RiO5 METHOD (5)

Environmental Measurements Laboratory
US Department of Energy

Plutonium in water

The following method is an excerpt method Pu-10 from the HASL-300 Procedures Manual from February 1997. The manual is available in its entirety, including many additional methods at the following link:
https://www.orau.org/ptp/PTP%20Library/library/DOE/eml/hasl300/HASL300TOC.htm

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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This report supersedes: HASL-300, 27th edition issued November 1990, revised 1992; 26th edition issued 1983; 25th edition issued 1982; supplement 8 issued 1981; supplement 7 issued 1979; supplement 6 issued 1978; supplement 5 issued 1977; supplement 4 issued 1976; supplement 3 issued 1972 (revised 1975). NYO-4700 issued 1957 (revised 1960, 1962); supplements 1 and 2 issued 1963.

Preface

This 28th edition supersedes all previous versions. The Manual was originally issued in 1957 as a U. S. Atomic Energy Commission Report (NYO-4700). In 1972, substantive changes were made, and the designation became HASL-300 (Health and Safety Laboratory). When the Laboratory's name was changed in 1977 to the Environmental Measurements Laboratory (EML), the Manual was retitled to the EML Procedures Manual (Supplement 6, issued 1978). In subsequent years, parts of HASL-300 were updated as necessary.

Volume I of this edition covers existing technology and procedures currently in use at EML. Some older procedures have been updated and new procedures have been added. Procedures no longer in use at EML, but still valid and used at other laboratories, appear in Volume II.

The Manual includes contributions from most of the EML scientific staff. We welcome any questions, corrections or information you may have concerning this publication. Points of contact are listed in the text. Additional information and updates are also available on EML's Web Site (www.eml.doe.gov). The special contributions of Sylvia Kendall for desktop publishing support and of Jenny May-Maiello for graphic design are also acknowledged.

Pu-10-RC

PLUTONIUM IN WATER

Contact Person(s) : Anna Berne

APPLICATION

This procedure is used for all types of water samples (i.e., sea water, lake water, tap water, etc.). If the sample contains suspended particulates, they must be removed by filtration. Large volume samples are analyzed after evaporation in an acidic medium.

The sample is heated in HNO_3 and then in 3:1 HNO_3:HCl. The volume is then reduced to near dryness and finally the volume of the sample is adjusted with 1:1 HNO_3. The sample is then ready to be purified by ion exchange separation (see Procedure Pu-11-RC).

SPECIAL REAGENT

1. ^{236}Pu tracer - a standard solution containing 0.2 Bq g^{-1} in a dispensing bottle. The purity of the tracer is measured by α spectrometry.

SAMPLE PREPARATION

1. Transfer 100-1000 mL of a H_2O sample to a beaker.

2. To the sample add ~ 0.05 Bq (or appropriate amount) of ^{236}Pu tracer.

3. Add an equal amount of concentrated HNO_3, cover the beaker with a watch glass and place on a hot plate. Reflux the solution for 4-8 h.

4. Replace the watch glass with a ribbed watch glass and evaporate the solution to near dryness. When the volume is reduced to ~ 100 mL, allow the solution to cool to room temperature and transfer to an appropriate size beaker.

5. Continue evaporating the sample to near dryness. Cool, add 75 mL of concentrated HNO_3 and 25 mL of concentrated HCl. Cover with a watch glass. Allow to react for 30 min. Then place the sample on a hot plate and bring to a boil. After the solution has boiled for 30 min, reduce heat and continue heating overnight. Do not allow the sample to evaporate to dryness.

6. Remove the sample from the hot plate and add 100 mL of H_2O to the sample. Allow the sample to cool to room temperature and filter under reduced pressure using a Buchner funnel with a Whatman No. 42 filter paper.

7. Wash with 50 mL of 1:1 HNO_3 and then 50 mL of H_2O.

8. Transfer the filtrate to a 250-mL beaker, cover the beaker with a ribbed watch glass.

9. (**Note**: If the filter paper contains a moderate amount of precipitate, it must be treated with HF.) Transfer the filter paper containing the residue from the HNO_3/HCl digestion to a platinum dish. Place the platinum dish in a muffle furnace and heat at 100°C, raise the temperature by increments of 100°C every hour until a final temperature of 450°C is reached. Continue heating at this temperature overnight. Turn off the muffle furnace and let the sample in the platinum dish cool sufficiently to remove it from the furnace. Add 15 mL of 1:1 HNO_3 and 15 mL of concentrated HF. Heat the sample to neardryness.

