AN-1700-COV

Eichrom Technologies
Application Notes 2018

Rapid Methods

New Matrices

Improved Separations

AN-1700-TOC

Eichrom Technologies Application Notes

Summary of Application Notes: The Application Notes presented in this book represent two page summaries of works published by respected scientist in the radiochemical community. These notes represent an extension to the basic methods available from the Eichrom Published Methods. The Application Notes contain some significant changes to the chemistry used in preparation and/or separation than the Eichrom methods. Application Notes will also have methodologies for different matrices or significant changes in sample size. These include new analytes or combination of analytes.

Application Notes

AN-1700-TOC

Eichrom Technologies Application Notes

eichrom®

AN-1401-10

Rapid Determination of ^{226}Ra in Emergency Urine and Water

Summary of Method ^{226}Ra is isolated from 100mL urine samples or up to 1 liter water samples and measured by alpha spectrometry as described by Maxwell, et al.[1] Radium is precipitated from samples with calcium phosphate. The calcium phosphate precipitate is dissolved in hydrochloric acid, and cation exchange chromatography is used to purify radium and barium from matrix ions. Eichrom DGA Resin is used to remove other alpha emitting nuclides from radium. Samples are prepared for radium measurement by alpha spectrometry via barium sulfate microprecipitation onto Eichrom Resolve® Filters. Sample preparation, including alpha spectrometry source preparation, for batches of 12 samples can be completed by a single operator in as little as 3-4 hours, with >90% yield of Radium. Yields can be traced with ^{133}Ba by gamma spectrometry or ^{225}Ra(^{229}Th) by alpha spectrometry. If tracing with ^{225}Ra, at least 8 hours of ingrowth time are required for the alpha emitting ^{217}At daughter of ^{225}Ra prior to alpha spectrometry measurements.

Reagents

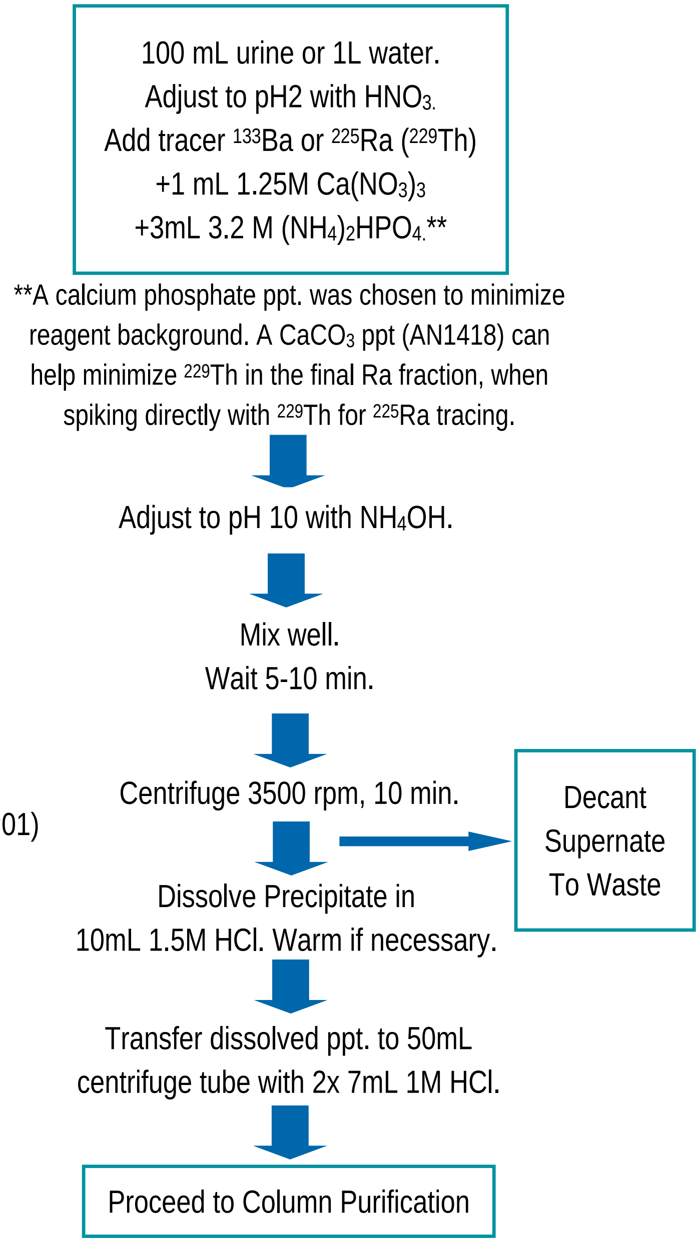
Cation Exchange Resin (Eichrom C8-B500-F-H)
DGA Resin, Normal 2mL Cartridges (Eichrom DN-R50-S)
Ammonium Hydroxide (Listed as 28% NH_3 or 56% NH_4OH)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Deionized Water
Hydrogen Peroxide (30%)
^{133}Ba or ^{225}Ra(^{229}Th) Tracer*
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
Barium Carrier (1mg/mL)
Isopropyl Alcohol
Ammonium Sulfate
Denatured Ethanol

*^{133}Ba allows immediate counting. ^{225}Ra(^{229}Th) requires >8hrs ingrowth before alpha meas. Ba/Ra recoveries can differ by up to 10% in difficult matrices.

Equipment

Plastic Chromatography Column (Eichrom AC-50E-5M)
Column Extension Funnel (Eichrom AC-20X-20M)
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 Filter in Disposable Funnel (Eichrom RF-DF-25-25PP01)
50mL and 250mL Centrifuge Tubes
Centrifuge
Hotplate
150mL Glass beakers
Vacuum Pump
Heat Lamp
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Gamma Spectrometry System (if ^{133}Ba tracer used)

Figure 1. Sample Preparation



100 mL urine or 1L water.
Adjust to pH2 with HNO_3.
Add tracer ^{133}Ba or ^{225}Ra (^{229}Th)
+1 mL 1.25M $Ca(NO_3)_3$
+3mL 3.2 M $(NH_4)_2HPO_4$.**

**A calcium phosphate ppt. was chosen to minimize reagent background. A $CaCO_3$ ppt (AN1418) can help minimize ^{229}Th in the final Ra fraction, when spiking directly with ^{229}Th for ^{225}Ra tracing.

Adjust to pH 10 with NH_4OH.

Mix well.
Wait 5-10 min.

Centrifuge 3500 rpm, 10 min.

Decant Supernate To Waste

Dissolve Precipitate in 10mL 1.5M HCl. Warm if necessary.

Transfer dissolved ppt. to 50mL centrifuge tube with 2x 7mL 1M HCl.

Proceed to Column Purification

Figure 2. Column Purification and Alpha Source Preparation

(1) Prewash 5.0g 50Wx8 200-400 mesh, cation exchange resin column[1]:

-10mL deionized water

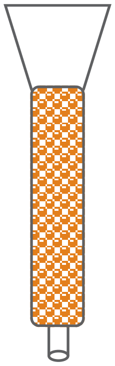
-20mL 6M HCl

-10mL 0.5M HCl

(2) Load Sample[2]

(3) Rinse 30mL 3M HCl

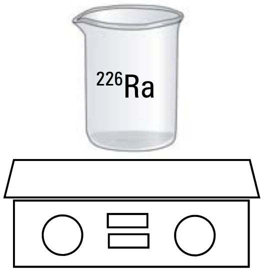
(4) Add 2mL DGA cartridge below cation exchange column.

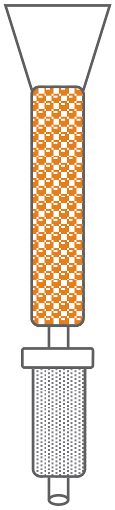


(5) Strip Ra/Ba with 25mL 5M HNO_3.

(6) Add 2mL 30% H_2O_2. Evaporate to dryness.

(7) Dissolve residue in 10mL 1.5M HCl.





(8) Add 50ug Ba carrier. Mix well.

(9) Add 3g $(NH_4)_2SO_4$ and 5mL iso-propanol. Mix well.

(10) Place in ice bath for 30 minutes.

(11) Set up Resolve® Filter Funnel on vacuum box.

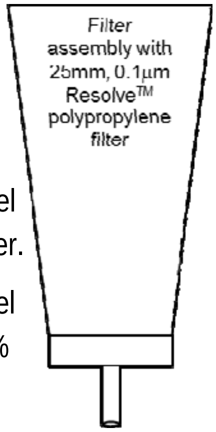
(12) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(13) Filter sample.

(14) Rinse sample tube with 5mL DI water and add to filter.

(15) Rinse filter funnel with 3mL DI water.

(16) Rinse filter funnel with 1-2mL 100% ethanol.

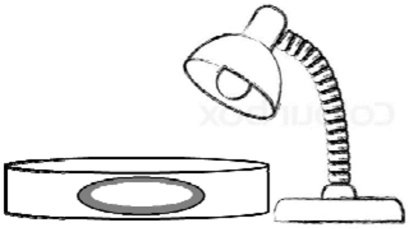


(17) Draw vacuum until filter is dry.

(18) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.

(19) Dry filter under heat lamp for 3-5 minutes.

(20) Measure ^{226}Ra and ^{225}Ra(^{217}At) by alpha spectrometry after >8 hours ^{217}At ingrowth. (^{133}Ba by gamma, if necessary.)



[1] If using ^{133}Ba tracer, 3.0g of cation exchange resin and proportionally smaller rinse volumes may be used.

[2] If tracing with ^{229}Th, a 20mL 1M HCl-1M H_3PO_4 rinse following the sample load can improve purity of final ^{226}Ra fraction.

Table 1. ^{226}Ra Analysis Results from 100mL Spiked Urine Samples

	Tracer ^{133}Ba % Recovery				^{226}Ra Measured Value (mBq/sample)			
Replicates	**Average**		**SD**	**^{226}Ra Reference Value (mBq/sample)**	**Average**		**SD**	**% Bias**
6	93	±	3	73.7	76.5	±	4.7	3.9
6	98	±	3	18.4	17.9	±	0.8	-2.7
6	92	±	5	Blank*	0.15	±	0.12	

*Calculated MDA 15 mBq/L (4 hr count, 100 mL sample)

*Calculated MDA 5 mBq/L (16 hr count, 100 mL sample)

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, Robin C. Utsey and Daniel R. McAlister, "Rapid Determination of ^{226}Ra in Emergency Urine Samples," *J. Radioanal. Nucl. Chem.*, 300(3), 1159-1166 (2014).

eichrom®

AN-1402-10

Rapid Determination of Sr in Emergency Milk Samples

Summary of Method Strontium is separated and concentrated from 100mL milk samples using a calcium phosphate precipitation. The precipitate is dissolved with nitric acid and centrifuged to remove residual protein and fat. The supernate, containing Sr, is wet ashed with HNO_3-H_2O_2 and then heated in a muffle furnace at 550°C for 30-60 minutes to destroy any residual organic matter. The muffled residue is wet ashed again with HNO_3-H_2O_2 and dissolved in HNO_3-$Al(NO_3)_3$. Strontium is separated from matrix impurities and potentially interfering radionuclides in the sample using stacked 2mL and 1mL cartridges of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter or liquid scintillation counter. Chemical yield of strontium is determined by gravimetric recovery of stable strontium or by ICP-AES measurement. Average chemical recovery of strontium is 75 ± 17%. Measured values of ^{90}Sr agreed to within 3.2% and 0.5% of reference values for 20 minute count times and 60 minute count times, respectively. The lower limit of detection for 100mL samples with 20 minute count times is 0.5Bq/L and with 60 minute count times is 0.16Bq/L. A single operator can prepare batches of 12-24 samples for ^{90}Sr measurement in less than 8 hours.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Nitric Acid (70%)
Hydrogen Peroxide (30%)
Deionized Water
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
Strontium Carrier (10mg/mL)
2M $Al(NO_3)_3$
^{90}Sr standard
Oxalic acid

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)
50mL and 250mL Centrifuge Tubes
Centrifuge
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Muffle Furnace
Hot Plate
Analytical Balance
250mL Glass Beakers
Vacuum Pump

Figure 1. Sample Preparation

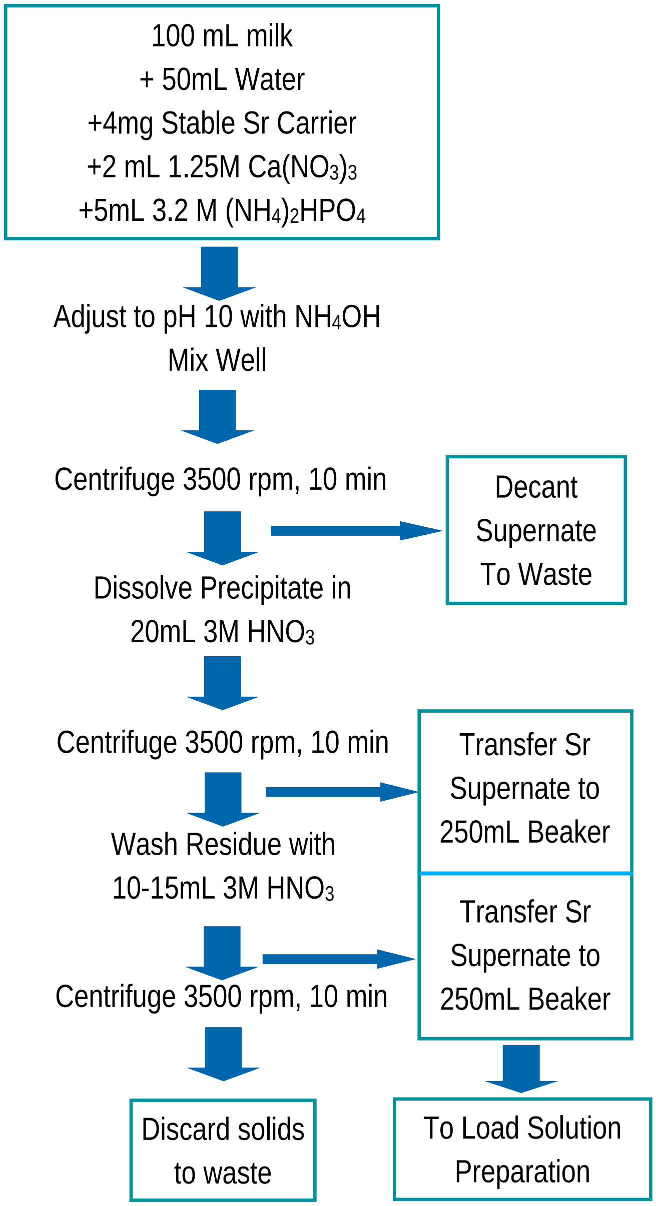


Figure 2. Load Solution Preparation and Strontium Separation

Evaporate Supernate to Dryness

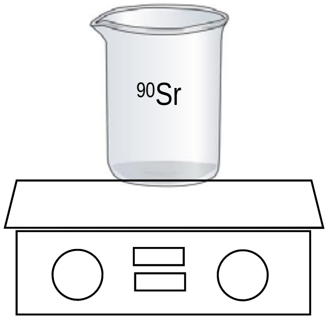
Wet ash with 5mL 70% HNO_3 and 5mL of 30% H_2O_2

Muffle at 550°C for 30-60 min.

Wet ash with 5mL 70% HNO_3 and 5mL of 30% H_2O_2

Dissolve residue in 5mL 70% HNO_3 and 5mL 2M $Al(NO_3)_3$ (warm to facilitate dissolution)

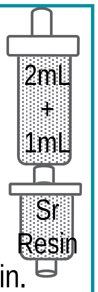
Cool to room temperature.



(1) Precondition Sr Resin with 5mL 8M HNO_3.

(2) Load sample.

(3) Rinse sample beaker with 5mL 8M HNO_3.



(4) Add beaker rinse to Sr Resin.

(5) Rinse Sr Resin sequentially with:

- 15 mL 8M HNO_3
- 5mL 3M HNO_3 - 0.05 oxalic acid
- 7mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

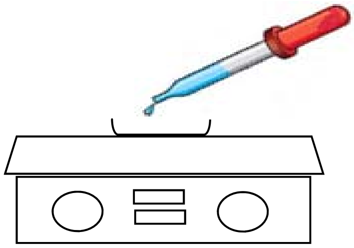
(7) Strip Sr with 13mL 0.05M HNO_3.

Gas Flow Proportional Counting*:

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 8M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.

(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.



(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Discrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr can then be measured by adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

Performance of Radiostrontium in Milk Method

20 Minute Count Times			
^{90}Sr, reference (Bq/L)	^{90}Sr,measured (Bq/L)	Uncertainty %, k = 2	% Bias
0	0.26	98.9	N/A
0	0.26	81.9	N/A
2.86	2.66	24.1	-7.0
2.86	3.96	24.7	38
2.86	3.31	20.2	15.7
2.86	2.67	18.7	-6.6
5.7	6.11	16.7	7.2
5.7	5.71	13.1	0.2
5.7	5.16	13.9	-9.5
14.3	12.8	9.1	-11
14.3	15.2	8.5	6.3
14.3	14.1	8.6	-1.4

60 Minute Count Times			
^{90}Sr, reference (Bq/L)	^{90}Sr,measured (Bq/L)	Uncertainty %, k = 2	% Bias
0	0.11	130	N/A
0	0.27	59	N/A
2.86	3.09	13.2	8.0
2.86	3.11	16.7	8.7
2.86	2.67	13.6	-6.6
2.86	2.67	11.3	-6.6
5.7	5.85	10.4	2.6
5.7	5.75	8.3	0.9
5.7	6.04	8.2	5.9
14.3	13.6	6.1	-4.9
14.3	14.0	6.1	-2.1
14.3	14.2	6.1	-0.7

References

1) Sherrod L. Maxwell, Brian K. Culligan, “Rapid method for the determination of radiostrontium in emergency milk samples,” *J. Radioanal. Nucl. Chem., 279(3), 757-760* (2009).

AN-1403-10

Rapid Determination of Sr in 50g Soil Samples

Summary of Method Strontium is separated and concentrated from 50 gram soil samples. Soils are leached with concentrated nitric and hydrochloric acid. The leachate is evaporated to dryness, and the residue is dissolved in 1M HCl. A ferric hydroxide-calcium phosphate precipitate concentrates strontium and removes matrix components leached from the soil. A calcium fluoride precipitate further concentrates and purifies the strontium fraction. Strontium is separated from matrix impurities and potentially interfering radionuclides in the sample using two stacked 2mL cartridges of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter. Average chemical recovery of strontium, determined by gravimetric yield of stable strontium carrier, is 91 $\pm$ 4%. Measured values of ^{90}Sr agreed to within 2% of reference values for 90 minute count times. The minimum detectable activity for ^{90}Sr in 50g samples with 90 minute count times is 0.41Bq/g. A single operator can prepare batches of 12 samples for the measurement of ^{90}Sr in less than 16 hours.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Sodium Fluoride
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Deionized Water
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
Strontium Carrier (10mg/mL)
2M $Al(NO_3)_3$
Sr-90 standard
Oxalic acid
Boric acid

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)
50mL and 250mL Centrifuge Tubes
Centrifuge
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Muffle Furnace
Hot Plate
Analytical Balance
600mL Glass Beakers
Vacuum Pump

Figure 1. Sample Preparation

Dry soil at 110°C. Blend and Size.
Remove 50g aliquot into 600mL glass beaker .

↓

Muffle at 550°C for 1-2 hours.

↓

Add 6mg Sr Carrier*, 50mL 70% HNO_3,
and 25mL 37% HCl.
*may need to adjust Sr carrier amount to
account for native Sr content in soil.

↓

Heat to dryness on hot plate, medium setting.

↓

Add 50mL 70% HNO_3. Warm sample. Transfer
solids and liquid to 250mL centrifuge tube.

↓

Centrifuge 3500 rpm, 10 min.
Transfer supernate to 600mL beaker.

↓

Add 25mL 70% HNO_3 to Solids. Mix and
Centrifuge. Transfer supernate to same
600mL beaker. Repeat once.
Discard solids to waste.

↓

Evaporate supernate in 600mL beaker to dryness.
Dissolve residue in 15-20mL 1M HCl.
Transfer to 250mL centrifuge tube.

↓

Dilute to 160mL. Add 1mL 1.25M $Ca(NO_3)_3$, 2mL
3.2M $(NH_4)_2HPO_4$, and 25mL 57% NH_4OH. Mix.
Centrifuge. Decant supernate to waste.

↓

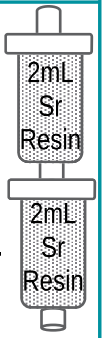
Continue to load solution preparation.

Figure 2. Load Solution Preparation and Strontium Separation

Dissolve residue in 40mL 1.5M HCl.
Dilute to 170mL with H_2O.
Add 25mL 49% HF.

Mix well. Centrifuge 10 min.

Discard Supernate.
Dissolve residue in 7mL 70% HNO_3,
7mL 3M HNO_3-0.25M Boric Acid,
7mL 2M $Al(NO_3)_3$.



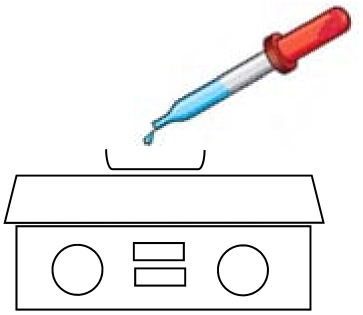
(1) Precondition Sr Resin with 10mL 8M HNO_3.
(2) Load sample.
(3) Rinse sample tube with 5mL 8M HNO_3.
(4) Add tube rinse to Sr Resin.
(5) Rinse Sr Resin sequentially with:
- 15 mL 8M HNO_3
- 10mL 3M HNO_3 - 0.05 oxalic acid
- 10mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

(7) Strip Sr with 18mL 0.05M HNO_3.

Gas Flow Proportional Counting*:

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.
(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Discrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr can then be measured by adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

Method Performance for 50g Soils Spiked with ^{90}Sr

Sample replicates	^{90}Sr Reference Value (mBq/g)	^{90}Sr Measured Value (mBq/g)	% Bias	Sr Carrier % Yield
7	5.92	5.95 ± 0.22	5.0	94.0 ± 2.6
7	11.8	11.5 ± 0.7	-2.5	89.6 ± 2.7
7	59.2	57.8 ± 1.7	-2.4	89.3 ± 4.7

MDA ^{90}Sr, 90 minute count, 50g Soil = 0.41 mBq/g

References

1) Sherrod L. Maxwell, Brian K. Culligan, Patrick J. Shaw “Rapid determination of radiostrontium in large soil samples,” *J. Radioanal. Nucl. Chem., 295(2), 965-971* (2013).

AN-1404-10

Rapid Determination of Sr in 1-2 Liter Seawater Samples

Summary of Method Strontium is separated and concentrated from 1-2L samples of seawater with a calcium phosphate precipitation, enhanced with 200mg of iron. Strontium is separated from matrix impurities and potentially interfering radionuclides in the sample using two stacked 2mL cartridges of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter or liquid scintillation counter. Chemical yield of strontium is determined by gravimetric recovery of native stable strontium in the seawater or by ICP-AES measurement. Average chemical recovery of strontium is 89 ± 5% for 1L samples and 82 ± 4% for 2L samples. Measured values of ^{90}Sr agreed to within 1% and 4% of reference values, for 1L and 2L, respectively, with two hour count times. The minimum detectable activity for ^{90}Sr for 2L samples with a two hour count time is 9.1Bq/L. A single operator can prepare batches of 12-24 samples for measurement of radiostrontium in less than 8 hours.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Nitric Acid (70%)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Deionized Water
Iron Carrier (50mg/mL Fe, as ferric nitrate)
3.2M $(NH_4)_2HPO_4$
2M $Al(NO_3)_3$
^{90}Sr standard
Oxalic acid

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)
50mL Centrifuge Tubes
250-500mL Centrifuge Tubes
Centrifuge
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Hot Plate
Analytical Balance
Vacuum Pump

Figure 1. Sample Preparation

1-2L Sample of Seawater. Add 200mg Fe(III), and 25mL 3.2M $(NH_4)_2HPO_4$.

Mix Well.

Adjust to pH 10 with ~30mL of 56% NH_4OH while stirring.

Centrifuge for 10 minutes in 250 or 500mL tubes at 3000rpm, decanting supernate to waste, until entire sample has been processed.

Wash precipitate with 50mL of pH 9 NH_4OH.

Centrifuge for 10 minutes at 3000rpm. Decant supernate to waste.

Dissolve precipitate in 25mL 70% HNO_3. Transfer to 50mL centrifuge tube.

Rinse centrifuge tube with 8mL 2M $Al(NO_3)_3$ and 5mL 3M HNO_3. Transfer tube rinse to Sr samples in 50mL centrifuge tube.

Load Solution to Sr Resin Separation.

Figure 2. Strontium Resin Separation (Optional ^{90}Y Ingrowth)

(1) Precondition Sr Resin with 10mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

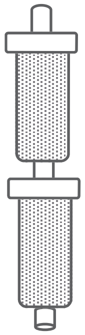
(3) Rinse sample tube with 5mL 8M HNO_3.

(4) Add tube rinse to Sr Resin. Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:
- 15 mL 8M HNO_3
- 10mL 3M HNO_3 - 0.05 oxalic acid
- 10mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

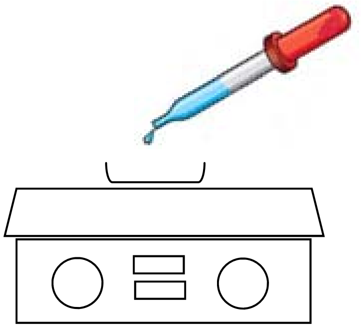
(7) Strip Sr with 20mL 0.05M HNO_3 at 1mL/min.



Gas Flow Proportional Counting*:

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Discrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr can then be measured by adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

Performance of ^{90}Sr Method for 1L and 2L Seawater Samples

Sample Replicates	Sample Volume, L	^{90}Sr, Reference Value (mBq/L)	^{90}Sr, Measured Value (mBq/L)	% Bias	Sr carrier % Recovery
11	1	148	150 ± 11	1.2	89 ± 5
4	2	148	154 ± 5	4.2	82 ± 4

2 hour count times
MDA = 9.1 mBq/L for 2L sample

References

1) Sherrod L. Maxwell, Brian K. Culligan, Robin C. Utsey, “Rapid determination of radiostrontium in seawater samples,” *J. Radioanal. Nucl. Chem., 298(2), 867-875* (2013).

AN-1405-10

Rapid Determination of Sr in Vegetation Samples

Summary of Method Strontium is separated and concentrated from 5-10 gram vegetation samples. Samples are muffled in zirconium crucibles 2-4 hours to destroy organic content. The residue is wet ashed with HNO_3-H_2O_2 and then fused with 15g NaOH at 600°C for ten minutes. The fusion cakes are dissolved in water, transferred to 250mL centrifuge tubes and precipitated with calcium phosphate to facilitate matrix removal. Strontium is separated from matrix impurities and potentially interfering radionuclides in the sample using stacked 2mL and 1mL cartridges of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter or liquid scintillation counter. Chemical yield of strontium is determined by gravimetric recovery of stable strontium or ICP-AES measurement. Average chemical recovery of strontium is 64 ± 4% for 5g samples and 70 ± 8% for 10g samples. Measured values of ^{90}Sr agreed to within 12% of reference values for 90 minute count times. The average time to complete the sample preparation is <8 hours.

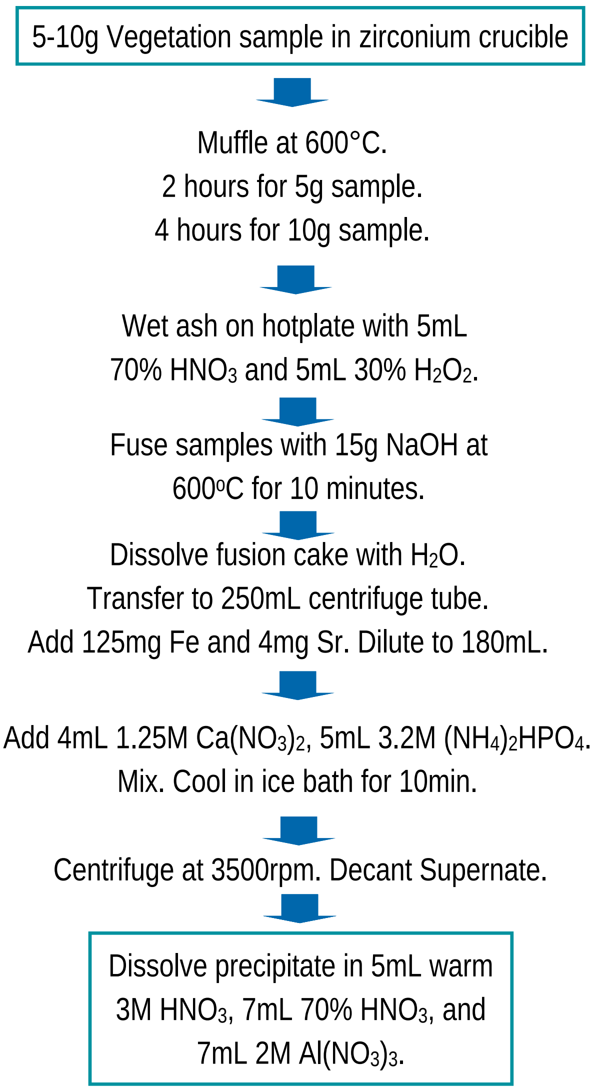
Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)
Nitric Acid (70%)
Hydrogen Peroxide (30%)
Deionized Water
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Strontium Carrier (10mg/mL)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
^{90}Sr standard
3.2M $(NH_4)_2HPO_4$
Sodium Hydroxide
Oxalic acid

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)
50mL and 250mL Centrifuge Tubes
Centrifuge
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Muffle Furnace
Hot Plate
Analytical Balance
250mL Zirconium crucibles with zirconium lids
Vacuum Pump

Figure 1. Sample Preparation



5-10g Vegetation sample in zirconium crucible

Muffle at 600°C.
2 hours for 5g sample.
4 hours for 10g sample.

Wet ash on hotplate with 5mL
70% HNO_3 and 5mL 30% H_2O_2.

Fuse samples with 15g NaOH at
600°C for 10 minutes.

Dissolve fusion cake with H_2O.
Transfer to 250mL centrifuge tube.
Add 125mg Fe and 4mg Sr. Dilute to 180mL.

Add 4mL 1.25M $Ca(NO_3)_2$, 5mL 3.2M $(NH_4)_2HPO_4$.
Mix. Cool in ice bath for 10min.

Centrifuge at 3500rpm. Decant Supernate.

Dissolve precipitate in 5mL warm
3M HNO_3, 7mL 70% HNO_3, and
7mL 2M $Al(NO_3)_3$.

Figure 2. Strontium Resin Separation (Optional ^{90}Y Ingrowth)

(1) Precondition Sr Resin with 10mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

(3) Rinse sample tube with 5mL 8M HNO_3.

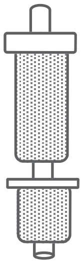
(4) Add tube rinse to Sr Resin. Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:

- 15 mL 8M HNO_3
- 10mL 3M HNO_3 - 0.05 oxalic acid
- 10mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

(7) Strip Sr with 20mL 0.05M HNO_3 at 1mL/min.



2mL + 1mL

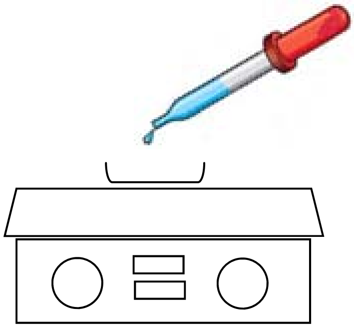
Sr Resin

Cartridges

Gas Flow Proportional Counting:*

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Discrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr can then be measured by adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

*Actinides may also be measured by adding a 2mL TEVA, TRU and DGA cartridges above Sr Resin and following the separation scheme in Eichrom application note AN-1406, “Rapid Determination of Actinides in Vegetation Samples.”

Performance of ^{90}Sr Method 5-10g Vegetation Samples

Sample Replicates	Sample Mass, g	^{90}Sr, Reference Value (Bq/g)	^{90}Sr, Measured Value (Bq/g)	% Bias	Sr carrier % Recovery
6	5.0	0.255	0.285 ± 0.03	12	64 ± 4
2	10.0	0.156	0.156 ± 0.001	0.0	69 ± 2
2	10.0	0.110	0.109 ± 0.003	-0.1	70 ± 7

90 minute count times

References

1) Sherrod L. Maxwell, Brian K. Culligan, Gary W. Noyes, “Rapid separation of actinides and radiostrontium in vegetation samples,” *J. Radioanal. Nucl. Chem., 286(1), 273-282* (2010).

Rapid Determination of Actinides in Vegetation Samples

AN-1406-10

Summary of Method U, Pu, Am and Cm are separated and concentrated from 5-10 gram vegetation samples. Samples are muffled in zirconium crucibles 2-4 hours to destroy organic content. The residue is wet ashed with HNO_3-H_2O_2 and then fused with 15g NaOH at 600ºC for ten minutes. The fusion cakes are dissolved in water, transferred to 250mL centrifuge tubes and precipitated twice to facilitate matrix removal. Actinides are separated on stacked 2mL cartridges of Eichrom TEVA, TRU and DGA resins. Actinides are measured by alpha spectrometry following CeF_3 microprecipitation onto Eichrom Resolve® Filters. Chemical yields of tracers ranged from 90-101% for ^{242}Pu, 84-93% for ^{243}Am, and 81-87% for ^{232}U. Measured values agreed to within 1-3% of reference values for Pu isotopes, 3-9% for Am and Cm isotopes, and 2-15% for U isotopes for 16 hour count times. A single operator can prepare batches of 12 samples for the measurement of actinides in less than 8 hours.

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)
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Lanthanum and Cerium Carriers (10mg/mL)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
Hydrofluoric Acid (49%) or Sodium Fluoride
3.2M $(NH_4)_2HPO_4$
10% (w:w)$TiCl_3$
Sodium Hydroxide
Denature Ethanol
Ascorbic Acid
Nitric Acid (70%)
Deionized Water
2M $Al(NO_3)_3$
Boric acid
Sodium Nitrite
Sulfamic Acid
Hydrogen Peroxide (30%)
Hydrochloric Acid (37%)
1.25M $Ca(NO_3)_2$

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
250mL Zirconium crucibles with zirconium lids
Alpha Spectrometry System
Centrifuge
Hot Plate
Analytical Balance
Muffle Furnace
Heat Lamp
Vacuum Pump

Figure 1. Sample Preparation

5-10g Vegetation sample +
Tracers in zirconium crucible.

Muffle at 600°C.
2 hours if 5g sample.
4 hours if 10g sample.

Wet ash on hotplate with 5mL
70% HNO_3 and 5mL 30% H_2O_2.

Fuse samples with 15g NaOH at 600ºC for 10 minutes.

Dissolve fusion cake with H_2O and
transfer to 250mL centrifuge tube.

Add 10mL 3M HNO_3 to crucible and heat to dissolve
residue. Transfer to the same 250mL centrifuge tube.

Add 125mg Fe, and 4mg La. Dilute to 180mL. Mix.

Add 4mL 1.25M $Ca(NO_3)_2$, 5mL 3.2M $(NH_4)_2HPO_4$,
and 10mL 10% $TiCl_3$. Mix. Cool in ice bath for 10min.

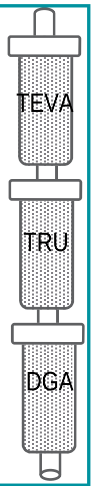
Centrifuge at 3500rpm. Decant Supernate.

Dissolve precipitate in 150mL 1M HCl.
Add 1mg La, 1mL 1.25M $Ca(NO_3)_3$, 6mL
10% $TiCl_3$. Mix. Add 20mL 49% HF.

Centrifuge at 3500rpm. Decant Supernate.

Dissolve precipitate in 5mL 3M HNO_3-0.25M Boric acid,
7mL 70% HNO_3, and 7mL 2M $Al(NO_3)_3$. Adjust valence
states with 0.5mL 1.5M Sulfamic acid, 10uL 50mg/mL
Fe carrier, and 1.25mL 1M Ascorbic acid. Mix.
Add 1mL 3.5M $NaNO_2$ and 1.5mL 70% HNO_3. Mix.

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 $TiCl_3$.

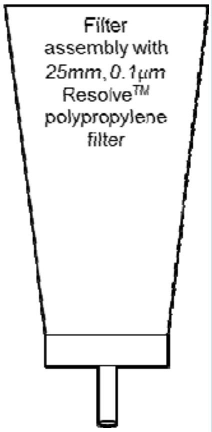
(8) Rinse DGA with 8mL 0.1M HNO_3.
(9) Place TRU cartridge above DGA .
(10) Strip Am/Cm from TRU onto DGA with 15mL 3M HCl.
(11) Separate TRU and DGA. Set TRU aside for U recovery.
(12) Rinse DGA with:
- -5mL 3M HCl
- -3mL 1M HNO_3
- -15mL 0.05M HNO_3

(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.

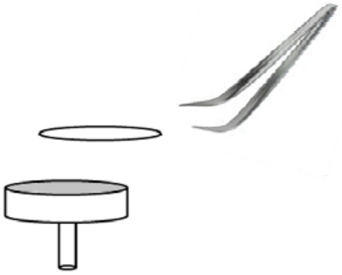
(16) Add 0.5mL 10% $TiCl_3$ to U samples, 0.5mL 30% H_2O_2 to Pu, and 0.2mL 30% H_2O_2 Am/Cm samples.

(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.

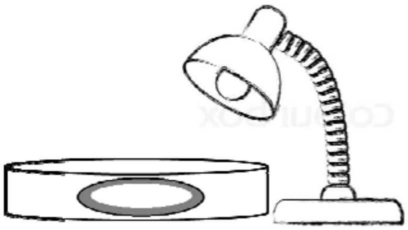


(19) Wet filter with 3mL 80% ethanol followed by 3mL DI water.
(20) Filter sample.
(21) Rinse sample tube with 5mL DI water and add to filter.
(22) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(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.



*Radiostrontium may also be measured by adding a 2mL + 1mL Sr Resin cartridge below DGA and following separation scheme in Eichrom application note AN-1405, "Rapid Determination of Sr in Vegetation Samples."
**Adding 50uL of 30% H_2O_2 to the tube rinse can help improve U recovery and decontamination in Pu/Np fractions.

