

THE LEADER IN ENVIRONMENTAL TESTING

# Solving Analytical Challenges with Eichrom "Tools"

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#### **Purpose of Presentation**

- Explore a couple of challenges/opportunities for utilizing Eichrom as a partner in analyses
- Stimulate others to utilize Eichrom resources to help solve chemistry issues in order to supply clients with better data
- May seem like a "sales pitch" for Eichrom
- It got me here!



#### **Challenge #1**

- DOE Client "support of characterization of uranium salvage solutions for disposal determination."
- Estimated Rad: pCi/g
   231000 pCi/L Tc-99; 3780 pCi/L U-235; 2.72% U-235
   231 pCi/g (3,780,000 pCi/L)
- Analytical requested:
  - ~ Total U, U-235, %U-235 (ICP-MS)
  - ~ Am-241, Np-237, Iso-Pu, Iso-Th (~5 pCi/L)
  - ~ Gross α/β, Gamma Spec



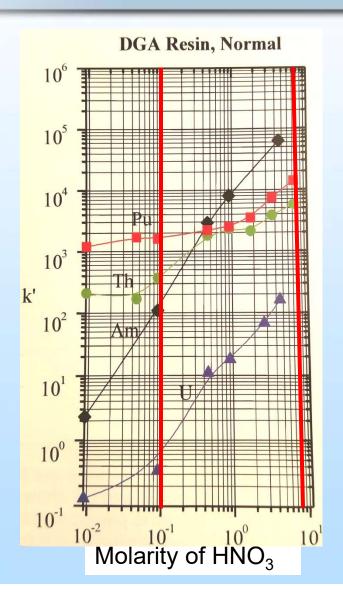
#### **High Uranium Sample**

- Separate relatively small activity of alpha emitters (looking for ~5 pCi/L MDC) from a large amount of uranium.
- Suggest 5 mL aliquot
- Original estimate: 380 mg U in 5 mL (76 mg/mL)
  - ~ 5 samples actual results (ICP-MS):
    - Total U: 73 130 mg/mL
    - U-235 enrichment: 2.2% 5.4%
    - U-234: 89 398 uCi/L
    - U-238: 24 42 uCi/L



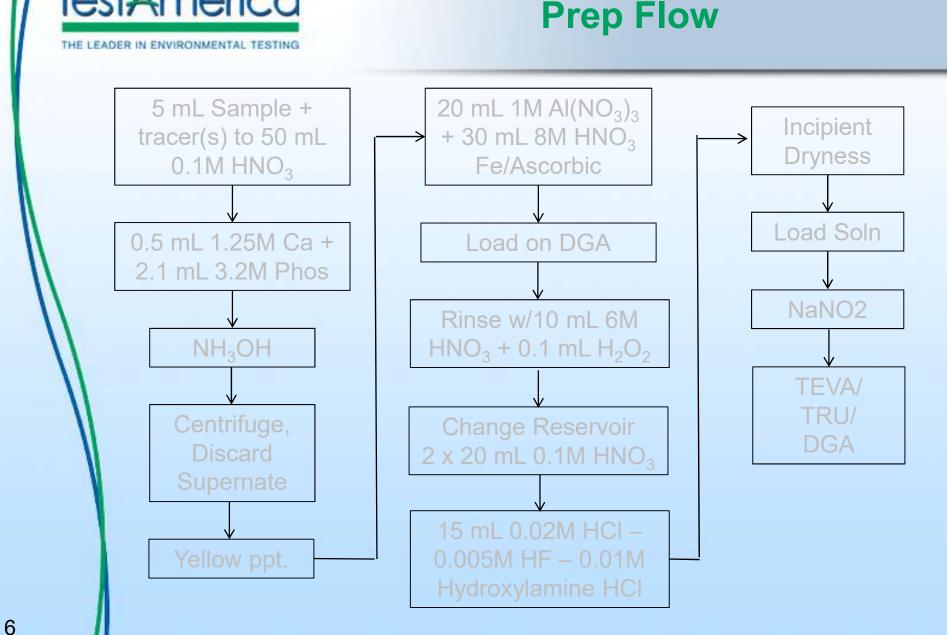
#### Tool(s)

- Technical Resources
  - ~ Product Catalog
- Resin(s)
  - ~ DGA
- Personnel
  - Dan McAlister and Dominic Silvestri



