# Polonium

Environmental Measurements Laboratory
U.S. Department of Energy

*Po-01-RC*

**POLONIUM IN WATER AND URINE**

---

Contact Person(s) : Isabel M. Fisenne

## APPLICATION

This procedure is applicable to water and urine (Hursh, 1958). Organic materials which can be converted to $Cl^-$ solutions should also lend themselves to analysis by the procedure given. Reagent blanks must be analyzed along with the samples. [**Note:** It has been shown (Fellman et al., 1989) that urine samples must be wet ashed to release polonium from metabolically labeled organic compounds. The procedure has been modified to incorporate the destruction of organic matter.]

Polonium is quantitatively deposited on a nickel disc from a strong HCl solution. This is a very specific separation and therefore can be carried out while many other radionuclides are present in the sample.

The plated disc is $\alpha$ counted on a scintillation counter. It is also possible to use a $^{208}Po$ or $^{209}Po$ tracer and count on an $\alpha$ spectrometer to measure chemical yield and the activity of the sample.

## SPECIAL APPARATUS

1. Nickel discs - made of 0.064-cm thick "commercial pure" nickel sheets. Discs are 2.2 cm in diameter with a 0.16-cm hole set 0.16 cm in from the edge. [**Note:** Coating the disc on one side with an acid resistant paint allows counting time to be cut in half.]

## SAMPLE PREPARATION

### A. Water.

1. To 1000 mL of tap water in a 1500-mL beaker, add 50 mL of HCl.

2. Evaporate to a volume of 20 mL and transfer to a 250-mL beaker. Add 100 mL of water and 100 mg of ascorbic acid.

3. Proceed to **Determination**.

### B. Urine.

1. If the time between sample collection and analysis is much greater than 1 h, the urine samples should be preserved by adding 1 mg of sulfamic acid per mL of urine and storing in a refrigerator at 3°C.

2. Measure 100 mL of urine in a graduated cylinder and transfer to a 250-mL beaker. Rinse the graduated cylinder with 20 mL of 1:1 $HNO_3$ and add to the urine.

3. Evaporate the solution to near dryness and add 5 mL portions of $HNO_3$ to destroy organic matter.

4. Convert the sample to the $Cl^-$ form by evaporating to near dryness with three successive 5-mL portions of HCl.

5. Add 20 mL of 1:1 HCl and 100 mg of ascorbic acid to the beaker.

## DETERMINATION

1. Place the beaker in a constant temperature bath at 55°C.

2. Degrease a nickel disc by dipping in $HNO_3$, followed by dipping in HCl and rinsing in water. Repeat until the surfaces of the disc are bright and shiny.

3. Suspend the disc on a glass stirring hook in the solution and stir for 2.5 h at a speed giving maximum agitation without splashing.

4. Remove the disc, rinse the stirring rod and disc with water and let dry in air.

5. Alpha count each side of the disc. Subtract background from each count and sum the two net cps.

6. Standardize the counter with a known quantity of any $\alpha$ emitter on a metal disc. Natural U plated on a similar disc is a convenient standard.

## LOWER LIMIT OF DETECTION (LLD)

| | | A | B | C |
|---|---|---|---|---|
| Counter Efficiency | (%) | 50 | 50 | 25 |
| Counter Background | (cps) | $1.675 \times 10^{-5}$ | $1.67 \times 10^{-5}$ | $8.33 \times 10^{-5}$ |
| Yield | (%) | 70 | 70 | 70 |
| Blank | (cps) | - | - | - |
| LLD (400 min) | (mBq) | 0.5 | 0.33 | 1.5 |
| LLD (1000 min) | (mBq) | 0.33 | 0.17 | 1.0 |
| LLD (5000 min) | (mBq) | 0.17 | 0.10 | 0.5 |

A = alpha scintillation counter (both sides)
B = alpha scintillation counter (one side)
C = solid-state alpha spectrometer (one side)

# REFERENCES

Fellman, A., L. Ralston, D. Hickman, L. Ayres, N. Cohen, H. Spitz and B. Robinson "The Importance Acid Digestion of Urine Prior to Spontaneous Deposition of $^{210}$Po" Health Physics, 57, 615-621 (1989)

Hursh, J. B. (Editor)
USAEC Report AECU-4024, November (1958)

*Po-02-RC*

# POLONIUM IN WATER, VEGETATION, SOIL, AND AIR FILTERS

---

Contact Person(s): Isabel M. Fisenne

## 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 $\alpha$ 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 $\alpha$ 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) | $8.33 \times 10^{-5}$ | $8.33 \times 10^{-5}$ | $8.33 \times 10^{-5}$ | $8.33 \times 10^{-5}$ |
| 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