Performance of Actinides in Vegetation Method

5 gram Samples

Nuclide	Replicates	Reference (mBq/g)	Measured (mBq/g)	% Bias	% Tracer Recovery
^{238}Pu	6	29.4	30.1 ± 3.7	2.4	101 ± 6
^{239}Pu	6	56.8	57.0 ± 4.8	0.3	101 ± 6
^{241}Am	6	48.0	48.5 ± 4.6	1.0	93 ± 7
^{244}Cm	6	6.28	5.9 ± 0.6	-6.1	93 ± 7
^{234}U	6	69.2	81 ± 7	17	87 ± 7
^{238}U	6	71.8	83 ± 10	16	87 ± 7

10 gram Samples

Nuclide	Replicates	Reference (mBq/g)	Measured (mBq/g)	% Bias	% Tracer Recovery
^{238}Pu	2	27.4	28.1 ± 0.4	2.6	90 ± 15
^{239}Pu	2	32.8	32.4 ± 0.9	-1.2	90 ± 15
^{241}Am	2	31.2	30.8 ± 0.0	-1.3	84 ± 12
^{234}U	2	41.6	41.3 ± 1.3	-0.7	81 ± 12
^{238}U	2	43.2	42.0 ± 0.3	-2.8	81 ± 12

References

1) Sherrod L. Maxwell, Brian K. Culligan, Gary W. Noyes, "Rapid separation of actinides and radiostrontium in vegetation samples," *J. Radioanal. Nucl. Chem., 286(1), 273-282* (2010).

AN-1407-10

Rapid Determination of Sr in Animal Tissue Samples

Summary of Method Strontium is separated and concentrated from up to 200g tissue samples. Samples are digested with aqua regia, wet ashed with HNO_3-H_2O_2 and muffled over night at 550°C to destroy organic content. Strontium is separated from matrix impurities and potentially interfering radionuclides in the sample using stacked 2mL and 1mL cartridges of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter or liquid scintillation counter. Chemical yield of strontium is determined by gravimetric recovery of stable strontium or ICP-AES measurement. Average chemical recoveries of strontium are 74-89% for 200g samples of catfish, bass, red drum, mullet, sea trout. Average strontium recoveries for 100 gram samples of deer, hog, bream and shellfish are 83-96%. A single operator can complete the sample preparation, including 16 hours for muffling, for 12-24 samples in less than 24 hours.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrogen Peroxide (30%)
Deionized Water
Strontium Carrier (10mg/mL)
Aluminum Nitrate, Nonahydrate
Sr-90 standard
Oxalic acid

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)
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Muffle Furnace
Hot Plate
Analytical Balance
600mL Glass Beakers
Vacuum Pump

Figure 1. Sample Preparation

Up to 200g Tissue in 600mL glass beaker

Add 4mg Sr carrier

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 furnace 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.

Load sample to Sr Resin Separation.

Figure 2. Strontium Resin Separation (Optional ^{90}Y Ingrowth)

(1) Precondition Sr Resin with 10mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

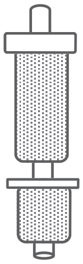
(3) Rinse sample beaker with 5mL 8M HNO_3.

(4) Add beaker rinse to Sr Resin. Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:
- 10 mL 8M HNO_3
- 10mL 3M HNO_3 - 0.05 oxalic acid
- 10mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

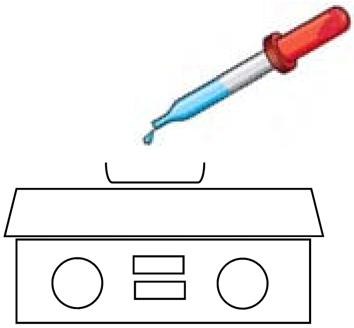
(7) Strip Sr with 20mL 0.05M HNO_3 at 1mL/min.



Gas Flow Proportional Counting:*

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Descrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). 89/90Sr may then be measured after adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

Actinides may also be measured by adding 2mL TEVA, TRU and DGA cartridges above Sr Resin and following the separation scheme in Eichrom application note AN-1408, “Rapid Determination of Actinides in Animal Tissue Samples.”

Sr Carrier Recovery for 100-200g Tissue Samples

Sample	grams	replicate	% Recovery Sr carrier
Beef	100	6	96.3 ± 0.5
Deer	100	59	83.4 ± 3.5
Fish-Bass	200	72	89.0 ± 16
Fish-Bream	100	57	91.7 ± 10
Fish-Catfish	200	69	89.4 ± 17
Fish-Mullet	200	6	85.6 ± 17
Fish-Red Fish	200	6	77.7 ± 21
Fish-Sea Trout	200	6	74.4 ± 25
Hog	100	17	86.0 ± 7.1
Shellfish	100	5	97.5 ± 0.9

References

1) 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).

eichrom®

AN-1408-10

Rapid Determination of Actinides in Animal Tissue Samples

Summary of Method Uranium, Plutonium, and Americium-Curium are separated and concentrated from up to 200g tissue samples. Samples are digested with aqua regia, wet ashed with HNO_3-H_2O_2 and muffled over night at 550°C to destroy organic content. Actinides are separated from matrix impurities and potentially interfering radionuclides in the sample using stacked 2mL cartridges of Eichrom TEVA, TRU and DGA Resin. Actinides are measured via alpha spectrometry following cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Average chemical recoveries of Pu for 100-200g samples are 93-101%. Typical americium recoveries are 93-105%. Typical uranium recoveries are 82-96%. A single operator can complete the sample preparation for 12-24 samples, including 16 hours for muffling, in less than 24 hours.

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%)
Hydrogen Peroxide (30%)
Cerium Carrier (1mg/mL)
Sodium nitrite
Ascorbic acid
Denatured Ethanol
Hydrochloric Acid (37%)
Deionized Water
2M $Al(NO_3)_3$
Sulfamic acid
10% (w:w) $TiCl_3$

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

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.

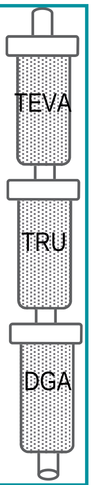
Wet ash samples with 5mL 70% HNO_3
and 5mL 30% H_2O_2, until residue is white.
Additional muffling at 550°C may be necessary.

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,
1mL 3.5M $NaNO_2$, and 1.5mL 70% HNO_3.

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.



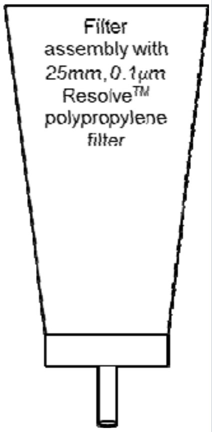
(6) Rinse TEVA cartridge with:
  -10mL 3M HNO_3
  -20mL 9M HCl(remove Th)
  -5mL 3M HNO_3
(7) Strip Pu(Np) from TEVA with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

(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.

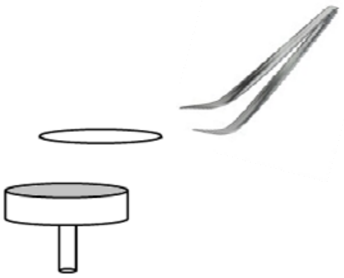
(14) Rinse TRU cartridge with 15mL 4M HCl-0.2M HF-0.002M $TiCl_3$.
(15) Strip U from TRU with 15mL of 0.1M ammonium bioxalate.
(16) Add 0.5mL 10% $TiCl_3$ to U samples, 0.5mL 30% H_2O_2 to Pu and 0.2mL 30% H_2O_2 to Am/Cm samples.

(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

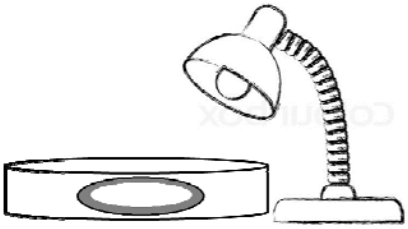


(22) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(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.



*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).

AN-1409-10

Rapid Determination of Sr in Building Materials

Summary of Method Strontium is separated and concentrated from 1.5 gram samples of concrete or brick. Samples are finely ground and fused in a zirconium crucible for 15 minutes at 600°C with 15 grams of sodium hydroxide. The fusion cake is dissolved in water and strontium is concentrated and separated from the matrix using a calcium phosphate precipitate enhanced with iron. A secondary precipitation with calcium fluoride removes additional matrix and decreases the volume of precipitate. The calcium fluoride precipitate is dissolved with nitric acid-boric acid-aluminum nitrate to form the Sr Resin load solution. Strontium is separated from remaining matrix and potentially interfering radionuclides using stacked 2mL and 1mL Sr Resin cartridges. Batches of 12-24 samples can be prepared for analysis in less than 8 hours. Radiostrontium is measured by gas flow proportional counting or liquid scintillation counting. Chemical yield of strontium is determined by gravimetric yield of stable strontium or ICP-AES measurement.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Sodium Fluoride
Deionized Water
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
2M $Al(NO_3)_3$
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Strontium Carrier (10mg/mL)
^{90}Sr standard
Oxalic acid
Boric acid
Sodium Hydroxide

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)
50mL and 250mL Centrifuge Tubes
Centrifuge
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Muffle Furnace
Hot Plate
Analytical Balance
250mL Zirconium crucibles with zirconium lids
Vacuum Pump

Figure 1. Sample Preparation

1.5g finely ground sample in zirconium crucible

↓

Add 4mg of Sr carrier.

↓

Heat samples to dryness on hot plate.

↓

Add 15g of NaOH.

↓

Cover crucibles with zirconium lid and place in muffle furnace at 600°C for 15 minutes.

↓

Carefully remove samples from furnace and cool in fume hood.

↓

Add 25-50mL of water and heat on hot plate to dissolve fusion cake.

↓

Transfer to a 250mL centrifuge tube. Rinse crucible with water. Dilute to 150mL with water.

↓

Add 2mL 1.25M $Ca(NO_3)_3$, 1mL 50mg/mL Fe, 5mL 3.2M $(NH_4)_2HPO_4$. Mix well. Centrifuge 3500rpm. Decant supernate.

↓

Dissolve precipitate in 50mL 1.5M HCl. Dilute to 170mL with water. Add 22mL 49%HF. Mix. Cool in ice bath 10min. Centrifuge 3500rpm. Decant supernate.

↓

Dissolve precipitate in 5mL warm 3M HNO_3-0.25M Boric acid, 7mL 70% HNO_3, and 7mL 2M $Al(NO_3)_3$.

Figure 2. Strontium Resin Separation (Optional ^{90}Y Ingrowth)

(1) Precondition Sr Resin with 10mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

(3) Rinse sample tube with 5mL 8M HNO_3.

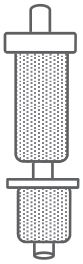
(4) Add tube rinse to Sr Resin. Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:

- 15 mL 8M HNO_3
- 10mL 3M HNO_3 - 0.05 oxalic acid
- 10mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

(7) Strip Sr with 20mL 0.05M HNO_3 at 1mL/min.

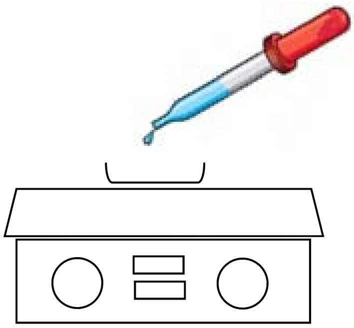


2mL + 1mL Sr Resin Cartridges

Gas Flow Proportional Counting:*

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Descrimination)

When necessary to obtain ^{89}Sr and ^{90}Sr data:

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (without LSC cocktail).

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

References

1)“Rapid radiochemical method for total radiostrontium (Sr-90) in building materials for environmental remediation following radiological incidents,” U.S. Environmental Protection Agency, National Analytical Radiation Environmental Laboratory, EPA 402-R14-001.

2)“Rapid method for sodium hydroxide fusion of concrete and brick matrices prior to americium, plutonium, strontium, radium, and uranium analyses for environmental remediation following radiological incidents,”U.S. Environmental Protection Agency, National Analytical Radiation Environmental Laboratory, EPA 402-R-14-004.

AN-1410-10

Rapid Determination of Sr in Emergency Urine Samples

Summary of Method Strontium is separated and concentrated from 100mL urine samples using calcium phosphate precipitation. An optional wet-ashing step with HNO_3-H_2O_2 destroys residual organic material. The precipitate or wet-ashed residue is dissolved in nitric acid and aluminum nitrate. Strontium is then separated from matrix impurities and potentially interfering radionuclides in the sample using a 2mL cartridge of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter or liquid scintillation counter. Chemical yield of strontium is determined by gravimetric recovery of stable strontium or ICP-AES measurement. Typical chemical recovery of strontium is >80%. Measured values of ^{90}Sr agreed to within 1.7% of reference values for 10 minute count times, although longer count times can be used to improve detection limits and uncertainty. A single operator can complete the separation method for batches of 12-24 samples in as little as 3-4 hours.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Nitric Acid (70%)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Hydrogen Peroxide (30%)
Deionized Water
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
Sr Carrier (10mg/mL)
2M $Al(NO_3)_3$
^{90}Sr standard
Oxalic acid

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)
50mL and 250mL Centrifuge Tubes
Centrifuge
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Hot Plate
Analytical Balance
250mL Glass Beakers
Vacuum Pump

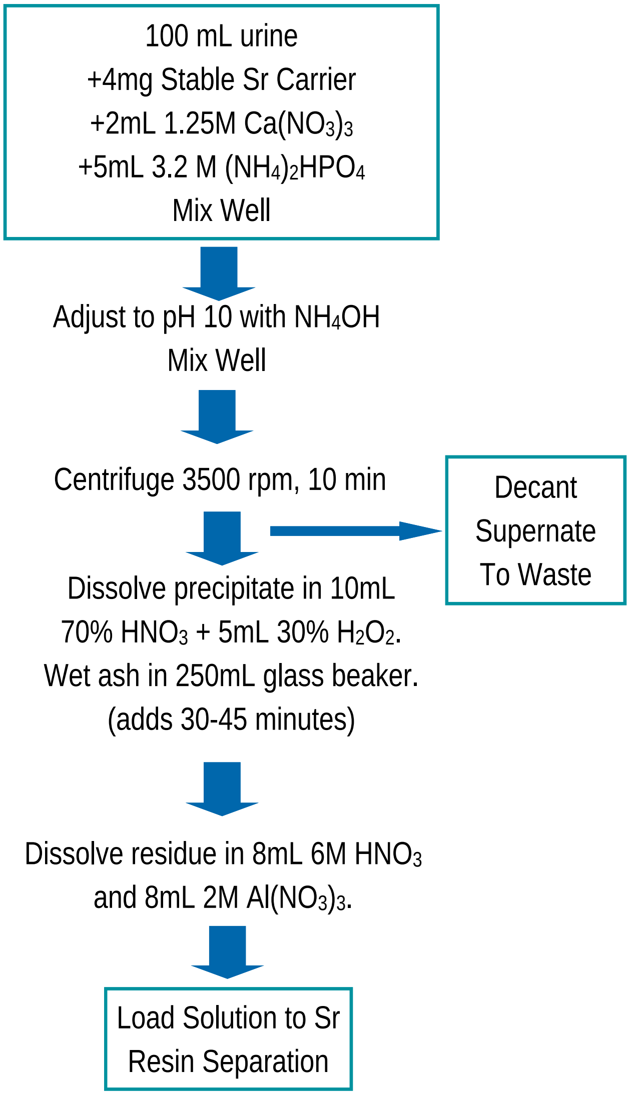


Figure 1. Sample Preparation

100 mL urine
+4mg Stable Sr Carrier
+2mL 1.25M $Ca(NO_3)_3$
+5mL 3.2 M $(NH_4)_2HPO_4$
Mix Well

Adjust to pH 10 with NH_4OH
Mix Well

Centrifuge 3500 rpm, 10 min

Decant Supernate To Waste

Dissolve precipitate in 10mL 70% HNO_3 + 5mL 30% H_2O_2. Wet ash in 250mL glass beaker. (adds 30-45 minutes)

Dissolve residue in 8mL 6M HNO_3 and 8mL 2M $Al(NO_3)_3$.

Load Solution to Sr Resin Separation

Figure 2. Load Solution Preparation and Strontium Separation

(1) Precondition Sr Resin with
10mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

(3) Rinse sample tube with 3mL
3M HNO_3.

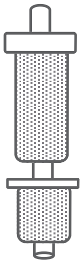
(4) Add tube rinse to Sr Resin.
Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:

- 10 mL 8M HNO_3
- 5mL 3M HNO_3 - 0.05 oxalic acid
- 5mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

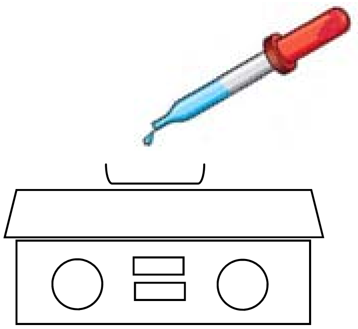
(7) Strip Sr with 15mL
0.05M HNO_3 at
1mL/min.



Gas Flow Proportional Counting:*

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Descrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr may then be measured after adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

Actinides may also be measured by adding a 2mL TEVA, TRU and DGA cartridges above Sr Resin and following the separation scheme in Eichrom application note AN-1412, “Rapid Determination of Actinides in Emergency Urine Samples.”

References

1) Sherrod L. Maxwell, Brian K. Culligan, “Rapid separation method for emergency water and urine samples,” *J. Radioanal. Nucl. Chem., 279(3), 901-907* (2009).

AN-1411-10

Rapid Determination of Sr in Emergency Water Samples

Summary of Method Strontium is separated and concentrated from up to 400mL water samples using calcium phosphate precipitation. The precipitate is dissolved in nitric acid and aluminum nitrate. Strontium is then separated from matrix impurities and potentially interfering radionuclides in the sample using a 2mL cartridge of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter or liquid scintillation counter. Chemical yield of strontium is determined by gravimetric recovery of stable strontium or ICP-AES measurement. Typical chemical recovery of strontium is >80%. Measured values of ^{90}Sr agreed to within 14% of reference values for 10 minute count times, although longer count times can be used to improve detection limits and uncertainty. A single operator can complete the separation method for batches of 12-24 samples in as little as 3-4 hours.

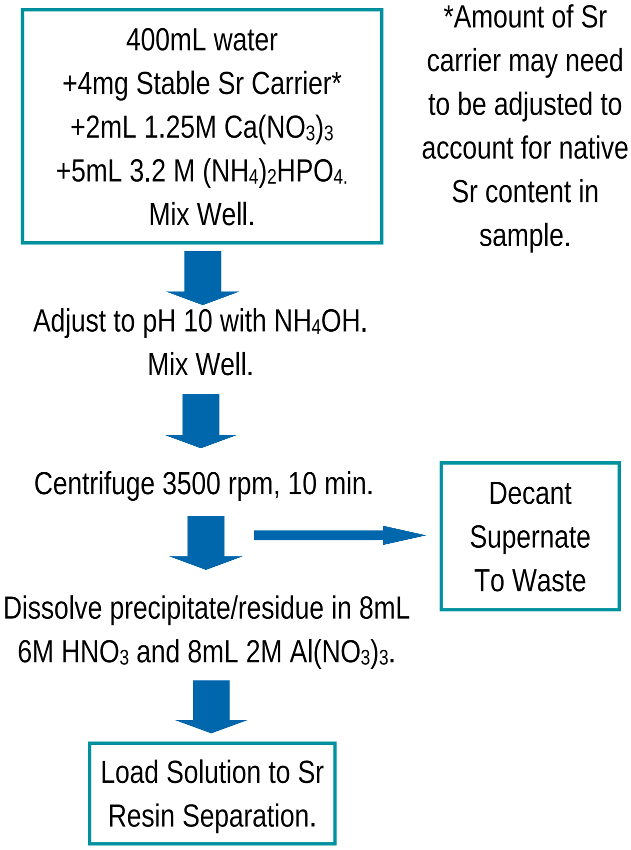
Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Nitric Acid (70%)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Deionized Water
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
Strontium Carrier (10mg/mL)
2M $Al(NO_3)_3$
^{90}Sr standard
Oxalic acid

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)
50mL and 250mL Centrifuge Tubes
Centrifuge
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Analytical Balance
Vacuum Pump

Figure 1. Sample Preparation



*Amount of Sr carrier may need to be adjusted to account for native Sr content in sample.

Figure 2. Load Solution Preparation and Strontium Separation

(1) Precondition Sr Resin with
10mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

(3) Rinse sample tube with 3mL
3M HNO_3.

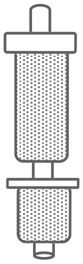
(4) Add tube rinse to Sr Resin.
Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:

- 10 mL 8M HNO_3
- 5mL 3M HNO_3 - 0.05 oxalic acid
- 5mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

(7) Strip Sr with 15mL
0.05M HNO_3 at
1mL/min.

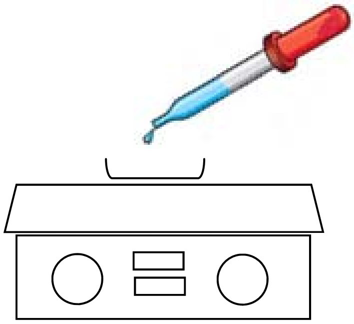


2mL + 1mL
Sr Resin
Cartridges

Gas Flow Proportional Counting:*

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}Sr$ Descrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}Sr$ may then be measured after adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}Sr$ can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

Actinides may also be measured by adding a 2mL TEVA, TRU and DGA cartridges above Sr Resin and following the separation scheme in Eichrom application note AN-1413, “Rapid Determination of Actinides in Emergency Water Samples.”

References

1) Sherrod L. Maxwell, Brian K. Culligan, “Rapid separation method for emergency water and urine samples,” *J. Radioanal. Nucl. Chem., 279(3), 901-907* (2009).

AN-1412-10

Rapid Determination of Actinides in Emergency Urine Samples

Summary of Method Uranium, Plutonium, and Americium-Curium are separated and concentrated from 100mL urine samples using calcium phosphate precipitation. The precipitate is dissolved in HNO_3-H_2O_2 and wet ashed to destroy residual organic material. The wet-ashed residue is dissolved in nitric acid and aluminum nitrate. Actinides are separated from matrix impurities and potentially interfering radionuclides in the sample using 2mL cartridges of Eichrom TEVA and TRU Resins. Actinides are measured by alpha spectrometry following source preparation by cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Chemical yields are determined by recovery of ^{232}U, ^{243}Am, and ^{242}Pu (or ^{236}Pu, if measuring ^{237}Np) tracers. Typical chemical recoveries are >90%. A single operator can complete the separation method for batches of 12-24 samples in as little as 4-5 hours.

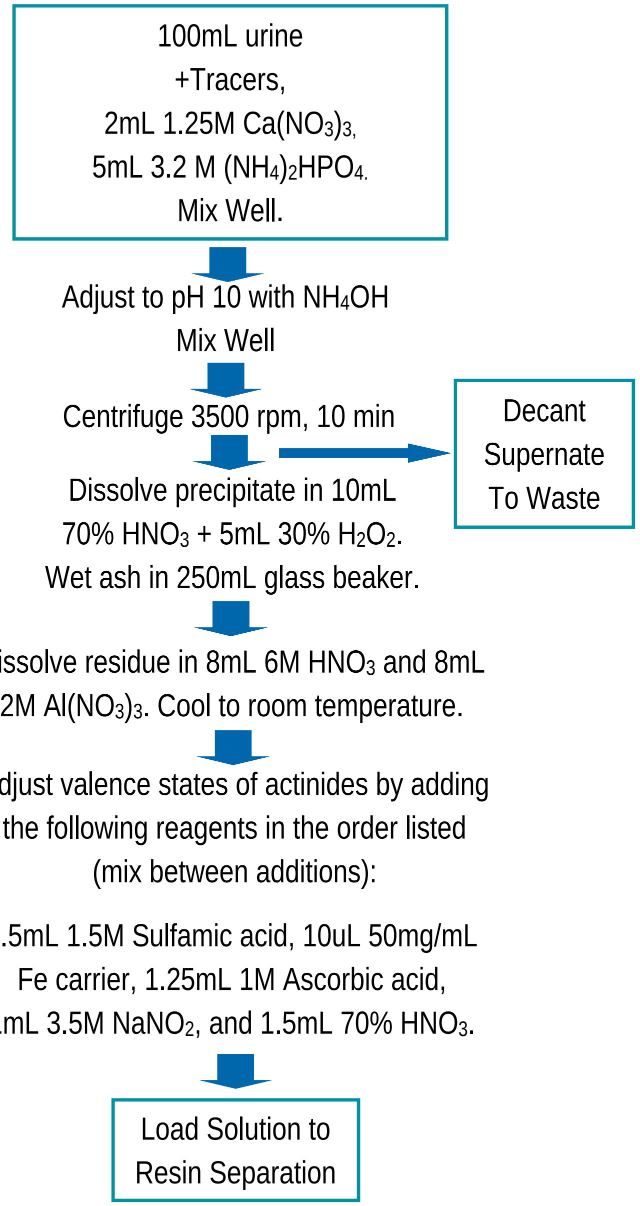
Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
Hydrofluoric Acid (49%) or Sodium Fluoride
Nitric Acid (70%)
Hydrogen Peroxide (30%)
Iron Carrier (50mg/mL)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
Sulfamic Acid
Sodium Nitrite
Hydrochloric Acid (37%)
Deionized Water
Cerium Carrier (1mg/mL)
3.2M $(NH_4)_2HPO_4$
10% (w:w) $TiCl_3$
Ascorbic Acid
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
Hot Plate
Analytical Balance
250mL Glass Beakers
Alpha Spectrometry System
Vacuum Pump
Heat Lamp

Figure 1. Sample Preparation



100mL urine
+Tracers,
2mL 1.25M $Ca(NO_3)_3$,
5mL 3.2 M $(NH_4)_2HPO_4$.
Mix Well.

Adjust to pH 10 with NH_4OH
Mix Well

Centrifuge 3500 rpm, 10 min

Decant
Supernate
To Waste

Dissolve precipitate in 10mL
70% HNO_3 + 5mL 30% H_2O_2.
Wet ash in 250mL glass beaker.

Dissolve residue in 8mL 6M HNO_3 and 8mL
2M $Al(NO_3)_3$. Cool to room temperature.

Adjust valence states of actinides by adding
the following reagents in the order listed
(mix between additions):

0.5mL 1.5M Sulfamic acid, 10uL 50mg/mL
Fe carrier, 1.25mL 1M Ascorbic acid,
1mL 3.5M $NaNO_2$, and 1.5mL 70% HNO_3.

Load Solution to
Resin Separation

Figure 2. Actinide Separation on TEVA - TRU*

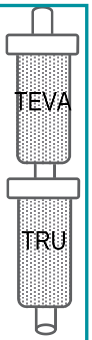
(1) Precondition stacked 2mL TEVA-TRU with 10mL 3M HNO_3.

(2) Load sample solution.

(3) Rinse sample tube with 5mL 6M HNO_3. Add tube rinse to cartridges.**

(4) Rinse cartridges with 5mL 3M HNO_3.

(5) Separate TEVA and TRU cartridges.



(6) Rinse TEVA cartridge with:

-15mL 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) Strip Am/Cm from TRU with 15mL 4M HCl. Dilute to 30mL prior to CeF_3 ppt.

(9) Rinse TRU with 15mL 4M HCl-0.2M HF. (Th removal)

(10) Strip U from TRU with 15mL 0.1M ammonium bioxalate.

(11) Add 0.5mL 30% H_2O_2 to Pu and 0.2mL 30% H_2O_2 to Am/Cm samples for additional U decon. during CeF_3 ppt.

(12) Add 0.5mL of 10% $TiCl_3$ to each U sample for CeF_3 ppt.

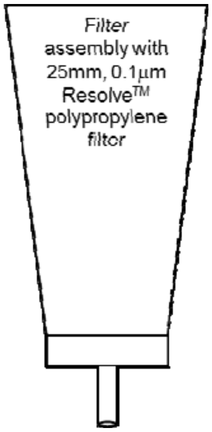
(13) Add 50-100ug Ce carrier to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box.

(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(16) Filter sample.

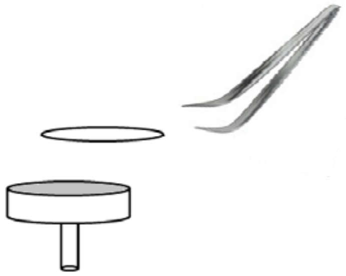
(17) Rinse sample tube with 5mL DI water and add to filter.



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

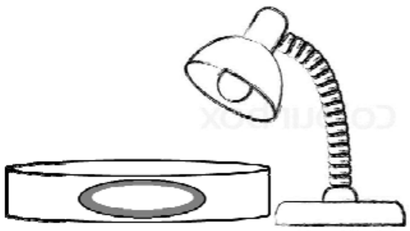
(19) Draw vacuum until filter is dry.

(20) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(21) Dry filter under heat lamp for 3-5 minutes.

(22) Measure actinides by alpha spectrometry.



*Strontium may also be measured by adding a 2mL Sr Resin Cartridge below DGA and following the separation scheme in Eichrom application note AN-1410, "Rapid Determination of Sr in Emergency Urine Samples."

**Adding 50uL of 30% H_2O_2 to the 6M HNO_3 tube rinse can help improve U recoveries and decontamination in the Pu/Np fraction.

References

1) Sherrod L. Maxwell, Brian K. Culligan, "Rapid separation method for emergency water and urine samples," *J. Radioanal. Nucl. Chem., 279(3), 901-907* (2009).

AN-1413-10

Rapid Determination of Actinides in Emergency Water Samples

Summary of Method Uranium, Plutonium and Americium-Curium are separated and concentrated from up to 400mL water samples using calcium phosphate precipitation. The precipitate is dissolved in nitric acid and aluminum nitrate. Actinides are separated from matrix impurities and potentially interfering radionuclides in the sample using 2mL cartridges of Eichrom TEVA and TRU Resins. Actinides are measured by alpha spectrometry following source preparation by cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Chemical yields are determined by recovery of ^{232}U, ^{243}Am, and ^{242}Pu (or ^{236}Pu, if measuring ^{237}Np) tracers. Typical chemical recoveries are >90%. A single operator can complete the separation method for batches of 12-24 samples in as little as 4-5 hours.

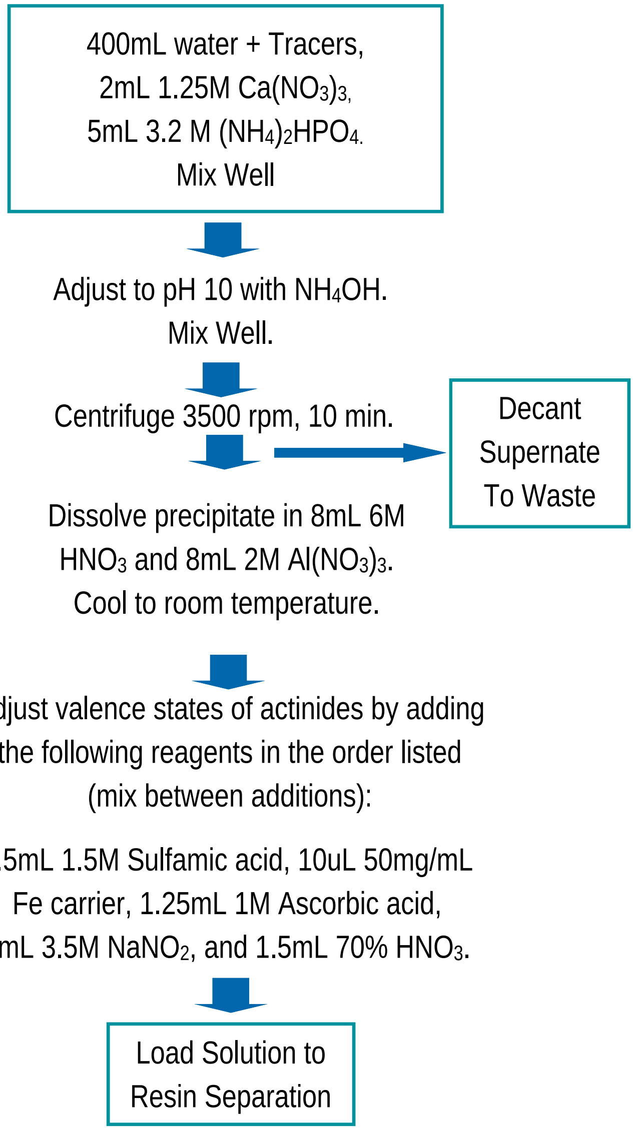
Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Sodium Fluoride
Deionized Water
Iron Carrier (50mg/mL)
Cerium Carrier (1mg/mL)
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
2M $Al(NO_3)_3$
10% (w:w) $TiCl_3$
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
Sulfamic Acid
Ascorbic Acid
Sodium Nitrite
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
Analytical Balance
Alpha Spectrometry System
Vacuum Pump

Figure 1. Sample Preparation



400mL water + Tracers,
2mL 1.25M $Ca(NO_3)_3$,
5mL 3.2 M $(NH_4)_2HPO_4$.
Mix Well

Adjust to pH 10 with NH_4OH.
Mix Well.

Centrifuge 3500 rpm, 10 min.

Decant Supernate To Waste

Dissolve precipitate in 8mL 6M HNO_3 and 8mL 2M $Al(NO_3)_3$.
Cool to room temperature.

Adjust valence states of actinides by adding the following reagents in the order listed (mix between additions):

0.5mL 1.5M Sulfamic acid, 10uL 50mg/mL Fe carrier, 1.25mL 1M Ascorbic acid, 1mL 3.5M $NaNO_2$, and 1.5mL 70% HNO_3.

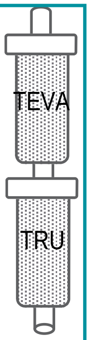
Load Solution to Resin Separation

Figure 2. Actinide Separation on TEVA - TRU*

(1) Precondition stacked 2mL TEVA-TRU 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 5mL 3M HNO_3.

(5) Separate TEVA and TRU cartridges.



(6) Rinse TEVA cartridge with:
- -15mL 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) Strip Am/Cm from TRU with 15mL 4M HCl. Dilute to 30mL prior to CeF_3 ppt.

(9) Rinse TRU with 15mL 4M HCl-0.2M HF. (Th removal)

(10) Strip U from TRU with 15mL 0.1M ammonium bioxalate.

(11) Add 0.5mL 30% H_2O_2 to Pu and 0.2mL 30% H_2O_2 to Am/Cm samples for additional U decon. during CeF_3 ppt.

(12) Add 0.5mL of 10% $TiCl_3$ to each U sample for CeF_3 ppt.

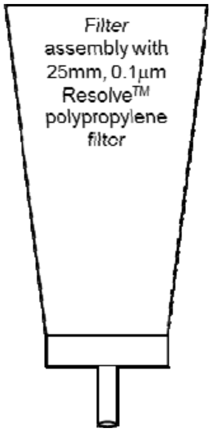
(13) Add 50-100ug Ce carrier to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box.

(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(16) Filter sample.

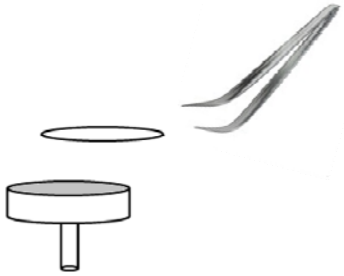
(17) Rinse sample tube with 5mL DI water and add to filter.



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

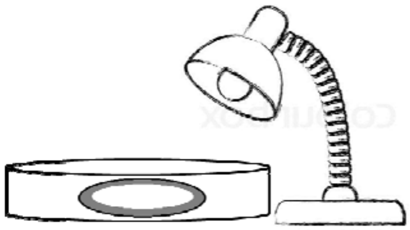
(19) Draw vacuum until filter is dry.

(20) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(21) Dry filter under heat lamp for 3-5 minutes.

(22) Measure actinides by alpha spectrometry.



*Strontium may also be measured by adding a 2mL Sr Resin Cartridge below DGA and following the separation scheme in Eichrom application note AN-1411, “Rapid Determination of Sr in Emergency Water Samples.”

**Adding 50uL of 30% H_2O_2 to the 6M HNO_3 tube rinse can help improve U recoveries and decontamination in the Pu/Np fraction.

References

1) Sherrod L. Maxwell, Brian K. Culligan, “Rapid separation method for emergency water and urine samples,” *J. Radioanal. Nucl. Chem., 279(3), 901-907* (2009).

AN-1414-10

Rapid Determination of ^{90}Sr in up to 40 Liter Seawater Samples

Summary of Method Yttrium-90, the daughter product of ^{90}Sr decay, is separated and concentrated from up to 40L samples of seawater. A ferric hydroxide precipitate enhanced with 10mg of lanthanum and 1mg of yttrium concentrates ^{90}Y, while rejecting much of the salt content of the seawater sample. A second precipitation with lanthanum fluoride removes additional matrix ions. Yttrium is separated from potentially interfering radionuclides in the sample, including rare earths such as ^{138}La and $^{139/144}$Ce, using a 2mL cartridge of Eichrom DGA Resin. ^{90}Y is measured on a low background gas flow proportional counter following cerium fluoride microprecipitation onto an Eichrom Resolve® Filter. Chemical yield of stable yttrium is determined by ICP-MS or ICP-AES. Average chemical recovery of yttrium is 84 ± 7% for 40L samples. Measured values of ^{90}Sr(^{90}Y) agree to within 5% of reference values, with two hour count times. The minimum detectable activity for ^{90}Sr for 40L samples with a two hour count time is 0.35mBq/L. The average time to complete the method is 8 hours. While standard methods targeting Sr are limited by the ~8mg/L native Sr content in seawater, targeting ^{90}Y directly allows for the efficient processing of very large seawater samples to achieve very low minimum detectible activities. However, interference by the fission product ^{91}Y ($t_{1/2}$ = 58.51 days) precludes application of this method for the measurement of ^{90}Sr(^{90}Y) immediately following a radiological incident involving the release of un-aged nuclear fuel or fission products.

Figure 1. Sample Preparation

Up to 40L Sample of Seawater.
Acidify to pH 2 with 37% HCl.
Add 1mg Yttrium carrier.

↓

Add 10mg La carrier. Add 50mg Fe carrier per liter of sample. Mix Well.