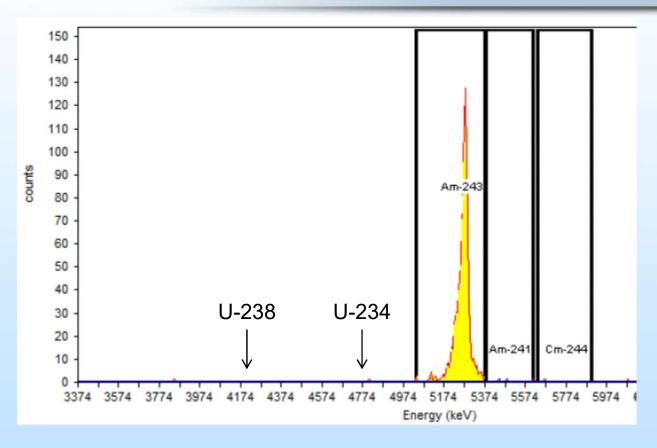


#### **Prep Flow**





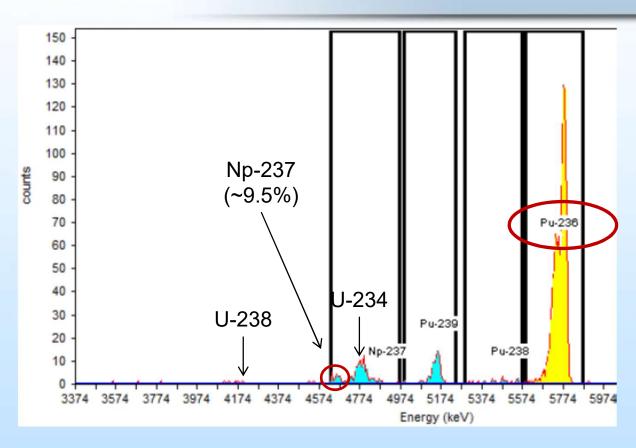
#### **Am-241 Results**



- MDC: 7 10 pCi/L; Tracer Recoveries > 90%
- Activity seen: < MDC 4740 pCi/L</li>



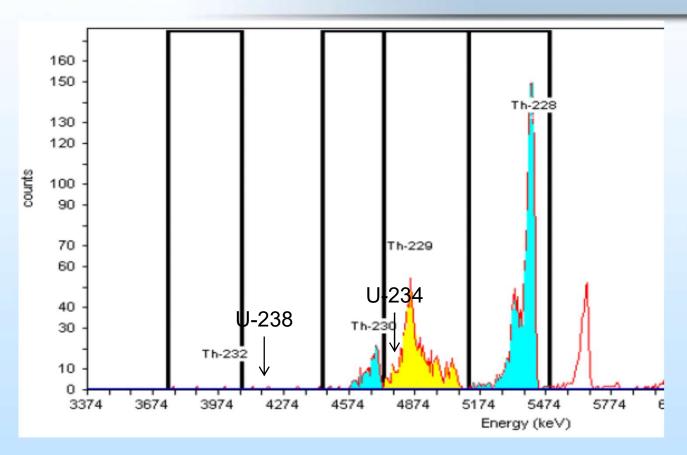
#### Pu/Np Results



- MDC: 4 6 pCi/L; Tracer Recoveries > 90%
- Activity seen: < MDC 35,600 pCi/L</li>



#### **Th-230**, **Th-232** Results



- MDC: 9 15 pCi/L; Tracer Recoveries > 90%
- Activity seen: 273 8,260 pCi/L



#### **Notes for Future**

- Np-237 Spike (LCS) was about 50% recovery
  - ~ Pu-238 and Pu-239 recoveries ~100%
  - Likely due to valence of Np(V) instead of Np(IV)
    - Taking to dryness might oxidize to Np(V)
    - Next time use both the Fe/Ascorbic and NaNO2 adjustments before the DGA load as well as after bringing up in load solution for the 3-stack.





