Polonium

Environmental Measurements Laboratory
U.S. Department of Energy

Po-02-RC

POLONIUM IN WATER, VEGETATION, SOIL, AND AIR FILTERS

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APPLICATION

This procedure has been tested for water, vegetation, soil, and Dynaweb filters. Reagent blanks must be analyzed along with the samples.

Polonium is equilibrated with ^{208}Po or ^{209}Po tracer and isolated from most other elements by coprecipitation with lead sulfide. The sulfide precipitate is dissolved in weak HCl solution. Polonium is quantitatively deposited on a nickel disc. The deposition is very specific and can be carried out in the presence of other radionuclides.

The plated disc is counted on an α spectrometer to measure chemical yield and activity of the sample. The solution from the deposition may be retained and analyzed for ^{210}Pb.

SPECIAL APPARATUS

1. Nickel discs - 1.75 cm diameter x 0.06 cm thick "commercial pure" nickel. Degrease in acetone, dip in HCl and rinse with water.

2. Electrolytic cell - see Specification 7.16.

3. Teflon stirring rods.

SPECIAL REAGENTS

1. Standardized ^{208}Po or ^{209}Po tracer solution - about 2 Bq g^{-1} in a dispensing bottle.

2. Lead carrier solution: 10 mg Pb mL^{-1} - 15.98 g $Pb(NO_3)_2$ L^{-1} of 1:99 HNO_3.

3. Thioacetamide solution - 100 g CH_3CSNH_2 L^{-1} of water.

4. Saturated ascorbic acid solution.

SAMPLE PREPARATION

A. Tap water.

1. Transfer 2.5 L of tap water to a 3-L beaker.

2. Add 50 mL of HNO_3 and 1 mL of Pb carrier solution. Add a weighed aliquot (30-80 mBq) of the ^{208}Po or ^{209}Po tracer solution.

3. Evaporate and add additional aliquots of tap water until a 10-L collection has been obtained. Evaporate gently to about 25 mL.

4. Transfer the solution to a 90-mL centrifuge tube with H_2O. Continue with **Determination**.

B. Vegetation.

1. Weigh 100 g of dried (105-110°C) material into a 400-mL beaker.

2. Add 1 mL of Pb carrier solution and a weighed aliquot (30-80 mBq) of ^{208}Po or ^{209}Po tracer solution.

3. Add 100 mL of HNO_3 with magnetic stirring using a Teflon-coated bar. Digest with gentle heat and stirring for 1 h.

4. Reduce the volume of the solution to about 25 mL and transfer the solution to a 90-mL centrifuge tube with water. Continue with **Determination**.

C. Soil.

1. Weigh 1 to 5 g of soil into a 40-mL platinum dish. Add 1 mL of Pb carrier and a weighed aliquot (30-80 mBq) of ^{208}Po or ^{209}Po tracer solution.

2. Add 10 mL of HNO_3 and 10 mL of 48% HF. Heat on a medium hot plate. Repeat the additions of HNO_3 and HF until no further dissolution takes place.

3. Add 10 mL of HNO_3 and reduce the volume to about 5 mL.

4. If insoluble material remains, filter the slurry by gravity through a Whatman No. 42 filter paper into a 90-mL centrifuge tube. Wash the filter with hot water. Discard the residue. Continue with **Determination**.

C. Dynaweb filter.

1. To a 8.9 cm diameter or 1/4 of an 20.3 cm diameter Dynaweb filter in a 600-mL beaker, add 1 mL of lead carrier and a weighed aliquot (30-80 mBq) of ^{208}Po or ^{209}Po tracer solution.

2. Add 300 mL of HNO_3 and digest on a medium hot plate.

3. Evaporate to about 25 mL. If the solution is not clear, repeat the evaporation with additional HNO_3.

4. Add about 200 mL of water to polymerize the Dynaweb material.

5. Filter with suction through a Millipore filter and wash with water. Discard the filter and polymerized Dynaweb material.

6. Transfer the filtrate back into the original beaker.

7. Reduce the volume to 25 mL. Repeat Steps 4-6 until the Dynaweb material is completely removed.

8. Transfer the solution to a 90-mL centrifuge tube. Continue with **Determination**.

DETERMINATION

1. Reduce the volume to about 5 mL in a steam bath. Add 50 mL of water.

2. Adjust the pH to 3.5-4 with NH_4OH. Add 5 mL of thioacetamide solution. Digest in a steam bath for 1 h.

3. Cool, centrifuge, and decant the supernate. Discard the supernate.

4. Dissolve the precipitate in 2 mL of HCl. Add 50 mL of water.

5. Adjust the pH to 3.5-4 with NH_4OH. Add 2 mL of thioacetamide solution. Digest in a steam bath for 1 h.

6. Cool, centrifuge, and decant the supernate. Discard the supernate.

7. Dissolve the precipitate in 1 mL of HCl. Dilute the solution to 25 mL with water.

8. Filter the solution by gravity through a Whatman No. 41 filter paper into a prepared deposition cell. Wash the filter with hot 0.5N HCl. Discard the filter.

9. Add 1 mL of saturated ascorbic acid solution to the cell.

10. Place the cell in an 80°C water bath.

11. Stir with a Teflon stirrer for 4 h at a speed giving maximum agitation without splashing. Occasional small additions of 0.5N HCl are necessary to make up for evaporation of the solution.

12. Remove the cell from the water bath and pour off the solution into a beaker. Reserve for ^{210}Pb determination if required.

13. Dismantle the cell, rinse the disc with water, then ethanol. Air dry the disc.

14. Place the disc on a warm hotplate to dry.

15. Count the disc on an α spectrometer to resolve the ^{208}Po or ^{209}Po tracer and ^{210}Po.

LOWER LIMIT OF DETECTION (LLD)

		A	B	C	D
Counter Efficiency	(%)	40	40	40	40
Counter Background	(cps)	8.33×10^{-5}	8.33×10^{-5}	8.33×10^{-5}	8.33×10^{-5}
Yield	(%)	80	75	60	60
Blank	(cps)	0.01	0.01	0.01	0.01
LLD (400 min)	(mBq)	1.5	2.0	2.0	2.0
LLD (1000 min)	(mBq)	1.0	1.3	1.3	1.3
LLD (5000 min)	(mBq)	0.4	0.6	0.6	0.6

Solid-state alpha spectrometer:

A = H_2O

B = Vegetation

C = Soil

D = Dynaweb filter