10. Repeat Step 9 two times.

11. Add 20 mL of 1:1 HNO_3 to the sample and heat on a hot plate under a low setting until near dryness (to remove traces of HF).

12. Repeat Step 11 two times.

13. Add 20 mL of 1:1 HNO_3 to sample.

14. Using a conical funnel, filter the sample by gravity through an 18.5 cm Whatman No. 42 filter paper into a beaker containing the filtrate from Step 9. Wash well with 1:1 HNO_3.

15. Reduce the volume of the solution to near dryness on a hot plate.

16. Adjust the volume to 100 mL by the addition of 1:1 HNO_3.

17. Proceed to Plutonium Purification - Ion Exchange Technique, Procedure *Pu-11-RC*.

Pu-11-RC

PLUTONIUM PURIFICATION - ION EXCHANGE TECHNIQUE

Contact Person(s) : Anna Berne

APPLICATION

This procedure has been applied to the leachates derived from the plutonium sample preparation methods described in this Manual. Ion exchange chromatography is used to remove the large amounts of impurities contained in these leachates.

SPECIAL APPARATUS

Ion exchange columns - see Specifications 7.5 and 7.6.

SPECIAL REAGENTS

1. 1:1 HNO_3 - 500 mL HNO_3 diluted to 1 L.

2. Hydroxylamine hydrochloride - $NH_2OH{\cdot}HCl$

3. 0.3M hydroxylamine hydrochloride-0.5M HNO_3 - 20.85 g of $NH_2OH{\cdot}HCl$ diluted to 1 L with 0.5M HNO_3.

4. Anion exchange resin - Bio-Rad AG 1-X8 (100-200 mesh, Cl^- form), see Specification 7.4

ION EXCHANGE SEPARATION

1. Cool the sample in an ice bath, add 1 g of $NH_2OH \cdot HCl$, stir, and let stand in an ice bath for 15 min. Remove the sample from the ice bath and heat to boiling on a hot plate with medium heat for 1-3 min. Cool the sample to room temperature.

2. Prepare the ion exchange resin column (see **Note**).

3. Pass the sample through the resin bed at a flow of ~ 1 mL min^{-1}. Wash the beaker and the column with 30 mL 1:1 HNO_3 three times. Allow the liquid to flow until the level reaches the top of the resin bed prior to each wash. Reserve the sample and wash the effluent for ^{241}Am determination (or until yield has been determined as satisfactory).

4. Elute the plutonium with 10 mL of 0.5 M HNO_3 twice - then with 100 mL of 0.3M hydroxylamine hydrochloride - 0.5M HNO_3 into a 250-mL beaker. Discard the resin.

5. Slowly add 25-30 mL HNO_3 until effervescence begins, then place on a hot plate and evaporate the eluate to dryness.

6. Dissolve the residue in 30 mL of 1:1 HNO_3 and cool in an ice bath. Add 500-600 mg of $NH_2OH \cdot HCl$ and repeat Steps 1-3 using a small column (see Specification 7.6) for all samples.

7. Wash the resin with 100 mL of HCl (two 10-mL portions followed by two 40 mL portions). Wash the resin with two 10-mL portions, followed by one 40-mL portion 1:1 HNO_3. Save the effluent until yield determination.

8. Repeat Steps 4 and 5. Discard the resin.

9. Convert the residue to the Cl^- form by adding 5 mL of HCl and evaporating to dryness three times at a low temperature.

10. See Procedure G-03 for microprecipitation for α spectrometry.

Note: Preparation of Columns

1. When preparing a large soil sample use a large column (Specification 7.5), otherwise use the column described in Specification 7.6.

2. Position a plug of glass wool in the base of the column so that no resin will drain out.

3. Add sufficient resin to form a resin bed of 10 cm in length. Wash the column with sufficient 1:1 HNO_3 to remove the Cl^- ion from the resin. Test the effluent with a dilute silver nitrate solution.