

# Rapid Determination of Actinides and $^{210}\text{Po}$ in Water

**Summary of Method** A method for the measurement of  $^{210}\text{Po}$  and actinides 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.  $^{210}\text{Po}$  and actinides are concentrated from up to 1L samples of ground water or 2L samples of drinking water using a calcium phosphate precipitate.  $^{210}\text{Po}$  and actinides are then separated from matrix ions and potentially interfering radionuclides using stacked 2mL cartridge of Eichrom TRU and DGA Resin.  $^{210}\text{Po}$  and actinides are measured using alpha spectrometry following bismuth phosphate and cerium fluoride microprecipitation, respectively, onto Eichrom Resolve® Filters. Tracer recoveries averaged  $81.5 \pm 2.6\%$  for  $^{209}\text{Po}$ ,  $93.4 \pm 6.8\%$  for  $^{242}\text{Pu}$ ,  $100.2 \pm 6.9\%$  for  $^{243}\text{Am}$  and  $96.6 \pm 2.5$  for  $^{232}\text{U}$ . Measured values typically agreed to within 3-5% of reference values. A single operator can prepare batches of 12-24 samples for alpha counting in 4-6 hours. Alpha spectrometry count times will vary depending on desired detection limit and data quality objectives.

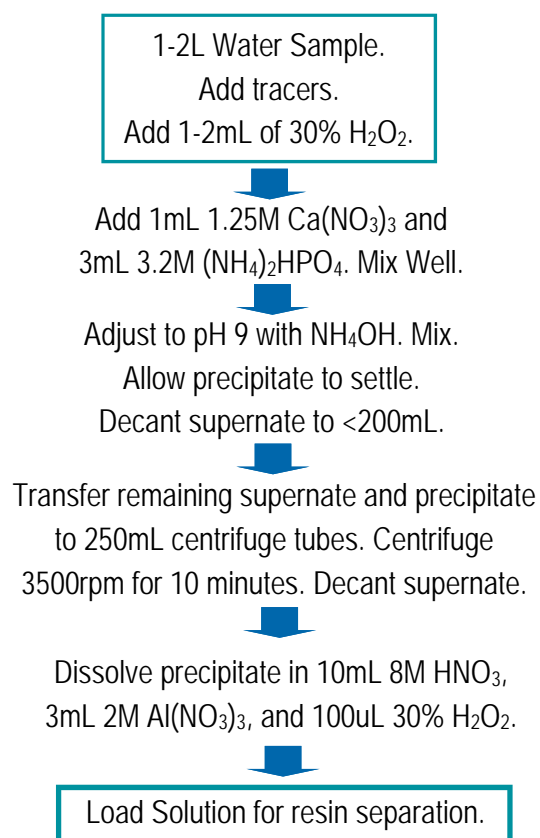
## Reagents

TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)  
 DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)  
 Ammonium Hydroxide (Listed as 28%  $\text{NH}_3$  or 56%  $\text{NH}_4\text{OH}$ )  
 $^{209}\text{Po}$ ,  $^{232}\text{U}$ ,  $^{243}\text{Am}$ ,  $^{242}\text{Pu}$  tracers  
 Bi and Ce carriers (1mg/mL)  
 Nitric Acid (70%)                      Hydrochloric Acid (37%)  
 Hydrofluoric Acid (49%)            Hydrogen Peroxide (30%)  
 Deionized Water                      3.2M  $(\text{NH}_4)_2\text{HPO}_4$   
 1.25M  $\text{Ca}(\text{NO}_3)_2$                   2M  $\text{Al}(\text{NO}_3)_3$   
 10% (w:w)  $\text{TiCl}_3$                     Denatured Ethanol  
 Oxalic acid/Ammonium Oxalate


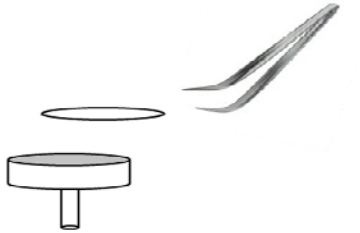
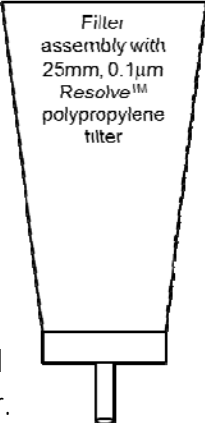
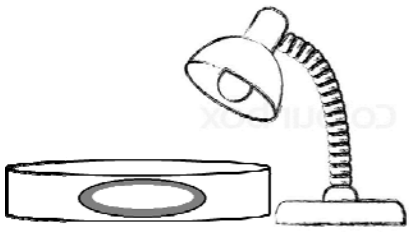
## Equipment