↓

Adjust to pH 9 with 56% NH_4OH. Mix.
Allow precipitate to settle.
Decant supernate until ~2L remains.

↓

Transfer remaining supernate and precipitate to 500mL centrifuge tubes. Centrifuge 3000rpm for 10 minutes. Decant supernate. Repeat until entire sample centrifuged.

↓

Wash precipitate with 100mL water.
Centrifuge. Decant supernate.

↓

Dissolve precipitate in 100mL 1.5M HCl.
Add 75mg Ca and 50mL 49% HF. Mix.
Wait 15 minutes. Centrifuge. Decant supernate.

↓

Dissolve precipitate in 10mL 3M HNO_3-0.25M Boric acid, 10mL 70% HNO_3, and 10mL 2M $Al(NO_3)_3$.

↓

Load Solution for Sr separation.

Reagents

DGA Resin, Normal 2mL Cartridges (Eichrom DN-R50-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Sodium Fluoride
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Deionized Water
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Yttrium and Cerium Carriers (1mg/mL)
Lanthanum Carrier (10mg/mL)
1.25M $Ca(NO_3)_2$
^{90}Sr standard
2M $Al(NO_3)_3$
Boric acid

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 500mL Centrifuge Tubes
Centrifuge
Gas Flow Proportional Counter
Analytical Balance
Vacuum Pump
Heat Lamp

Figure 2. Yttrium Separation on DGA and CeF_3 Microcprecipitation

(1) Precondition DGA Resin with 5mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

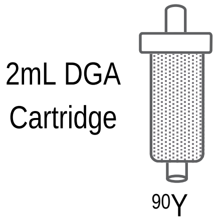
(3) Rinse sample tube with 5mL 8M HNO_3.

(4) Add tube rinse to DGA Resin. Elute at 1-2mL/min.

(5) Rinse DGA Resin sequentially with:
- 15 mL 8M HNO_3 (Ca,Sr,Pb)
- 20mL 0.05M HNO_3 (La,Ce,Sr,U)
- 15mL 3M HNO_3-0.25M HF (U,Th)
- 10mL 3M HCl (Ca,La,Pb)

(6) Dispose of (1) to (5) as waste.

(7) Strip Y with 20mL 0.25M HCl at 1mL/min.



(8) Remove 0.1-1.0mL aliquot for stable Y recovery by ICP-MS or ICP-AES. Dilute aliquot as appropriate.

(9) To remaining sample:
- Add 100ug Ce carrier.
- Mix well.
- Add 2mL 49% HF.
- Mix well. Wait 15-20 minutes.

(10) Set up Resolve® Filter Funnel on vacuum box.

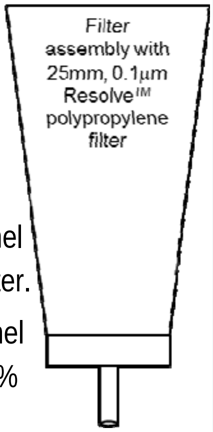
(11) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(12) Filter sample.

(13) Rinse sample tube with 5mL DI water and add to filter.

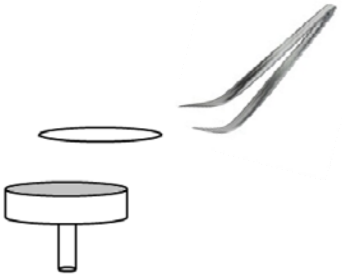
(14) Rinse filter funnel with 3mL DI water.

(15) Rinse filter funnel with 1-2mL 100% ethanol.



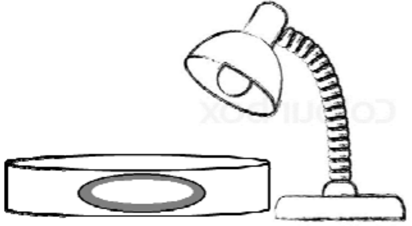
(16) Draw vacuum until filter is dry.

(17) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.



(18) Dry filter under heat lamp for 10-15 minutes.

(19) Measure ^{90}Y on low background gas flow proportional counter.



Method Performance 10-40L Spike Seawater Samples

Sample Volume, L	% Recovery Y carrier	^{90}Sr (mBq/L) Reference	^{90}Sr (mBq/L) Measured	% Bias
10	85.5	296	310	4.7
20	89.2	28.2	28.1	-0.4
30	72.3	18.8	18.5	-1.6
40	87.6	14.1	13.7	-2.8
40	86.5	14.1	13.9	-1.4

MDA for 40L sample = 0.35 mBq/L for 2 hour count time
MDA for 40L sample = 0.20 mBq/L for 8 hour count time

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, Robin C. Utsey, Daniel R. McAlister, “Rapid determination of ^{90}Sr in seawater samples,” *J. Radioanal. Nucl. Chem.*, 303, 709-717 (2015).

AN-1415-10

Rapid Determination of ^{210}Po in Water Samples

Summary of Method A method for the measurement of ^{210}Po 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}Po is concentrated from up to 1L samples of ground water or 2L samples of drinking water using a calcium phosphate precipitate. ^{210}Po is then separated from matrix ions and potentially interfering radionuclides using a 2mL cartridge of Eichrom DGA Resin. ^{210}Po is measured using alpha spectrometry following bismuth phosphate microprecipitation onto an Eichrom Resolve® Filter. Chemical recoveries of polonium, determined with a ^{209}Po tracer, were typically 80-90%. ^{210}Po 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).

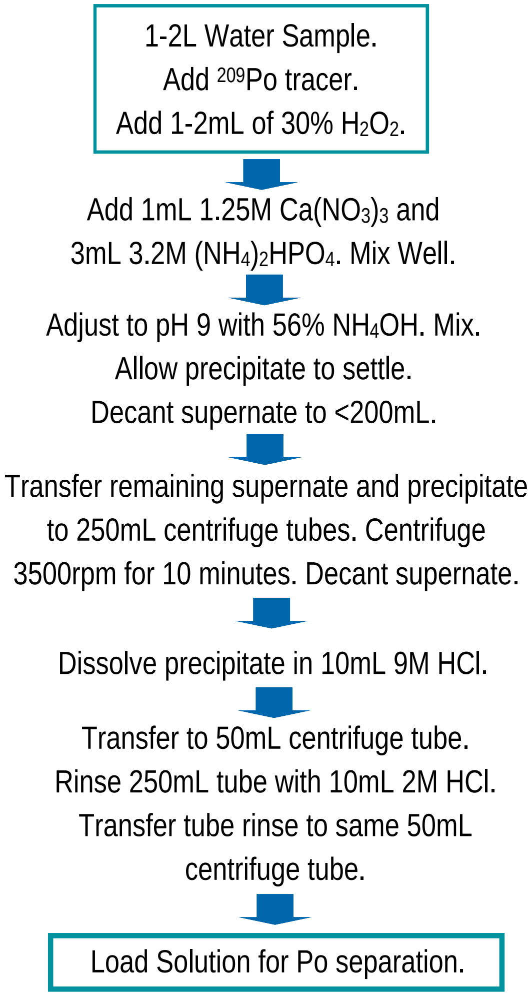
Reagents

DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Hydrogen Peroxide (30%)
Deionized Water
1.25M $Ca(NO_3)_2$
3.2M $(NH_4)_2HPO_4$
^{209}Po 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 ^{209}Po tracer.
Add 1-2mL of 30% H_2O_2.

Add 1mL 1.25M $Ca(NO_3)_3$ and
3mL 3.2M $(NH_4)_2HPO_4$. Mix Well.

Adjust to pH 9 with 56% NH_4OH. 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 HCl.

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

Load Solution for Po separation.

Figure 2. Polonium Separation on DGA and $BiPO_4$ Microprecipitation

(1) Precondition DGA Resin with 5mL 2M HCl.

(2) Load ^{210}Po sample at 1-2mL/min.

(3) Rinse sample tube with 5mL 2M HCl.

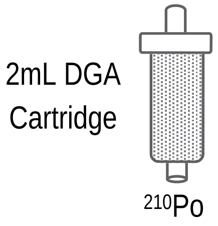
(4) Add tube rinse to DGA Resin. Elute at 1-2mL/min.

(5) Rinse DGA Resin sequentially with:

- 5mL 2M HCl
- 15mL 0.25M HCl
- 5mL 6M HNO_3

(6) Dispose of (1) to (5) as waste.

(7) Strip Po with 15mL 0.05M HNO_3 at 1mL/min.



(8) To polonium sample:

- Add 125ug Bi carrier.
- Add 0.1mL 30% H_2O_2.
- 0.75mL 3.2M $(NH_4)_2HPO_4$.
- Mix well.
- Add 200uL 56% NH_4OH.
- Mix well. Wait 15-20 minutes.

(9) Set up Resolve® Filter Funnel on vacuum box.

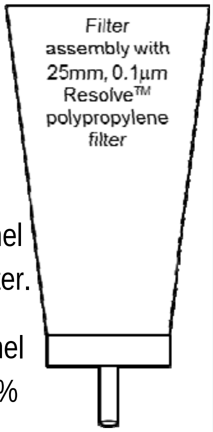
(10) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(11) Filter sample.

(12) Rinse sample tube with 5mL DI water and add to filter.

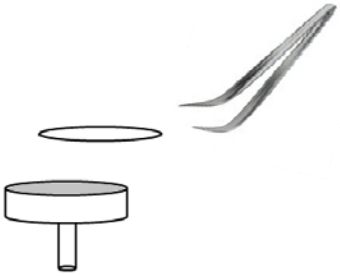
(13) Rinse filter funnel with 3mL DI water.

(14) Rinse filter funnel with 1-2mL 100% ethanol.



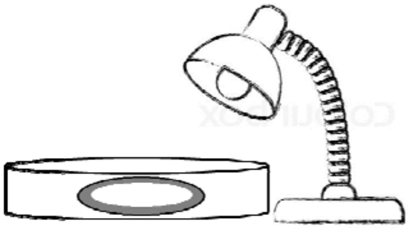
(15) Draw vacuum until filter is dry.

(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 ^{210}Po and ^{209}Po tracer by alpha spectrometry.



Method Performance ^{210}Po in Water

Sample	Volume mL	Replicates	% Recovery ^{209}Po tracer	^{210}Po (mBq/L) Reference	^{210}Po (mBq/L) Measured	% Bias
Ground Water	200	6	87.4 ± 5.8	316	308 ± 5	-2.5
Ground Water	200	7	82.3 ± 3.9	1262	1289 ± 6	2.1
Ground Water	1000	6	85.0 ± 8.2	63.3	61.5 ± 5.1	-2.8
Drinking Water	2000	4	80.0 ± 9.6	63.3	61.1 ± 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 ^{210}Po in water samples,” *J. Radioanal. Nucl. Chem.*, 298(3), 1977-1989 (2014).

AN-1416-10

Rapid Determination of Actinides and ^{210}Po in Water

Summary of Method A method for the measurement of ^{210}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}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}Po and actinides are then separated from matrix ions and potentially interfering radionuclides using stacked 2mL cartridge of Eichrom TRU and DGA Resin. ^{210}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 ± 2.6% for ^{209}Po, 93.4 ± 6.8% for ^{242}Pu, 100.2 ± 6.9% for ^{243}Am and 96.6 ± 2.5 for ^{232}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.

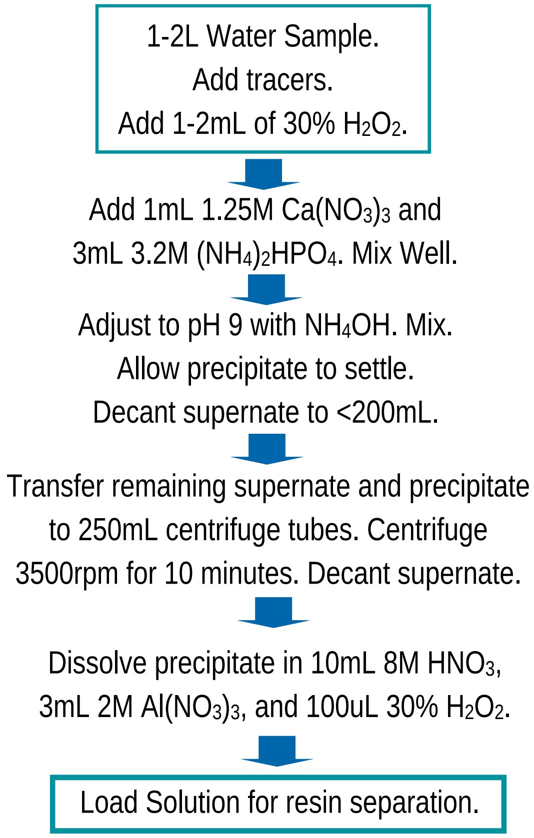
Reagents

TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Ammonium Hydroxide (Listed as 28% NH_3 or 56% NH_4OH)
^{209}Po, ^{232}U, ^{243}Am, ^{242}Pu tracers
Bi and Ce carriers (1mg/mL)
Nitric Acid (70%)
Hydrofluoric Acid (49%)
Deionized Water
1.25M $Ca(NO_3)_2$
10% (w:w) $TiCl_3$
Oxalic acid/Ammonium Oxalate
Hydrochloric Acid (37%)
Hydrogen Peroxide (30%)
3.2M $(NH_4)_2HPO_4$
2M $Al(NO_3)_3$
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)
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



1-2L Water Sample.
Add tracers.
Add 1-2mL of 30% H_2O_2.

Add 1mL 1.25M $Ca(NO_3)_3$ and
3mL 3.2M $(NH_4)_2HPO_4$. Mix Well.

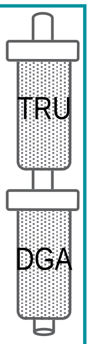
Adjust to pH 9 with NH_4OH. Mix.
Allow precipitate to settle.
Decant supernate to <200mL.

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

Dissolve precipitate in 10mL 8M HNO_3,
3mL 2M $Al(NO_3)_3$, and 100uL 30% H_2O_2.

Load Solution for resin separation.

Figure 2. TRU/DGA Separation and Source Preparation



(1) Precondition TRU/DGA Resin with 5mL 8M HNO_3.

(2) Load samples.

(3) Rinse sample tube with 5mL 8M HNO_3, and add tube rinse to TRU/DGA.*

(4) Rinse TRU/DGA with:
- -10mL 10M HNO_3
- -15mL 4M HCl.

(5) Separate TRU and DGA.

(6) Strip Pu from TRU with 12mL 3M HCl-0.02M $TiCl_3$. Add 0.5mL 30% H_2O_2.

(7) Rinse TRU with:
- -5mL 8M HNO_3
- -20mL 1.5M HCl-0.15M HF.

(8) Strip U from TRU with 15mL 0.1M ammonium bioxalate. Add 0.5mL $TiCl_3$ for CeF_3 ppt.

(9) Rinse DGA:
- -5mL 3M HCl
- -12mL 3M HNO_3-0.25M HF
- -5mL 4M HCl

(10) Strip Am/Cm from DGA with 12mL 0.25M HCl. Add 0.2mL 30% H_2O_2.

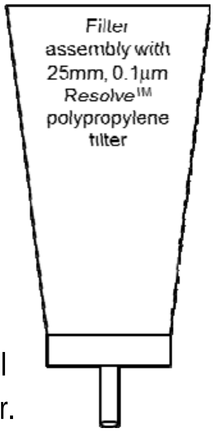
(11) Rinse DGA with 6M 8M HNO_3.

(12) Strip Po from DGA with 15mL 0.05M HNO_3. Add 0.1mL 30% H_2O_2.

(13) Po samples: Add 125ug Bi carrier, 0.75mL 3.2M $(NH_4)_2HPO_4$. Mix well. Add 0.2mL 56% NH_4OH. Mix well. Wait 15-20 minutes.

Actinide samples: Add 50-100ug Ce carrier. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box. Wet filter with 3mL 80% ethanol and 3mL DI H_2O.



(15) Filter sample.

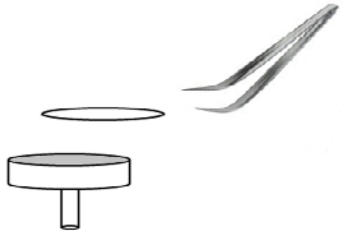
(16) Rinse sample tube with 5mL DI water and add to filter.

(17) Rinse filter funnel with 3mL DI water.

(18) Rinse filter funnel with 2mL 100% ethanol.

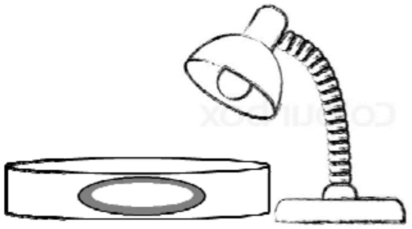
(19) Draw vacuum until filter is dry.

(20) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.



(21) Dry filter under heat lamp for 3-5 minutes.

(22) Measure Po and actinides by alpha spectrometry.



*Adding 50uL 30% H_2O_2 to the tube rinse can improve U recoveries and decontamination in Pu(Np) fractions.

Method Performance ^{210}Po and Actinides in Water

Analyte	Tracer	% Recovery of tracer	Analyte (mBq/L) Reference	Analyte (mBq/L) Measured	% Bias
^{210}Po	^{209}Po	81.5 ± 2.6	1584	1660 ± 3	4.8
^{238}Pu	^{242}Pu	93.4 ± 6.8	370	381 ± 4	3.0
^{241}Am	^{243}Am	100.2 ± 6.9	370	381 ± 3	3.0
^{244}Cm	^{243}Am	100.2 ± 6.9	328	328 ± 4	0.1
^{238}U	^{232}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 ^{210}Po in water samples," *J. Radioanal. Nucl. Chem.*, 298(3), 1977-1989 (2014).

eichrom®

AN-1417-11

Rapid Determination of $^{226/228}$Ra in Water Samples

Summary of Method Ra isotopes are separated and measured from 1.0-1.5 liter samples of terrestrial waters. Radium is concentrated from samples on MnO_2 Resin. After a >36 hour ingrowth period for ^{228}Ac from ^{228}Ra, radium and ^{228}Ac are separated from matrix ions and potentially interfering radionuclides using stacked 2mL cartridges of Eichrom LN and DGA Resins. ^{228}Ac is prepared for gas flow proportional counting using a cerium fluoride microprecipitation onto Eichrom Resolve® Filters. ^{226}Ra is prepared for alpha spectrometry using a barium sulfate microprecipitation onto Eichrom Resolve® Filters. Chemical yield of radium is determined by adding a ^{133}Ba tracer. A single operator can process batches of 12-24 samples in 4-5 hours. Results for ^{226}Ra and ^{228}Ra can be obtained in 48 hours, including >36 hour ingrowth time for ^{228}Ac. Results for ^{226}Ra and ^{228}Ra in spiked river and ground water samples typically agreed to within 5% of reference values.

Reagents

MnO_2 Resin (Eichrom MN-B100-A)
LN Resin (Eichrom LN-R50-S)
DGA Resin, Normal 2mL Cartridges (Eichrom DN-R50-S)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
^{133}Ba Tracer
Barium and Cerium Carriers (1mg/mL)
Nitric Acid (70%)
Hydrochloric Acid (37%)
1.25M $Ca(NO_3)_2$
Denatured Ethanol
Deionized Water
Hydrofluoric Acid (50%)
Hydrogen Peroxide (30%)
Ammonium Sulfate
Isopropyl Alcohol

Equipment

Plastic Chromatography Column (Eichrom AC-50E-5M)
Column Extension Funnel (Eichrom AC-20X-20M)
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 Filter in Disposable Funnel (Eichrom RF-DF-25-25PP01)
50mL Centrifuge Tubes
Stainless Steel Planchets with Adhesive Tape
Alpha Spectroscopy System
Gamma Spectroscopy System (if ^{133}Ba tracer used)
Low Background Gas Flow Proportional Counter
150mL Glass beakers
Vacuum Pump
Hot Plate
Heat Lamp

Figure 1. Sample Preparation

1.0-1.5 Liter Water Samples
Adjust to pH2 with HNO_3
+Tracer ^{133}Ba
+1 mL 1.25M $Ca(NO_3)_3$

↓

Adjust to pH 6-7 with NH_4OH

↓

1.5g column of MnO_2 Resin, slurry packed from water in 5mL column. (Precondition column with 25mL DI water.) Load sample at up to 15mL/min.

↓

Rinse column with 50mL DI water.

↓

Strip MnO_2 with 12mL of 4M HCl-1.5% H_2O_2. Bubbling will occur. Resin will turn from dark brown to white as MnO_2 is dissolved.

↓

If measuring ^{228}Ra, allow >36 hours for ^{228}Ac ingrowth.

↓

Proceed to LN/DGA Separation

Figure 2. LN-DGA Separation and Alpha Source Preparation

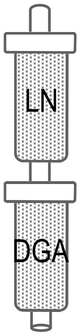
(1) Precondition LN + DGA Resin with 5mL 4M HCl.

(2) Load samples at 1mL/min.

(3) Rinse sample tube with 5mL 4M HCl.

(4) Add tube rinse to LN + DGA Resin.

(5) Rinse LN + DGA with 5mL 4M HCl.



(6) Separate LN and DGA cartridges.

(7) Evaporate radium fraction from steps (2) to (5) to dryness.

(8) Dissolve residue in 10mL of 0.1M HCl + 50uL 30% H_2O_2.

(9) Pass Ra solution through same LN resin cartridge (Ra not retained). Rinse beaker with 5mL 0.1M HCl. Add beaker rinse to LN resin. Collect load/rinse for step (12).

(10) Rinse DGA Resin cartridge with 15mL 4M HCl.

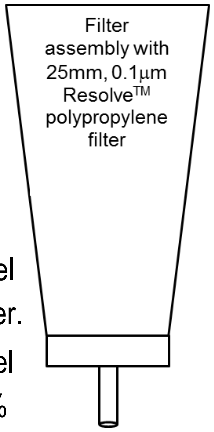
(11) Strip ^{228}Ac from DGA with 10mL of 0.5mL HCl.

(12) <u>^{226}Ra samples:</u> Add 50ug Ba carrier. Mix well. Add 3g $(NH_4)_2SO_4$. Mix well. Add 5mL isopropanol. Place in ice bath for 30 minutes.

<u>^{228}Ra(Ac) samples:</u> Add 50ug Ce carrier. Mix well. Add 1mL 49% HF. Mix well. Wait 30 minutes.

(13) Set up Resolve® Filter Funnel on vacuum box.

(14) Wet filter with 3mL 80% ethanol followed by 3mL DI water.



(15) Filter sample.

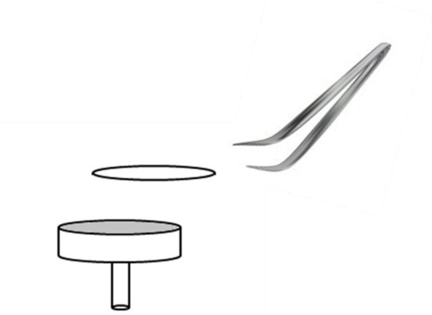
(16) Rinse sample tube with 5mL DI water and add to filter.

(17) Rinse filter funnel with 3mL DI water.

(18) Rinse filter funnel with 1-2mL 100% ethanol.

(19) Draw vacuum until filter is dry.

(20) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.



(21) Dry filter under heat lamp for 3-5 minutes.

(22) Measure ^{228}Ra(^{228}Ac) by gas flow proportional counting. Measure ^{226}Ra and ^{225}Ra(^{217}At) by alpha spectrometry after >8 hour ^{217}At ingrowth. (^{133}Ba in ^{226}Ra fraction by gamma if necessary.)

Method Performance $^{226/228}$Ra in Water

Sample	Volume Liters	Replicates	^{133}Ba Tracer % Recovery	% Recovery ^{226}Ra	% Recovery ^{228}Ra
River Water	1.5	3	101 ± 5	103 ± 1	103 ± 7
Ground Water	1.0	5	95 ± 4	104 ± 1	102 ± 8

1040pCi ^{133}Ba, 5.0pCi ^{226}Ra, 20pCi ^{228}Ra

References

1) Sherrod L. Maxwell, "Rapid Method for ^{226}Ra and ^{228}Ra in Water Samples," *J. Radioanal. Nucl. Chem.*, 270(3), 651-655 (2006).

AN-1418-10

Rapid Determination of ^{226}Ra in Water Samples

Summary of Method ^{226}Ra is separated from up to 1 liter water samples and measured by alpha spectrometry. Radium is precipitated from samples with calcium carbonate. The calcium carbonate precipitate is dissolved in hydrochloric acid, and cation exchange chromatography is used to purify radium and barium from matrix ions. Eichrom DGA Resin is used to remove other alpha emitting nuclides from radium. Samples are prepared for radium measurement by alpha spectrometry using barium sulfate micro-precipitation method onto Eichrom® Resolve Filters. Sample preparation, including alpha spectrometry source preparation, for batches of 12-24 samples can be completed by a single operator in as little as 3-4 hours. Yields can be traced with ^{133}Ba by gamma spectrometry or ^{225}Ra(^{229}Th) by alpha spectrometry. If tracing with ^{225}Ra, >8 hours of ingrowth time for the alpha emitting ^{217}At daughter of ^{225}Ra is required prior to measurement by alpha spectrometry.

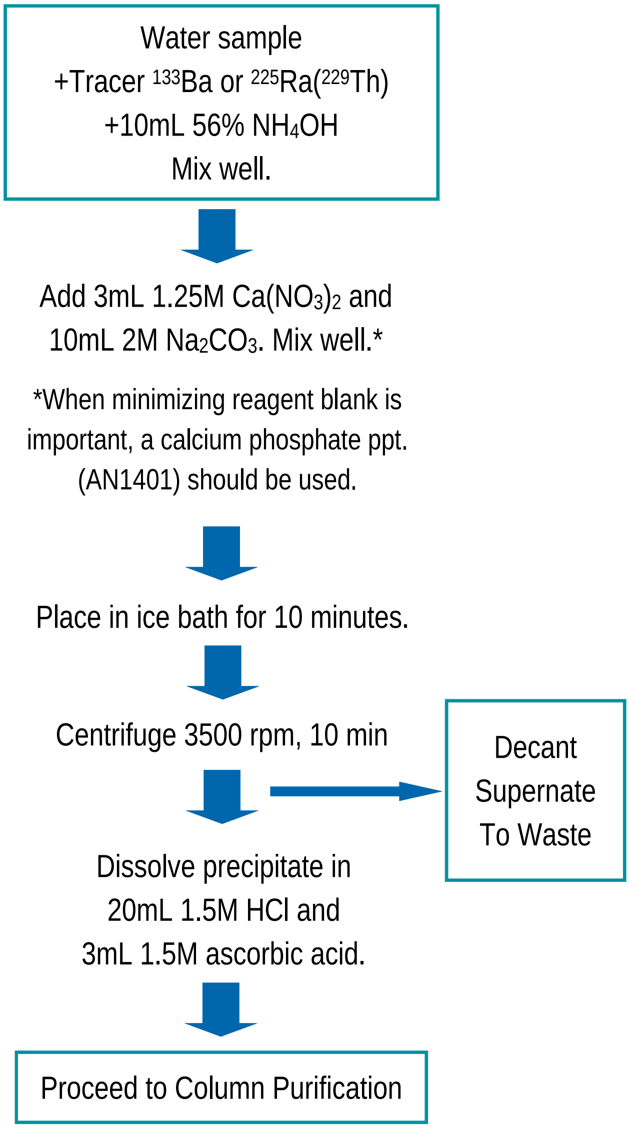
Reagents

Cation Exchange Resin (Eichrom C8-B500-F-H)
DGA Resin, Normal 2mL Cartridges (Eichrom DN-R50-S)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
^{133}Ba or ^{225}Ra(^{229}Th) Tracer
Nitric Acid (70%)
1.25M $Ca(NO_3)_2$
Barium Carrier (1mg/mL)
Ammonium Sulfate
Denatured Ethanol
H_2O_2 (30%)
Hydrochloric Acid (37%)
2M Na_2CO_3
Isopropyl Alcohol
Ascorbic Acid
Deionized Water

Equipment

Plastic Chromatography Column (Eichrom AC-50E-5M)
Column Extension Funnel (Eichrom AC-20X-20M)
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 Filter in Disposable Funnel (Eichrom RF-DF-25-25PP01)
50mL and 250mL Centrifuge Tubes
Centrifuge
Stainless Steel Planchets with adhesive tape
Hotplate
Alpha Spectrometry System
Gamma Spectrometry System (if ^{133}Ba tracer used)
150mL Glass beakers
Vacuum Pump
Heat Lamp

Figure 1. Sample Preparation



Water sample
+Tracer ^{133}Ba or ^{225}Ra(^{229}Th)
+10mL 56% NH_4OH
Mix well.

Add 3mL 1.25M $Ca(NO_3)_2$ and
10mL 2M Na_2CO_3. Mix well.*

*When minimizing reagent blank is
important, a calcium phosphate ppt.
(AN1401) should be used.

Place in ice bath for 10 minutes.

Centrifuge 3500 rpm, 10 min

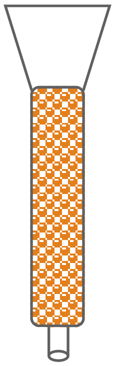
Decant
Supernate
To Waste

Dissolve precipitate in
20mL 1.5M HCl and
3mL 1.5M ascorbic acid.

Proceed to Column Purification

Figure 2. Column Purification and Alpha Source Preparation

(1) Prewash 5.0g 50Wx8 200-400 mesh, cation exchange resin column[1]:
- -10mL deionized water
- -20mL 6M HCl
- -10mL 0.5M HCl



(2) Load Sample[2]

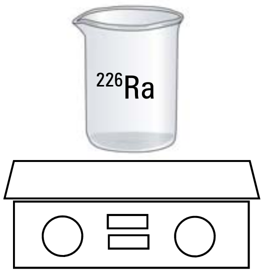
(3) Rinse 30mL 3M HCl

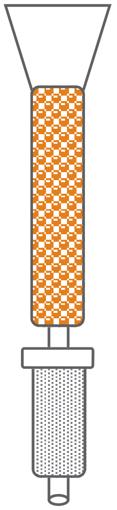
(4) Add 2mL DGA cartridge below cation exchange column.

(5) Strip Ra/Ba with 25mL 5M HNO_3.

(6) Add 2mL 30% H_2O_2. Evaporate to dryness.

(7) Dissolve residue in 10mL 1.5M HCl.





(8) Add 50ug Ba carrier. Mix well.

(9) Add 3g $(NH_4)_2SO_4$ and 5mL iso-propanol. Mix well.

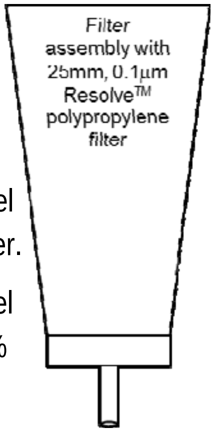
(10) Place in ice bath for 30 minutes.

(11) Set up Resolve® Filter Funnel on vacuum box.

(12) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(13) Filter sample.

(14) Rinse sample tube with 5mL DI water and add to filter.



(15) Rinse filter funnel with 3mL DI water.

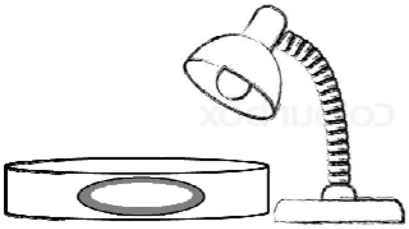
(16) Rinse filter funnel with 1-2mL 100% ethanol.

(17) Draw vacuum until filter is dry.

(18) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.

(19) Dry filter under heat lamp for 3-5 minutes.

(20) Measure ^{226}Ra and ^{225}Ra(^{217}At) by alpha spectrometry after >8 hours ^{217}At ingrowth. (^{133}Ba by gamma, if necessary.)



[1] If using ^{133}Ba tracer, 3.0g of cation exchange resin and proportionally smaller rinse volumes may be used.

[2] If tracing with ^{229}Th, a 20mL 1M HCl-1M H_3PO_4 rinse following the sample load can improve purity of final ^{226}Ra fraction.

Method Performance ^{226}Ra in Water

Sample	^{225}Ra(^{217}At) %Yield*	^{226}Ra(mBq/L) Reference	^{226}Ra(mBq/L) Measured	%Bias
1	84.8	73.8	69.6	-5.7
2	87.3	73.8	75.7	2.6
3	86.2	73.8	71.3	-3.4
4	98.7	73.8	66.9	-9.3
AVG	89 ± 6	73.8	70.9 ± 3.7	-3.9

*^{225}Ra tracer is added as ^{229}Th in equilibrium with its daughters and measured by its alpha emitting ^{217}At daughter (7.066MeV) after >8 hr ingrowth.

References

1) Sherrod L. Maxwell, Brian K. Culligan, "Rapid Determination of ^{226}Ra in Environmental Samples," *J. Radioanal. Nucl. Chem.*, 293(1), 149-155 (2012).

AN-1419-10

Rapid Determination of ^{226}Ra in Concrete and Brick

Summary of Method ^{226}Ra is separated from 1 gram samples of concrete and brick and measured by alpha spectrometry. Samples are fused with sodium hydroxide at 600°C. The fusion cake is dissolved in water, and radium is precipitated from samples with calcium carbonate. The calcium carbonate precipitate is dissolved in hydrochloric acid, and cation exchange chromatography is used to purify radium and barium from matrix ions. Barium is removed from samples using Eichrom Sr Resin. Eichrom DGA Resin is used to separate other alpha emitting nuclides from radium. Samples are prepared for radium measurement by alpha spectrometry via barium sulfate micro-precipitation onto Eichrom® Resolve Filters. Sample preparation including alpha spectrometry source preparation for batches of 12 samples can be completed by a single operator in as little as 6 hours, with 85-90% yield of Radium. Yields are traced with ^{225}Ra(^{229}Th) by alpha spectrometry. At least 8 hours of ingrowth time for the alpha emitting ^{217}At daughter of ^{225}Ra is required prior to measurement by alpha spectrometry.

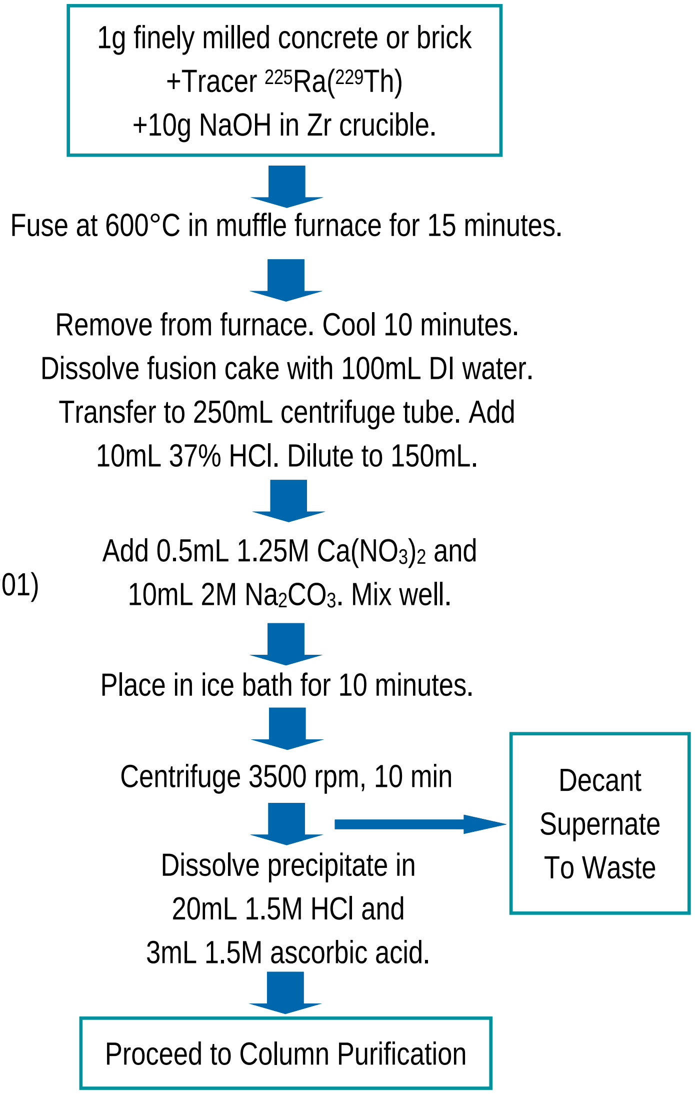
Reagents

Cation Exchange Resin (Eichrom C8-B500-F-H)
Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
DGA Resin, Normal 2mL Cartridges (Eichrom DN-R50-S)
Nitric Acid (70%)
Deionized Water
1.25M Ca(NO$_3$)$_2$
Barium Carrier (1mg/mL)
Ammonium Sulfate
Ascorbic Acid
H$_2$O$_2$(30%)
Hydrochloric Acid (37%)
^{225}Ra(^{229}Th) Tracer
2M Na$_2$CO$_3$
Isopropyl Alcohol
Sodium Hydroxide
Denatured Ethanol

Equipment

Plastic Chromatography Column (Eichrom AC-50E-5M)
Column Extension Funnel (Eichrom AC-20X-20M)
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)
Resolve Filter in Disposable Funnel (Eichrom RF-DF-25-25PP01)
Yellow Outer Tips (Eichrom AC-1000-OT)
50mL and 250mL Centrifuge Tubes
Centrifuge
Stainless Steel Planchets with adhesive tape
Hotplate
Alpha Spectrometry System
150mL Glass beakers
Vacuum Pump
250mL Zirconium Crucible w/ lid
Muffle Furnace
Heat Lamp

Figure 1. Sample Preparation



1g finely milled concrete or brick
+Tracer ^{225}Ra(^{229}Th)
+10g NaOH in Zr crucible.

Fuse at 600°C in muffle furnace for 15 minutes.

Remove from furnace. Cool 10 minutes.
Dissolve fusion cake with 100mL DI water.
Transfer to 250mL centrifuge tube. Add
10mL 37% HCl. Dilute to 150mL.

Add 0.5mL 1.25M Ca(NO$_3$)$_2$ and
10mL 2M Na$_2$CO$_3$. Mix well.

Place in ice bath for 10 minutes.

