RiO5 METHOD (27)

Junwen Wu
College of Ocean and Earth Sciences, Xiamen University
qdwjunwen@163.com

^{238}Pu, ^{239,240}Pu—Alpha Counting—Sediment sample

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 1

2 EQUIPMENT CHEMICAL REAGENTS 1

2.1 EQUIPMENT 1
2.2 TRACERS 1
2.3 CHEMICAL REAGENTS 1
2.4 SOLUTIONS 1

3 PROCEDURE 2

4 REFERENCES 2

5 FLOW CHART 3

6 IMAGES ERROR! BOOKMARK NOT DEFINED.

1 SCOPE

This method specifies the minimum requirements and laboratory methods for measuring ^{238}Pu and $^{239,240}Pu$ in sediment samples via alpha-counting.

Before digestion, the sample should be ashed at 480 ℃ for 48 h in order to remove the organic matter. This method will meet difficulties when the counting statistics are low or when the alpha spectrum is disturbed by natural radioactivities in the sample or background (Hakanen et al., 1984). It is thus necessary to collect the enough sample volume (about 100 g) due to low ^{238}Pu activity. Interference is especially due to ^{228}Th, indicated by the presence of ^{224}Ra in the alpha spectrum. Meanwhile, this method cannot separate ^{239}Pu and ^{240}Pu because of their similar energy.

2 EQUIPMENT CHEMICAL REAGENTS

2.1 Equipment

- alpha spectrometry
- Muffle furnace
- Hot plate
- Analytical balance
- Anion-exchange resin(Bio-Rad): AG 1-X8 and AG MP-1M

2.2 Tracers

- ^{242}Pu (IRMM-085):9.464(14)×10^{-9} kg/kg solution
- Soil reference material (IAEA-soil-6) (International Atomic Energy Agency, IAEA).

2.3 Chemical reagents

- Analytical reagent grade: nitric acid (HNO_3), hydrochloric acid (HCl), ammonium hydroxide (NH_4OH), hydrogen peroxide (H_2O_2), HBr, $FeCl_3$, NH_4I.
- Ultrapure grade HNO_3 (Tama Chemicals, Tokyo, Japan)
- Milli-Q purified water (18.2 MΩ cm).

2.4 Solutions

- 8M HNO_3,
- 9 HCl, 10 HCl, 12 HCl
- 0.1M NH_4I-12M HCl
- aqua regia :Conc.HNO3:Conc. HCl=1:3

- HCl-H_2O_2(10mL Conc. HCl+0.01 mL 30% H_2O_2)

3 PROCEDURE

1. Weigh 100 g dry sample and ash at 480 ℃ for 48 h.
2. Sample leach in hot 400 mL HNO_3 and 100 mL HCl, add ^{242}Pu spike.
3. Co-precipitation with $Fe(OH)_3$ at pH 8.5.
4. Load AG 1-X8 column (precondition with 8M HNO_3), and then wash the column using 40 mL 8M HNO_3, 100 mL 9 HCl.
5. Leach the column with 2*40 mL 0.1M NH_4I-12M HCl. The column was rinsed with 5 mL 12 M HCl. evaporate to dryness, add 1 mL aqua regia and evaporate to dryness again (repeated twice).
6. Add 2 mL conc. HCl and evaporate to dryness again (repeated twice), add 4 ml HCl-H_2O_2 (freshly prepared) and heat at 40 ℃ for 30 min.
7. Load the AG MP-1M (precondition with 8 mL HCl-H_2O_2). And then wash the column using 20 mL 8M HNO_3, 8 mL 10 HCl, 16 mL HBr (Pu solution), evaporate to dryness, add 1 ml conc. HNO_3 and evaporate to dryness, dissolve.
8. Electroplate final sample solution onto stainless steel discs.
9. Alpha spectrometry counting.