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  
 Heat Lamp  
 Stainless steel planchets (1.25 inch) with adhesive tape

**Figure 1. Sample Preparation**



**Figure 2. TRU/DGA Separation and Source Preparation**

<p>(1) Precondition TRU/DGA Resin with 5mL 8M HNO<sub>3</sub>.</p> <p>(2) Load samples.</p> <p>(3) Rinse sample tube with 5mL 8M HNO<sub>3</sub>, and add tube rinse to TRU/DGA.*</p> <p>(4) Rinse TRU/DGA with: -10mL 10M HNO<sub>3</sub> -15mL 4M HCl.</p> <p>(5) Separate TRU and DGA.</p>		<p>(10) Strip Am/Cm from DGA with 12mL 0.25M HCl. Add 0.2mL 30% H<sub>2</sub>O<sub>2</sub>.</p> <p>(11) Rinse DGA with 6M 8M HNO<sub>3</sub>.</p> <p>(12) Strip Po from DGA with 15mL 0.05M HNO<sub>3</sub>. Add 0.1mL 30% H<sub>2</sub>O<sub>2</sub>.</p> <p>(13) <u>Po samples</u>: Add 125ug Bi carrier, 0.75mL 3.2M (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>. Mix well. Add 0.2mL 56% NH<sub>4</sub>OH. Mix well. Wait 15-20 minutes.</p> <p><u>Actinide samples</u>: Add 50-100ug Ce carrier. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.</p> <p>(14) Set up Resolve® Filter Funnel on vacuum box. Wet filter with 3mL 80% ethanol and 3mL DI H<sub>2</sub>O.</p> <p>(15) Filter sample.</p> <p>(16) Rinse sample tube with 5mL DI water and add to filter.</p> <p>(17) Rinse filter funnel with 3mL DI water.</p>	<p>(18) Rinse filter funnel with 2mL 100% ethanol.</p> <p>(19) Draw vacuum until filter is dry.</p> <p>(20) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.</p>
<p>(6) Strip Pu from TRU with 12mL 3M HCl-0.02M TiCl<sub>3</sub>. Add 0.5mL 30% H<sub>2</sub>O<sub>2</sub>.</p> <p>(7) Rinse TRU with: -5mL 8M HNO<sub>3</sub> -20mL 1.5M HCl-0.15M HF.</p> <p>(8) Strip U from TRU with 15mL 0.1M ammonium bioxalate. Add 0.5mL TiCl<sub>3</sub> for CeF<sub>3</sub> ppt.</p> <p>(9) Rinse DGA: -5mL 3M HCl -12mL 3M HNO<sub>3</sub>-0.25M HF -5mL 4M HCl</p>			<p>(21) Dry filter under heat lamp for 3-5 minutes.</p> <p>(22) Measure Po and actinides by alpha spectrometry.</p>
			

\*Adding 50uL 30% H<sub>2</sub>O<sub>2</sub> to the tube rinse can improve U recoveries and decontamination in Pu(Np) fractions.

**Method Performance <sup>210</sup>Po and Actinides in Water**

Analyte	Tracer	% Recovery		Analyte (mBq/L) Reference	Analyte (mBq/L) Measured	% Bias
		of tracer				
<sup>210</sup> Po	<sup>209</sup> Po	81.5 ± 2.6		1584	1660 ± 3	4.8
<sup>238</sup> Pu	<sup>242</sup> Pu	93.4 ± 6.8		370	381 ± 4	3.0
<sup>241</sup> Am	<sup>243</sup> Am	100.2 ± 6.9		370	381 ± 3	3.0
<sup>244</sup> Cm	<sup>243</sup> Am	100.2 ± 6.9		328	328 ± 4	0.1
<sup>238</sup> U	<sup>232</sup> U	96.6 ± 2.5		655	627 ± 4	-4.4

200mL ground water samples, 6 replicates  
8-16 hour count time

**References**

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