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Welcome

Eichrom Technologies User's Group Meeting

Held at the 61th RRMC Conference – Wednesday, 28 October 2015
Sheraton Iowa City Hotel
Iowa City, IA

Eichrom User's Group Meeting

- **Quality, A Brief History** – Sarah McAlister
- **Purity of DGA Normal for Po Separations** – Daniel McAlister
- **Rapid Methods for Ra-226 and Ra-228: An Update**

  – Sherrod Maxwell
- **Pb-Resin: New Approaches, Challenges, and Troubleshooting**

  – Dustin May, Andrew Nelson, Michael Schultz
- **Uranium Valence Control for Analytical Separations**

  – Daniel McAlister
- **Removal of Tc-99 Interference from Ni-63 Analysis of Water Sample** – Terry Romanko
- **Additional Questions and Answers** – You, Our Customers

Eichrom Technologies

Michael Fern	President
Shari Tegel	Director of Finance and Administration
Joel Williamson	Director of Operations
J van de Linde	Director of Sales
Jill Bryant	Quality System Coordinator
Daniel McAlister, Ph.D.	Senior Chemist
Sarah McAlister	Quality Manager
Terence O'Brien	Technical Sales Scientist
Phil	

Thank you for collaborating, asking, questioning, testing, pushing, buying and joining us today.

What's NEW with Eichrom

- 25th Anniversary Celebration – Founded February 1990

- Revised and New Methods

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New and Revised Methods

Analytical Procedure

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AMERICIUM, NEPTUNIUM, PLUTONIUM, THORIUM, CURIUM, AND URANIUM IN WATER

(WITH VACUUM BOX SYSTEM)

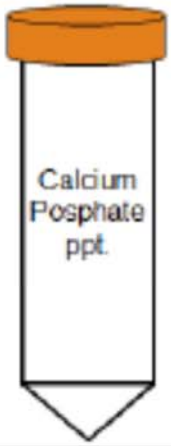
1. SCOPE

1.1. This is a method for the separation of americium, neptunium, plutonium, thorium, curium and uranium in water. After completing this method, source preparation for measurement of actinides by alpha spectrometry is performed by electrolytic deposition onto stainless steel planchets (Eichrom Method SPA02) or by rare earth fluoride microprecipitation onto polypropylene filters (Eichrom Method SPA01).

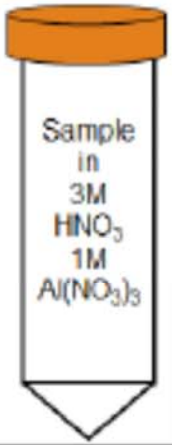
Flow charts for easy method application

1) Aliquot up to 1000mL of water into glass beaker.
2) Add 5mL concentrated HNO_3 and add yield tracers.
3) Add 1mL of 1.25M $Ca(NO_3)_2$.
4) Heat samples at medium setting for 30-60 minutes.
5) Remove samples from heat.
6) Add 0.75mL of phenolphthalein and 3mL of 3.2M $(NH_4)_2HPO_4$.
7) While stirring sample, slowly add conc. NH_4OH until reaching pH 9.
8) Cool to room temperature. Allow precipitate to settle or centrifuge.
9) Decant supernate and discard as waste.
10) Transfer precipitate to centrifuge tube with DI water.
11) Centrifuge ~10minutes at 2000rpm. Decant supernate.
12) Add 10mL DI water to ppt. Mix well. Centrifuge. Decant supernate. Dissolve ppt with 5mL conc. HNO_3. Transfer to 100mL beaker.
13) Rinse centrifuge tube with 2-3mL conc. HNO_3. Transfer to 100mL beaker. Evaporate to dryness.
14) Dissolve reside in 16mL 3M HNO_3-1M $Al(NO_3)_3$. Add 1mL 1.5M Sulfamic Acid, 0.5 mL Fe, and 1mL 1M Ascorbic Acid. Swirl to mix. Wait 3-5 minutes.
15) Add 1mL 3.5M $NaNO_2$. Swirl to mix.

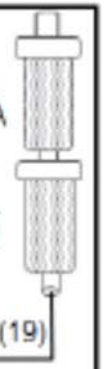
Water Sample in glass beaker. Acidify pH 2.



Centrifuge. Decant Supernate. Wash ppt with H_2O. Centrifuge. Decant.



16) Precondition TEVA-TRU with 5mL 3M HNO_3.
17) Load sample onto TEVA-TRU. Allow liquid to drain. TEVA retaines U. TRU retains Am and Pu.
18) Rinse sample tube with 5mL 3M HNO_3. Add rinse to TEVA-TRU. Allow liquid to drain.
19) Rinse TEVA-TRU with 5mL 3M HNO_3. Allow liquid to drain.
20) Separate TEVA and TRU cartridges.