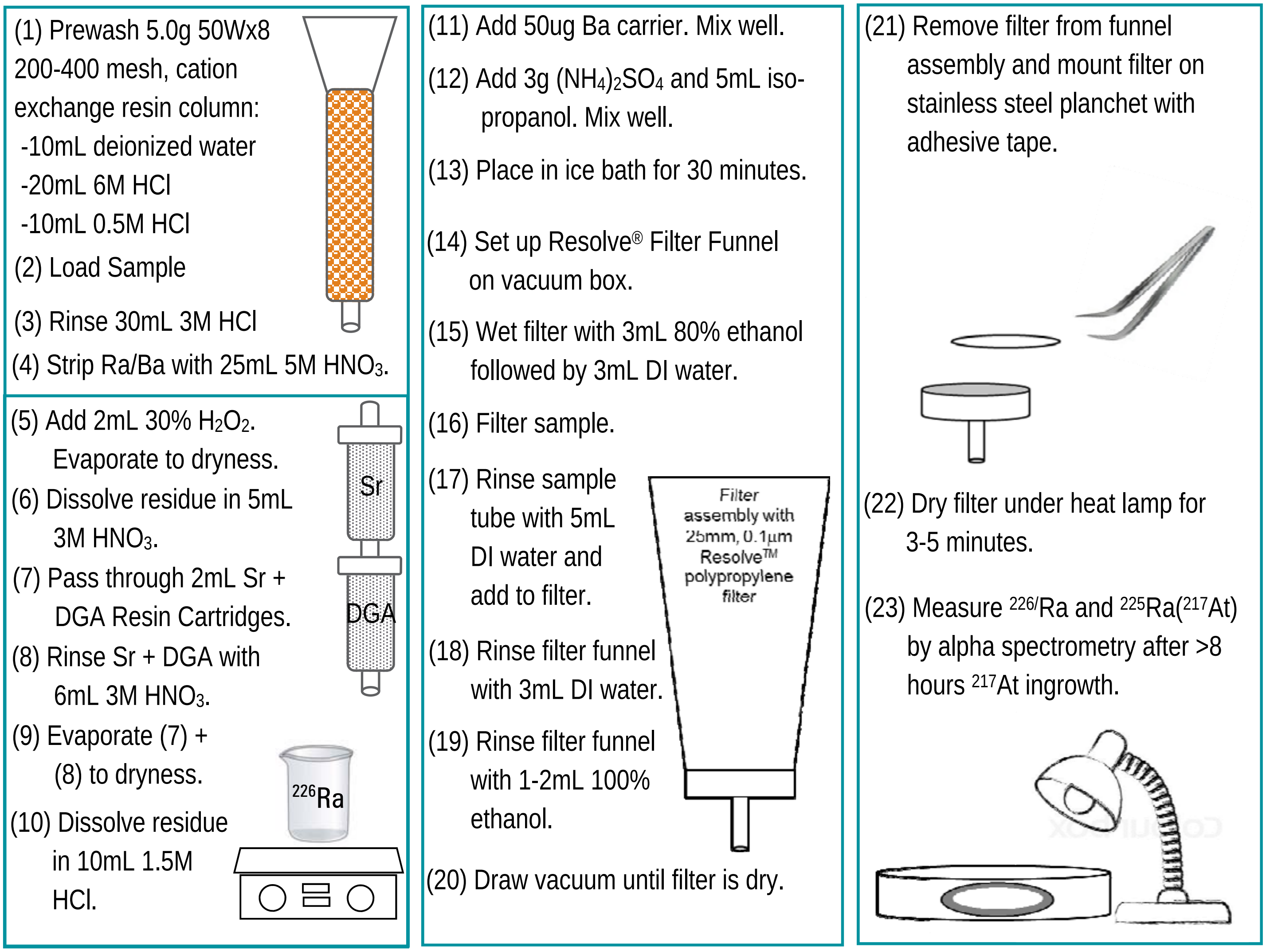
Centrifuge 3500 rpm, 10 min

Decant
Supernate
To Waste

Dissolve precipitate in
20mL 1.5M HCl and
3mL 1.5M ascorbic acid.

Proceed to Column Purification

Figure 2. Column Purification and Alpha Source Preparation



(1) Prewash 5.0g 50Wx8 200-400 mesh, cation exchange resin column:
-10mL deionized water
-20mL 6M HCl
-10mL 0.5M HCl

(2) Load Sample

(3) Rinse 30mL 3M HCl

(4) Strip Ra/Ba with 25mL 5M HNO_3.

(5) Add 2mL 30% H_2O_2. Evaporate to dryness.

(6) Dissolve residue in 5mL 3M HNO_3.

(7) Pass through 2mL Sr + DGA Resin Cartridges.

(8) Rinse Sr + DGA with 6mL 3M HNO_3.

(9) Evaporate (7) + (8) to dryness.

(10) Dissolve residue in 10mL 1.5M HCl.

(11) Add 50ug Ba carrier. Mix well.

(12) Add 3g $(NH_4)_2SO_4$ and 5mL iso-propanol. Mix well.

(13) Place in ice bath for 30 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box.

(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(16) Filter sample.

(17) Rinse sample tube with 5mL DI water and add to filter.

(18) Rinse filter funnel with 3mL DI water.

(19) Rinse filter funnel with 1-2mL 100% ethanol.

(20) Draw vacuum until filter is dry.

(21) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.

(22) Dry filter under heat lamp for 3-5 minutes.

(23) Measure ^{226}Ra and ^{225}Ra(^{217}At) by alpha spectrometry after >8 hours ^{217}At ingrowth.

[1] If using ^{133}Ba tracer, 3.0g of cation exchange resin and proportionally smaller rinse volumes may be used.

[2] If tracing with ^{229}Th, a 20mL 1M HCl-1M H_3PO_4 rinse following the sample load can improve purity of final ^{226}Ra fraction.

Method Performance ^{226}Ra in Concrete and Brick

Sample	Replicates	^{225}Ra(^{217}At) %Yield*	^{226}Ra(mBq/g) Reference	^{226}Ra(mBq/g) Measured	%Bias
Concrete	6	85 ± 7	184.5	181 ± 4	-1.9
Brick	6	87 ± 7	73.8	77.8 ± 4.6	5.4

*^{225}Ra tracer is added as ^{229}Th in equilibrium with its daughters and measured by its alpha emitting ^{217}At daughter (7.066MeV) after >8 hr ingrowth.

References

1) Sherrod L. Maxwell, Brian K. Culligan, "Rapid Determination of ^{226}Ra in Environmental Samples," *J. Radioanal. Nucl. Chem., 293(1), 149-155* (2012).

AN-1420-10

Rapid Determination of ^{226}Ra in Glass Fiber Air Filters

Summary of Method ^{226}Ra is separated from 47mm glass fiber air filters and measured by alpha spectrometry. Samples are fused with sodium hydroxide at 600°C. The fusion cake is dissolved in water, and radium is precipitated from samples with calcium carbonate. The calcium carbonate precipitate is dissolved in hydrochloric acid, and cation exchange chromatography is used to purify radium and barium from matrix ions. Barium is removed from samples using Eichrom Sr Resin. Eichrom DGA Resin is used to separate radium from other alpha emitting nuclides. Samples are prepared for alpha spectrometry by barium sulfate micro-precipitation onto Eichrom® Resolve Filters. Sample preparation, including alpha spectrometry source preparation, for batches of 12 samples can be completed by a single operator in as little as 6 hours, with 85-90% yield of Radium. Yields are traced with ^{225}Ra(^{229}Th) by alpha spectrometry. At least 8 hours of ingrowth time for the alpha emitting ^{217}At daughter of ^{225}Ra is required prior to measurement by alpha spectrometry.

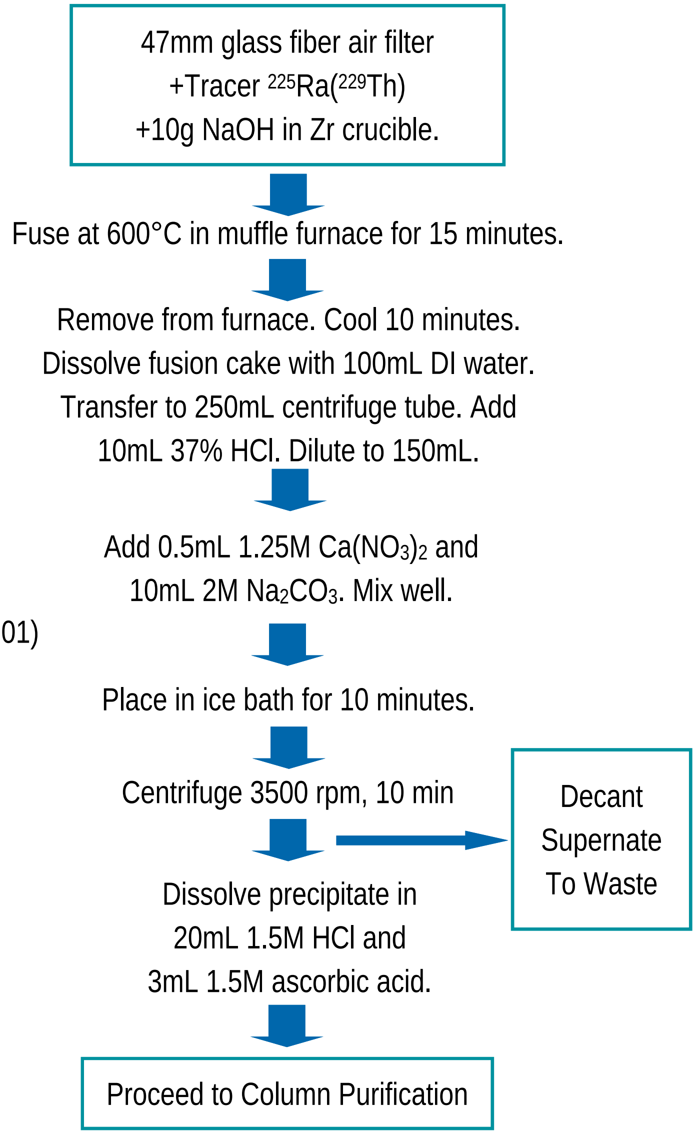
Reagents

Cation Exchange Resin (Eichrom C8-B500-F-H)
Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
DGA Resin, Normal 2mL Cartridges (Eichrom DN-R5S)
Nitric Acid (70%)
Deionized Water
1.25M $Ca(NO_3)_2$
Barium Carrier (1mg/mL)
Ammonium Sulfate
Sodium Hydroxide
H_2O_2(30%)
Hydrochloric Acid (37%)
^{225}Ra(^{229}Th) Tracer
2M Na_2CO_3
Isopropyl Alcohol
Denatured Ethanol
Ascorbic Acid

Equipment

Plastic Chromatography Column (Eichrom AC-50E-5M)
Column Extension Funnel (Eichrom AC-20X-20M)
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 Filter in Disposable Funnel (Eichrom RF-DF-25-25PP01)
50mL and 250mL Centrifuge Tubes
Centrifuge
Stainless Steel Planchets with adhesive tape
Hotplate
Alpha Spectrometry System
150mL Glass beakers
Vacuum Pump
250mL Zirconium Crucible w/ lid
Muffle Furnace
Heat Lamp

Figure 1. Sample Preparation



47mm glass fiber air filter
+Tracer ^{225}Ra(^{229}Th)
+10g NaOH in Zr crucible.

Fuse at 600°C in muffle furnace for 15 minutes.

Remove from furnace. Cool 10 minutes.
Dissolve fusion cake with 100mL DI water.
Transfer to 250mL centrifuge tube. Add
10mL 37% HCl. Dilute to 150mL.

Add 0.5mL 1.25M $Ca(NO_3)_2$ and
10mL 2M Na_2CO_3. Mix well.

Place in ice bath for 10 minutes.

Centrifuge 3500 rpm, 10 min

Decant
Supernate
To Waste

Dissolve precipitate in
20mL 1.5M HCl and
3mL 1.5M ascorbic acid.

Proceed to Column Purification

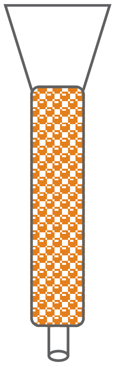
Figure 2. Column Purification and Alpha Source Preparation

(1) Prewash 5.0g 50Wx8 200-400 mesh, cation exchange resin column:
- -10mL deionized water
- -20mL 6M HCl
- -10mL 0.5M HCl

(2) Load Sample

(3) Rinse 30mL 3M HCl

(4) Strip Ra/Ba with 25mL 5M HNO_3.

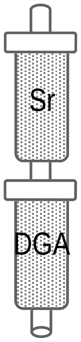


(5) Add 2mL 30% H_2O_2. Evaporate to dryness.

(6) Dissolve residue in 5mL 3M HNO_3.

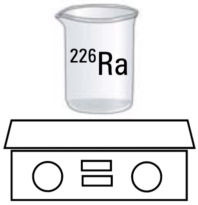
(7) Pass through 2mL Sr + DGA Resin Cartridges.

(8) Rinse Sr + DGA with 6mL 3M HNO_3.



(9) Evaporate (7) + (8) to dryness.

(10) Dissolve residue in 10mL 1.5M HCl.



(11) Add 50ug Ba carrier. Mix well.

(12) Add 3g $(NH_4)_2SO_4$ and 5mL iso-propanol. Mix well.

(13) Place in ice bath for 30 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box.

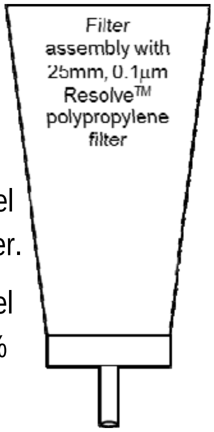
(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(16) Filter sample.

(17) Rinse sample tube with 5mL DI water and add to filter.

(18) Rinse filter funnel with 3mL DI water.

(19) Rinse filter funnel with 1-2mL 100% ethanol.

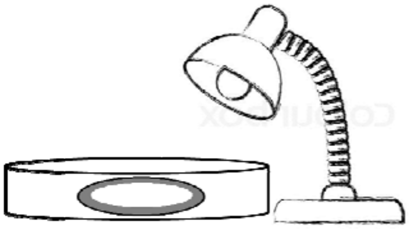


(20) Draw vacuum until filter is dry.

(21) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.

(22) Dry filter under heat lamp for 3-5 minutes.

(23) Measure $^{226/}$Ra and ^{225}Ra(^{217}At) by alpha spectrometry after >8 hours ^{217}At ingrowth.



[1] If using ^{133}Ba tracer, 3.0g of cation exchange resin and proportionally smaller rinse volumes may be used.

[2] If tracing with ^{229}Th, a 20mL 1M HCl-1M H_3PO_4 rinse following the sample load can improve purity of final ^{226}Ra fraction.

Method Performance ^{226}Ra in 47mm Glass Fiber Air Filter

Sample	^{225}Ra(^{217}At) %Yield*	^{226}Ra(mBq/filter) Reference	^{226}Ra(mBq/filter) Measured	%Bias
1	80.7	73.8	70.5	-4.5
2	79.9	73.8	80.8	9.5
3	78.6	73.8	77.0	4.3
4	73.0	73.8	79.5	7.7
5	71.5	73.8	77.7	5.3
AVG	77 ± 4	73.8	77 ± 4	4.3

*^{225}Ra tracer is added as ^{229}Th in equilibrium with its daughters and measured by its alpha emitting ^{217}At daughter (7.066MeV) after >8 hr ingrowth.

References

1) Sherrod L. Maxwell, Brian K. Culligan, "Rapid Determination of ^{226}Ra in Environmental Samples," *J. Radioanal. Nucl. Chem.*, 293(1), 149-155 (2012).

eichrom®

AN-1421-10

Rapid Determination of ^{226}Ra in 1g Soil Samples

Summary of Method ^{226}Ra is separated from 1 gram samples of soil and measured by alpha spectrometry. Samples are fused with sodium hydroxide at 600°C. The fusion cake is dissolved in water, and radium is precipitated from samples with calcium carbonate. The calcium carbonate precipitate is dissolved in hydrochloric acid, and cation exchange chromatography is used to purify radium and barium from matrix ions. Barium is removed from samples using Eichrom Sr Resin. Eichrom DGA Resin is used to reomve other alpha emitting nuclides from radium. Samples are prepared for alpha spectrometry by barium sulfate micro-precipitation onto Eichrom® Resolve Filters. Sample preparation, including alpha spectrometry source preparation, for batches of 12-24 samples can be completed by a single operator in as little as 6 hours. Yields are traced with ^{225}Ra(^{229}Th) by alpha spectrometry. At least 8 hours of ingrowth time for the alpha emitting ^{217}At daughter of ^{225}Ra is required prior to measurement by alpha spectrometry.

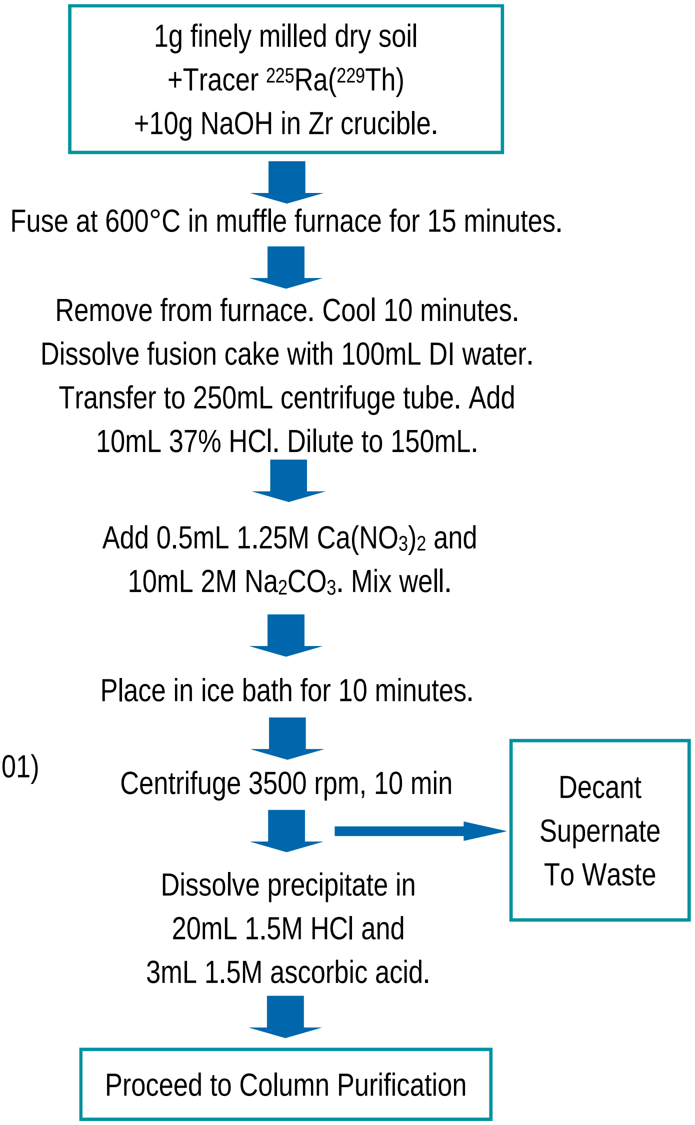
Reagents

Cation Exchange Resin (Eichrom C8-B500-F-H)
Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
DGA Resin, Normal 2mL Cartridges (Eichrom DN-R50-S)
Nitric Acid (70%)
Deionized Water
1.25M $Ca(NO_3)_2$
Barium Carrier (1mg/mL)
Ammonium Sulfate
Ascorbic Acid
H_2O_2(30%)
Hydrochloric Acid (37%)
^{225}Ra(^{229}Th) Tracer
2M Na_2CO_3
Isopropyl Alcohol
Denatured Ethanol
Sodium Hydroxide

Equipment

Plastic Chromatography Column (Eichrom AC-50E-5M)
Column Extension Funnel (Eichrom AC-20X-20M)
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 Filter in Disposable Funnel (Eichrom RF-DF-25-25PP01)
50mL and 250mL Centrifuge Tubes
Centrifuge
Stainless Steel Planchets with adhesive tape
Hotplate
Alpha Spectrometry System
150mL Glass beakers
Vacuum Pump
250mL Zirconium Crucible w/ lid
Muffle Furnace

Figure 1. Sample Preparation



1g finely milled dry soil
+Tracer ^{225}Ra(^{229}Th)
+10g NaOH in Zr crucible.

Fuse at 600°C in muffle furnace for 15 minutes.

Remove from furnace. Cool 10 minutes.
Dissolve fusion cake with 100mL DI water.
Transfer to 250mL centrifuge tube. Add
10mL 37% HCl. Dilute to 150mL.

Add 0.5mL 1.25M $Ca(NO_3)_2$ and
10mL 2M Na_2CO_3. Mix well.

Place in ice bath for 10 minutes.

Centrifuge 3500 rpm, 10 min

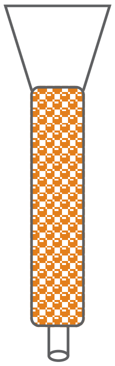
Decant
Supernate
To Waste

Dissolve precipitate in
20mL 1.5M HCl and
3mL 1.5M ascorbic acid.

Proceed to Column Purification

Figure 2. Column Purification and Alpha Source Preparation

(1) Prewash 5.0g 50Wx8 200-400 mesh, cation exchange resin column:
- -10mL deionized water
- -20mL 6M HCl
- -10mL 0.5M HCl



(2) Load Sample

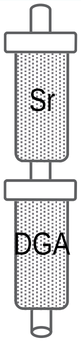
(3) Rinse 30mL 3M HCl

(4) Strip Ra/Ba with 25mL 5M HNO_3.

(5) Add 2mL 30% H_2O_2. Evaporate to dryness.

(6) Dissolve residue in 5mL 3M HNO_3.

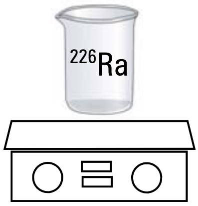
(7) Pass through 2mL Sr + DGA Resin Cartridges.



(8) Rinse Sr + DGA with 6mL 3M HNO_3.

(9) Evaporate (7) + (8) to dryness.

(10) Dissolve residue in 10mL 1.5M HCl.



(11) Add 50ug Ba carrier. Mix well.

(12) Add 3g $(NH_4)_2SO_4$ and 5mL iso-propanol. Mix well.

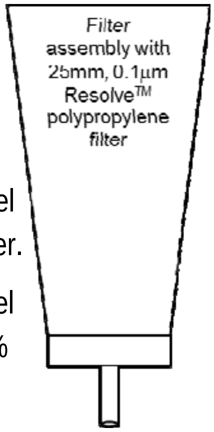
(13) Place in ice bath for 30 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box.

(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(16) Filter sample.

(17) Rinse sample tube with 5mL DI water and add to filter.



(18) Rinse filter funnel with 3mL DI water.

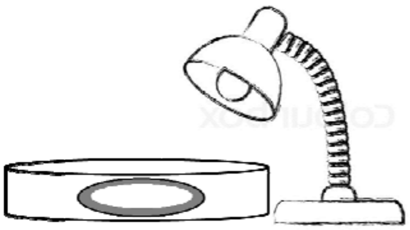
(19) Rinse filter funnel with 1-2mL 100% ethanol.

(20) Draw vacuum until filter is dry.

(21) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.

(22) Dry filter under heat lamp for 3-5 minutes.

(23) Measure ^{226}Ra and ^{225}Ra(^{217}At) by alpha spectrometry after >8 hours ^{217}At ingrowth.



[1] If using ^{133}Ba tracer, 3.0g of cation exchange resin and proportionally smaller rinse volumes may be used.

[2] If tracing with ^{229}Th, a 20mL 1M HCl-1M H_3PO_4 rinse following the sample load can improve purity of final ^{226}Ra fraction.

Method Performance ^{226}Ra in 1g Soil Samples

Sample	^{225}Ra(^{217}At) %Yield*	^{226}Ra(mBq/g) Reference	^{226}Ra(mBq/g) Measured	%Bias
1	75.2	184.5	185.9	0.8
2	77.9	184.5	192.0	4.1
3	74.8	184.5	176.9	-4.1
4	73.3	184.5	184.7	0.1
AVG	75 ± 2	184.5	185 ± 6	0.3

*^{225}Ra tracer is added as ^{229}Th in equilibrium with its daughters and measured by its alpha emitting ^{217}At daughter (7.066MeV) after >8 hr ingrowth.

References

1) Sherrod L. Maxwell, Brian K. Culligan, "Rapid Determination of ^{226}Ra in Environmental Samples," *J. Radioanal. Nucl. Chem.*, 293(1), 149-155 (2012).

eichrom®

AN-1422-10

Rapid Determination of ^{226}Ra in 5g Vegetation Samples

Summary of Method ^{226}Ra is separated from 5 gram samples of vegetation and measured by alpha spectrometry. Samples are fused with sodium hydroxide at 600°C. The fusion cake is dissolved in water, and radium is precipitated from samples with calcium carbonate. The calcium carbonate precipitate is dissolved in hydrochloric acid, and cation exchange chromatography is used to purify radium and barium from matrix ions. Barium is removed from samples using Eichrom Sr Resin. Eichrom DGA Resin is used to separate other alpha emitting nuclides from radium. Samples are prepared for alpha spectrometry by barium sulfate micro-precipitation onto Eichrom® Resolve Filters. Sample preparation, including alpha spectrometry source preparation, for batches of 12 samples can be completed by a single operator in as little as 6 hours. Yields are traced with ^{225}Ra(^{229}Th) by alpha spectrometry. At least 8 hours of ingrowth time for the alpha emitting ^{217}At daughter of ^{225}Ra is required prior to measurement by alpha spectrometry.

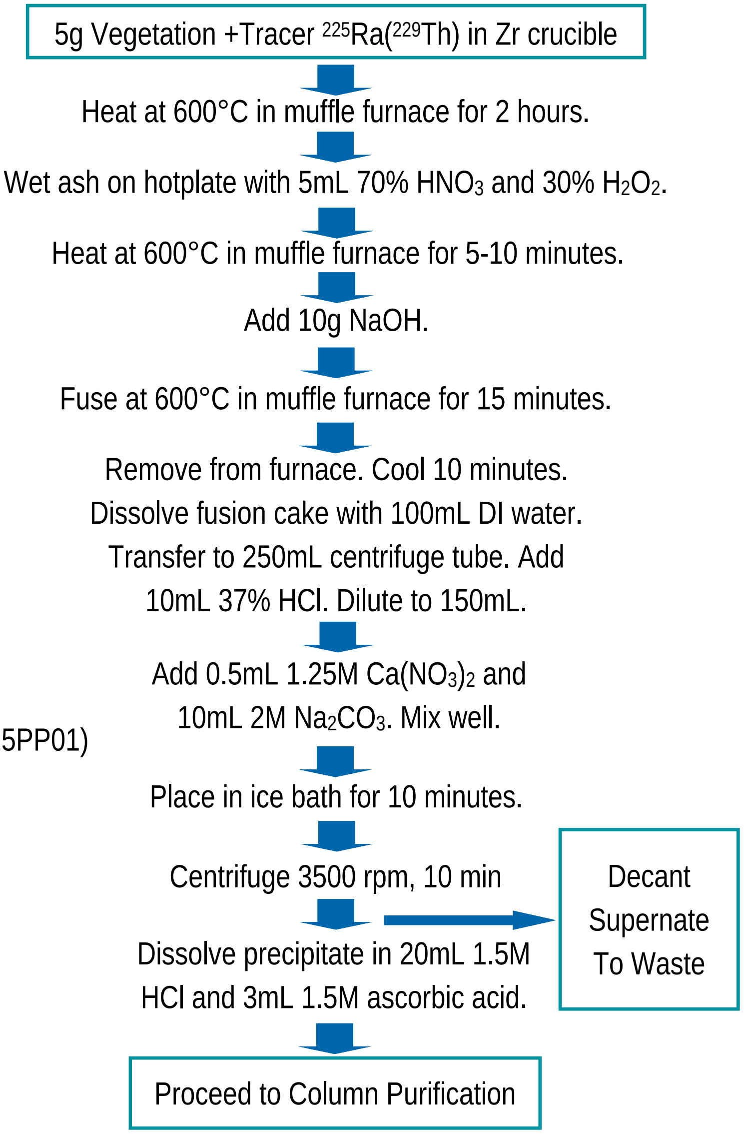
Reagents

Cation Exchange Resin (Eichrom C8-B500-F-H)
Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
DGA Resin, Normal 2mL Cartridges (Eichrom DN-R50-S)
Nitric Acid (70%)
^{225}Ra(^{229}Th) Tracer
2M Na_2CO_3
Isopropyl Alcohol
Denatured Ethanol
Sodium Hydroxide
Hydrochloric Acid (37%)
1.25M $Ca(NO_3)_2$
Barium Carrier (1mg/mL)
Ammonium Sulfate
Ascorbic Acid
Hydrogen Peroxide (30%)

Equipment

Plastic Chromatography Column (Eichrom AC-50E-5M)
Column Extension Funnel (Eichrom AC-20X-20M)
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 Filter in Disposable Funnel (Eichrom RF-DF-25-25PP01)
50mL and 250mL Centrifuge Tubes
Centrifuge
Stainless Steel Planchets with adhesive tape
Hotplate
Alpha Spectrometry System
150mL Glass beakers
Vacuum Pump
250mL Zirconium Crucible w/ lid
Muffle Furnace
Heat Lamp

Figure 1. Sample Preparation



5g Vegetation +Tracer ^{225}Ra(^{229}Th) in Zr crucible

Heat at 600°C in muffle furnace for 2 hours.

Wet ash on hotplate with 5mL 70% HNO_3 and 30% H_2O_2.

Heat at 600°C in muffle furnace for 5-10 minutes.

Add 10g NaOH.

Fuse at 600°C in muffle furnace for 15 minutes.

Remove from furnace. Cool 10 minutes.
Dissolve fusion cake with 100mL DI water.
Transfer to 250mL centrifuge tube. Add
10mL 37% HCl. Dilute to 150mL.

Add 0.5mL 1.25M $Ca(NO_3)_2$ and
10mL 2M Na_2CO_3. Mix well.

Place in ice bath for 10 minutes.

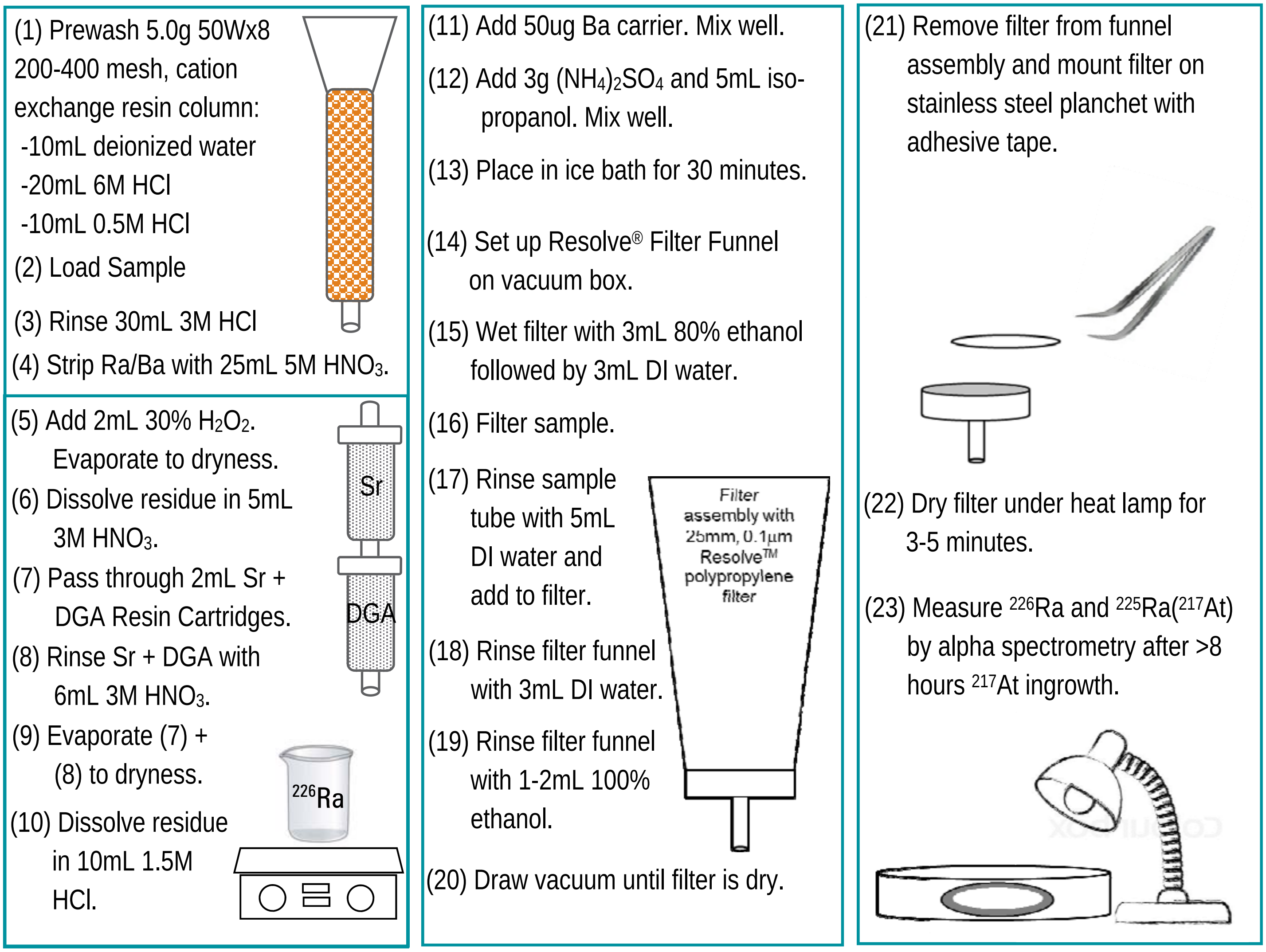
Centrifuge 3500 rpm, 10 min

Decant Supernate To Waste

Dissolve precipitate in 20mL 1.5M
HCl and 3mL 1.5M ascorbic acid.

Proceed to Column Purification

Figure 2. Column Purification and Alpha Source Preparation



(1) Prewash 5.0g 50Wx8 200-400 mesh, cation exchange resin column:
- -10mL deionized water
- -20mL 6M HCl
- -10mL 0.5M HCl

(2) Load Sample

(3) Rinse 30mL 3M HCl

(4) Strip Ra/Ba with 25mL 5M HNO_3.

(5) Add 2mL 30% H_2O_2. Evaporate to dryness.

(6) Dissolve residue in 5mL 3M HNO_3.

(7) Pass through 2mL Sr + DGA Resin Cartridges.

(8) Rinse Sr + DGA with 6mL 3M HNO_3.

(9) Evaporate (7) + (8) to dryness.

(10) Dissolve residue in 10mL 1.5M HCl.

(11) Add 50ug Ba carrier. Mix well.

(12) Add 3g $(NH_4)_2SO_4$ and 5mL iso-propanol. Mix well.

(13) Place in ice bath for 30 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box.

(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(16) Filter sample.

(17) Rinse sample tube with 5mL DI water and add to filter.

(18) Rinse filter funnel with 3mL DI water.

(19) Rinse filter funnel with 1-2mL 100% ethanol.

(20) Draw vacuum until filter is dry.

(21) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.

(22) Dry filter under heat lamp for 3-5 minutes.

(23) Measure ^{226}Ra and ^{225}Ra(^{217}At) by alpha spectrometry after >8 hours ^{217}At ingrowth.

[1] If using ^{133}Ba tracer, 3.0g of cation exchange resin and proportionally smaller rinse volumes may be used.

[2] If tracing with ^{229}Th, a 20mL 1M HCl-1M H_3PO_4 rinse following the sample load can improve purity of final ^{226}Ra fraction.

Method Performance ^{226}Ra in 5g Vegetation Samples

Sample**	^{225}Ra(^{217}At) %Yield*	^{226}Ra(mBq/g) Reference	^{226}Ra(mBq/g) Measured	%Bias
1	91.5	73.8	70.8	-4.1
2	88.3	73.8	73.8	0.0
3	93.1	73.8	69.8	-5.4
4	82.2	73.8	68.5	-7.2
5	80.2	73.8	81.4	10.3
AVG	87 ± 6	73.8	73 ± 5	-1.1

*^{225}Ra tracer is added as ^{229}Th in equilibrium with its daughters and measured by its alpha emitting ^{217}At daughter (7.066MeV) after >8 hours ingrowth.

**5 grams of blank hay matrix spiked with ^{226}Ra

References

1) Sherrod L. Maxwell, Brian K. Culligan, "Rapid Determination of ^{226}Ra in Environmental Samples," *J. Radioanal. Nucl. Chem.*, 293(1), 149-155 (2012).

AN-1423-10

Rapid Determination of Pu, Np, and U in 1-8L Seawater Samples

Summary of Method Plutonium, Neptunium, and Uranium are separated and concentrated from up to 8L samples of seawater with a hydrous titanium oxide precipitation, enhanced with 5mg of lanthanum and 125mg of ferric iron. A second precipitation with lanthanum fluoride removes additional matrix ions, and Uranium and Pu+Np are separated from potentially interfering radionuclides in the sample using stacked 2mL cartridges of Eichrom TEVA and TRU Resins. Isotopic U and Pu+Np are measured by alpha spectrometry following cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Chemical yields are determined by recovery of ^{232}U and ^{242}Pu (or ^{236}Pu if measuring ^{237}Np) tracers. Recoveries of ^{232}U average 95 ± 6%, while ^{236}Pu average 90 ± 9%. Measured values of ^{238}U, ^{239}Pu, and ^{237}Np typically agree to within 10% of reference value. A single operator can process batches of 12 samples through alpha source preparation in 6-8 hours. Alpha spectrometry count times will vary depending on desired detection limit and data quality objectives.

