

AN-1415-10

Rapid Determination of ²¹⁰Po in Water Samples

Summary of Method A method for the measurement of ²¹⁰Po in terrestrial water samples is described, offering significant advantages in detection limit, processing time, and resistance to chemical and radiochemical interferences over standard methods where polonium is determined following spontaneous deposition onto metal planchets. ²¹⁰Po is concentrated from up to 1L samples of ground water or 2L samples of drinking water using a calcium phosphate precipitate. ²¹⁰Po is then separated from matrix ions and potentially interfering radionuclides using a 2mL cartridge of Eichrom DGA Resin. ²¹⁰Po is measured using alpha spectrometry following bismuth phosphate microprecipitation onto an Eichrom Resolve® Filter. Chemical recoveries of polonium, determined with a ²⁰⁹Po tracer, were typically 80-90%. ²¹⁰Po measurements typically agreed to reference values to within 3-5%. A single operator can prepare batches of 12-24 samples for alpha counting in 3-4 hours. Alpha spectrometry count times will vary depending on desired detection limit and data quality objectives. Polonium determination may also be integrated into methods for the determination of actinides (Eichrom Application Note AN-1416).

Reagents

DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)

Nitric Acid (70%)

Hydrochloric Acid (37%)

Ammonium Hydroxide (listed as 28% NH₃ or 56% NH₄OH)

Hydrogen Peroxide (30%)

Deionized Water

1.25M Ca(NO₃)₂

3.2M (NH₄)₂HPO₄

²⁰⁹Po tracer

Bi standard solution (1mg/mL)

Denatured Ethanol

Equipment

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)

Cartridge Reservoir, 20mL (Eichrom AR-200-RV20)

Inner Support Tubes-PE (Eichrom AR-1000-TUBE-PE)

Yellow Outer Tips (Eichrom AR-1000-OT)

Resolve Filters in Funnel (Eichrom RF-DF25-25PP01)

50mL and 250mL Centrifuge Tubes

Centrifuge

Alpha Spectrometry System

Analytical Balance

Vacuum Pump

Stainless steel planchets (1.25 inch) with adhesive tape

Heat Lamp

Figure 1. Sample Preparation

1-2L Water Sample.
Add ²⁰⁹Po tracer.

Add 1-2mL of 30% H₂O₂.

Add 1mL 1.25M Ca(NO₃)₃ and 3mL 3.2M (NH₄)₂HPO₄. Mix Well.

Adjust to pH 9 with 56% NH₄OH. Mix.
Allow precipitate to settle.
Decant supernate to <200mL.

Transfer remaining supernate and precipitate to 250mL centrifuge tubes. Centrifuge 3500rpm for 10 minutes. Decant supernate.

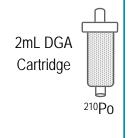
Dissolve precipitate in 10mL 9M HCI.

Transfer to 50mL centrifuge tube.
Rinse 250mL tube with 10mL 2M HCI.
Transfer tube rinse to same 50mL
centrifuge tube.

Load Solution for Po separation.

Figure 2. Polonium Separation on DGA and BiPO₄ Microprecipitation

- (1) Precondition DGA Resin with 5mL 2M HCI
- (2) Load ²¹⁰Po sample at 1-2mL/min.
- (3) Rinse sample tube with 5mL 2M HCI
- (4) Add tube rinse to DGA Resin. Elute at 1-2mL/min.
- (5) Rinse DGA Resin sequentially with:
- 5mL 2M HCI
- 15mL 0.25M HCI
- 5mL 6M HNO₃
- (6) Dispose of (1) to (5) as waste.
- (7) Strip Po with 15mL 0.05M HNO₃ at 1mL/min.



- (8) To polonium sample:
- Add 125ug Bi carrier.
- Add 0.1mL 30% H₂O₂.
- 0.75mL 3.2M (NH₄)₂HPO₄.
- Mix well.
- Add 200uL 56% NH₄OH.
- Mix well. Wait 15-20 minutes.
- (9) Set up Resolve® Filter Funnel on vacuum box.
- (10) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

Filter

assembly with

25mm, 0.1µm

Resolve™ polypropylene

- (11) Filter sample.
- (12) Rinse sample tube with 5mL DI water and add to filter.
- (13) Rinse filter funnel with 3mL DI water.
- (14) Rinse filter funnel with 1-2mL 100% ethanol.

- (15) Draw vacuum until filter is dry.
- (16) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.



- (17) Dry filter under heat lamp for 3-5 minutes.
- (18) Measure ²¹⁰Po and ²⁰⁹Po tracer by alpha spectrometry.



Method Performance ²¹⁰Po in Water

Volume			% Recovery	²¹⁰ Po (mBq/L)	²¹⁰ Po (mBq/L)	
Sample	mL	Replicates	²⁰⁹ Po tracer	Reference	Measured	% Bias
Ground Water	200	6	87.4 <u>+</u> 5.8	316	308 <u>+</u> 5	-2.5
Ground Water	200	7	82.3 <u>+</u> 3.9	1262	1289 <u>+</u> 6	2.1
Ground Water	1000	6	85.0 <u>+</u> 8.2	63.3	61.5 <u>+</u> 5.1	-2.8
Drinking Water	2000	4	80.0 <u>+</u> 9.6	63.3	61.1 <u>+</u> 6.2	-3.5

6-12 hour count time

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, Robin C. Utsey, Daniel R. McAlister, "Rapid determination of ²¹⁰Po in water samples," *J. Radioanal. Nucl. Chem.*, 298(3), 1977-1989 (2014).