(16) (17) (18) (19) → Waste

21) Rinse TEVA column with 10mL 3M HNO_3.
22) Place clean centrifuge tube below TEVA. Strip Th with 15mL 9M HCl.
23) Rinse TEVA column with 20mL 5M HCl-0.05M oxalic acid. Discard to Waste.
24) Place clean centrifuge tube below each TEVA. Strip Pu-Np with 20mL 0.1M HCl-0.05M HF-0.03M $TiCl_3$.

(21) (23) → Waste
(22) → Th sample to source preparation
(24) → Pu-Np sample to source preparation

25) Place clean centrifuge tubes below TRU. Strip Am with 15mL of 4M HCl.
26) Rinse TRU with 12mL 4M HCl-0.1M HF. Discard as waste.
27) Place a clean centrifgue tube below each cartridge. Strip U with 15mL 0.1M ammonium bioxalate.

(26) → Waste
(25) → Am sample to source preparation
(27) → Pu sample to source preparation

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Accountability involves taking personal responsibility for the **successful outcome** of an action, task or project.

It requires a focus, from the very beginning, on doing **what is necessary** to achieve success regardless of what others may do or fail to do.

ACCOUNTABILITY

Agenda for the Eichrom User's Group Meeting

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What's NEW with Eichrom

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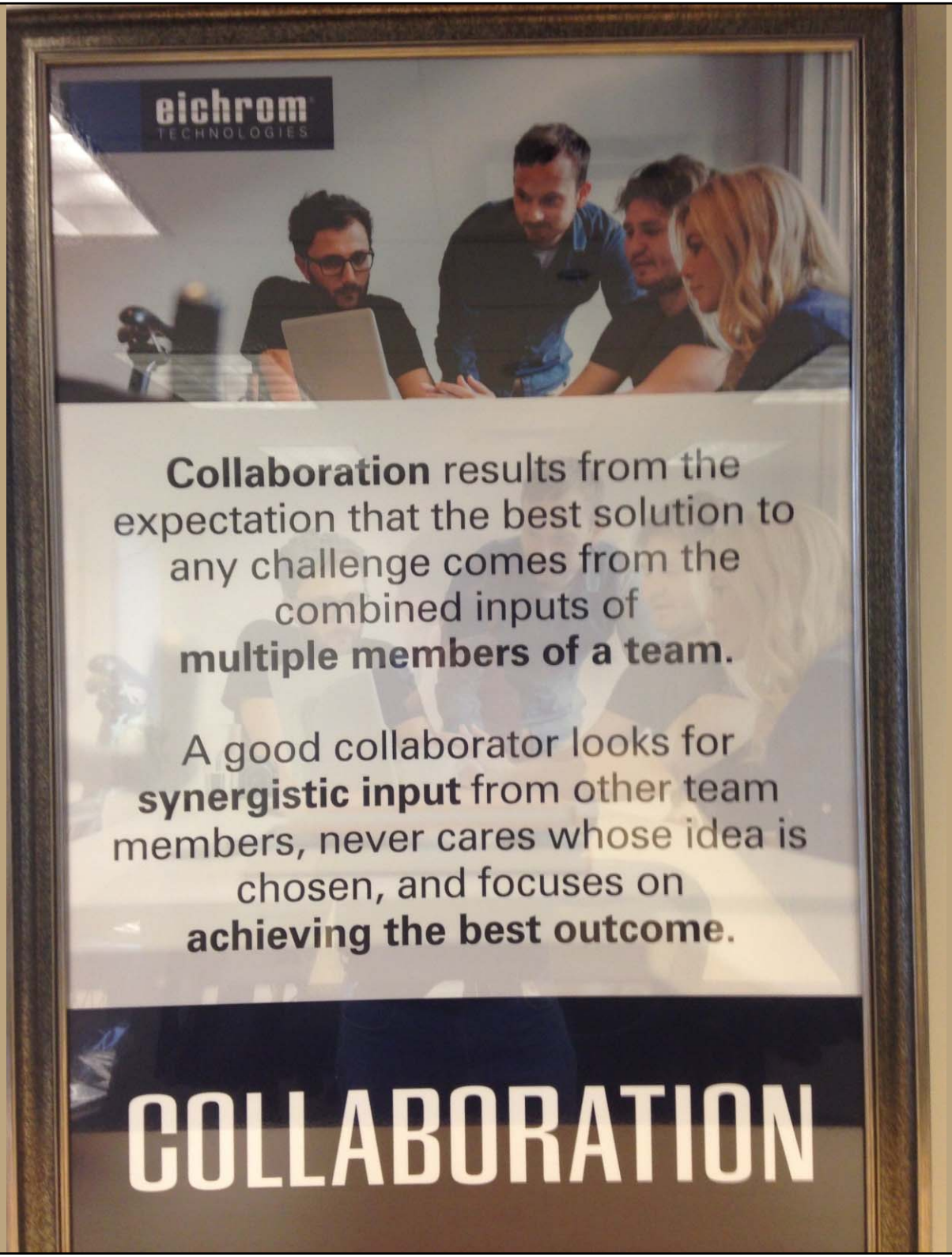
- New Application Notes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TECHNOLOGIES
Collaboration results from the expectation that the best solution to any challenge comes from the combined inputs of multiple members of a team.
A good collaborator looks for synergistic input from other team members, never cares whose idea is chosen, and focuses on achieving the best outcome.
COLLABORATION