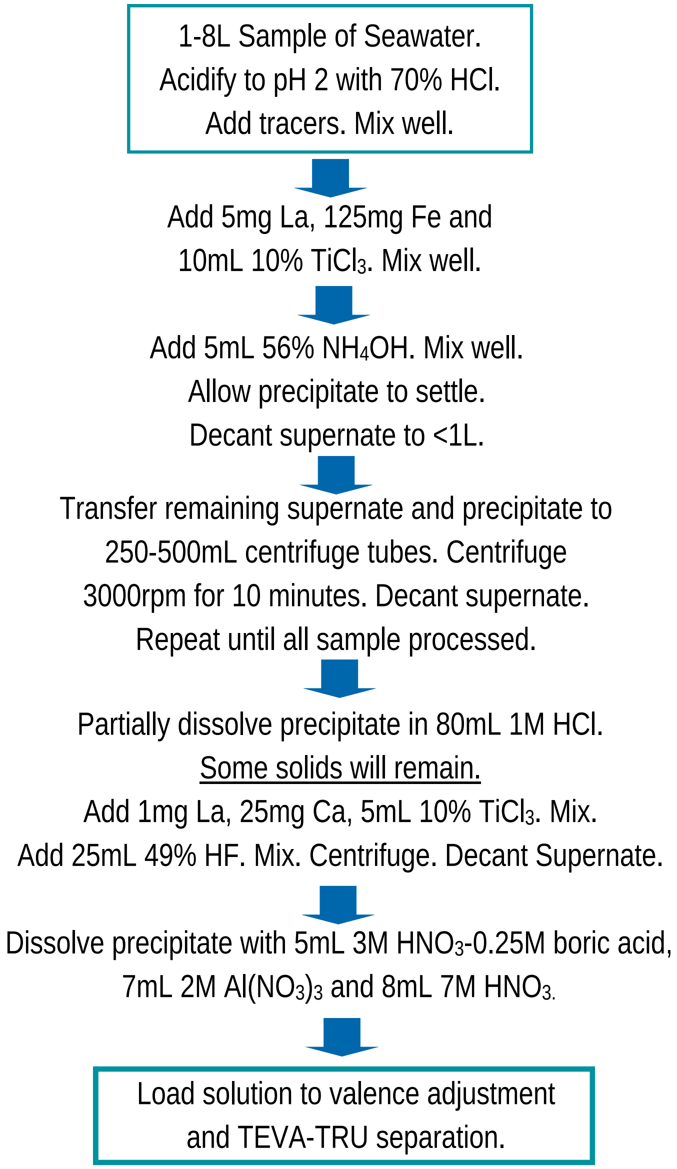
Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Sodium Fluoride
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Lanthanum and Cerium Carriers (1mg/mL)
^{232}U and ^{242}Pu(or ^{236}Pu if meas. ^{237}Np) tracers
Oxalic acid/Ammonium Oxalate
Deionized Water
10% (w:w) $TiCl_3$
Boric acid
$NaNO_2$
Denatured Ethanol
H_2O_2(30%)
2M $Al(NO_3)_3$
Sulfamic Acid
Ascorbic Acid
1.25M $Ca(NO_3)_2$

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 250-500mL Centrifuge Tubes
Centrifuge
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Analytical Balance
Vacuum Pump
Heat Lamp

Figure 1. Sample Preparation



1-8L Sample of Seawater.
Acidify to pH 2 with 70% HCl.
Add tracers. Mix well.

Add 5mg La, 125mg Fe and
10mL 10% $TiCl_3$. Mix well.

Add 5mL 56% NH_4OH. Mix well.
Allow precipitate to settle.
Decant supernate to <1L.

Transfer remaining supernate and precipitate to
250-500mL centrifuge tubes. Centrifuge
3000rpm for 10 minutes. Decant supernate.
Repeat until all sample processed.

Partially dissolve precipitate in 80mL 1M HCl.
<u>Some solids will remain.</u>
Add 1mg La, 25mg Ca, 5mL 10% $TiCl_3$. Mix.
Add 25mL 49% HF. Mix. Centrifuge. Decant Supernate.

Dissolve precipitate with 5mL 3M HNO_3-0.25M boric acid,
7mL 2M $Al(NO_3)_3$ and 8mL 7M HNO_3.

Load solution to valence adjustment
and TEVA-TRU separation.

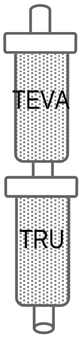
Figure 2. TEVA-TRU Separation and Alpha Source Preparation

(1) Adjust valence states of actinides by adding the following reagents in the order listed (mix between steps):

- 0.2mL 1.5M Sulfamic acid
- 0.01mL 50mg/mL Fe carrier
- 1.5mL 1M Ascorbic acid
- 1mL 3.5M $NaNO_2$

(2) Precondition stacked 2mL TEVA + TRU cartridges with 5mL 3M HNO_3.

(3) Load sample solution at ~1mL/min.



(4) Rinse sample tube with 5mL 3M HNO_3. Add tube rinse to cartridges.

(5) Rinse cartridges with 10mL 3M HNO_3.*

(6) Separate TEVA and TRU cartridges.

(7) Rinse TEVA with 15mL 9M HCl.

(8) Rinse TEVA with 5mL 3M HNO_3.

(9) Strip Pu and Np from TEVA with 20mL 0.1M HCl-0.05M HF-0.01M $TiCl_3$.

(10) Rinse TRU with 20mL 4M HCl-0.2M HF.

(11) Rinse TRU with 12mL 10M HNO_3.

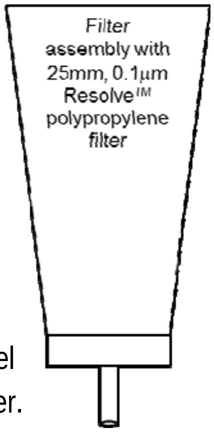
(12) Strip U from TRU with 15mL 0.1M ammonium bioxalate.

(13) Add 0.5mL 10% $TICl_3$ to U samples and 0.5mL 30% H_2O_2 to Pu/Np samples.

(14) Add 50-100ug Ce carrier to each sample. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(15) Set up Resolve® Filter Funnel on vacuum box.

(16) Wet filter with 3mL 80% ethanol followed by 3mL DI water.



(17) Filter sample.

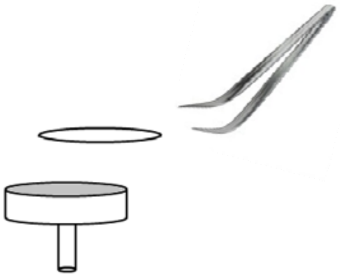
(18) Rinse sample tube with 5mL DI water and add to filter.

(19) Rinse filter funnel with 3mL DI water.

(20) Rinse filter funnel with 1-2mL 100%ethanol.

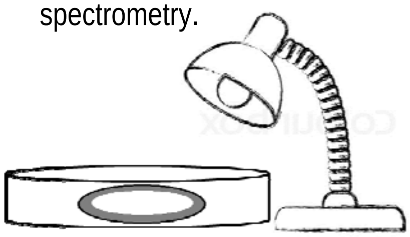
(21) Draw vacuum until filter is dry.

(22) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.



(23) Dry filter under heat lamp for 3-5 minutes.

(24) Measure actinides by alpha spectrometry.



*Adding 50uL of 30% H_2O_2 to tube rinse can improve Uranium recoveries and decontamination in Pu(Np) fractions.

Method Performance Pu, Np and U from Seawater

Analyte	Volume, L	Replicates	Tracer	% Tracer Recovery	Analyte(mBq/L) Reference	Analyte(mBq/L) Measured	% Bias
^{239}Pu	2	5	^{236}Pu	91 ± 9	33.8	32.6 ± 1.4	-3.6
^{239}Pu	4	1	^{236}Pu	86	16.9	16.2	-4.1
^{239}Pu	8	2	^{236}Pu	87 ± 3	27.8	27.6 ± 0.5	-0.7
^{237}Np	2	5	^{236}Pu	91 ± 9	17.4	17.7 ± 1.5	1.7
^{237}Np	4	1	^{236}Pu	86	8.7	7.2	-17
^{237}Np	8	2	^{236}Pu	87 ± 3	4.4	4.2 ± 0.4	-4.5
^{238}U	2	5	^{232}U	99 ± 2	51.8	49.3 ± 1.5	-4.8
^{238}U	4	1	^{232}U	86	25.9	25.0	-3.6
^{238}U	8	2	^{232}U	92 ± 5	96.3	94 ± 3	-2.4

16 hour count times

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, Robin C. Utsey, Daniel R. McAlister, “Rapid determination of actinides in seawater samples,” *J. Radioanal. Nucl. Chem., 300(3), 1175-1189* (2014).

AN-1424-10

Rapid Determination of Pu, Am, and Cm in 80L Seawater Samples

Summary of Method Plutonium, Americium, and Curium are separated and concentrated from up to 80L samples of seawater with a hydrous titanium oxide precipitation, enhanced with lanthanum and ferric iron. A second precipitation with lanthanum fluoride removes additional matrix ions, and Pu and Am+Cm are separated from potentially interfering radionuclides in the sample using stacked 2mL cartridges of Eichrom TEVA and DGA Resins. Isotopic Pu and Am+Cm are measured by alpha spectrometry following cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Chemical yields are determined by recovery of ^{243}Am and ^{242}Pu tracers. Recoveries of ^{243}Am average 94 ± 3%, while ^{242}Pu average 86 ± 4%. Measured values of ^{239}Pu, ^{241}Am, and ^{244}Cm typically agree to within 10% of reference values. A single operator can process batches of 12 samples through alpha source preparation in 6-8 hours. Alpha spectrometry count times will vary depending on desired detection limit and data quality objectives.

Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Ammonium Hydroxide (listed as 28% NH_3 or 56% NH_4OH)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Sodium Fluoride
Deionized Water
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Lanthanum and Cerium Carriers (1mg/mL)
^{243}Am and ^{242}Pu tracers
10% (w:w) $TiCl_3$
2M $Al(NO_3)_3$
Sulfamic Acid
$NaNO_2$
H_2O_2(30%)
Boric acid
Ascorbic Acid
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 250-500mL Centrifuge Tubes
Centrifuge
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Analytical Balance
Vacuum Pump
Heat Lamp

Figure 1. Sample Preparation

1-80L Sample of Seawater.
Acidify to pH 2 with 70% HCl.
Add tracers. Mix well.

↓

Add 15mg La, 50mg Fe/L sample and
1mL 10% $TiCl_3$/L sample. Mix well.

↓

Add 2.5mL 56% NH_4OH/L sample.
Mix well. Allow precipitate to settle.
Decant supernate to <1-2L.

↓

Transfer remaining supernate and precipitate to
250-500mL centrifuge tubes. Centrifuge
3000rpm for 10 minutes. Decant supernate.
Repeat until all sample processed.

↓

Partially dissolve precipitate in 120mL 1M HCl.
<u>Some solids will remain.</u>
Add 15mL 10% $TiCl_3$. Mix. Add 50mL 49% HF. Mix.
Wait 15 minutes. Centrifuge. Decant Supernate.

↓

Dissolve precipitate with 10mL 3M HNO_3-0.25M boric
acid, 12mL 2M $Al(NO_3)_3$ and 9mL 7M HNO_3.

↓

Load solution to valence adjustment
and TEVA-DGA separation.

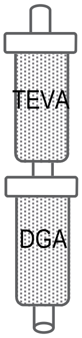
Figure 2. TEVA-DGA Separation and Alpha Source Preparation

(1) Adjust valence states of actinides by adding the following reagents in the order listed (mix between steps):

- 0.2mL 1.5M Sulfamic acid
- 0.01mL 50mg/mL Fe carrier
- 1.5mL 1M Ascorbic acid
- 1mL 3.5M $NaNO_2$

(2) Precondition stacked 2mL TEVA + DGA cartridges with 5mL 3M HNO_3.

(3) Load sample solution at ~1mL/min.



(4) Rinse sample tube with 5mL 6M HNO_3.* Add tube rinse to cartridges.

(5) Rinse cartridges with 10mL 3M HNO_3.

(6) Separate TEVA and DGA cartridges.

(7) Rinse TEVA with 15mL 9M HCl.

(8) Rinse TEVA with 12mL 3M HNO_3.

(9) Strip Pu and Np from TEVA with 20mL 0.1M HCl-0.05M HF-0.01M $TiCl_3$. Add 0.5mL 30% H_2O_2.

(10) Rinse DGA with 15mL 3M HCl.

(11) Rinse DGA with 4mL 1M HNO_3.

(12) Rinse DGA w/ 30mL 0.05M HNO_3.

(13) Rinse DGA with 16mL 3M HNO_3-0.25M HF.

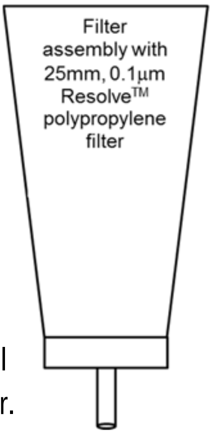
(14) Rinse DGA with 8mL 3M HCl.

(15) Strip Am+Cm from DGA with 20mL 0.25M HCl. Add 0.2mL 30% H_2O_2.

(16) Add 50-100ug Ce carrier. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(17) Set up Resolve® Filter Funnel on vacuum box.

(18) Wet filter with 3mL 80% ethanol followed by 3mL DI water.



(19) Filter sample.

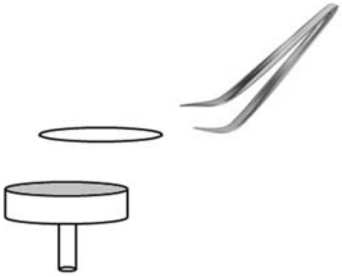
(20) Rinse sample tube with 5mL DI water and add to filter.

(21) Rinse filter funnel with 3mL DI water.

(22) Rinse filter funnel with 1-2mL 100%ethanol.

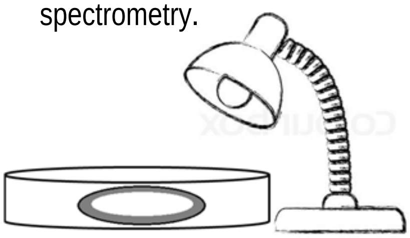
(23) Draw vacuum until filter is dry.

(24) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.



(25) Dry filter under heat lamp for 3-5 minutes.

(26) Measure actinides by alpha spectrometry.



*Adding 50uL of 30% H_2O_2 to the tube rinse can improve Uranium recoveries and decontamination in Pu(Np) fractions.

Method Performance Pu, Am and Cm from Seawater

Analyte	Volume, L	Replicates	Tracer	% Tracer Recovery	Analyte(mBq/L) Reference	Analyte(mBq/L) Measured	% Bias
^{239}Pu	16	2	^{242}Pu	90 ± 1	4.22	4.67 ± 0.05	11
^{239}Pu	25	2	^{242}Pu	84.6 ± 0.2	3.22	3.3 ± 0.1	2.5
^{239}Pu	40	2	^{242}Pu	86 ± 2	0.81	0.82 ± 0.02	1.2
^{239}Pu	80	2	^{242}Pu	85 ± 5	0.40	0.37 ± 0.01	-7.5
^{241}Am	16	2	^{243}Am	95 ± 4	3.31	3.1 ± 0.1	-6.3
^{241}Am	25	2	^{243}Am	93.1 ± 0.1	2.12	1.9 ± 0.1	-10
^{241}Am	40	2	^{243}Am	96 ± 2	0.53	0.51 ± 0.02	-3.8
^{241}Am	80	2	^{243}Am	93 ± 4	0.27	0.25 ± 0.01	-7.4
^{244}Cm	16	2	^{243}Am	95 ± 4	2.16	2.1 ± 0.2	-2.8
^{244}Cm	25	2	^{243}Am	93.1 ± 0.1	1.35	1.3 ± 0.1	-3.7
^{244}Cm	40	2	^{243}Am	96 ± 2	0.85	0.78 ± 0.04	-8.2
^{244}Cm	80	2	^{243}Am	93 ± 4	0.42	0.41 ± 0.01	-2.3

16 hour count times

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, Robin C. Utsey, Daniel R. McAlister, “Rapid determination of actinides in seawater samples,” *J. Radioanal. Nucl. Chem., 300(3), 1175-1189* (2014).

eichrom®

AN-1425-10

Rapid Determination of Actinides in 10g Emergency Food Samples

Summary of Method U, Pu, Np, Am and Cm are separated and concentrated from 10 gram food samples. Samples are muffled at 600°C in zirconium crucibles 2 hours to destroy organic content. The residue is wet ashed with HNO_3-H_2O_2 and then fused with 15g NaOH at 600°C for ten minutes. The fusion cakes are dissolved in water, transferred to 250mL centrifuge tubes and precipitated sequentially with hydrous titanium oxide and lanthanum fluoride to facilitate matrix removal. Actinides are separated on stacked 2mL cartridges of Eichrom TEVA, TRU and DGA resins. Actinides are measured by alpha spectrometry following CeF_3 microprecipitation onto Eichrom Resolve® Filters. Chemical yields of tracers ranged from 93-98% for ^{236}Pu, 85-93% for ^{243}Am, and 78-89% for ^{232}U. Measured values typically agreed to within 10% of reference values. Sample preparation for batches of 12 samples can be completed by a single operator in <8 hours. Alpha spectrometry count times will depend on detection limit and data quality objectives.

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)
Deionized Water
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
La and Ce carriers (1mg/mL)
3.2M $(NH_4)_2HPO_4$
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid

1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
$NaNO_2$
Sulfamic Acid

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
Heat Lamp
Muffle Furnace
Hot Plate
Analytical Balance
250mL Zirconium crucibles with zirconium lids
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Vacuum Pump

Figure 1. Sample Preparation

10g Food sample + tracers in zirconium crucible .

↓

Muffle at 600°C for 2 hours.

↓

Wet ash on hotplate with 5mL 70% HNO_3 and 5mL 30% H_2O_2.

↓

Fuse samples with 15g NaOH at 600°C for 10minutes.

↓

Dissolve fusion cake with H_2O. Transfer to 250mL c-tube.

↓

Add 10mL 3M HNO_3 to crucible. Heat to dissolve residue. Transfer to same 25mL c-tube.

↓

Add 125mg Fe and 5mg La to c-tube. Dilute to 180mL.

↓

Add 4mL 1.25M $Ca(NO_3)_2$, 5mL 3.2M $(NH_4)_2HPO_4$, 5mL 10% $TiCl_3$. Mix. Cool in ice bath for 10min.

↓

Centrifuge at 3500rpm. Decant Supernate.

↓

Partially dissolve precipitate in 60mL 1M HCl. Some solids will remain. Dilute to 170mL. Add 1mg La, 1mL 1.25M $Ca(NO_3)_3$, 3mL 10% $TiCl_3$. Mix. Add 20mL 49% HF.

↓

Centrifuge at 3500rpm. Decant Supernate.

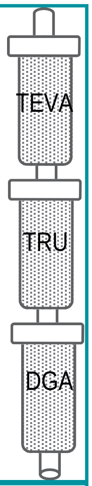
↓

Dissolve precipitate in 5mL 3M HNO_3-0.25M Boric acid, 7mL 70% HNO_3, and 7mL 2M $Al(NO_3)_3$.

↓

Fix valence states. Mix between each addition of: 0.5mL 1.5M sulfamic acid, 10uL 50mg/mL Fe, 1.5mL 1M ascorbic acid, 1mL 3.5M $NaNO_2$, 1.5mL 70% HNO_3.

Figure 2. Actinide Separation on TEVA - TRU - DGA* and Source Preparation



(1) Precondition stacked 2mL TEVA, TRU, DGA cartridges with 10mL 3M HNO_3.
(2) Load sample solution.
(3) Rinse sample tube with 5mL 3M 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
- -5mL 3M HNO_3

(7) Strip Pu (and Np) from TEVA cartridge with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

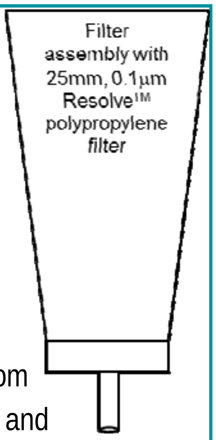
(8) Rinse DGA cartridge with 10mL 0.1M HNO_3.
(9) Place TRU cartridge above DGA.
(10) Strip Am/Cm from TRU onto DGA with 15mL 3M HCl .
(11) Separate TRU cartridge from DGA cartridge.

(12) Rinse DGA cartridge sequentially with:
- -5mL 3M HCl
- -3mL 1M HNO_3
- -15mL 0.05M HNO_3

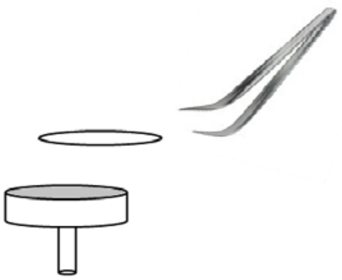
(13) Strip Am and Cm from DGA with 10mL 0.25M HCl.

(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 filter.



(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.

*Adding 50uL 30% H_2O_2 to the tube rinse can improve Uranium recoveries and decontamination in Pu(Np) fractions.

Method Performance Actinides in 10 Gram Food Samples (16 hour count times)

Sample	Replicates	Analyte	Tracer	% Tracer Recovery	Analyte (mBq/g) Reference	Analyte (mBq/g) Measured	% Bias
Baby Food	5	^{238}Pu	^{236}Pu	93.5 ± 7.5	2.9	2.9 ± 0.1	-0.7
	5	^{239}Pu	^{236}Pu	93.5 ± 7.5	3.6	3.3 ± 0.4	-7.9
	5	^{237}Np	^{236}Pu	93.5 ± 7.5	3.7	3.4 ± 0.2	-8.1
	5	^{241}Am	^{243}Am	84.6 ± 6.3	5.1	5.0 ± 0.1	-3.5
	5	^{244}Cm	^{243}Am	84.6 ± 6.3	3.5	3.7 ± 0.3	4.4
	5	^{238}U	^{232}U	78 ± 10	5.7	5.6 ± 0.4	-1.5
	5	^{234}U	^{232}U	78 ± 10	5.9	5.9 ± 0.2	-0.3

Sample	Replicates	Analyte	Tracer	% Tracer Recovery	Analyte (mBq/g) Reference	Analyte (mBq/g) Measured	% Bias
Apples	5	^{238}Pu	^{236}Pu	98 ± 12	2.9	2.9 ± 0.1	-0.5
	5	^{239}Pu	^{236}Pu	98 ± 12	3.6	3.6 ± 0.4	-0.9
	5	^{237}Np	^{236}Pu	98 ± 12	3.7	3.3 ± 0.1	-11.5
	5	^{241}Am	^{243}Am	93.4 ± 8.5	5.1	4.9 ± 0.3	-2.8
	5	^{244}Cm	^{243}Am	93.4 ± 8.5	3.5	3.7 ± 0.5	6.3
	5	^{238}U	^{232}U	89 ± 10	5.7	5.6 ± 0.3	-1.2
	5	^{234}U	^{232}U	89 ± 10	5.9	5.5 ± 0.4	-6.4

References

1) Sherrod L. Maxwell, Brian K. Culligan, Angel Kelsy-Wall, Patrick J. Shaw, “Rapid separation of actinides and in emergency food samples,” *J. Radioanal. Nucl. Chem.*, 292(1), 339-347 (2011).

AN-1426-10

Rapid Determination of Actinides in 100g Emergency Food Samples

Summary of Method U, Pu, Np, Am and Cm are separated and concentrated from 100gram food samples. Samples are muffled at 600°C in zirconium crucibles 2 hours to destroy organic content. The residue is wet ashed with HNO_3-H_2O_2 and then fused with 15g NaOH at 600°C for ten minutes. The fusion cakes are dissolved in water, transferred to 250mL centrifuge tubes and precipitated sequentially with hydrous titanium oxide and lanthanum fluoride to facilitate matrix removal. Actinides are separated on stacked 2mL cartridges of Eichrom TEVA, TRU and DGA resins. Actinides are measured by alpha spectrometry following CeF_3 microprecipitation onto Eichrom Resolve® Filters. Chemical yields of tracers ranged from 93-98% for ^{236}Pu, 85-93% for ^{243}Am, and 78-89% for ^{232}U. Measured values typically agreed to within 10% of reference values. Sample preparation for batches of 12 samples can be completed by a single operator in <16hours. Alpha spectrometry count times will depend on detection limit and data quality objectives.

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)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
La and Ce carriers (1mg/mL)
Deionized Water
3.2M $(NH_4)_2HPO_4$
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
$NaNO_2$
Sulfamic Acid

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
250mL Zirconium crucibles with zirconium lids
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Centrifuge
Analytical Balance
Vacuum Pump
Muffle Furnace
1L Glass Beakers
Heat Lamp

Figure 1. Sample Preparation

100g Food sample + tracers in 1L glass beaker

Muffle at 550°C for 12 hours.

Transfer ash to Zr-crucible. Rinse beaker with 10mL 70% HNO_3 and 10mL 30% H_2O_2.Transfer to crucible. Wet ash.

Fuse samples with 15g NaOH at 600°C for 10minutes.

Dissolve fusion cake with H_2O. Transfer to 250mL c-tube.

Add 10mL 3M HNO_3 to crucible. Heat to dissolve residue. Add to same c-tube.

Add 125mg Fe and 5mg La to c-tube. Dilute to 180mL.

Add 4mL 1.25M $Ca(NO_3)_2$, 5mL 3.2M $(NH_4)_2HPO_4$, 5mL 10% $TiCl_3$. Mix. Cool in ice bath for 10min.

Centrifuge at 3500rpm. Decant Supernate.

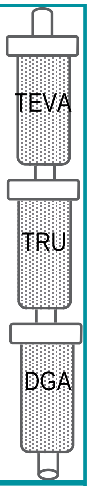
Partillay dissolve precipitate in 60mL 1M HCl. Some solids will remain. Dilute to 170mL. Add 1mg La, 1mL 1.25M $Ca(NO_3)_3$, 3mL 10% $TiCl_3$. Mix. Add 20mL 49% HF.

Centrifuge at 3500rpm. Decant Supernate.

Dissolve precipitate in 5mL 3M HNO_3-0.25M Boric acid, 7mL 70% HNO_3, and 7mL 2M $Al(NO_3)_3$.

Fix valence states. Mix between each addition of: 0.5mL 1.5M sulfamic acid, 10uL 50mg/mL Fe, 1.5mL 1M ascorbic acid, 1mL 3.5M $NaNO_2$, 1.5mL 70% HNO_3.

Figure 2. Actinide Separation on TEVA - TRU - DGA* and Source Preparation



(1) Precondition stacked 2mL TEVA, TRU, DGA cartridges with 10mL 3M HNO_3.

(2) Load sample solution.

(3) Rinse sample tube with 5mL 3M 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
- -5mL 3M HNO_3

(7) Strip Pu (and Np) from TEVA cartridge with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

(8) Rinse DGA cartridge with 10mL 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 cartridge from DGA cartridge.

(12) Rinse DGA cartridge sequentially 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.

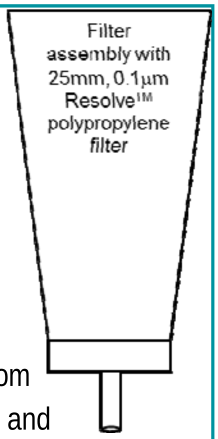
(17) Add 50-100ug Ce carrier to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(18) Set up Resolve® Filter Funnel on vacuum box.

(19) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(20) Filter sample.

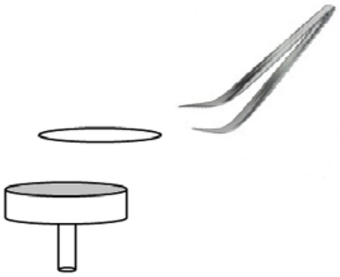
(21) Rinse sample tube with 5mL DI water and add to filter.



(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.

*Adding 50uL 30% H_2O_2 to tube rinse can improve Uranium recoveries and decontamination in Pu(Np) fractions.

Method Performance 100g Apple Samples (16 hr count times)

Sample	Replicates	Analyte	Tracer	% Tracer Recovery	Analyte (mBq/g) Reference	Analyte (mBq/g) Measured	% Bias
Apples	5	^{238}Pu	^{236}Pu	78 ± 8	0.29	0.30 ± 0.02	3.1
	5	^{239}Pu	^{236}Pu	78 ± 8	0.36	0.37 ± 0.05	4.0
	5	^{237}Np	^{236}Pu	78 ± 8	0.37	0.36± 0.02	-3.3
	5	^{241}Am	^{243}Am	76 ± 3	0.25	0.25 ± 0.02	-2.3
	5	^{244}Cm	^{243}Am	76 ± 3	0.35	0.41 ± 0.03	16
	5	^{238}U	^{232}U	71 ± 5	0.57	0.56 ± 0.04	-1.4
	5	^{234}U	^{232}U	71 ± 5	0.59	0.58 ± 0.05	-2.7

References

1) Sherrod L. Maxwell, Brian K. Culligan, Angel Kelsy-Wall, Patrick J. Shaw, “Rapid separation of actinides and in emergency food samples,” *J. Radioanal. Nucl. Chem.*, 292(1), 339-347 (2011).

AN-1427-10

Rapid Determination of Plutonium in Large Rice Samples

Summary of Method Plutonium is separated and measured from up to 1.5kg rice samples. Rice samples are muffled and wet ashed to reduce volume and destroy organic content. The residue is then fused with sodium hydroxide. Precipitation steps remove additional matrix and prepare plutonium for separation on Eichrom TEVA resin. Plutonium is measured by alpha spectrometry following cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Plutonium recovery through the method, determined using ^{242}Pu tracer, was 87 ± 4% for 1kg samples. Measured values for ^{239}Pu and ^{238}Pu agreed within 6% of reference values, even when refractory ^{239}Pu was present in the sample. Sample preparation can be completed in less than 48 hours.

Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
La carrier (10mg/mL)
Ce carrier (1mg/mL)
Deionized Water
3.2M $(NH_4)_2HPO_4$
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
$NaNO_2$
Sulfamic Acid
^{242}Pu tracer

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
250mL Zirconium crucibles with zirconium lids
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Centrifuge
Muffle Furnace
Analytical Balance
600mL glass beakers
Heat Lamp
Hot Plate
Vacuum Pump

Figure 1. Sample Preparation

Rice Sample + tracers in 600mL glass beaker(s). Multiple beakers may be needed for large samples.

↓

Muffle for 5 hours at 350°C, then 550°C for 12 hours.

↓

Carefully wet ash with enough 1:1 (v:v) 70% HNO_3:30% H_2O_2 to cover sample.

↓

Heat to dryness. Muffle 550°C for 6-12 hours.

↓

Transfer residue to Zr crucible. Rinse beaker with 70% HNO_3. Add rinse to crucible. Wet ash with 1:1 70% HNO_3:30% H_2O_2. Heat to dryness.

↓

Repeat wet ash until no black char remains (violet residue common).

↓

Fuse samples with 15g NaOH at 600°C for 20-30 minutes.

↓

Dissolve fusion cake with H_2O. Transfer to 250mL c-tube.

↓

Add 10mL 3M HNO_3 to crucible. Heat to dissolve residue. Transfer to same 25mL c-tube.

↓

Add 125mg Fe and 10mg La to c-tube. Dilute to 180mL.

↓

Add 2mL 1.25M $Ca(NO_3)_2$, 1mL 3.2M $(NH_4)_2HPO_4$, 6mL 10% $TiCl_3$. Mix. Cool in ice bath for 10min.

↓

Centrifuge at 3500rpm. Decant Supernate.

↓

Partially dissolve precipitate in 100mL 1.5M HCl. Some solids will remain. Dilute to 170mL. Add 5mL 10% $TiCl_3$ and 22mL 49% HF. Mix.

↓

Centrifuge at 3500rpm. Decant Supernate.

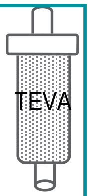
↓

Dissolve precipitate in 5mL 3M HNO_3-0.25M Boric acid, 7mL 6M HNO_3, and 7mL 2M $Al(NO_3)_3$. Cool to room temperature.

↓

Fix valence states. Mix between each addition of: 0.5mL 1.5M sulfamic acid, 40uL 50mg/mL Fe, 1.5mL 1M ascorbic acid, 1mL 3.5M $NaNO_2$.

Figure 2. Plutonium Separation on TEVA Resin and Source Preparation



(1) Precondition 2mL TEVA, 5mL 3M HNO_3.

(2) Load sample solution.

(3) Rinse sample tube with 5mL 3M HNO_3.* Add tube rinse to cartridges.

(4) Rinse TEVA cartridge with:
- -15mL 3M HNO_3
- -20mL 9M HCl (Th removal)
- -5mL 3M HNO_3

(5) Strip Pu from TEVA cartridge with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

-If measuring Pu by ICP-MS, Pu may be stripped from TEVA with 20mL of 0.05M HCl-0.025M HF-0.02M hydroxylamine-HCl.

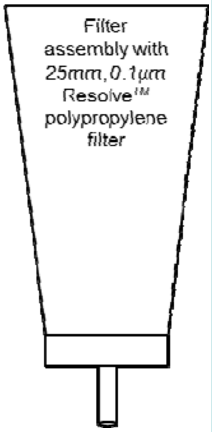
-If preparing Pu sources for alpha spectrometry by electrodeposition, strip Pu with 20mL 0.1M HCl-0.025M HF-0.02M rongalite (sodium-hydroxymethanesulfinate).

(6) Add 0.5mL 30% H_2O_2 for Uranium decontamination in rare earth fluoride precipitation alpha source

(7) Add 50ug Ce carrier to all samples. Mix well.

(8) Add 1mL 49% HF. 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.

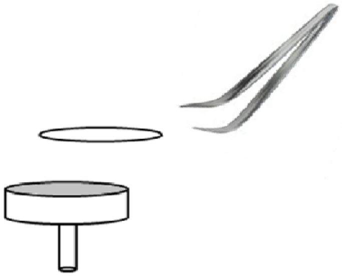
(11) Filter sample.

(12) Rinse sample tube with 5mL DI water and add to filter.

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

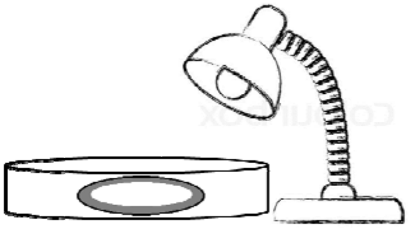
(14) Draw vacuum until filter is dry.

(15) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(16) Dry filter under heat lamp for 3-5 minutes.

(17) Measure plutonium by alpha spectrometry.



*Adding 50uL of 30% H_2O_2 to tube rinse can improve Uranium decontamination.

Method Performance

		^{242}Pu Tracer	Reference (mBq/kg)		Measured (mBq/kg)		% Bias	
Sample (kg)	Replicates	% Yield	^{239}Pu	^{238}Pu	^{239}Pu	^{238}Pu	^{239}Pu	^{238}Pu
1.0	8	87 ± 4	12.5	10.6	11.8 ± 1.0	10.5 ± 0.7	-5.6	-0.7

MDA for 1 kg sample, 30hours count time, 0.37uBq/kg

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, “Rapid fusion method for determination of plutonium isotopes in large rice samples,” *J. Radioanal. Nucl. Chem.*, 298(2), 1367-1374 (2013).

AN-1428-10

Rapid Determination of Actinides in Fecal Samples

Summary of Method Actinides are separated and measured from fecal samples. Fecal samples are muffled and wet ashed prior to fusion with sodium hydroxide. Sequential precipitation steps remove sample matrix prior to actinide separation on 2mL cartridges of Eichrom TEVA, TRU and DGA resins. Actinides are measured by alpha spectrometry following cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Samples can be prepared for measurement in less than 24 hours.

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)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
La carrier (10mg/mL)
Deionized Water
3.2M $(NH_4)_2HPO_4$
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid
Ce carrier (1mg/mL)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
$NaNO_2$
Sulfamic Acid

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
250mL Ceramic crucibles
250mL Zirconium crucibles with zirconium lids
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Centrifuge
Analytical Balance
Vacuum Pump
Hot Plate
Vellum paper
Muffle Furnace
1L Glass Beakers
Heat Lamp

Figure 1. Sample Preparation

Fecal sample + tracers in 1L glass beaker lined with vellum paper.

Muffle at 250°C ~20 min. to dry. Muffle at 350°C for 20 min. Muffle 450°C for 20 min. Muffle 550°C for 45 min.

Transfer char to 250mL ceramic crucible. Muffle at 850°C for 1.5 hours. Transfer solids to 250mL Zr crucible. Rinse ceramic crucible with 70% HNO_3. Add to Zr crucible.

Wet ash material remaining in beaker with 5mL 70% HNO_3 and 5mL 30% H_2O_2. Heat to dryness. Transfer residue to Zr Crucible with 70% HNO_3.

Heat Zr crucibles to dryness on hotplate. Muffle at 600°C until solids turn white/lavender.

Fuse samples with 20g NaOH at 600°C for 15 minutes.

Dissolve fusion cake with H_2O. Transfer to 250mL c-tube.

Add 10mL 3M HNO_3 to crucible. Heat to dissolve residue. Add to same c-tube. Cool to room temp.

Add 125mg Fe and 6mg La to c-tube. Dilute to 180mL.

Add 1mL 1.25M $Ca(NO_3)_2$, 2mL 3.2M $(NH_4)_2HPO_4$, 6mL 10% $TiCl_3$. Mix. Cool in ice bath for 10min.

Centrifuge at 3500rpm. Decant Supernate.

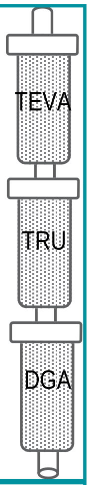
Partially dissolve precipitate in 120mL 1.5M HCl. Some solids will remain. Dilute to 170mL. Add 1mg La, 1mL 1.25M $Ca(NO_3)_3$, and 5mL 10% $TiCl_3$. Mix. Add 22mL 49% HF.

Centrifuge at 3500rpm. Decant Supernate.

Dissolve precipitate in 5mL 3M HNO_3-0.25M Boric acid, 7mL 70% HNO_3, and 7mL 2M $Al(NO_3)_3$.

Fix valence states. Mix between each addition of: 0.5mL 1.5M sulfamic acid, 40uL 50mg/mL Fe, 1.5mL 1M ascorbic acid, 1mL 3.5M $NaNO_2$, 1.5mL 70% HNO_3.

Figure 2. Actinide Separation on TEVA - TRU - DGA* and Source Preparation



(1) Precondition stacked 2mL TEVA, TRU, DGA cartridges with 10mL 3M HNO_3.

(2) Load sample solution.

(3) Rinse sample tube with 5mL 3M 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 (and Np) from TEVA cartridge with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

(8) Add 0.5mL 30% H_2O_2 for Uranium decon. in alpha source preparation.

(9) Rinse DGA cartridge with 10mL 0.1M HNO_3. (U removal).

(10) Place TRU cartridge above DGA.

(11) Strip Am/Cm from TRU onto DGA with 15mL 3M HCl at 1-2mL/min.