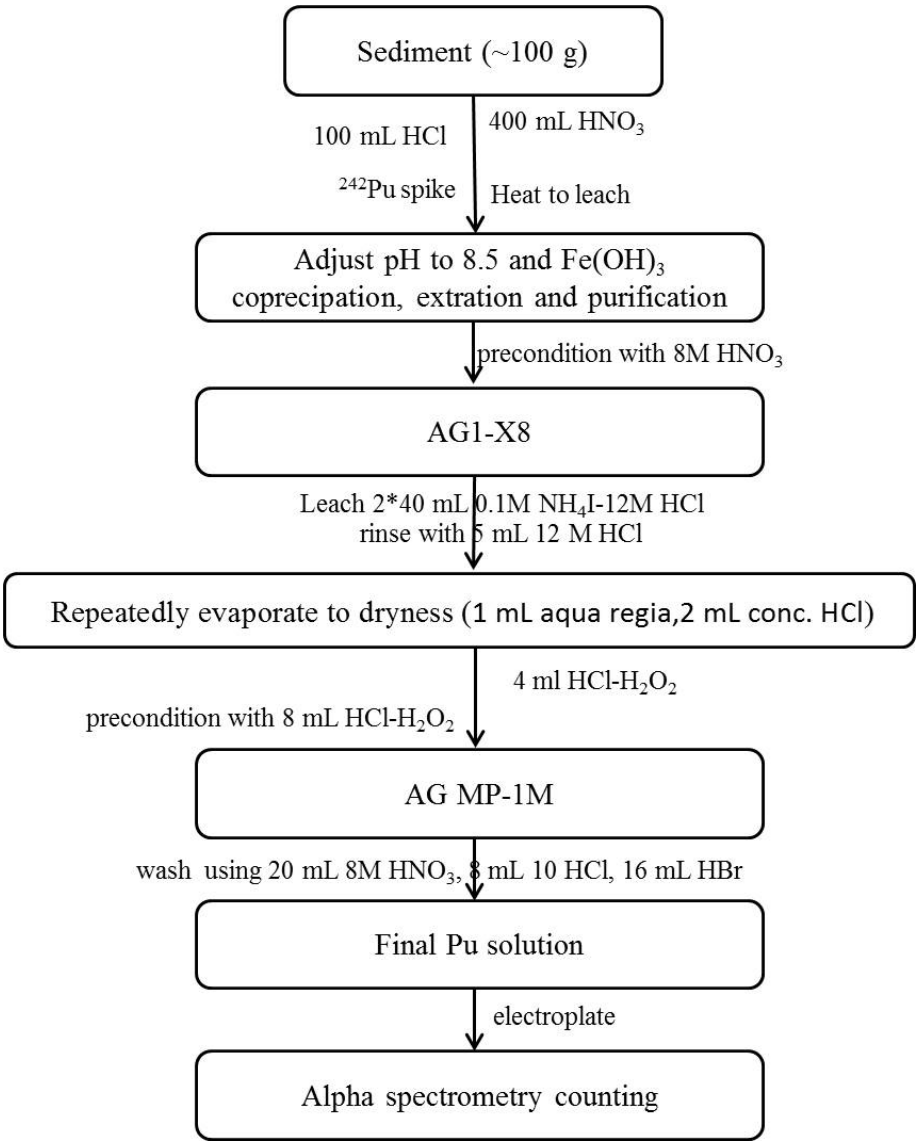
4 REFERENCES

Hakanen, M., Jaakkola, T., Korpela, H., 1984. Simultaneous determination of ^{241}Pu, ^{238}Pu and $^{239,240}Pu$ in low activity environmental samples. Nuclear Instruments and Methods in Physics Resarch 223, 382-385.

Lansard, B., Charmasson, S., Gascó, C., Antón, M. P., Grenz, C., & Arnaud, M. (2007). Spatial and temporal variations of plutonium isotopes (^{238}Pu and $^{239,240}Pu$) in sediments off the Rhone river mouth (NW Mediterranean). Science of the Total Environment, 376(1–3), 215-227.

Lee, S.H., Povinec, P.P., Wyse, E., Pham, M.K., Hong, G.H., Chung, C.S., Kim, S.H., Lee, H.J. (2005). Distribution and inventories of ^{90}Sr, ^{137}Cs, ^{241}Am and Pu isotopes in sediments of the Northwest Pacific Ocean. Mar. Geol. 216, 249-263.

5 FLOW CHART


Sediment (~100 g)
100 mL HCl
^{242}Pu spike
400 mL HNO_3
Heat to leach
Adjust pH to 8.5 and $Fe(OH)_3$ coprecipation, extration and purification
precondition with 8M HNO_3
AG1-X8
Leach 2*40 mL 0.1M NH_4I-12M HCl
rinse with 5 mL 12 M HCl
Repeatedly evaporate to dryness (1 mL aqua regia,2 mL conc. HCl)
4 ml HCl-H_2O_2
precondition with 8 mL HCl-H_2O_2
AG MP-1M
wash using 20 mL 8M HNO_3, 8 mL 10 HCl, 16 mL HBr
Final Pu solution
electroplate
Alpha spectrometry counting