#### **Challenge #2**

- High quench in Fe-55 LSC samples
  - ~ Due to HNO3
    - Chemical quench due to the NO3
    - Color quench due to yellowing





#### Tool(s)

- Technical Resources
  - Application Notes
- Personnel
  - Dan McAlister and Dominic Silvestri

### eichrom<sup>\*</sup>

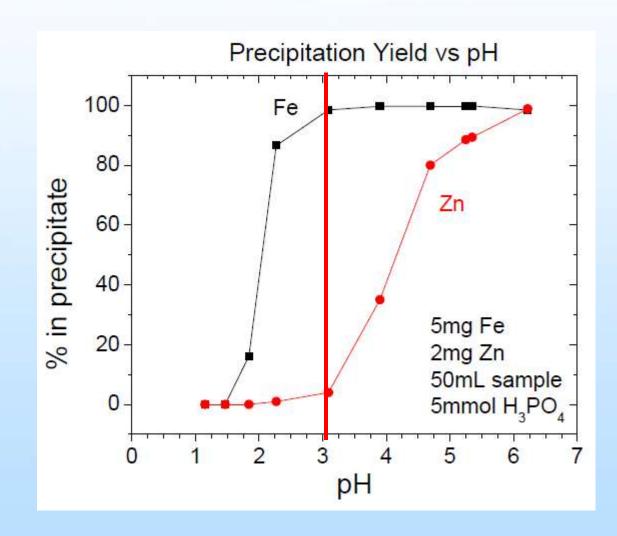
## Measurement of <sup>55</sup>Fe in Water (TRU Separation)

AN-1612-10

**Summary of Method** <sup>55</sup>Fe is separated and measured from up to 500mL aliquots of water. Samples are preconcentrated by evaporation or ferric hydroxide precipitation and purified on 2mL cartridges of TRU Resin. Holdback carriers, 0.1-1mg each of Zn, Mn, Cs, Nb, Zr, and Co are added to improve separation from these nuclides of these elements. An iron phosphate precipitate at is used to prepare samples for liquid scintillation counting. Chemical recovery of iron is determined by ICP-AES measurement of 2mg of stable iron carrier. <sup>55</sup>Fe may also be determined from chloride media using TEVA resin (Eichrom AN-1611). AN-1612 provides higher Zn decontamination and can be incorporated into TEVA-TRU actinide separations, but is limited to 2mg total Fe per 2mL cartridge. AN-1611 can process 5-6mg of Fe, but is less rugged for Zn decontamination.

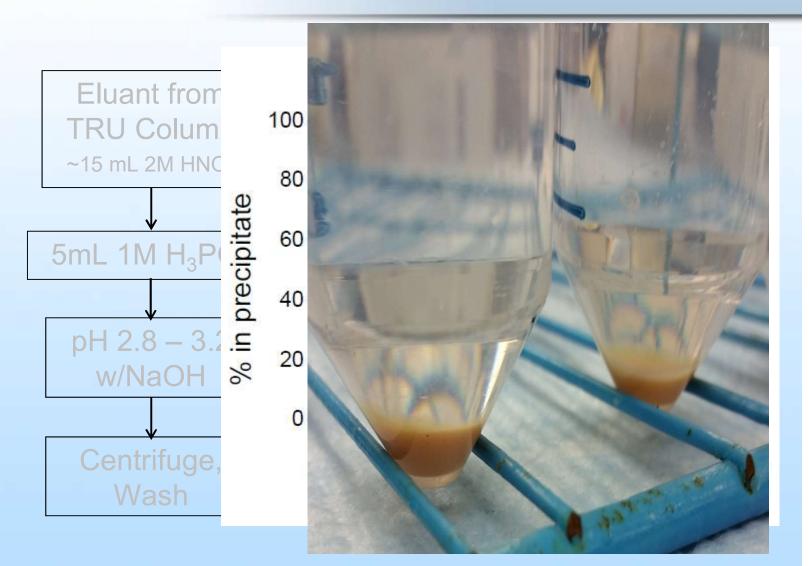


#### **Iron Phosphate**





#### **Prep Flow**





#### **Final LSC Vial**



tSIE = 260.12

Eff = 0.205



tSIE = 170.77 tSIE = 14.37

Eff = 0.131 Eff = Off Curve



#### **Acknowledgements/Contacts**

- Dan McAlister Eichrom
- Chelsea Mazariegos TAL Prep Analyst
- Paul Mallinckrodt TAL Prep Analyst
- Amanda Dick TAL Count Analyst
- Sherrod Maxwell SRNS
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### Questions?