(12) Separate TRU cartridge from DGA cartridge. Set TRU aside for U recovery.

(13) Rinse DGA cartridge with:
- -5mL 3M HCl
- -3mL 1M HNO_3
- -15mL 0.05M HNO_3

(14) Strip Am and Cm from DGA with 10mL 0.25M HCl. Add 0.2mL 30% H_2O_2.

(15) Rinse TRU cartridge with:
- -15mL 4M HCl-0.2M HF-2mM $TiCl_3$
- -5mL 8M HNO_3.

(16) Strip U from TRU with 15mL of 0.1M ammonium bioxalate.

(17) Add 0.5mL 10% $TiCl_3$ to U samples.

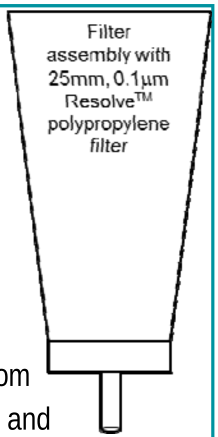
(18) Add 50-100ug Ce carrier to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(19) Set up Resolve® Filter Funnel on vacuum box.

(20) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(21) Filter sample.

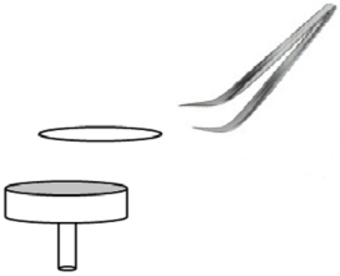
(22) Rinse sample tube with 5mL DI water and add to filter.



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

(24) Draw vacuum until filter is dry.

(25) 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.

*Adding 50uL 30% H_2O_2 can improve Uranium recoveries and decontamination in Pu(Np) fractions.

Method Performance

Analyte	Samples	Tracer	% Tracer Recovery	Reference (Bq/sample)	Measurement (Bq/sample)	% Bias
$^{239/240}$Pu	5	^{242}Pu	95 ± 9	0.085 - 0.204	0.081 - 0.198	-11 to -1.5
^{238}Pu	5	^{242}Pu	95 ± 9	0.066 - 0.156	0.071 - 0.146	-5.3 to 3.0
^{241}Am	5	^{243}Am	83 ± 4	0.199 - 0.476	0.201 - 0.464	-11 to 1.0
^{238}U	5	^{232}U	63 ± 7	0.226 - 0.541	0.196 - 0.592	-9.0 to 2.9
^{234}U	5	^{232}U	63 ± 7	0.218 - 0.521	0.206 - 0.536	-13 to 9.4

6 hour count time

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, Ronie B. Spencer “Rapid fusion method for determination of actinides in fecal samples,” *J. Radioanal. Nucl. Chem.*, 298(3), 1533-1542 (2013).

AN-1429-10

Rapid Determination of Actinides in Asphalt Samples

Summary of Method Actinides are separated and measured from 1g samples of asphalt. Asphalt samples are fused in zirconium crucibles with sodium hydroxide. Sequential precipitations remove matrix prior to separation of actinides on 2mL cartridges of Eichrom TRU and DGA resins. Actinides are measured by alpha spectrometry following cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Chemical recoveries averaged 91±6%, 84±12%, and 86±7%, respectively, for ^{242}Pu, ^{243}Am and ^{232}U tracers. Measured values typically agreed to within 2-6% of reference values. Batches of 12 samples can be prepared for measurement in as little as 4 hours.

Reagents

TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
La carrier (10mg/mL)
Deionized Water
3.2M $(NH_4)_2HPO_4$
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid
Ce carrier (1mg/mL)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
$NaNO_2$
Sulfamic Acid

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
Muffle Furnace
Analytical Balance
250mL Zirconium crucibles with zirconium lids
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Vacuum Pump
Heat Lamp

Figure 1. Sample Preparation

1g finely ground asphalt + tracers in 250mL Zr-crucible

Fuse samples with 15g NaOH at 600°C for 15 minutes.

Dissolve fusion cake with H_2O. Transfer to 250mL c-tube.

Add 10mL 3M HNO_3 to crucible. Heat to dissolve residue. Add to same c-tube.

Add 125mg Fe and 5mg La to c-tube. Dilute to 180mL.

Add 2mL 1.25M $Ca(NO_3)_2$, 5mL 3.2M $(NH_4)_2HPO_4$, 10mL 10% $TiCl_3$. Mix. Cool in ice bath for 10min.

Centrifuge at 3500rpm. Decant Supernate.

Partially dissolve precipitate in 60-80mL 1.5M HCl.*
Dilute to 170mL with 0.01M HCl.
Add 1mg La and 5mL 10% $TiCl_3$. Mix.
Add 20mL 49% HF. Mix. Wait 10 min.

*The entire precipitate will not dissolve in HCl. Dissolution will be completed with the HF addition.

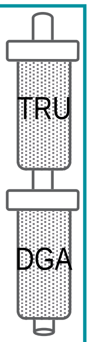
Centrifuge at 3500rpm. Decant Supernate.

Dissolve precipitate in 5mL 3M HNO_3-0.25M Boric acid, 7mL 70% HNO_3, and 7mL 2M $Al(NO_3)_3$.

Fix valence states. Mix between each addition of: 0.5mL 1.5M sulfamic acid, 10uL 50mg/mL Fe, 1.5mL 1M ascorbic acid, 1mL 3.5M $NaNO_2$, 1.5mL 70% HNO_3.

Figure 2. Actinide Separation on TRU/DGA and Source Preparation

(1) Precondition TRU/DGA resin with 5mL 8M HNO_3.
(2) Load samples.
(3) Rinse sample tube with 5mL 8M HNO_3, and add tube rinse to TRU/DGA.*
(4) Rinse TRU/DGA with:
-10mL 10M HNO_3
-15mL 4M HCl
(5) Separate TRU and DGA.



(6) Strip Pu from TRU w/ 15mL 3M HCl-0.02M $TiCl_3$. Add 0.5mL 30% H_2O_2.

(7) Rinse TRU with:
-5mL 8M HNO_3 + 50uL 30% H_2O_2
-10mL 4M HCl-0.2M HF
-10mL 4M HCl-0.2M HF-2mM $TiCl_3$
-3mL 8M HNO_3

(8) Strip U from TRU with 15mL 0.1M ammonium bioxalate. Add 0.5mL $TiCl_3$ for CeF_3 ppt.

(9) Rinse DGA with:
-12mL 3M HCl
-20mL 0.05M HNO_3
-12mL 3M HNO_3-0.25M HF

(10) Rinse DGA with 5mL 3M HCl.
(11) Strip Am/Cm from DGA with 12mL 0.25M HCl. Add 0.2mL 30% H_2O_2.

(12) Add 50-ug Ce carrier to each sample. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(13) Set up Resolve® Filter Funnel on vacuum box.

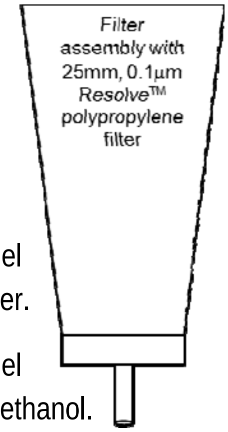
(14) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(15) Filter sample.

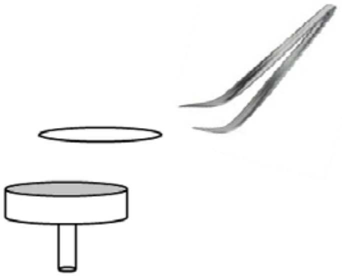
(16) Rinse sample tube with 5mL DI water and add to filter.

(17) Rinse filter funnel with 3mL DI water.

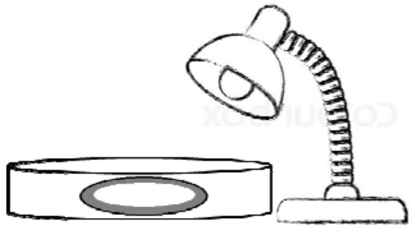
(18) Rinse filter funnel with 2mL 100% ethanol.



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



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



*Adding 50uL of 30% H_2O_2 to the tube rinse can help improve U recoveries and decontamination in Pu(Np) fractions.

Method Performance

Analyte	Replicates	Tracer	% Tracer Recovery	Analyte Reference (mBq/g)	Analyte Measured (mBq/g)	% Bias
^{239}Pu	8	^{242}Pu	91 ± 6	39.2	40 ± 2	2.0
^{241}Am	8	^{243}Am	84 ± 13	24.4	23 ± 3	-5.7
^{244}Cm	8	^{243}Am	84 ± 13	35.5	37 ± 5	4.2
^{238}U	8	^{232}U	86 ± 7	73.6	72 ± 8	-2.1
^{234}U	8	^{232}U	86 ± 7	73.6	72 ± 9	-2.1

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, “Rapid determination of actinides and in asphalt samples,” *J. Radioanal. Nucl. Chem.*, 299(3), 1891-1901 (2014).

AN-1430-10

Rapid Determination of Actinides in Soil Samples

Summary of Method Actinides are separated and measured from 1-2g samples of soil. Soil samples are fused in zirconium crucibles with sodium hydroxide. Sequential precipitations remove matrix prior to separation of actinides on 2mL cartridges of Eichrom TRU and DGA resins. Actinides are measured by alpha spectrometry following cerium fluoride microprecipitation onto Eichrom Resolve® Filters. Chemical recoveries averaged 97±9%, 96±7%, and 91±4%, respectively, for ^{242}Pu, ^{243}Am and ^{232}U tracers. Measured values typically agreed to within 3% of reference values. Batches of 12 samples can be prepared for measurement in as little as 4 hours.

Reagents

TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
La carrier (10mg/mL)
Deionized Water
3.2M $(NH_4)_2HPO_4$
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid
Ce carrier (1mg/mL)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
$NaNO_2$
Sulfamic Acid

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
Muffle Furnace
Analytical Balance
250mL Zirconium crucibles with zirconium lids
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Vacuum Pump
Heat Lamp

Figure 1. Sample Preparation

1-2g finely ground soil + tracers in 250mL Zr-crucible

Fuse samples with 15g NaOH at 600°C for 15 minutes.

Dissolve fusion cake with H_2O. Transfer to 250mL c-tube.

Add 10mL 3M HNO_3 to crucible. Heat to dissolve residue. Add to same c-tube.

Add 125mg Fe and 5mg La to c-tube. Dilute to 180mL.

Add 2mL 1.25M $Ca(NO_3)_2$, 5mL 3.2M $(NH_4)_2HPO_4$, 10mL 10% $TiCl_3$. Mix. Cool in ice bath for 10min.

Centrifuge at 3500rpm. Decant Supernate.

Partially dissolve precipitate in 60-80mL 1.5M HCl.*
Dilute to 170mL with 0.01M HCl.
Add 1mg La and 5mL 10% $TiCl_3$. Mix.
Add 20mL 49% HF. Mix. Wait 10 min.

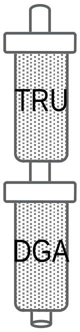
*The entire precipitate will not dissolve in HCl. Dissolution will be completed with the HF addition.

Centrifuge at 3500rpm. Decant Supernate.

Dissolve precipitate in 5mL 3M HNO_3-0.25M Boric acid, 7mL 70% HNO_3, and 7mL 2M $Al(NO_3)_3$.

Fix valence states. Mix between each addition of: 0.5mL 1.5M sulfamic acid, 10uL 50mg/mL Fe, 1.5mL 1M ascorbic acid, 1mL 3.5M $NaNO_2$, 1.5mL 70% HNO_3.

Figure 2. Actinide Separation on TRU/DGA and Source Preparation



(1) Precondition TRU/DGA resin with 5mL 8M HNO_3.

(2) Load samples.

(3) Rinse sample tube with 5mL 8M HNO_3, and add tube rinse to TRU/DGA.*

(4) Rinse TRU/DGA with:
-10mL 10M HNO_3
-15mL 4M HCl

(5) Separate TRU and DGA.

(6) Strip Pu from TRU w/ 15mL 3M HCl-0.02M $TiCl_3$. Add 0.5mL 30% H_2O_2.

(7) Rinse TRU with:
-5mL 8M HNO_3 + 50uL 30% H_2O_2
-10mL 4M HCl-0.2M HF
-10mL 4M HCl-0.2M HF-2mM $TiCl_3$
-3mL 8M HNO_3

(8) Strip U from TRU with 15mL 0.1M ammonium bioxalate. Add 0.5mL $TiCl_3$ for CeF_3 ppt.

(9) Rinse DGA with:
-12mL 3M HCl
-20mL 0.05M HNO_3
-12mL 3M HNO_3-0.25M HF

(10) Rinse DGA with 5mL 3M HCl.

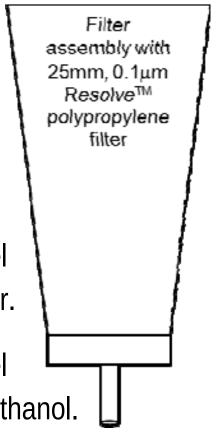
(11) Strip Am/Cm from DGA with 12mL 0.25M HCl. Add 0.2mL 30% H_2O_2.

(12) Add 50-ug Ce carrier to each sample. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(13) Set up Resolve® Filter Funnel on vacuum box.

(14) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(15) Filter sample.



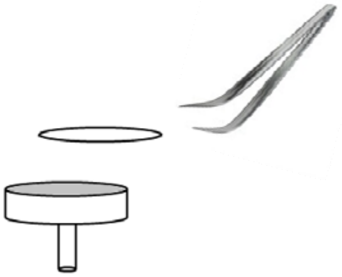
(16) Rinse sample tube with 5mL DI water and add to filter.

(17) Rinse filter funnel with 3mL DI water.

(18) Rinse filter funnel with 2mL 100% ethanol.

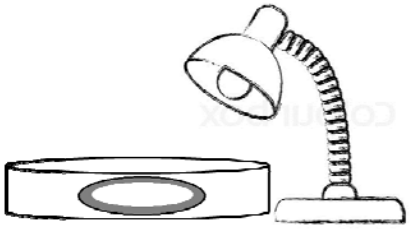
(19) Draw vacuum until filter is dry.

(20) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.



(21) Dry filter under heat lamp for 3-5 minutes.

(22) Measure actinides by alpha spectrometry.



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

Method Performance

Analyte	Replicates	Tracer	% Tracer Recovery	Analyte Reference (mBq/g)	Analyte Measured (mBq/g)	% Bias
^{239}Pu	7	^{242}Pu	97 ± 9	98.0	95 ± 3	-3.1
^{241}Am	7	^{243}Am	96 ± 7	61.1	59 ± 4	-3.4
^{238}U	7	^{232}U	91 ± 4	184	183 ± 6	-0.5

16 hour counts

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchinson, “Rapid determination of actinides and in asphalt samples,” *J. Radioanal. Nucl. Chem.*, 299(3), 1891-1901 (2014).

eichrom®

AN-1431-10

Rapid Determination of Pu, Np, Am and Cm in 100g Soil Samples

Summary of Method Pu(Np) and Am-Cm are separated and concentrated from 100-200 gram soil samples. Samples are muffled at 550°C to destroy organic content and wet ashed and leached with HNO_3 and HCl. The filtered leachates are evaporated to dryness and fused with NaOH in Zr crucibles. Sequential precipitations facilitate matrix removal. Actinides are separated on stacked 2mL cartridges of Eichrom TEVA, TRU, and DGA resins. Native rare earths from the samples are removed from Am-Cm using TEVA Resin and ammonium thiocyanate. Actinides are measured by alpha spectrometry following CeF_3 microprecipitation onto Eichrom Resolve® Filters. Chemical yields of tracers ranged from 93-98% for ^{236}Pu and 85-93% for ^{243}Am. Measured values typically agreed to within 10% of reference values. Sample preparation for batches of 12 samples can be completed by a single operator in <8 hours.

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)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), and ^{243}Am tracers
La carrier (10mg/mL)
Deionized Water
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid
Formic Acid
Ce carrier (1mg/mL)
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
$NaNO_2$
Sulfamic Acid
Ammonium Thiocyanate

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)
1L and 600mL Glass beakers
250mL Zirconium crucibles with lids
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
50mL and 250mL Centrifuge Tubes
25mm 0.45um filters
Centrifuge
Muffle Furnace
Analytical Balance
Heat Lamp
Hot Plate
Vacuum Pump

Figure 1. Sample Preparation

Dry 100-200g soil at 110°C. Blend and Size.

↓

Aliquot sample to 1L glass beaker. Add tracers.

↓

Muffle at 550°C for 4 hours.

↓

Cool. Add 75mL 70% HNO_3 and 25mL 37% HCl.
Heat to dryness on hotplate.

↓

Add 75mL 70% HNO_3. Warm sample. Transfer solids and liquid to 250mL centrifuge tube.

↓

Add 25mL 70% HNO_3 to beaker. Warm beaker. Transfer solids and liquid to 250mL centrifuge tube.

↓

Centrifuge 3500 rpm, 10 min.

↓

Filter leachate through 25mm 0.45um filter. Transfer leachate to 600mL beaker.

↓

Add 25mL 70% HNO_3 to solids. Mix. Centrifuge. Filter leachate into same 600mL beaker. Repeat once.

↓

Add 25mL warm 4M HCl to solids. Mix. Centrifuge. Filter leachate into same 600mL beaker. Repeat once.

↓

Evaporate supernate in 600mL beaker to dryness. Wet ash with 15mL 70% HNO_3 to dryness. Repeat once.

↓

Transfer solids to 250mL Zr crucibles. Rinse beakers with 70% HNO_3. Transfer to same crucible. Heat crucibles to dryness.

↓

Add 20g NaOH to each crucible. Fuse at 600°C for 20min.

↓

Dissolve fusion cakes with water. Transfer to 250mL c-tubes. Rinse crucibles with 10mL 3M HNO_3. Heat to dissolve residue. Transfer to same c-tube. Continue to precipitation steps.

Figure 2. Actinide Separation on TEVA - TRU - DGA and Source Preparation

Dilute samples to 180mL.
Add 7mg La and 20mL 10% $TiCl_3$.
Mix. Cool to room temperature.

↓

Centrifuge 3500 rpm. 5min.
Decant supernate

↓

Partially dissolve in 60mL 1.5M HCl.
Solids will remain. Dilute to 170mL.
Add 2mg La and 10mL 30% H_2O_2. Mix.
Add 22mL 49% HF. Mix.

↓

Centrifuge 3500 rpm. 5min.
Decant supernate

↓

Dissolve solids in 5mL 3M HNO_3-0.25M Boric acid, 6mL 7M HNO_3, and 7.5mL 2M $Al(NO_3)_3$. Warming samples can improve dissolution.

↓

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 $NaNO_2$

(1) Precondition 2mL TEVA, TRU, DGA cartridges with 10mL 8M HNO_3.
(2) Load Sample.
(3) Rinse c-tube with 5mL 6M HNO_3.*
Add to stacked cartridges.

(4) Rinse cartridges w/ 5mL 6M HNO_3.
(5) Separate TEVA from TRU-DGA.

(6) Rinse TEVA with:
-10mL 6M HNO_3
-10mL 3M HNO_3
-20mL 9M HCl (Th removal)
-5mL 3M HNO_3
(7) Strip Pu from TEVA with 20mL 0.1M HCl-0.05M HF-0.03M $TiCl_3$.

(8) Rinse TRU-DGA with 15mL 4M HCl.
(9) Discard TRU cartridge.
(10) Rinse DGA w/ 20mL 0.05M HNO_3.
(11) Strip Am/Cm w/ 10mL 0.25M HCl.

(12) Add 2mL 70% HNO_3 + 50uL 10% H_2SO_4 to Am/Cm. Evaporate to dryness.
(13) Ash to dryness with 3mL 70% HNO_3 + 2mL 30% H_2O_2.
(14) Dissolve Am/Cm in 5mL 4M NH_4SCN-0.1M Formic acid.
(15) Precondition 2mL TEVA with 5mL 4M NH_4SCN-0.1M Formic acid.
(16) Load Am/Cm on TEVA.
(17) Rinse Am/Cm beaker with 5mL 4M NH_4SCN-0.1M Formic acid. Add to TEVA.
(18) Rinse TEVA w/ 10mL 1.5M NH_4SCN-0.1M Formic acid.
(19) Strip Am/Cm from TEVA with 20mL 1M HCl.

(20) Add 50ug Ce carrier to all samples. Add 0.5mL 30% H_2O_2 to Pu samples and 0.2mL 30% H_2O_2 to Am/Cm samples. Mix. Add 1mL 49% HF. Mix. Wait 15-20 minutes.
(21) Set up Resolve® Filter Funnel on vacuum box.
(22) Wet filter with 3mL 80% ethanol followed by 3mL DI water.
(23) Filter sample.
(24) Rinse sample tube with 5mL DI water and add to filter.
(25) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.
(26) Draw vacuum until filter is dry.
(27) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.
(28) Dry filter under heat lamp for 3-5 minutes.
(29) Measure actinides by alpha spectrometry.

*Adding 50uL of 30% H_2O_2 to tube rinse can help improve U decontamination.

Method Performance

Sample Size (g)	Replicates	^{242}Pu Tracer % Recovery	^{238}Pu Measured % Bias	^{243}Am Tracer % Recovery	^{241}Am Measured % Bias
100	3	86 ± 7	-3.0	94 ± 4	-10
100	3	81 ± 15	-6.0	80 ± 5	-13
200	2	82 ± 1	2.0	93 ± 5	-19
200	3	80 ± 8	-5.0	93 ± 5	-18

References

1) Sherrod L. Maxwell, “Rapid method for determination of plutonium, americium, and curium in large soil samples,” *J. Radioanal. Nucl. Chem.*, 275(2), 395-402 (2008).

AN-1432-10

Rapid Determination of Actinides in 1g Concrete and Brick Samples

Summary of Method U, Pu, Np, Am and Cm are separated and concentrated from 1 gram samples of concrete and brick. Samples are fused with NaOH at 600°C in zirconium crucibles. The fusion cakes are dissolved in water, transferred to 250mL centrifuge tubes and precipitated sequentially with iron-hydrous titanium oxide and lanthanum fluoride to facilitate matrix removal. Actinides are separated on stacked 2mL cartridges of Eichrom TEVA, TRU and DGA resins. Actinides are measured by alpha spectrometry following CeF_3 microprecipitation onto Eichrom Resolve® Filters. Chemical yields of tracers ranged from 79-98% for ^{236}Pu, 77-90% for ^{243}Am, and 72-81% for ^{232}U. Measured values typically agreed to within 10% of reference values. Sample preparation for batches of 12 samples can be completed by a single operator in <8 hours. Alpha spectrometry count times will depend on detection limit and data quality objectives.

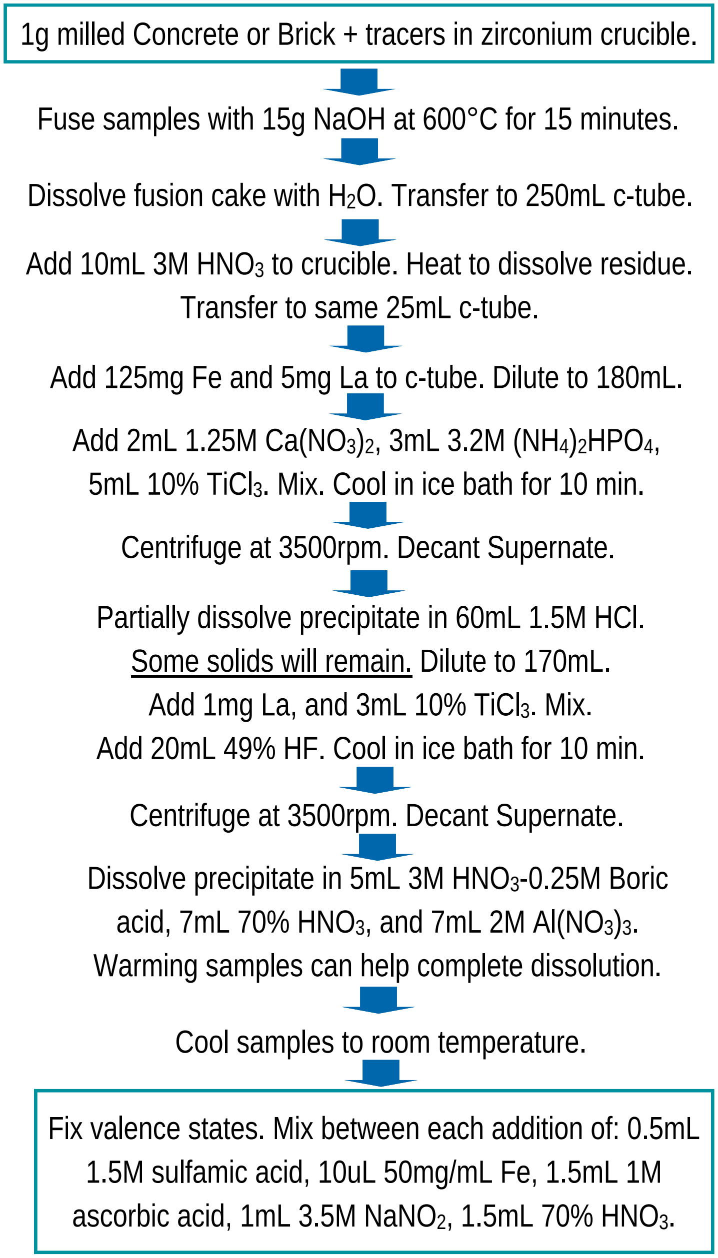
Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
La carrier (10mg/mL)
Deionized Water
3.2M $(NH_4)_2HPO_4$
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid
Ce carrier (1mg/mL)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
$NaNO_2$
Sulfamic Acid

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
Heat Lamp
Muffle Furnace
Hot Plate
Analytical Balance
250mL Zirconium crucibles with zirconium lids
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Vacuum Pump

Figure 1. Sample Preparation



1g milled Concrete or Brick + tracers in zirconium crucible.

Fuse samples with 15g NaOH at 600°C for 15 minutes.

Dissolve fusion cake with H_2O. Transfer to 250mL c-tube.

Add 10mL 3M HNO_3 to crucible. Heat to dissolve residue. Transfer to same 25mL c-tube.

Add 125mg Fe and 5mg La to c-tube. Dilute to 180mL.

Add 2mL 1.25M $Ca(NO_3)_2$, 3mL 3.2M $(NH_4)_2HPO_4$, 5mL 10% $TiCl_3$. Mix. Cool in ice bath for 10 min.

Centrifuge at 3500rpm. Decant Supernate.

Partially dissolve precipitate in 60mL 1.5M HCl. Some solids will remain. Dilute to 170mL. Add 1mg La, and 3mL 10% $TiCl_3$. Mix. Add 20mL 49% HF. Cool in ice bath for 10 min.

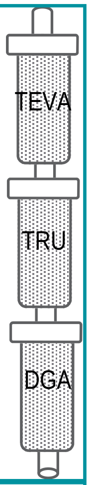
Centrifuge at 3500rpm. Decant Supernate.

Dissolve precipitate in 5mL 3M HNO_3-0.25M Boric acid, 7mL 70% HNO_3, and 7mL 2M $Al(NO_3)_3$. Warming samples can help complete dissolution.

Cool samples to room temperature.

Fix valence states. Mix between each addition of: 0.5mL 1.5M sulfamic acid, 10uL 50mg/mL Fe, 1.5mL 1M ascorbic acid, 1mL 3.5M $NaNO_2$, 1.5mL 70% HNO_3.

Figure 2. Actinide Separation on TEVA - TRU - DGA* and Source Preparation



(1) Precondition stacked 2mL TEVA, TRU, DGA cartridges with 10mL 3M HNO_3.

(2) Load sample solution.

(3) Rinse sample tube with 5mL 3M 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
  -5mL 3M HNO_3

(7) Strip Pu (and Np) from TEVA cartridge with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

(8) Add 0.5mL 30% H_2O_2 for Uranium decon. in alpha source preparation.

(9) Rinse DGA cartridge with 10mL 0.1M HNO_3.

(10) Place TRU cartridge above DGA.

(11) Strip Am/Cm from TRU onto DGA with 15mL 4M HCl .

(12) Separate TRU cartridge from DGA cartridge. Set TRU aside for U recovery.

(13) Rinse DGA cartridge sequentially with:
  -5mL 4M HCl
  -5mL 1M HNO_3
  -15mL 0.05M HNO_3

(14) Strip Am and Cm from DGA with 10mL 0.25M HCl. Add 0.2mL 30% H_2O_2.

(15) Rinse TRU cartridge with 15mL 4M HCl-0.2M HF-0.002M $TiCl_3$.

(16) Rinse TRU cartridge with 10mL 8M HNO_3 .

(17) Strip U from TRU with 15mL of 0.1M ammonium bioxalate.

(18) Add 0.5mL 10% $TiCl_3$.

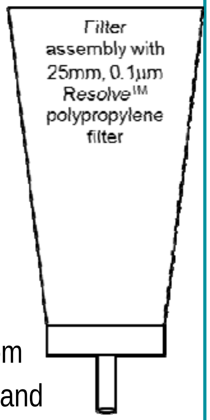
(19) Add 50ug Ce carrier to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(20) Set up Resolve® Filter Funnel on vacuum box.

(21) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(22) Filter sample.

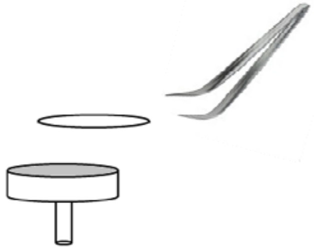
(23) Rinse sample tube with 5mL DI water and add to filter.



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

(25) Draw vacuum until filter is dry.

(26) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(27) Dry filter under heat lamp for 3-5 minutes.

(28) Measure actinides by alpha spectrometry.

*Adding 50uL 30% H_2O_2 to the tube rinse can improve Uranium recoveries and decontamination in the Pu/Np fraction.

Method Performance

Analyte	Replicates	Tracer	Tracer % Yield	Analyte Reference (mBq/g)	Analyte Measured (mBq/g)	% Bias
^{239}Pu	5	^{236}Pu	90 ± 7	18.0	18 ± 2	0.0
^{238}Pu	5	^{236}Pu	90 ± 7	14.8	15 ± 2	1.4
^{237}Np	5	^{236}Pu	90 ± 7	37.0	33 ± 1	-11
^{241}Am	5	^{243}Am	85 ± 6	25.4	24 ± 1	-5.5
^{244}Cm	5	^{243}Am	85 ± 6	35.0	35 ± 2	0.0
^{238}U	5	^{232}U	77 ± 3	29.6	31 ± 3	4.7
^{234}U	5	^{232}U	77 ± 3	28.4	26 ± 4	-8.5

References

1) Sherrod L. Maxwell, Brian K. Culligan, Angel Kelsey-Wall, Patrick J. Shaw, “Rapid radiochemical method for determination of actinides in emergency concrete and brick samples,” *Analytica Chimica Acta,* 701(1), 112-118 (2011).

eichrom®

AN-1433-10

Rapid Determination of Actinides in Emergency Air Filter Samples

Summary of Method U, Pu, Np, Am and Cm are separated and concentrated from air filters. Samples are digested in Teflon beakers once with HNO_3-H_2O_2-HF and then several times with HNO_3-H_2O_2. After evaporating to dryness from HNO_3-H_3BO_3 to complex any residual fluoride, actinides are valence adjusted and separated on stacked 2mL cartridges of Eichrom TEVA and TRU resins. Actinides are measured by alpha spectrometry following CeF_3 microprecipitation onto Eichrom Resolve® Filters. Chemical yields of tracers averaged from 94±12% for ^{242}Pu, 87±6% for ^{243}Am, and 67±32% for ^{232}U. Poor ^{232}U recoveries in some samples were traced to insufficient mass of Ce carrier in the source preparation step. Recovery of ^{232}U improved upon increasing to 100ug of Ce carrier. Measured values typically agreed to within 10% of reference values. Sample preparation for batches of 12 samples can be completed by a single operator in <8 hours. Alpha spectrometry count times will depend on detection limit and data quality objectives.

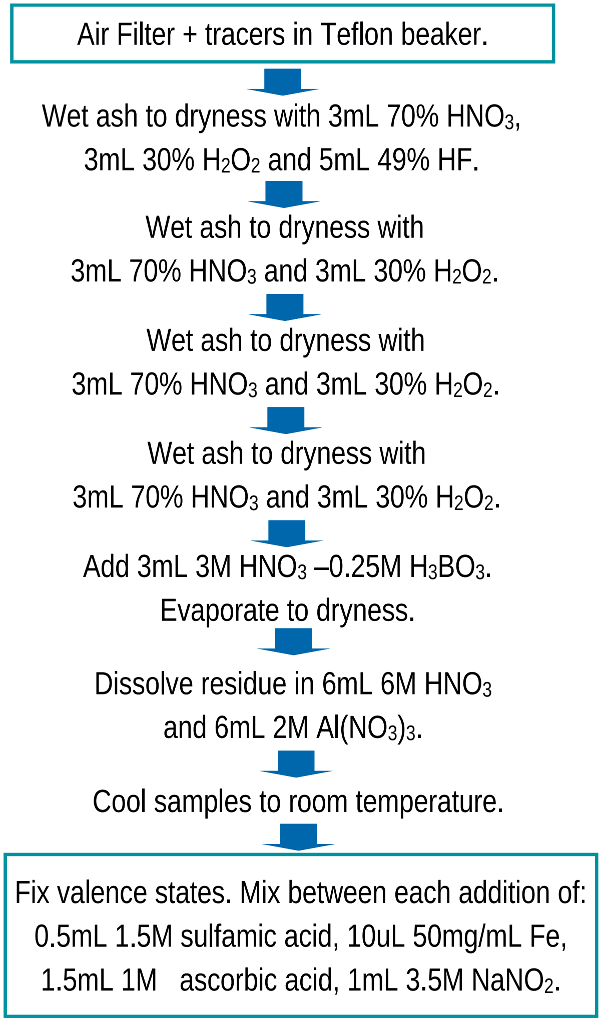
Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu (or ^{236}Pu if meas. Np), ^{243}Am and ^{232}U tracers
Oxalic acid/Ammonium oxalate
Ce carrier (1mg/mL)
Deionized water
10% (w:w) $TiCl_3$
HCl (37%)
Boric acid
$NaNO_2$
Sulfamic Acid
2M $Al(NO_3)_3$
HNO_3 (70%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid

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 Centrifuge Tubes
Centrifuge
Heat Lamp
Hot Plate
Analytical Balance
250mL Teflon beakers
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Vacuum Pump

Figure 1. Sample Preparation



Air Filter + tracers in Teflon beaker.

Wet ash to dryness with 3mL 70% HNO_3,
3mL 30% H_2O_2 and 5mL 49% HF.

Wet ash to dryness with
3mL 70% HNO_3 and 3mL 30% H_2O_2.

Wet ash to dryness with
3mL 70% HNO_3 and 3mL 30% H_2O_2.

Wet ash to dryness with
3mL 70% HNO_3 and 3mL 30% H_2O_2.

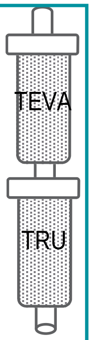
Add 3mL 3M HNO_3 –0.25M H_3BO_3.
Evaporate to dryness.

Dissolve residue in 6mL 6M HNO_3
and 6mL 2M $Al(NO_3)_3$.

Cool samples to room temperature.

Fix valence states. Mix between each addition of:
0.5mL 1.5M sulfamic acid, 10uL 50mg/mL Fe,
1.5mL 1M ascorbic acid, 1mL 3.5M $NaNO_2$.

Figure 2. Actinide Separation on TEVA - TRU*



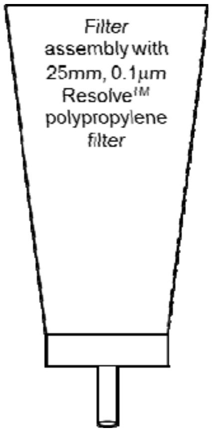
(1) Precondition stacked 2mL TEVA-TRU with 10mL 3M HNO_3.
(2) Load sample solution.
(3) Rinse sample tube with 5mL 6M HNO_3. Add tube rinse to cartridges.**
(4) Rinse cartridges with 5mL 3M HNO_3.
(5) Separate TEVA and TRU cartridges.

(6) Rinse TEVA cartridge with:
- -15mL 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 $TiCl_3$. Add 0.5mL 30% H_2O_2 for additional U decon. during CeF_3

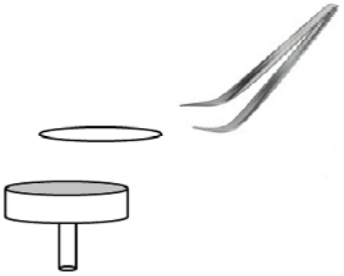
(8) Strip Am/Cm from TRU with 15mL 4M HCl. Dilute to 30mL and add 0.2mL 30% H_2O_2.
(9) Rinse TRU with 15mL 4M HCl-0.2M HF. (Th removal)
(10) Strip U from TRU with 15mL 0.1M ammonium bioxalate.

(11) Add 0.5mL of 10% $TiCl_3$ to each U sample for CeF_3 ppt.
(12) Add 50-100ug Ce carrier to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.
(13) Set up Resolve® Filter Funnel on vacuum box.

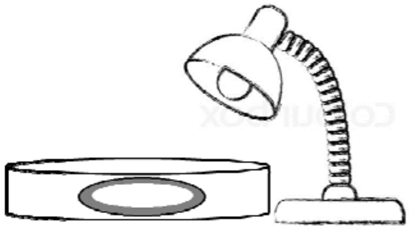


(14) Wet filter with 3mL 80% ethanol followed by 3mL DI water.
(15) Filter sample.
(16) Rinse sample tube with 5mL DI water and add to filter.
(17) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.
(18) Draw vacuum until filter is dry.

(19) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



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



*$^{89/90}$Sr can also be measured by placing a 2mL Sr Resin cartridge below DGA and following the separation scheme in application note AN-1434

**Adding 50uL of 30% H_2O_2 to the 6M HNO_3 tube rinse can help improve U recoveries and decontamination in the Pu/Np fraction.

References

1) Sherrod L. Maxwell, Brian K. Culligan, Gary W. Noyes, “Rapid separation method for actinides in emergency air filter samples,” *Applied Radiation and Isotopes,* 68(12), 2125-2131 (2010).