Application Notes

Number	Title
AN-1401	Rapid Determination of ^{226}Ra in Emergency Urine and Water
AN-1402	Rapid Determination of Sr in Emergency Milk Samples
AN-1403	Rapid Determination of Sr in 50g Soil Samples
AN-1404	Rapid Determination of Sr in 1-2 Liter Seawater Samples
AN-1405	Rapid Determination of Sr in Vegetation Samples
AN-1406	Rapid Determination of Actinides in Vegetation Samples
AN-1407	Rapid Determination of Sr in Animal Tissue Samples
AN-1408	Rapid Determination of Actinides in Animal Tissue Samples
AN-1409	Rapid Determination of Sr in Building Materials
AN-1410	Rapid Determination of Sr in Emergency Urine Samples
AN-1411	Rapid Determination of Sr in Emergency Water Samples
AN-1412	Rapid Determination of Actinides in Emergency Urine Samples
AN-1413	Rapid Determination of Actinides in Emergency Water Samples
AN-1414	Rapid Determination of ^{90}Sr in Up to 40 Liter Seawater Samples
AN-1415	Rapid Determination of ^{210}Po in Water Samples
AN-1416	Rapid Determination of Actinides and ^{210}Po in Water
AN-1417	Rapid Determination of $^{226/228}$Ra in Water Samples
AN-1418	Rapid Determination of ^{226}Ra in Water Samples
AN-1419	Rapid Determination of ^{226}Ra in Concrete and Brick

Example of layout of application notes

Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Hydrofluoric Acid (49%) or Sodium Fluoride
Iron Carrier (50mg/mL Fe, as ferric nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrogen Peroxide (30%)
Deionized Water
Cerium Carrier (1mg/mL)
2M $Al(NO_3)_3$
Sodium nitrite
Sulfamic acid
Ascorbic acid
10% (w:w) $TiCl_3$
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)
Muffle Furnace
Hot Plate
Analytical Balance
600mL Glass Beakers
Stainless Steel planchets with adhesive
Vacuum Pump
Alpha Spectrometry System
Heat Lamp

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Figure 1. Sample Preparation

Up to 200g Tissue in 600mL glass beaker

↓

Add tracers

↓

Add 80mL 70% HNO_3, and 20mL 37% HCl.

↓

Digest on hotplate medium setting until complete dryness.

↓

Remove from hot plate and cool.

↓

Carefully add 3mL 70% HNO_3 and 3mL 30% H_2O_2 (Foaming may occur).

↓

Evaporate to dryness on hot plate.

↓

Muffle at 200°C for 10 minutes, 300°C for 1 hour, and 550°C over night.

↓

Remove samples from muffle oven and cool.

↓

Wet ash samples with 5mL 70% HNO_3 and 5mL 30% H_2O_2, until residue is white. Additional muffling at 550°C may be necessary.

↓

Dissolve residue in 12mL 6M HNO_3 and 12mL 2M $Al(NO_3)_3$. Add 3M HNO_3 as necessary to complete dissolution.

↓

Adjust valence states of actinides by adding (mix between each addition):
0.5mL 1.5M Sulfamic acid, 10uL 50mg/mL Fe carrier, 1.25mL 1M Ascorbic acid,

Separation and filter preparation

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

(1) Precondition stacked 2mL TEVA, TRU, DGA with 10mL 3M HNO_3.
(2) Load sample solution.
(3) Rinse sample tube with 5mL 6M HNO_3.** Add tube rinse to cartridges.
(4) Rinse cartridges with 10mL 3M HNO_3.
(5) Separate TEVA, TRU, and DGA cartridges.

