

5 FLOW CHART


Sediment sample collection
Freeze-dried in laboratory
Add ^{209}Po spike
Leach sediment with HNO_3 three times
Combine leached solution
Auto-plating at 95°C for 4 h after adding ascorbic acid, hydroxylamine hydrochloride and sodium citrate
Measurement of Po isotopes using alpha-spectrometry