AN-1434-10

Rapid Determination of Sr in Emergency Air Filter Samples

Summary of Method Strontium is separated and concentrated from air filters. Samples are digested in Teflon beakers once with HNO_3-H_2O_2-HF and then several times with HNO_3-H_2O_2. After evaporating to dryness from HNO_3-H_3BO_3 to complex any residual fluoride, strontium is separated on a 2mL cartridges of Eichrom Sr resin. Radiostrontium is measured by low background gas flow proportional counting or liquid scintillation counting. Chemical yield of strontium, which averaged 86±5%, is determined by gravimetric recovery of stable strontium carrier or ICP-AES measurement. ^{90}Sr measurements agreed to within 10% of reference values. ^{89}Sr and ^{90}Sr activities can be determined by Cerenkov counting or by subsequent ^{90}Y ingrowth, separation and measurement. Sample preparation for batches of 12 samples can be completed by a single operator in <8 hours.

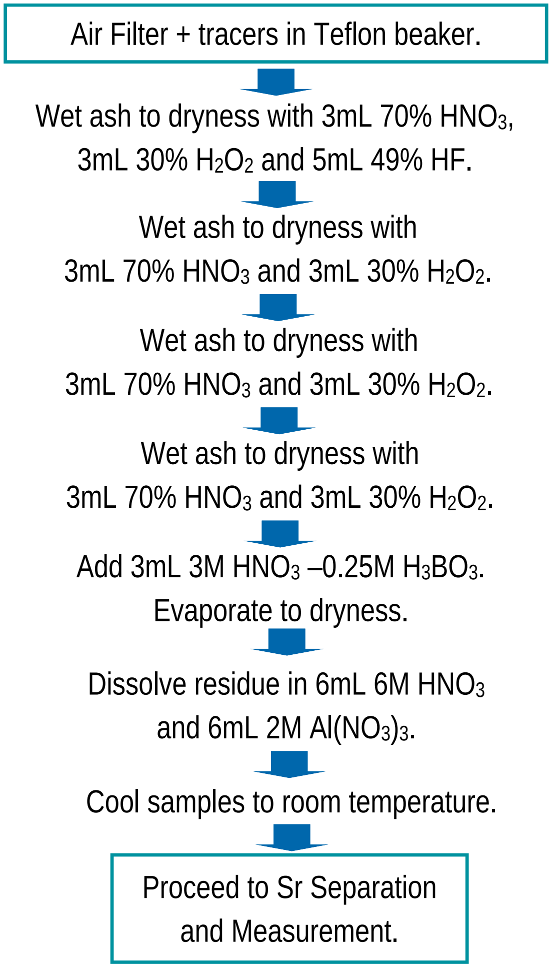
Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Oxalic acid/Ammonium oxalate
Sr carrier (10mg/mL)
Deionized Water
2M $Al(NO_3)_3$
HNO_3 (70%)
HF (49%) or NaF
Boric acid
H_2O_2 (30%)

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)
Hot Plate
Analytical Balance
250mL Teflon beakers
Cupped Stainless Steel Planchets (~5mL volume)
Low background gas flow proportional counter
Vacuum Pump

Figure 1. Sample Preparation



Air Filter + tracers in Teflon beaker.

Wet ash to dryness with 3mL 70% HNO_3, 3mL 30% H_2O_2 and 5mL 49% HF.

Wet ash to dryness with 3mL 70% HNO_3 and 3mL 30% H_2O_2.

Wet ash to dryness with 3mL 70% HNO_3 and 3mL 30% H_2O_2.

Wet ash to dryness with 3mL 70% HNO_3 and 3mL 30% H_2O_2.

Add 3mL 3M HNO_3 –0.25M H_3BO_3. Evaporate to dryness.

Dissolve residue in 6mL 6M HNO_3 and 6mL 2M $Al(NO_3)_3$.

Cool samples to room temperature.

Proceed to Sr Separation and Measurement.

Figure 2. Load Solution Preparation and Strontium Separation

(1) Precondition Sr Resin with
5mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

(3) Rinse sample tube with 3mL
3M HNO_3.

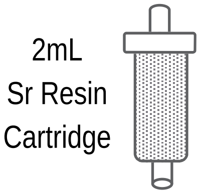
(4) Add tube rinse to Sr Resin.
Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:

- 10 mL 8M HNO_3
- 5mL 3M HNO_3 - 0.05 oxalic acid
- 5mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

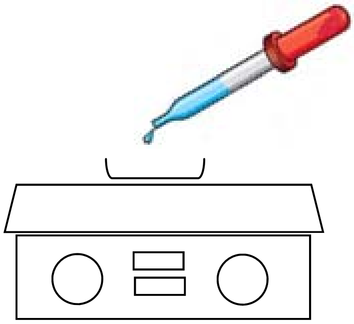
(7) Strip Sr with 15mL
0.05M HNO_3 at
1mL/min.



Gas Flow Proportional Counting:*

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Descrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr may then be measured after adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

*Actinides also be measured by placing 2mL cartridges of TEVA, TRU and DGA resin above Sr Resin and following the separation scheme in application note AN-1433.

References

1) Sherrod L. Maxwell, Brian K. Culligan, Gary W. Noyes, “Rapid separation method for actinides in emergency air filter samples,” *Applied Radiation and Isotopes,* 68(12), 2125-2131 (2010).

AN-1435-10

Rapid Determination of Np/Pu in 20-50g Soil Samples

Summary of Method Plutonium and Neptunium are separated and concentrated from 20-50 gram soil samples. Samples are leached with HNO_3 and HCl. The leachates are evaporated to dryness, and sequential precipitations with Fe/Ti-hydroxide and LaF_3 facilitate matrix removal. Pu-Np are separated on 2mL cartridges of Eichrom TEVA resin. Pu-Np are measured by alpha spectrometry following CeF_3 microprecipitation onto Eichrom Resolve® Filters. Chemical yields of the ^{236}Pu tracer ranged from 82-96%. Measured values typically agreed to within 10% of reference values. Sample preparation for batches of 12 samples can be completed by a single operator in <8 hours.

Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{236}Pu tracer
La carrier (10mg/mL)
Deionized Water
10% (w:w) $TiCl_3$
HCl (37%)
Boric acid
$NaNO_2$
Sulfamic Acid
NH_4OH (28% NH_3 or 56% NH_4OH)
Ce carrier (1mg/mL)
2M $Al(NO_3)_3$
HNO_3 (70%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ascorbic Acid

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)
600mL Glass beakers
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
50mL and 250mL Centrifuge Tubes
Centrifuge
Heat Lamp
Hot Plate
Analytical Balance
Vacuum Pump

Figure 1. Sample Preparation

Dry 20-50g soil at 110°C. Blend and Size.

↓

Aliquot sample to 600mL glass beaker. Add ^{236}Pu.

↓

Add 1.5mL 70% HNO_3 and 0.5mL 37% HCl per gram of sample. Heat to 80°C on hotplate.

↓

Transfer liquid to 250mL centrifuge tube.

↓

Add 20mL 70% HNO_3 to beaker. Warm beaker. Transfer liquid to same 250mL centrifuge tube. Repeat once.

↓

Centrifuge 3500 rpm, 10 min.

↓

Transfer leachate to 600mL beaker. Evaporate to dryness.

↓

Dissolve residue in 20mL 1M HCl. Warm if necessary.

↓

Dilute samples to 180mL.
Add 5mg La, 125mg of Fe, and 20mL 10% $TiCl_3$.
Mix. Add 25mL 56% NH_4OH. Mix.

↓

Centrifuge 3500 rpm. 5min. Decant supernate

↓

Partially dissolve in 60mL 1.5M HCl.
Solids will remain. Dilute to 170mL.
Add 3mg La and 20mL 10% $TiCl_3$. Mix.
Add 22mL 49% HF. Mix.
Place in ice bath for 10min.

↓

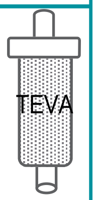
Centrifuge 3500 rpm. 5min. Decant supernate.

↓

Dissolve solids in 6mL 3M HNO_3-0.25M H_3BO_3, 8.5mL 7M HNO_3, and 8mL 2M $Al(NO_3)_3$. Warming samples can improve dissolution.

Figure 2. Actinide Separation on TEVA - TRU - DGA and Source Preparation

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 $NaNO_2$



(1) Precondition 2mL TEVA, cartridges with 5mL 8M HNO_3.

(2) Load Sample.

(3) Rinse centrifuge tube with 5mL 6M HNO_3.*
Add to TEVA.

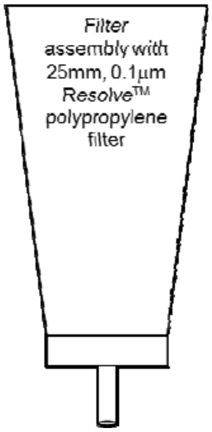
(4) Rinse cartridges with:
- -15mL 3M HNO_3
- -20mL 9M HCl
- -5mL 3M HNO_3.

(5) Strip Pu/Np from TEVA with 20mL 0.1M HCl-0.05M HF-0.03M $TiCl_3$.

(6) Add 0.5mL 30% H_2O_2 to each sample for additional uranium decontamination during CeF_3 ppt.

(7) 50ug Ce carrier. Mix.
Add 1mL 49% HF. Mix.
Wait 15-20 minutes.

(8) Set up Resolve® Filter Funnel on vacuum box.



(9) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

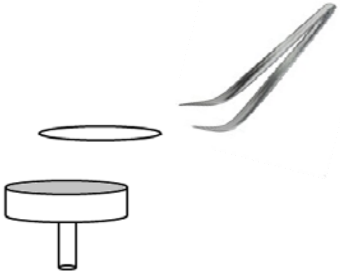
(10) Filter sample.

(11) Rinse sample tube with 5mL DI water and add to filter.

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

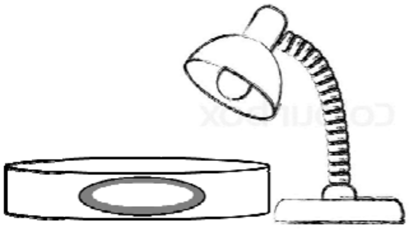
(13) Draw vacuum until filter is dry.

(14) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(15) Dry filter under heat lamp for 3-5 minutes.

(16) Measure actinides by alpha spectrometry.



*Adding 50uL 30% H_2O_2 to the tube rinse can improve Uranium decontamination.

Sample, g	replicates	^{236}Pu Tracer % Yield	^{239}Pu Reference (mBq)	^{239}Pu Measured (mBq)	^{239}Pu %Bias	^{238}Pu Reference (mBq)	^{238}Pu Measured (mBq)	^{238}Pu %Bias	^{237}Np Reference (mBq)	^{237}Np Measured (mBq)	^{237}Np %Bias
20	6	89±6	116.3	118±7	1.5	63.2	67±4	6.0	37.0	39±4	5.4
20	6	96±7	1.69	2.1±0.4	24	25.3	25±2	-1.2	37.0	35±2	-5.4
30	6	82±6	116.3	121±5	4.0	63.2	68±5	7.6	37.0	39±4	5.4
50	6	88±5	116.3	114±3	-2.0	63.2	64±2	1.3	37.0	21±11	-43

References

1) Sherrod L. Maxwell, Brian K. Culligan, Gary W. Noyes, “Rapid separation method for ^{237}Np and Pu isotopes in large soil samples,” *Applied Radiation and Isotopes,* 69(7), 917-925 (2011).

AN-1436-10

Rapid Determination of Np/Pu in 20-75g Soil Samples (ICP-MS)

Summary of Method Plutonium and Neptunium are separated and concentrated from 20-75 gram soil samples. Samples are leached with HNO_3 and HCl. The leachates are evaporated to dryness, and sequential precipitations with Fe/Ti-hydroxide and LaF_3 facilitate matrix removal. Pu-Np are separated on 2mL cartridges of Eichrom TEVA and DGA resins. Pu-Np are measured by ICP-MS. Chemical yields of the ^{242}Pu tracer were 87±4%, 75±6%, and 70±3% for 20, 50 and 75g samples, respectively. Measured values for ^{239}Pu agreed to within 1% of reference values, while ^{237}Np agreed to within 15%. Decontamination factors of $>10^6$ were achieved for Pu over U (^{238}U-H can interfere with the measurement of ^{239}Pu by ICP-MS). Sample preparation for batches of 12 samples can be completed by a single operator in <8 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}Pu tracer
Deionized Water
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
$NaNO_2$
Ascorbic Acid
La carrier (10mg/mL)
2M $Al(NO_3)_3$
HNO_3 (70%)
NH_4OH (28% HN_3 or 56% NH_4OH)
Boric acid
Sulfamic 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
ICP-MS system
Centrifuge
Hot Plate
Analytical Balance
Vacuum Pump

Figure 1. Sample Preparation

Dry 20-75g soil at 110°C. Blend and Size.

Aliquot sample to 600mL glass beaker. Add ^{242}Pu.

Add 1.5mL 70% HNO_3 and 0.5mL 37% HCl per gram of sample. Heat to 80°C on hotplate.

Transfer liquid to 250mL centrifuge tube.

Add 20mL 70% HNO_3 to beaker. Warm beaker.
Transfer liquid to same 250mL centrifuge tube.
Repeat once.

Centrifuge 3500 rpm, 10 min.

Transfer leachate to 600mL beaker.
Evaporate to dryness.

Dissolve residue in 20mL 1M HCl. Warm if necessary.

Dilute samples to 180mL.
Add 5mg La, 125mg of Fe, and 20mL 10% $TiCl_3$.
Mix. Add 25mL 56% NH_4OH. Mix.

Centrifuge 3500 rpm. 5min. Decant supernate

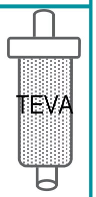
Partially dissolve in 60mL 1.5M HCl.
Solids will remain. Dilute to 170mL.
Add 3mg La and 20mL 10% $TiCl_3$. Mix.
Add 22mL 49% HF. Mix.
Place in ice bath for 10min.

Centrifuge 3500 rpm. 5min. Decant supernate.

Dissolve solids in 6mL 3M HNO_3-0.25M H_3BO_3, 8.5mL 7M HNO_3, and 8mL 2M $Al(NO_3)_3$. Warming samples can improve dissolution.

Figure 2. Actinide Separation on TEVA - DGA

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 $NaNO_2$

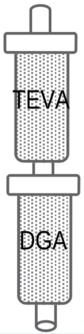


(1) Precondition 2mL TEVA, cartridges with 5mL 8M HNO_3.

(2) Load Sample.

(3) Rinse centrifuge tube with 5mL 6M HNO_3. Add to TEVA.*

(4) Rinse cartridges with:
- -30mL 3M HNO_3
- -15mL 9M HCl (Th removal)



(5) Add 2mL DGA cartridge below TEVA.**

(6) Strip Pu from TEVA onto DGA with 15mL 3M HNO_3-0.1M Ascorbic Acid-0.02M Fe^{2+}.

(7) Separate TEVA and DGA.

(8) Rinse TEVA with 5mL 3M HNO_3.

(9) Strip Np from TEVA with 14mL 0.25M HCl-0.005M HF-0.01M Hydroxylamine hydrochloride.

(10) Rinse DGA with:
- -5mL 8M HNO_3
- -20mL 0.1M HNO_3
- -10mL 0.05M HNO_3.

(11) Strip Pu from DGA with 11mL 0.02M HCl-0.005M HF-0.01M Hydroxylamine hydrochloride.

(12) Measure ^{237}Np and Pu by ICP-MS.

*Adding 50uL of 30% H_2O_2 to the 6M HNO_3 tube rinse can further improve uranium decontamination factors.

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

References

1) Sherrod L. Maxwell, Brian K. Culligan, Gary W. Noyes, “Rapid separation method for ^{237}Np and Pu isotopes in large soil samples,” *Applied Radiation and Isotopes,* 69(7), 917-925 (2011).
2) Sherrod L. Maxwell, Brian K. Culligan, Vernon D. Jones, Sheldon T. Nichols, Gary W. Noyes, Maureen A. Bernard, “Rapid Determination of ^{237}Np and Plutonium Isotopes by ICP-MS and Alpha Spectrometry,” *Health Physics*, 101(2), 180-186 (2011).

AN-1437-10

Rapid Determination of Actinides in Urine by ICP-MS + Alpha Spec.

Summary of Method Actinides are separated and concentrated from 100mL urine samples. Actinides are concentrated from urine samples using a calcium phosphate precipitation. Pu, Np, Am-Cm, and U are separated on 2mL cartridges of Eichrom TEVA, TRU and DGA resins. Pu-Np are measured by ICP-MS. Measured values for ^{239}Pu and ^{237}Np agreed to within 1-2% of reference values, while ^{241}Am and ^{244}Cm agreed to within 2-3%. Decontamination factors of $>10^6$ were achieved for Pu over U (^{238}U-H can interfere with the measurement of ^{239}Pu by ICP-MS). Sample preparation for batches of 12 samples can be completed by a single operator in <8 hours.

Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
DGA Resin, Normal, 2mL Cartridges (Eichrom DN-R50-S)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu (ICP-MS) or ^{236}Pu (alpha) tracer
^{233}U (ICP-MS) or U^{232} (alpha) tracer
^{243}Am tracer
1.25M $Ca(NO_3)_2$
Deionized Water
HNO_3 (70%)
NH_4OH
$NaNO_2$
Sulfamic Acid
Ce carrier (1mg/mL)
3.2M $(NH_4)_2HPO_4$
2M $Al(NO_3)_3$
HCl (37%)
HF (49%) or NaF
Denatured ethanol
Ascorbic Acid
Oxalic acid/Ammonium oxalate
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)
Resolve Filters in Funnel (Eichrom RF-DF25-25PP01)
600mL Glass beakers
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
ICP-MS System
50mL and 250mL Centrifuge Tubes
Centrifuge
Heat Lamp
Hot Plate
Analytical Balance
Vacuum Pump

Figure 1. Sample Preparation

Urine Sample + Tracers

Add 1mL 1.25M $Ca(NO_3)_2$ and
3mL 3.2M $(NH4)_2HPO_4$. Mix well.
Heating to near boiling can improve
tracer/analyte equilibration.

Adjust to pH 9 with NH_4OH.
Cool to room temperature.

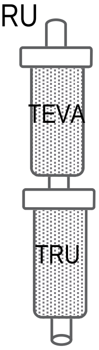
Centrifuge 3500 rpm, 5 min.

Decant Supernate.

Dissolve precipitate in 8mL 6M HNO_3
and 8mL 2M $Al(NO_3)_3$.

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 $NaNO_2$

Figure 2. Actinide Separation on TEVA - DGA



(1) Precondition 2mL TEVA+TRU with 10mL 6M HNO_3.

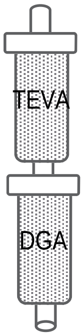
(2) Load Sample.

(3) Rinse centrifuge tube with 5mL 6M HNO_3. Add to TEVA-TRU.*

(4) Rinse TEVA+TRU with 5mL 3M HNO_3.

(5) Separate TEVA and TRU.

(6) Rinse TEVA with:
-30mL 3M HNO_3
-15mL 9M HCl (Th removal)



(7) Add DGA below TEVA.**

(8) Strip Pu/Np from TEVA to DGA with 15mL 3M HNO_3-0.1M Ascorbic acid-0.02M Fe^{2+}.

(9) Separate TEVA and DGA.

(10) Rinse TEVA with 5mL 3M HNO_3.

(11) Strip Np from TEVA with 14mL 0.25M HCl-0.005M HF-0.01M Hydroxylamine hydrochloride.

(12) Rinse DGA with:
-5mL 8M HNO_3
-20mL 0.1M HNO_3
-10mL 0.05M HNO_3.

(13) Strip Pu from DGA with 11mL 0.02M HCl-0.005M HF-0.01M Hydroxylamine hydrochloride.

(14) Strip Am/Cm from TRU with 15mL 4M HCl.

(15) Rinse TRU resin with 15mL 4M HCl-0.2MHF.

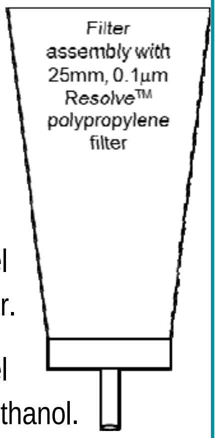
(16) Strip U from TRU with 15mL 0.01M ammonium bioxalate.

(17) Measure Np, Pu and U by ICP-MS.

(18) Add 50ug Ce carrier and 0.5mL 30% H_2O_2 to each Am-Cm sample. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 min.

(19) Set up Resolve® Filter Funnel on vacuum box.

(20) Wet filter with 3mL 80% ethanol followed by 3mL DI water.



(21) Filter sample.

(22) Rinse sample tube with 5mL DI water and add to filter.

(23) Rinse filter funnel with 3mL DI water.

(24) Rinse filter funnel with 2mL 100% ethanol.

(25) Draw vacuum until filter is dry.

(26) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.

(27) Dry filter under heat lamp for 3-5 minutes.

(28) Measure actinides by alpha spectrometry.

* Adding 50uL of 30% H_2O_2 to the 6M HNO_3 tube rinse can help improve U recoveries and decontamination in the Np and Pu fractions.

** Adding a 1mL UTEVA cartridge between TEVA and DGA can help improve uranium decontamination.

References

1) Sherrod L. Maxwell, Vernon D. Jones, “Rapid determination of Actinides in urine by ICP-MS and alpha spectrometry: A hybrid approach,” *Talanta,* 80(1), 143-150 (2009).

2) Sherrod L. Maxwell, Brian K. Culligan, Vernon D. Jones, Sheldon T. Nichols, Gary W. Noyes, Maureen A. Bernard, “Rapid Determination of ^{237}Np and Plutonium Isotopes by ICP-MS and Alpha Spectrometry,” *Health Physics*, 101(2), 180-186 (2011).

eichrom®

AN-1438-10

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}Pu, ^{242}Pu, and ^{237}Np agreed to within 1-4% of reference values, while ^{237}Np agreed to within 15%. Decontamination factors of >10^6 were achieved for Pu over U (^{238}U-H can interfere with the measurement of ^{239}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}Pu tracer
3.2M $(NH_4)_2HPO_4$
2M $Al(NO_3)_3$
HCl (37%)
HF (49%) or NaF
Sulfamic Acid
Hydroxylamine Hydrochloride
1.25M $Ca(NO_3)_2$
Deionized Water
HNO_3 (70%)
NH_4OH
$NaNO_2$
Ascorbic Acid

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

Water Sample + ^{242}Pu Tracer

↓

Add 1mL 1.25M $Ca(NO_3)_2$ and
3mL 3.2M $(NH4)_2HPO_4$. Mix well.
Heating to near boiling can improve
tracer/analyte equilibration.

↓

Adjust to pH 9 with NH_4OH.
Cool to room temperature.

↓

Centrifuge 3500 rpm, 5 min.

↓

Decant Supernate.

↓

Dissolve precipitate in 8mL 6M HNO_3
and 8mL 2M $Al(NO_3)_3$.

Figure 2. Actinide Separation on TEVA - DGA

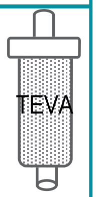
Cool samples to room temp. Fix valence by adding: (mix between steps)
-0.5mL 1.5M sulfamic acid
-40uL 50mg/mL Fe carrier
-1.5mL 1M ascorbic acid (Wait 3 min)
-1mL 3.5M $NaNO_2$

(1) Precondition 2mL TEVA, cartridges with 5mL 8M HNO_3.

(2) Load Sample.

(3) Rinse centrifuge tube with 5mL 6M HNO_3. Add to TEVA.*

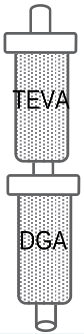
(4) Rinse cartridges with:
 -30mL 3M HNO_3
 -15mL 9M HCl (Th removal)



(5) Add 2mL DGA cartridge below TEVA.**

(6) Strip Pu from TEVA onto DGA with 15mL 3M HNO_3-0.1M Ascorbic Acid-0.02M Fe^{2+}.

(7) Separate TEVA and DGA.



(8) Rinse TEVA with 5mL 3M HNO_3.

(9) Strip Np from TEVA with 14mL 0.25M HCl-0.005M HF-0.01M Hydroxylamine hydrochloride.

(10) Rinse DGA with:
 -5mL 8M HNO_3
 -20mL 0.1M HNO_3
 -10mL 0.05M HNO_3.

(11) Strip Pu from DGA with 11mL 0.02M HCl-0.005M HF-0.01M Hydroxylamine hydrochloride.

(12) Measure ^{237}Np and Pu by ICP-MS.

* Adding 50uL of 30% H_2O_2 to 6M HNO_3 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 ^{237}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 ^{237}Np and Plutonium Isotopes by ICP-MS and Alpha Spectrometry,” *Health Physics*, 101(2), 180-186 (2011).

AN-1601-10

Determination of ^{227}Ac in Geological Samples

Summary of Method Soil or rock samples are pulverized to <1mm and dissolved, either by acid digestion or sodium hydroxide fusion. ^{227}Ac is separated from matrix ions using a ferric hydroxide precipitation step. Following dissolution in 4M HCl, ^{227}Ac is separated from radiometric impurities using a 2mL cartridge of DGA, Normal resin. ^{227}Ac is prepared for measurement using a CeF_3 microprecipitation onto Resolve$^{(R)}$ Filters. An ^{225}Ac(^{229}Th) tracer is used to measure chemical recovery of actinium. After a 30 minute ingrowth time, the ^{225}Ac tracer yield is measured via alpha spectrometry using the ^{221}Fr and ^{217}At daughters of ^{225}Ac. ^{227}Ac is measured via its ^{227}Th and ^{223}Ra daughters after a longer period of ingrowth (30-90 days). Ac yields are typically 70-90%. MDA for ^{227}Ac was 0.05Bq/kg for 3 day count times after 90 days ingrowth period.

Reagents

DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Cerium Carrier (10mg/mL)
^{229}Th(^{225}Ac) tracer
Hydrofluoric Acid (49%) or Sodium Fluoride
Boric acid
HCl (37%)
Deionized Water
HNO_3 (70%)
NaOH
H_2O_2 (30%)
Optional for additional Th/U removal:
TRU Resin, 2mL cartridges (Eichrom TR-R50-S)

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
Alpha Spectrometry System
Ball mill grinder or equivalent
Centrifuge
Heat Lamp
Vacuum Pump
Analytical Balance

Fusion Option

250mL Zirconium crucibles with zirconium lids
Muffle Furnace

Digestion Option

Hot Plate
Teflon Beakers

Sample Preparation

0.25-50g Soil or Rock

Pulverize to <1mm.

Aliquot Sample. Add ^{229}Th(^{225}Ac) tracer.

Acid Digestion Option

Digest in Teflon beaker on hotplate with 2:1 conc. HNO_3:HF to near dryness.

Digest in Teflon beaker on hotplate with conc. HNO_3 + Boric Acid.

Dissolve Residue in 4M HCl + 0.25M Boric acid. If solids remain. Repeat digestion. Proceed to ferric hydroxide precipitation.

Fusion Option

In Zr crucible. Add 10-15g NaOH

Muffle at 600^{o}C for 15-30 minutes.

Cool. Dissolve fusion cake with 50mL water. Heat as necessary. Rinse crucible with 50mL 4M HCl. Proceed to ferric hydroxide precipitation.

Ferric Hydroxide Precipitation

Transfer sample to 250mL centrifuge tube.

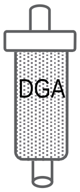
Dilute to 150mL with water. Add 25mg Fe carrier. Mix well.

Centrifuge 2500 rpm for 10 minutes. Decant Supernate.

Rinse ppt with 50mL water. Centrifuge. Decant Supernate.

Dissolve precipitate with 10mL conc. HCl. Dilute to 30mL.

Ac Separation on DGA



(1) Precondition 2mL DGA with 10mL 4M HCl.*

(2) Load sample solution.

(3) Rinse sample tube with 5mL 4M HCl. Add tube rinse to DGA. (If TRU cartridge is used, remove following this step.)

(4) Rinse DGA with 10mL 3M HNO_3.

(5) Rinse DGA with 20mL 0.5M HNO_3.

(6) Strip Ac from DGA with 20mL 2M HCl. (2M HCl is used to achieve additional decontamination from Th.)

* A 2mL cartridge of TRU resin may be added above DGA for additional decontamination from U/Th.

(7) Add 0.5mL 30% H_2O_2 to samples.

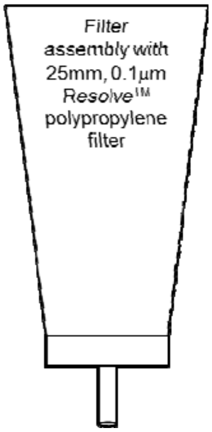
(8) Add 50ug Ce carrier to samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(9) Set up Resolve® Filter Funnel on vacuum box.

(10) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(11) Filter sample.

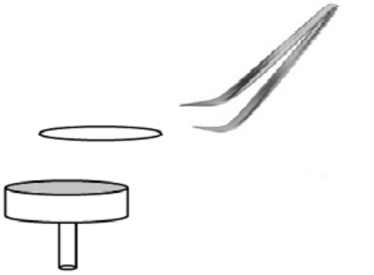
(12) Rinse sample tube with 5mL DI water and add to filter.



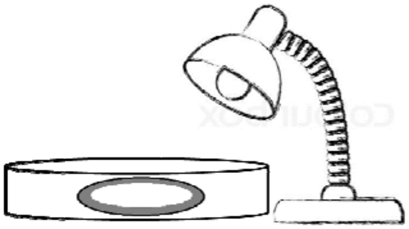
(13) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(14) Draw vacuum until filter is dry.

(15) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(16) Dry filter under heat lamp for 3-5 minutes.



(17) Measure actinium or daughters by alpha spectrometry.

Method Performance

Rock Standard	^{227}Ac Measured Bq/kg	^{227}Ac Reference Bq/kg	% Bias	Tracer Recovery
BCR-2	0.955 ± 0.083	0.967	-1.2	83
BHVO-1	0.299 ± 0.017	0.283	5.7	71
HK-018	0.965 ± 0.009	0.948	1.8	86
HK-019	0.962 ± 0.073	0.966	-0.4	91
HK-021	0.559 ± 0.055	0.572	-2.3	80
HK-022	0.887 ± 0.080	0.862	2.9	68
SAV B6	0.677 ± 0.067	0.680	-0.4	66

References

1) H. Dulaiova, K.W.W. Sims, M.A. Charette, J. Prytulak, J.S. Blusztajn "A new method for the determination of actinium-227 in geological samples," *J. Radioanal. Nucl. Chem., 296, 279-283* (2013).

AN-1602-10

Determination of ^{227}Ac in Water Samples

Summary of Method ^{227}Ac is preconcentrated from up to 1L of water sample using a ferric hydroxide precipitation. Following dissolution in 4M HCl, ^{227}Ac is separated from radiometric impurities using a 2mL cartridge of DGA, Normal resin. ^{227}Ac is prepared for measurement using a CeF$_3$ microprecipitation onto Resolve$^{(R)}$ Filters. An ^{225}Ac(^{229}Th) tracer is used to measure chemical recovery of actinium. After a 30 minute ingrowth time, the ^{225}Ac tracer yield is measured via alpha spectrometry. ^{227}Ac is measured via its ^{227}Th and ^{223}Ra daughters after a longer period of ingrowth (30-90 days). Actinium yields are typically 70-90%. MDA for ^{227}Ac was 0.05Bq/L for 3 day count times after 90 days ingrowth period.

Reagents

DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Cerium Carrier (10mg/mL)
^{229}Th(^{225}Ac) tracer
Hydrofluoric Acid (49%) or Sodium Fluoride
Nitric Acid (70%)
Hydrochloric Acid (37%)
Sodium Hydroxide
Deionized Water
H_2O_2 (30%)
Optional for additional Th/U removal:
TRU Resin, 2mL cartridges (Eichrom TR-R50-S)

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
Alpha Spectrometry System
Centrifuge
Vacuum Pump
Heat Lamp
Analytical Balance
Hot Plate
1L Glass beakers
pH meter or pH strips or pH indicator (pH 8-9)

Sample Preparation

Up to 1L Sample

Aliquot Sample. Add ^{229}Th(^{225}Ac) tracer and 25 mg of Fe carrier.

Heat sample to 80^{o}C and mix well to equilibrate sample and tracer.

Adjust pH to 8-9 with NaOH.

Cool sample and allow ppt to settle.

Decant supernate to ~200mL.

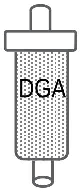
Transfer sample to 250mL centrifuge tube.

Centrifuge 2500 rpm for 10 minutes. Decant Supernate.

Rinse ppt with 50mL water. Centrifuge. Decant Supernate.

Dissolve precipitate with 10mL conc. HCl. Dilute to 30mL.

Ac Separation on DGA



(1) Precondition 2mL DGA with 10mL 4M HCl.*

(2) Load sample solution.

(3) Rinse sample tube with 5mL 4M HCl. Add tube rinse to DGA. (If TRU cartridge is used, remove following this step.)

(4) Rinse DGA with 10mL 3M HNO_3.

(5) Rinse DGA with 20mL 0.5M HNO_3.

(6) Strip Ac from DGA with 20mL 2M HCl. (2M HCl is used to achieve additional decontamination from Th.)

* A 2mL cartridge of TRU resin may be added above DGA for additional decontamination from U/Th.

(7) Add 0.5mL 30% H_2O_2 to samples.

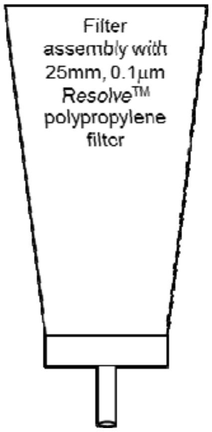
(8) Add 50ug Ce carrier to samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(9) Set up Resolve® Filter Funnel on vacuum box.

(10) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(11) Filter sample.

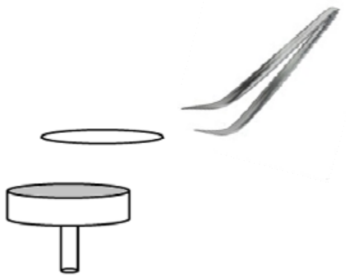
(12) Rinse sample tube with 5mL DI water and add to filter.



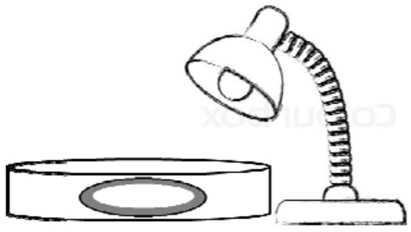
(13) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(14) Draw vacuum until filter is dry.

(15) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(16) Dry filter under heat lamp for 3-5 minutes.



(17) Measure actinides by alpha spectrometry.

Method Performance

Water Standard	^{227}Ac Measured Bq/kg	^{227}Ac Reference Bq/kg	% Bias	Tracer Recovery
IAEA Standard	333 ± 16	329 ± 16	1.2	75

References

1) H. Dulaiova, K.W.W. Sims, M.A. Charette, J. Prytulak, J.S. Blusztajn "A new method for the determination of actinium-227 in geological samples," *J. Radioanal. Nucl. Chem., 296, 279-283* (2013).

eichrom®

AN-1603-10

Rapid Determination of Actinides in Limestone and Marble

Summary of Method Actinides are separated and concentrated from 1.5 gram samples of limestone or marble. Samples are finely ground and fused in a zirconium crucible for 15 minutes at 600°C with 15 grams of sodium hydroxide. The fusion cake is dissolved in water, and actinides are concentrated and separated from the matrix using a calcium phosphate precipitate enhanced with iron. A secondary precipitation with calcium fluoride removes additional matrix (including silicates) and decreases the volume of precipitate. The calcium fluoride precipitate is dissolved with nitric acid-boric acid-aluminum nitrate to form the load solution. Analytes are separated from remaining matrix and potentially interfering radionuclides using stacked TEVA, TRU, and DGA Resin cartridges. Actinides are measured by alpha spectrometry after CeF_3 microprecipitation onto Resolve(R) Filters. Simultaneous separation of radiostrontium can be achieved by using the separation method in AN-1604.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)*
Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)*
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
Strontium*, Lanthanum and Cerium Carriers (10mg/mL)
Iron Carrier (50mg/mL Fe, as ferric nitrate)
^{242}Pu (or ^{236}Pu if Np is measured) tracer
^{243}Am and ^{232}U tracers
^{90}Sr standard*
30% H_2O_2
Hydrochloric Acid (37%)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
Boric acid
Ascorbic acid
$NaNO_2$
10% $TiCl_3$
HF(49%)
Nitric Acid (70%)
Deionized Water
3.2M $(NH_4)_2HPO_4$
Oxalic acid
Sodium Hydroxide
Sulfamic acid

*Only needed if Sr is measured.

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
Cupped Stainless Steel Planchets (~5mL volume)*
250mL Zirconium crucibles with zirconium lids
Alpha Spectrometry System
Stainless Steel planchets with two sided tape
Centrifuge
Muffle Furnace
Gas Flow Proportional Counter*
Hot Plate/Heat Lamp

Figure 1. Sample Preparation

1.5g finely ground sample in zirconium crucible

↓

Add 4mg Sr carrier* and Pu/U/Am tracers.

↓

Heat samples to dryness on hot plate.

↓

Add 15g of NaOH.

↓

Cover crucibles with zirconium lid and place in muffle furnace at 600°C for 15-20 minutes.

↓

Carefully remove samples from furnace and cool in fume hood.

↓

Add 25-50mL of water and heat on hot plate to dissolve fusion cake.

↓

Transfer to a 250mL centrifuge tube. Rinse crucible with water. Dilute to 160mL with water.

↓

Add 20mL conc. HCl (omit if Sr meas.), 125mg Fe, 5mg La. Mix. Add 5mL 3.2M $(NH_4)_2HPO_4$ (8.5mL if Sr meas.). Mix. Centrifuge 10min. Decant supernate.

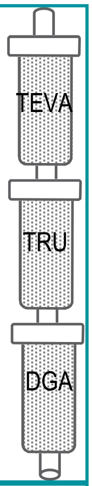
↓

Dissolve precipitate in 80mL 1.5M HCl. Dilute to 170mL. Add 4mL 10% $TiCl_3$ and 15mL 49%HF. Mix. Cool in ice bath 10min. Centrifuge 10min. Decant supernate.