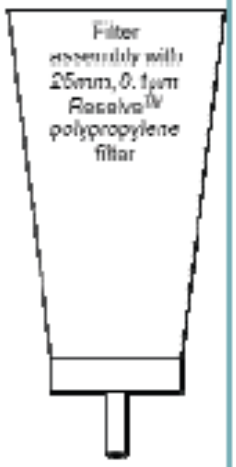
(6) Rinse TEVA cartridge with:
-10mL 3M HNO_3
-20mL 9M HCl(remove Th)
-5mL 3M HNO_3
(7) Strip Pu(Np) from TEVA with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

(8) Rinse DGA with 8mL 0.1M HNO_3.
(9) Place TRU cartridge above DGA .
(10) Strip Am/Cm from TRU onto DGA with 15mL 3M HCl.
(11) Separate TRU and DGA. Set TRU aside for U recovery.
(12) Rinse DGA with:
-5mL 3M HCl
-3mL 1M HNO_3
-15mL 0.05M HNO_3

(13) Strip Am and Cm from DGA with 10mL 0.25M HCl.

(14) Rinse TRU cartridge with 15mL 4M HCl-0.2M HF-0.002M $TiCl_3$.
(15) Strip U from TRU with 15mL of 0.1M ammonium bioxalate.
(16) Add 0.5mL 10% $TiCl_3$ to U samples, 0.5mL 30% H_2O_2 to Pu and 0.2mL 30% H_2O_2 to Am/Cm samples.

(17) Add 50-100ug Ce carrier to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.
(18) Set up Resolve® Filter Funnel on vacuum box.
(19) Wet filter with 3mL 80% ethanol followed by 3mL DI water.
(20) Filter sample.
(21) Rinse sample tube with 5mL DI water and add to



(22) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(23) Draw vacuum until filter is dry.
(24) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.

(25) Dry filter under heat lamp for 3-5 minutes.
(26) Measure actinides by alpha spectrometry.

Application Evaluation

*Radiostrontium may also be measured by adding a 2mL + 1mL Sr Resin cartridge below DGA and following separation scheme in Eichrom application note AN-1407, "Rapid Determination of Sr in Animal Tissue Samples."

**Adding 50uL of 30% H_2O_2 to tube rinse can improve U recoveries and decontamination in Pu/Np samples.

Method Performance for 100-200g Tissue Samples

			% Tracer Recovery		
Sample	mass, g	replicates	Pu-236	Am-243	U-232
Beef	100	6	98.7 ± 5.7	97.1 ± 8.4	93.4 ± 4.7
Deer	100	59	99.3 ± 12	93.4 ± 10	90.4 ± 8.0
Fish-Bass	200	72	96.2 ± 14	102 ± 13	95.1 ± 8.1
Fish-Bream	100	57	96.6 ± 12	98.4 ± 7.7	91.1 ± 6.3
Fish-Catfish	200	69	98.3 ± 12	103.7 ± 7.6	89 ± 12
Hog	100	17	93 ± 20	96.4 ± 9.7	86 ± 15
Shelfish	100	5	101.3 ± 2.2	97.4 ± 7.1	81.7 ± 3.2

Reference Sherrod L. Maxwell, Donald M. Faison, "Rapid column extraction method for actinides and strontium in fish and other animal tissue samples," *J. Radioanal. Nucl. Chem., 275(3), 605-612* (2007).

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Break

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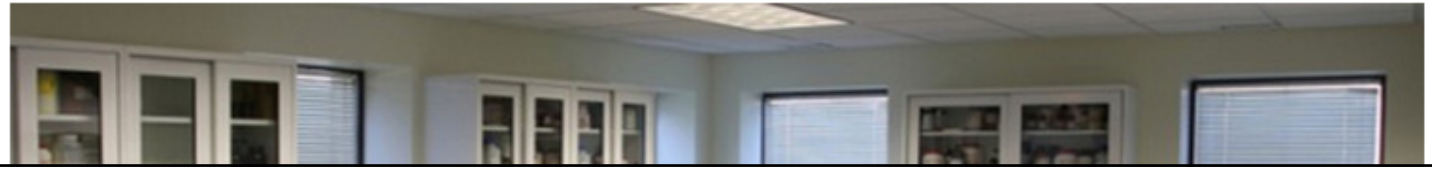
Nuclear Medicine

More than 25 years spent developing chromatography products and methods of separation and purification in the field of radiochemistry has created a high level of expertise at Eichrom. Our proprietary products are the global standard for laboratory analysis of actinides and beta-emitting fission products. They are routinely used for environmental monitoring and internal dosimetry programs at nuclear facilities in over 150 countries and on all 7 continents.

We are licensed to work with radioactive materials in our facility in suburban Chicago. All our products are manufactured under a quality management system that has been registered to the ISO 9001 standard since 1995.



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INTEGRITY

Continuing Agenda for the Eichrom UGM

- **Uranium Valence Control for Analytical Separations**

  – Daniel McAlister

- **Removal of Tc-99 Interference from Ni-63 Analysis of Water Sample** – Terry Romanko

- **Additional Questions and Answers** – You, Our Customers

- Thank You for attending the Eichrom User's Group Meeting at the 61th Radiobioassay and Radiochemical Measurements Conference

- Please take some time and discuss your work area needs

- Conference Dinner

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