

# Rapid Determination of Np/Pu in Water Samples by ICP-MS

**Summary of Method** Plutonium and Neptunium are separated and concentrated from 200mL water samples. Pu and Np are concentrated from the water sample using a calcium phosphate precipitation. Pu-Np are separated on 2mL cartridges of Eichrom TEVA and DGA resins. Pu-Np are measured by ICP-MS. Measured values for  $^{239}\text{Pu}$ ,  $^{242}\text{Pu}$ , and  $^{237}\text{Np}$  agreed to within 1-4% of reference values, while  $^{237}\text{Np}$  agreed to within 15%. Decontamination factors of  $>10^6$  were achieved for Pu over U ( $^{238}\text{U}$ -H can interfere with the measurement of  $^{239}\text{Pu}$  by ICP-MS). Sample preparation for batches of 12 samples can be completed by a single operator in <4 hours.

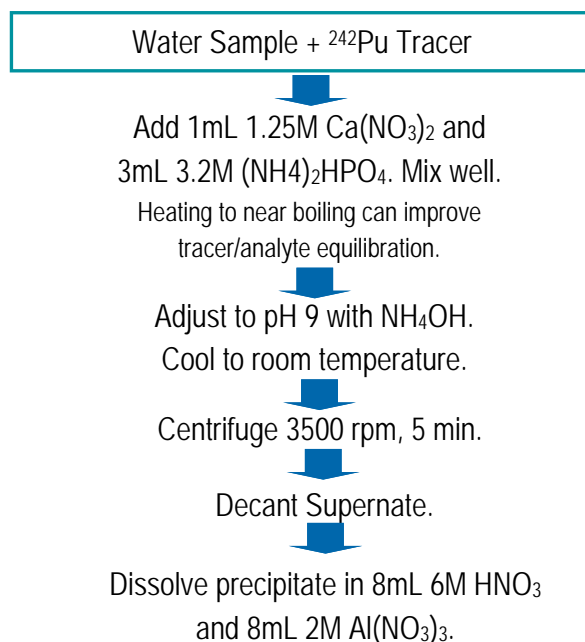
## Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)  
 DGA Resin, Normal, 2mL Cartridges (Eichrom DN-R50-S)  
 Iron carrier (50mg/mL Fe, as ferric iron nitrate)  
 $^{242}\text{Pu}$  tracer                      1.25M  $\text{Ca}(\text{NO}_3)_2$   
 3.2M  $(\text{NH}_4)_2\text{HPO}_4$               Deionized Water  
 2M  $\text{Al}(\text{NO}_3)_3$                        $\text{HNO}_3$  (70%)  
 HCl (37%)                               $\text{NH}_4\text{OH}$   
 HF (49%) or NaF                       $\text{NaNO}_2$   
 Sulfamic Acid                          Ascorbic Acid  
 Hydroxylamine Hydrochloride

## 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)  
 600mL Glass beakers  
 50mL and 250mL Centrifuge Tubes  
 Centrifuge  
 Hot Plate  
 Analytical Balance  
 Vacuum Pump  
 ICP-MS System

## Figure 1. Sample Preparation



**Figure 2. Actinide Separation on TEVA - DGA**

<p>Cool samples to room temp. Fix valence by adding: (mix between steps)          -0.5mL 1.5M sulfamic acid          -40uL 50mg/mL Fe carrier          -1.5mL 1M ascorbic acid (Wait 3 min)          -1mL 3.5M NaNO<sub>2</sub></p>	<p>(8) Rinse TEVA with 5mL 3M HNO<sub>3</sub>.          (9) Strip Np from TEVA with 14mL 0.25M HCl-0.005M HF-0.01M Hydroxylamine hydrochloride.</p>	
<p>(1) Precondition 2mL TEVA, cartridges with 5mL 8M HNO<sub>3</sub>.          (2) Load Sample.          (3) Rinse centrifuge tube with 5mL 6M HNO<sub>3</sub>. Add to TEVA.*          (4) Rinse cartridges with:          -30mL 3M HNO<sub>3</sub>          -15mL 9M HCl (Th removal)</p>	<p>(10) Rinse DGA with:          -5mL 8M HNO<sub>3</sub>          -20mL 0.1M HNO<sub>3</sub>          -10mL 0.05M HNO<sub>3</sub>.          (11) Strip Pu from DGA with 11mL 0.02M HCl-0.005M HF-0.01M Hydroxylamine hydrochloride.</p>	
<p>(5) Add 2mL DGA cartridge below TEVA.**          (6) Strip Pu from TEVA onto DGA with 15mL 3M HNO<sub>3</sub>-0.1M Ascorbic Acid-0.02M Fe<sup>2+</sup>.          (7) Separate TEVA and DGA.</p>	<p>(12) Measure <sup>237</sup>Np and Pu by ICP-MS.</p>	



\* Adding 50uL of 30% H<sub>2</sub>O<sub>2</sub> to 6M HNO<sub>3</sub> tube rinse can help improve U decontamination.

\*\* Adding a 1mL UTEVA cartridge between TEVA and DGA can provide additional uranium decontamination.

## References

- 1) Sherrod L. Maxwell, Brian K. Culligan, Vernon D. Jones, Sheldon T. Nichols, Gary W. Noyes, "Rapid determination of <sup>237</sup>Np and Pu isotopes in water by ICP-MS and alpha spectrometry," *J. Radioanal. Nucl. Chem.*, 287(1), 223-230 (2011).
- 2) Sherrod L. Maxwell, Brian K. Culligan, Vernon D. Jones, Sheldon T. Nichols, Gary W. Noyes, Maureen A. Bernard, "Rapid Determination of <sup>237</sup>Np and Plutonium Isotopes by ICP-MS and Alpha Spectrometry," *Health Physics*, 101(2), 180-186 (2011).