↓

Dissolve precipitate in 7mL 3M HNO_3-0.25M Boric acid, 7.5mL 7M HNO_3, and 7mL 2M $Al(NO_3)_3$. Adjust valence with 1mg Fe, 1.5mL 1M Ascorbic acid. Mix. Add 1mL 3.5M $NaNO_2$.

Figure 2. Actinide Separation on TEVA - TRU - DGA* and Source Preparation



(1) Precondition stacked 2mL TEVA, TRU, DGA cartridges with 10mL 3M HNO_3.*

(2) Load sample solution.

(3) Rinse sample tube with 5mL 3M 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
- -5mL 3M HNO_3

(7) Strip Pu (and Np) from TEVA cartridge with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

(8) Rinse DGA cartridge with 10mL 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 cartridge from DGA cartridge.

(12) Rinse DGA cartridge sequentially 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.

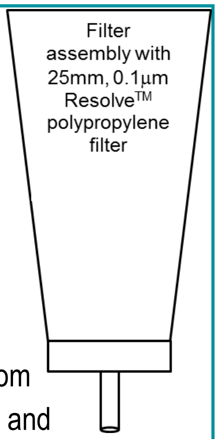
(17) Add 50ug Ce to Pu and Am/Cm samples, 100ug Ce to U samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(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 filter.



(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.

*Radiostrontium may also be measured by adding a 2mL + 1mL Sr Resin cartridge below DGA and following separation scheme in Eichrom Application note AN-1604-10.

**Adding 50uL 30% H_2O_2 to the tube rinse can improve Uranium recoveries and decontamination in Pu(Np) fractions.

Method Performance

Sample	replicates	analyte	tracer	% tracer recovery	mBq/g reference	mBq/g measured	% bias
1.5g limestone	6	$^{239/240}$Pu	^{242}Pu	100 ± 5	29.4	30 ± 2	± 5
1.5g limestone	6	$^{239/240}$Pu	^{236}Pu	93 ± 6	23.0	24 ± 1	± 5
1.5g limestone	6	^{238}Pu	^{236}Pu	93 ± 6	28.8	29 ± 2	± 5
1.5g limestone	6	^{237}Np	^{236}Pu	93 ± 6	37.0	39 ± 3	± 7
1.5 g marble	4	$^{239/240}$Pu	^{242}Pu	96 ± 3	29.4	30 ± 2	± 6
1.5 g marble	4	^{241}Am	^{243}Am	89 ± 4	29.1	29 ± 1	± 3
1.5 g marble	4	^{244}Cm	^{243}Am	89 ± 4	34.8	35 ± 3	± 6
1.5 g marble	7	^{238}U	^{232}U	93 ± 6	50.2	48 ± 1	± 4

References

1) Maxwell, Culligan, Hutchinson, Utsey, Sudowe, McAlister, "Rapid Method to Determine Actinides and Sr-89/90 in Limestone and Marble Samples," *J. Radioanal. Nucl. Chem.* 310, 377-388 (2016).

AN-1604-10

Rapid Determination of $^{89/90}$Sr in Limestone and Marble

Summary of Method Strontium is separated and concentrated from 1.5 gram samples of limestone or marble. Samples are finely ground and fused in a zirconium crucible for 15 minutes at 600°C with 15 grams of sodium hydroxide. The fusion cake is dissolved in water, and strontium is concentrated and separated from the matrix using a calcium phosphate precipitate enhanced with iron. A secondary precipitation with calcium fluoride removes additional matrix (including silicates) and decreases the volume of precipitate. The calcium fluoride precipitate is dissolved with nitric acid-boric acid-aluminum nitrate to form the load solution. Strontium is separated from remaining matrix and potentially interfering radionuclides using stacked 2mL + 1mL Sr Resin cartridges. Radiostrontium is measured by gas flow proportional counting or liquid scintillation counting. Chemical yields of strontium are determined by gravimetric yield or by ICP-AES. Batches of 12-24 samples can be prepared for analysis in less than 8 hours. Simultaneous separation of actinides can be achieved by using the separation method in AN-1603.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Sr Resin, 1mL Cartridges (Eichrom SR1ML-R50-S)
Strontium Carrier (10mg/mL)
Iron Carrier (50mg/mL Fe, as ferric nitrate)
^{90}Sr standard
Nitric Acid (70%)
Deionized Water
3.2M $(NH_4)_2HPO_4$
Oxalic acid
Sodium Hydroxide
HF(49%)
Hydrochloric Acid (37%)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
Boric acid

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)
50mL and 250mL Centrifuge Tubes
Cupped Stainless Steel Planchets (~5mL volume)
250mL Zirconium crucibles with zirconium lids
Centrifuge
Muffle Furnace
Analytical Balance
Gas Flow Proportional Counter
Hot Plate/Heat Lamp
Vacuum Pump

Figure 1. Sample Preparation

1.5g finely ground sample in zirconium crucible

Add 4mg Sr carrier.

Heat samples to dryness on hot plate.

Add 15g of NaOH.

Cover crucibles with zirconium lid and place
in muffle furnace at 600°C for 15-20 minutes.

Carefully remove samples from furnace
and cool in fume hood.

Add 25-50mL of water and heat on hot
plate to dissolve fusion cake.

Transfer to a 250mL centrifuge tube. Rinse
crucible with water. Dilute to 160mL with water.

Add 50mg Fe Mix.
Add 8.5mL 3.2M $(NH_4)_2HPO_4$. Mix.
Centrifuge 10min. Decant supernate.

Dissolve precipitate in 80mL 1.5M HCl. Dilute to 170mL.
Add 15mL 49%HF. Mix. Cool in ice bath 10min.
Centrifuge 10min. Decant supernate.

Dissolve precipitate in 7mL 3M HNO_3-0.25M Boric acid,
7.5mL 7M HNO_3, and 7mL 2M $Al(NO_3)_3$. Warm as needed.

Figure 2. Strontium Resin Separation (Optional ^{90}Y Ingrowth)*

(1) Precondition Sr Resin with
10mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

(3) Rinse sample tube with 5mL
8M HNO_3.

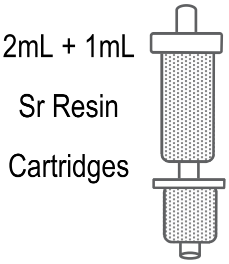
(4) Add tube rinse to Sr Resin.
Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:

- 10 mL 8M HNO_3
- 10mL 3M HNO_3 - 0.05 oxalic acid
- 10mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

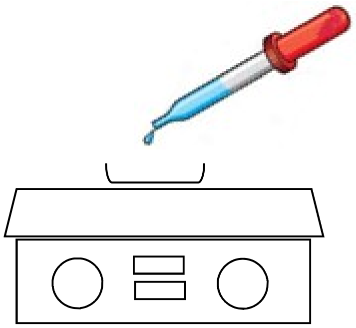
(7) Strip Sr with 20mL 0.05M HNO_3
at 1mL/min.



Gas Flow Proportional Counting:**

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

**(Options for $^{89/90}$Sr Discrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr may then be measured after adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

(c) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. ^{90}Y can be removed on DGA Resin. $^{89/90}$Sr will elute in DGA Resin load. Additional rinsing will remove remaining Sr. ^{90}Y can be eluted in 0.1M HCl and counted by gas flow proportional counting or liquid scintillation (Cerenkov).

*Actinides may also be measured by adding 2mL TEVA, TRU and DGA Resin cartridges above Sr Resin and following separation scheme in Eichrom Application note AN-1603.

**Additional discussion of $^{89/90}$Sr separation and measurement options can be found in Eichrom Application Note AN-1624-10.

Method Performance

Sample	**% Sr tracer recovery**	**^{90}Sr Bq/g reference**	**^{90}Sr Bq/g measured**	**% bias**
1	84.1	1.415	1.41	-0.1
2	84.8	1.415	1.42	0.4
3	84.8	1.415	1.38	-2.7
AVG	84.6 ± 0.4		1.40 ± 0.02	

References

1) Maxwell, Culligan, Hutchinson, Utsey, Sudowe, McAlister, “Rapid Method to Determine Actinides and Sr-89/90 in Limestone and Marble Samples,” *J. Radioanal. Nucl. Chem.* 310, 377-388 (2016).

AN-1605-10

Rapid Determination of $^{89/90}Sr$ in 5g Concrete Samples

Summary of Method Strontium is separated and concentrated from 5 gram concrete samples. Samples are finely ground and fused in a zirconium crucible for 30 minutes at 600°C with 30 grams of sodium hydroxide. The fusion cake is dissolved in water, and strontium is concentrated and separated from the matrix using a calcium phosphate precipitate enhanced with iron. A secondary precipitation with calcium fluoride removes additional matrix (including silicates) and decreases the volume of precipitate. The calcium fluoride precipitate is dissolved with nitric acid-boric acid -aluminum nitrate to form the load solution. Analytes are separated from remaining matrix and potentially interfering radionuclides using two stacked 2mL Sr Resin cartridges. Radiostrontium is measured by gas flow proportional counting or liquid scintillation counting. Chemical yields of strontium are determined by gravimetric yield or by ICP-AES. Batches of 12-24 samples can be prepared for analysis in less than 8 hours. For aged samples, where the shorter lived ^{89}Sr ($t_{1/2}$ = 50.55 days) is unlikely to be present, ^{90}Sr can be determined from the direct separation of its ^{90}Y daughter from up to 10g concrete samples, using Eichrom Application Note AN-1606.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Strontium Carrier (10mg/mL)
Iron Carrier (50mg/mL Fe, as ferric nitrate)
^{90}Sr standard
30% H_2O_2
Hydrochloric Acid (37%)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
Boric acid
HF(49%)
Nitric Acid (70%)
Deionized Water
3.2M $(NH_4)_2HPO_4$
Oxalic acid
Sodium Hydroxide

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)
50mL and 500mL Centrifuge Tubes
Cupped Stainless Steel Planchets (~5mL volume)
250mL Zirconium crucibles with zirconium lids
Centrifuge
Muffle Furnace
Analytical Balance
Gas Flow Proportional Counter
Hot Plate/Heat Lamp
Vacuum Pump

Figure 1. Sample Preparation

5g finely ground sample in zirconium crucible

Add 6mg Sr carrier.

Heat samples to dryness on hot plate.

Add 30g of NaOH.

Cover crucibles with zirconium lid and place in muffle furnace at 600°C for 30 minutes.

Carefully remove samples from furnace and cool in fume hood.

Add 50-100mL of water and heat on hot plate to dissolve fusion cake.

Transfer to a 500mL centrifuge tube. Rinse crucible with water. Dilute to 450mL with water.

Cool to room temp. Add 50mg Fe. Mix. Add 8.5mL 3.2M $(NH_4)_2HPO_4$. Mix. Centrifuge 10min. Decant supernate.

Dissolve precipitate in 200mL 1.5M HCl. Add 50mL 0.01M HCl and 15mL 49%HF. Mix. Cool in ice bath 10min. Centrifuge 10min. Decant supernate.

Dissolve precipitate in 7mL 3M HNO_3-0.25M Boric acid, 7mL conc. HNO_3, 7mL 8M HNO_3 and 7mL 2M $Al(NO_3)_3$. Warm as needed.

Figure 2. Strontium Resin Separation (Optional ^{90}Y Ingrowth)

(1) Precondition 2x2mL Sr Resin with 10mL 8M HNO_3.

(2) Load sample at 1-2mL/min.

(3) Rinse sample tube with 5mL 8M HNO_3.

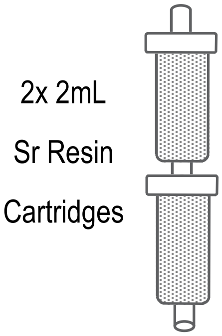
(4) Add tube rinse to Sr Resin. Elute at 1-2mL/min.

(5) Rinse Sr Resin sequentially with:

- 20 mL 8M HNO_3
- 10mL 3M HNO_3 - 0.05 oxalic acid
- 10mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.

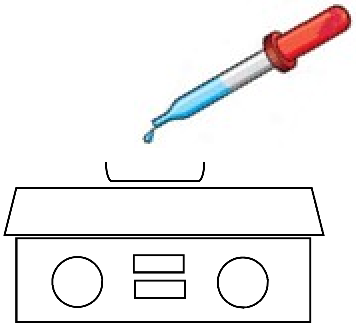
(7) Strip Sr with 20mL 0.05M HNO_3 at 1mL/min.



Gas Flow Proportional Counting:*

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Discrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr may then be measured after adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

(c) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. ^{90}Y can be removed on DGA Resin. $^{89/90}$Sr will elute in DGA Resin load. Additional rinsing will remove remaining Sr. ^{90}Y can be eluted in 0.1M HCl and counted by gas flow proportional counting or liquid scintillation (Cerenkov).

*Additional discussion of $^{89/90}$Sr separation and measurement options can be found in Eichrom Application Note AN-1624-10.

Method Performance (5g gram Concrete, Sr Resin Method)

Sample	% Sr tracer recovery	^{90}Sr Bq/g reference	^{90}Sr Bq/g measured	% bias
1	78.5	1.416	1.51	6.6
2	77.8	1.416	1.35	-4.6
3	80.5	1.416	1.42	0.2
4	62.2	1.416	1.49	5.2
AVG	75 ± 8		1.44 ± 0.07	

References

1) Maxwell, Culligan, Hutchinson, Utsey, Sudowe, McAlister, "Rapid Method to Determine 89/90Sr in Large Concrete Samples," *J. Radioanal. Nucl. Chem.* 310, 399-411 (2016).

eichrom®

AN-1606-10

Rapid Determination of ^{90}Sr in 10g Concrete Samples

Summary of Method ^{90}Sr is determined by the direct separation of its daughter ^{90}Y from 10 gram concrete samples. Samples are finely ground and fused in a zirconium crucible for 30 minutes at 600°C with 40 grams of sodium hydroxide. The fusion cake is dissolved in water, and strontium is concentrated and separated from the matrix using a ferric hydroxide precipitate. A secondary precipitation with Y/Ca-fluoride removes additional matrix (including silicates) and decreases the volume of precipitate. The Y/Ca-fluoride precipitate is dissolved with nitric acid-boric acid-aluminum nitrate to form the load solution. ^{90}Y is separated from remaining matrix and potentially interfering radionuclides using stacked 2mL TRU and DGA Resin cartridges. ^{90}Y is measured by gas flow proportional counting following microprecipitation onto Resolve® Filters. Chemical yields are determined by ICP-AES analysis. Batches of 12-24 samples can be prepared for analysis in less than 8 hours. This method is only suitable for aged samples, where the shorter lived ^{89}Sr ($t_{1/2}$ = 50.55 days) and fission products such as ^{91}Y are unlikely to be present. For samples not meeting this criterion, $^{89/90}$Sr can be determined from up to 5g concrete samples, using Eichrom Application Note AN-1605.

Reagents

DGA Resin, 2mL Cartridges (Eichrom DN-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
Yttrium Carrier (10mg/mL)
Iron Carrier (50mg/mL Fe, as ferric nitrate)
^{90}Sr standard
Nitric Acid (70%)
Hydrochloric Acid (37%)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
Boric acid
HF(49%)
Sodium Hydroxide
Deionized Water
3.2M $(NH_4)_2HPO_4$
Oxalic acid

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 with funnel (Eichrom RF-DF25-25PP01)
50mL and 500mL Centrifuge Tubes
Stainless Steel Planchets with two sided tape
250mL Zirconium crucibles with zirconium lids
Centrifuge
Muffle Furnace
Analytical Balance
Gas Flow Proportional Counter
Hot Plate/Heat Lamp
Vacuum Pump

Figure 1. Sample Preparation

10g finely ground sample in zirconium crucible

Add 2mg Y carrier.
Heat samples to dryness on hot plate.

Add 40g of NaOH.
Cover crucibles with zirconium lid and place in muffle furnace at 600°C for 30 minutes.

Carefully remove samples from furnace and cool in fume hood.

Add 50-100mL of water and heat on hot plate to dissolve fusion cake.

Transfer to a 500mL centrifuge tube. Rinse crucible with water. Dilute to 450mL with water. Cool to room temp. Add 125mg Fe Mix. Centrifuge 10min. Decant supernate.

Rinse precipitate with 150mL pH ~9 NaOH. Centrifuge. Decant Supernate. Repeat.

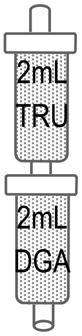
Dissolve precipitate in 200mL 1.5M HCl. Add 50mL 0.01M HCl and 15mL 49%HF. Mix. Cool in ice bath 10min. Centrifuge 10min. Decant supernate.

Dissolve precipitate in 7mL 3M HNO_3-0.25M Boric acid, 7mL conc. HNO_3, 7mL 8M HNO_3 and 7mL 2M $Al(NO_3)_3$. Warm as needed.

Figure 2. TRU-DGA Separation and Gas Flow Proportional Counting

(1) Precondition 2mL TRU* + DGA Resin with 10mL 8M HNO_3.

(2) Load sample at 1-2mL/min.



(3) Rinse sample tube with 5mL 8M HNO_3.

(4) Add tube rinse TRU + DGA. Elute 1-2 mL/min.

(5) Rinse sequentially with:
- 5mL 6M HNO_3
- 10mL 3M HNO_3
- 12mL 4M HCl

(6) Dispose of (1) to (5) as waste.

(7) Discard TRU Resin.

(8) Rinse DGA sequentially with:
- 10mL 8M HNO_3 (Ca)
- 15mL 0.1M HNO_3 (U)
- 25mL 3M HNO_3-0.25M HF (Th)

(9) Strip Y with 18mL 0.25M HCl.

(10) Dilute to 20mL with water.

(11) Take 1mL aliquot and dilute to 20mL for ICP-AES yield measurement.

(8) Add 100ug Ce carrier to samples.

(9) Mix well.

(10) Add 2mL 49% HF.

(11) Mix well.

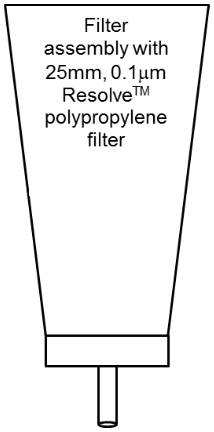
(12) Wait 15-20 minutes.

(13) Set up Resolve® Filter Funnel on vacuum box.

(14) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(15) Filter sample.

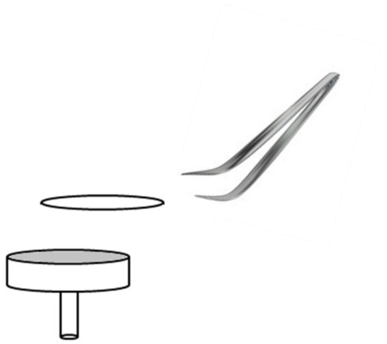
(16) Rinse sample tube with 5mL DI water and add to filter.



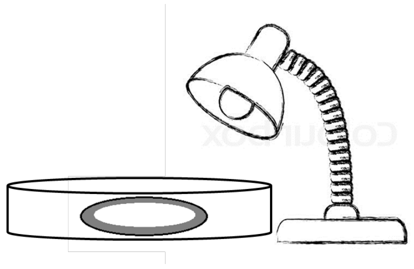
(17) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(18) Draw vacuum until filter is dry.

(19) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(20) Dry filter under heat lamp for 3-5 minutes.



(21) Measure Y-90 by gas flow proportional counting.

*TRU Resin improves decontamination factors for U, Th and Bi isotopes, which could interfere with the measurement of ^{90}Y by gas flow proportional counting.

Method Performance (10g gram Concrete, TRU/DGA Resin Method)

Sample	% Y tracer recovery	^{90}Sr Bq/g reference	^{90}Sr Bq/g measured	% bias
1	81.7	0.0327	0.031	-5.4
2	83.3	0.0327	0.033	1.2
3	83.7	0.0327	0.031	-5.0
4	86.3	0.0327	0.033	-0.6
AVG	84 ± 2		0.032 ± 0.001	

References

1) Maxwell, Culligan, Hutchinson, Utsey, Sudowe, McAlister, “Rapid Method to Determine 89/90Sr in Large Concrete Samples,” *J. Radioanal. Nucl. Chem.* 310, 399-411 (2016).

eichrom®

AN-1607-10

Rapid Determination of Np/Pu/Am in 10-20g Soil Samples

Summary of Method Plutonium, Neptunium, and Americium are separated and concentrated from 10 gram soil samples. Samples are fused in zirconium crucibles with 40g NaOH to facilitate complete dissolution. Actinides are separated from matrix using an iron/titanium hydroxide precipitate. A second precipitate with Ca/La-fluoride is used to remove additional matrix, particularly silicates, and decrease the volume of precipitate. Actinides are separated from potential radiometric impurities using 2mL cartridges of TEVA and DGA Resins. Am/Cm fractions may require additional purification using TEVA-NH_4SCN to remove native rare earths which can degrade alpha spectra. Pu/Np and Am/Cm are prepared for alpha spectrometry measurement via CeF_3 microprecipitation onto Resolve® Filters. To further lower detection limits, two 10g soil aliquots can be fused separately, combined following the Fe/Ti hydroxide precipitate, and then processed through the remaining steps of the method.

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}Pu (or ^{236}Pu if Np Measured) tracer
^{243}Am tracer
Ce Carrier (10mg/mL)
Deionized Water
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
$NaNO_2$
Ascorbic Acid
NH_4SCN (rare earth separation)
La carrier (10mg/mL)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
Sulfamic Acid
30% H_2O_2

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 with funnel (Eichrom RF-DF25-25PP01)
250mL Zirconium Crucibles with lids
50mL and 500mL Centrifuge Tubes
Alpha Spectrometry System
Stainless Steel Planchets with double sided tape
Centrifuge
Analytical Balance
Muffle Furnace
Hot Plate
Vacuum Pump

Figure 1. Sample Preparation

Dry soil at 110°C. Blend and Crush to fine powder.

Aliquot 10g sample to Zr crucible. Add $^{242}Pu(^{236}Pu)/^{243}Am$.

Dry on Hotplate. Place in Muffle furnace at 250°C.
Ramp to 600°C. Heat 2 hrs to destroy organics.

Remove from furnace. Add 40g NaOH.

Fuse at 600°C for 30 minutes.

Transfer to 500mL centrifuge tube with water.
Add 125mg Fe, 100mg Ca, 10mg La, 10mL 10% $TiCl_3$.
Dilute to 450 mL. Mix. Cool to room temp. in ice bath.

Centrifuge 10 min. Discard Supernate.

Rinse ppt with 150 mL pH ~9 NaOH.
Centrifuge 10min. Decant Supernate.

Partially dissolve in 200mL 1.5M HCl.
<u>Solids will remain.</u> Dilute to 250mL.
Add 6mL 10% $TiCl_3$ and 40mL 49% HF.
Mix. Place in ice bath for 10min.

Centrifuge 10min. Decant supernate.

Dissolve solids in 10mL 3M HNO_3-0.25M H_3BO_3, 6mL 7M HNO_3, 8mL 2M $Al(NO_3)_3$ and 3mL 3M HNO_3.
Warming samples can improve dissolution.

Cool to room temperature.
Adjust valence with 1mg Fe, 1.5mL 1M Ascorbic acid. Mix. Add 1mL 3.5M $NaNO_2$.

Figure 2. Actinide Separation on TEVA - DGA

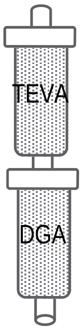
(1) Precondition 2mL TEVA + DGA cartridges with 5mL 8M HNO_3.

(2) Load Sample.

(3) Rinse centrifuge tube with 5mL 6M HNO_3. Add to TEVA + DGA.*

(4) Rinse TEVA + DGA with 10mL 3M HNO_3.

(5) Separate TEVA and DGA.



(6) Rinse TEVA with
- -15mL 3M HNO_3
- -20mL 9M HCl (Th)
- -5mL 3M HNO_3

(7) Strip Pu/Np from TEVA with 20mL 0.1M HCl-0.05M HF-0.01M $TiCl_3$.

(8) Rinse DGA with:
- -10mL 3M HCl (Ca)
- -3mL 1M HNO_3
- -15mL 0.1M HNO_3 (La, Ca)
- -25mL 3M HNO_3-0.25M HF (Th)
- -5mL 4M HCl

(9) Strip Am/Cm with 12mL 0.25M HCl.

<u>Optional: Rare Earth Removal Steps</u>

(10) Add 3mL 3M HNO_3 and 3mL 30% H_2O_2 to Am/Cm fraction.

(11) Wet ash on hotplate to dryness.

(12) Dissolve in 10mL 1.5M NH_4SCN.

(13) Precondition 2mL TEVA with 5mL 1.5M NH_4SCN.

(14) Load Am/Cm fraction.

(15) Rinse beaker with 5mL 1.5M NH_4SCN. Add to TEVA.

(16) Rinse TEVA with 5mL 1.5M NH_4SCN.

(17) Strip Am/Cm with 20mL 1M HCl.

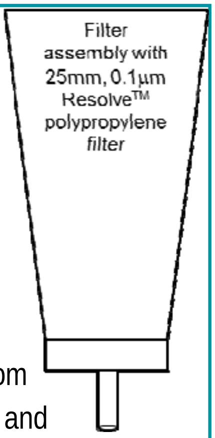
(18) Add 50ug Ce carrier and 0.5mL 30% H_2O_2 to all samples. Mix well. Add 1mL 49% HF. Mix well. Wait 15-20 minutes.

(19) Set up Resolve® Filter Funnel on vacuum box.

(20) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

(21) Filter sample.

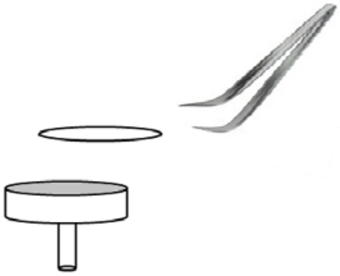
(22) Rinse sample tube with 5mL DI water and add to filter.



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

(24) Draw vacuum until filter is dry.

(25) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(26) Dry filter under heat lamp for 3-5 minutes.

(27) Measure actinides by alpha spectrometry.

*Adding 50uL of 30% H_2O_2 to the 6M HNO_3 tube rinse can further improve uranium decontamination factors.

Method Performance

Sample	replicates	analyte	tracer	% tracer recovery	mBq/g reference	mBq/g measured	% bias
10g Soil	10	$^{239/240}Pu$	^{236}Pu	85 ± 8	3.43	3.41 ± 0.22	± 5
10g Soil	6	^{237}Np	^{236}Pu	82 ± 4	3.99	4.19 ± 0.16	± 6
10g Soil	11	^{241}Am	^{243}Am	89 ± 4	2.14	2.07 ± 0.16	± 6

References

1) Maxwell, Culligan, Hutchinson, McAlister, “Rapid Fusion Method for the Determination of Pu, Np, and Am in Large Soil Samples,” *J. Radioanal. Nucl. Chem.* 305 : 599-608 (2015).

AN-1608-10

Rapid Fusion Method for Refractory Th, U, and Pu in Soils

Summary of Method U, Th and Pu are separated and concentrated from 1-2 gram soil samples. Samples are fused with NaOH at 600°C in zirconium crucibles. The fusion cakes are dissolved in water, transferred to 250mL centrifuge tubes and precipitated sequentially with Fe/Ti-hydroxide and lanthanum fluoride to facilitate matrix removal. U, Th, and Pu are separated on stacked 2mL cartridges of Eichrom TEVA and TRU resins. U, Th, and Pu are measured by alpha spectrometry following CeF_3 microprecipitation onto Eichrom Resolve® Filters. Batches of 12-24 samples can be prepared for alpha spectrometry in less than 8 hours. Method ruggedness has been demonstrated with successful analysis of high fired refractory material from MAPEP 30 soil standards. For one gram soil samples and 16 hour count times, MDA for this method is ~500uBq/g.

Reagents

TEVA Resin, 2mL Cartridges (Eichrom TE-R50-S)
TRU Resin, 2mL Cartridges (Eichrom TR-R50-S)
Iron carrier (50mg/mL Fe, as ferric iron nitrate)
^{242}Pu, ^{232}U and ^{229}Th tracers
Oxalic acid/Ammonium oxalate
La carrier (10mg/mL)
Deionized Water
3.2M $(NH_4)_2HPO_4$
10% (w:w) $TiCl_3$
HCl (37%)
HF (49%) or NaF
H_2O_2 (30%)
Denatured ethanol
Ce carrier (1mg/mL)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
HNO_3 (70%)
NaOH
Boric acid
$NaNO_2$
Ascorbic Acid

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
Heat Lamp
Muffle Furnace
Hot Plate
Analytical Balance
250mL Zirconium crucibles with zirconium lids
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Vacuum Pump

Figure 1. Sample Preparation

1-2g finely ground soil + tracers in zirconium crucible.

↓

Place in Furnace at 250°C. Ramp to 600°C. Heat 30 min.

↓

Cool. Add 15g NaOH. Fuse at 600°C for 15-20 minutes.

↓

Dissolve fusion cake with 2x 50mL H_2O. Transfer to 250mL c-tube.

↓

Add 10mL 3M HNO_3 to crucible. Heat to dissolve residue.
Transfer to same 250mL c-tube.

↓

Add 150mg Fe and 5mg La to c-tube. Dilute to 180mL.

↓

Add 2mL 1.25M $Ca(NO_3)_2$, 3mL 3.2M $(NH_4)_2HPO_4$,
10mL 10% $TiCl_3$. Mix. Cool in ice bath for 10 min.

↓

Centrifuge 10min. Decant Supernate.

↓

Partially dissolve precipitate in 80mL 1.5M HCl.
Some solids will remain. Dilute to 170mL.
Add 1mg La, 0.5mL 1.25M Ca, and 6mL 10% $TiCl_3$.
Mix. Add 25mL 49% HF. Cool in ice bath for 10 min.

↓

Centrifuge 10min. Decant Supernate.

↓

Dissolve precipitate in 10mL 3M HNO_3-0.25M Boric acid, 6mL 7M HNO_3, and 8.5mL 2M $Al(NO_3)_3$.
Warming samples can help complete dissolution.

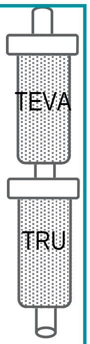
↓

Cool samples to room temperature.

↓

Fix valence states. Mix between each addition of:
20uL 50mg/mL Fe
1.5mL 1M ascorbic acid
1mL 3.5M $NaNO_2$.

Figure 2. Actinide Separation on TEVA - TRU - DGA* and Source Preparation



(1) Precondition stacked 2mL TEVA and TRU cartridges with 10mL 3M HNO_3.

(2) Load sample solution.

(3) Rinse sample tube with 5mL 3M HNO_3 + 30uL 30% H_2O_2. Add to cartridges.

(4) Rinse cartridges w/ 10mL 3M HNO_3.

(5) Separate TEVA-TRU cartridges.

(6) Rinse TEVA cartridge with10mL 3M HNO_3.

(7) Strip Th with 15mL 9M HCl. Dilute to 30mL. Add 0.5mL 30% H_2O_2 and 40ug Ce.

(8) Rinse TEVA with:

- 5mL 9M HCl
- 5mL 3M HNO_3

(9) Strip Pu (and Np) from TEVA with 20mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$. Add 0.5mL 30% H_2O_2 and 50ug Ce. Mix.

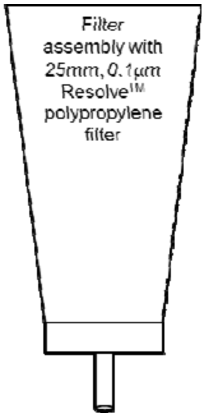
(10) Rinse TRU cartridge with:

- 5mL 8M HNO_3
- 20mL 4M HCl-0.2M HF-0.002M $TiCl_3$
- 10mL 8M HNO_3

(11) Strip U from TRU with 15mL of 0.1M ammonium bioxalate.

(12) Add 100ug Ce and 0.5mL 10% $TiCl_3$

(13) Add 1mL 49% HF to all samples. Mix well. Wait 15-20 minutes.



(14) Set up Resolve® Filter Funnel on vacuum box.

(15) Wet filter with 3mL 80% ethanol followed by 3mL DI water.

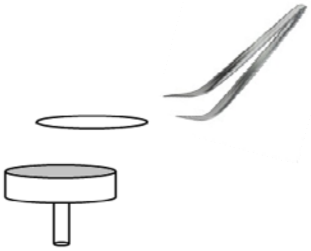
(16) Filter sample.

(17) Rinse sample tube with 5mL DI water and add to filter.

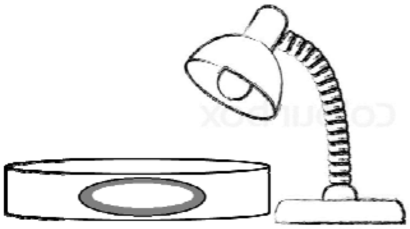
(18) Rinse filter funnel with 3mL DI water and 2mL 100% ethanol.

(19) Draw vacuum until filter is dry.

(20) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(21) Dry filter under heat lamp for 3-5 minutes.



(22) Measure actinides by alpha spectrometry.

Method Performance

Sample	replicates	analyte	tracer	% tracer recovery	mBq/g reference	mBq/g measured	% bias
1g Soil	12	^{238}U	^{232}U	86 ± 8	83.0	85 ± 3	± 3
1g Soil	12	^{234}U	^{232}U	86 ± 8	81.0	80 ± 2	± 2
1g Soil	12	^{228}Th	^{229}Th	91 ± 6	51.1	50 ± 2	± 4
1g Soil	12	^{230}Th	^{229}Th	91 ± 6	96.2	98 ± 6	± 5
1g Soil	12	^{232}Th	^{229}Th	91 ± 6	48.8	50 ± 3	± 6
1g Soil	3	^{239}Pu	^{242}Pu	91 ± 6	76.8	79 ± 3	± 4
1g Soil	3	^{239}Pu	^{242}Pu	91 ± 6	96.0	98 ± 5	± 4

References

1) Maxwell, Hutchinson, McAlister, "Rapid Fusion Method for the Determination of Refractory Thorium and Uranium Isotopes in Soil Samples" *Analytica Chimica Acta,* 701(1), 112-118 (2015).

Measurement of Tritium in Water

AN-1609-10

Summary of Method Tritium is measured in 5-10mL aliquots of water using liquid scintillation counting. An Eichrom Tritium column is used to remove potentially interfering nuclides and matrix which can cause quench in the liquid scintillation cocktail. Sample size will be limited by the amount of sample that can be effectively mixed with the liquid scintillation cocktail (typically 5-10mL) and the salt content of the sample which can impact the separation of difficult to remove nuclides, such as isotopes of Cs. For samples which this method is not adequate, distillation methods, such as ASTM D4107 are recommended.

Reagents

Tritium Column (Eichrom H3-C50-A)
Deionized Water
Liquid Scintillation Cocktail
Nitromethane (Or other quench agent)
^{3}H Standard (To measure LSC quench curve)
HCl (for sample pH adjustment)
NaOH or NH_4OH (for sample pH adjustment)

Equipment

Column Rack (Eichrom AC-103)
Extension Funnels (Eichrom AC-120)
Centrifuge Tubes - 50mL
20mL glass liquid scintillation tubes
Liquid scintillation counter
Calibrated pipets and disposable tips
pH meter or pH strips
Analytical balance

Table 1. Sample Processing on Tritium Column

	Tap Water	Ground Water	Sea Water
Sample mL	15	15	3
Dilution	none	none	3mL sample to 10mL
Discard mL	2.5	2.5	2.5
Collect mL	12.5	12.5	5.0
mL to LSC	10.0	10.0	5.0

Sample Preparation

1-25mL of water sample.

Dilute high salt samples as necessary (Table 1)

If necessary, filter sample.

Adjust sample pH to 6-8.

Remove tips from tritium columns.

Allow column storage solution to drain.

Rinse column with 10mL DI water.

Add sample to column.

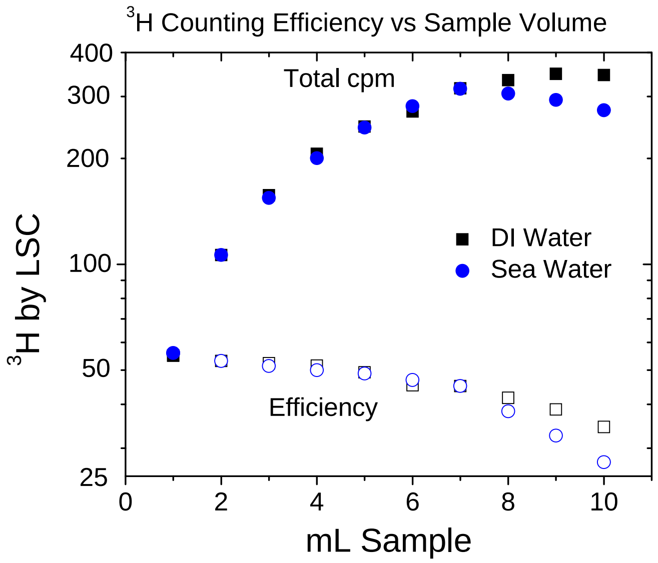
Allow column to flow by gravity.
Discard first 2.5mL.

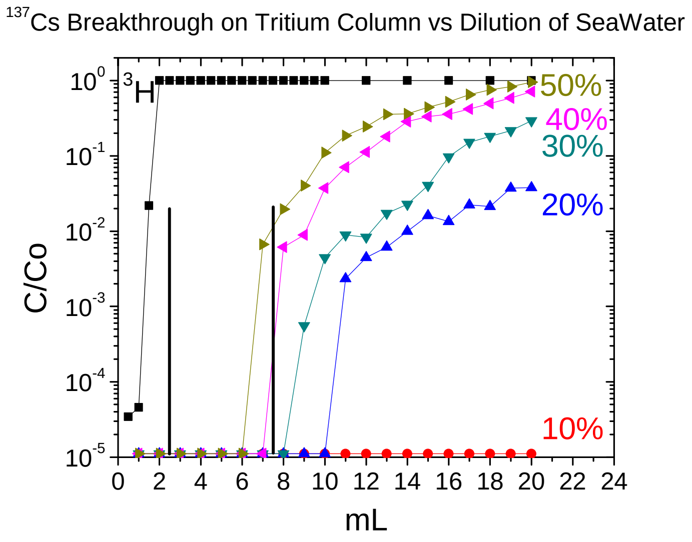
Collect remaining sample in
50mL centrifuge tube.

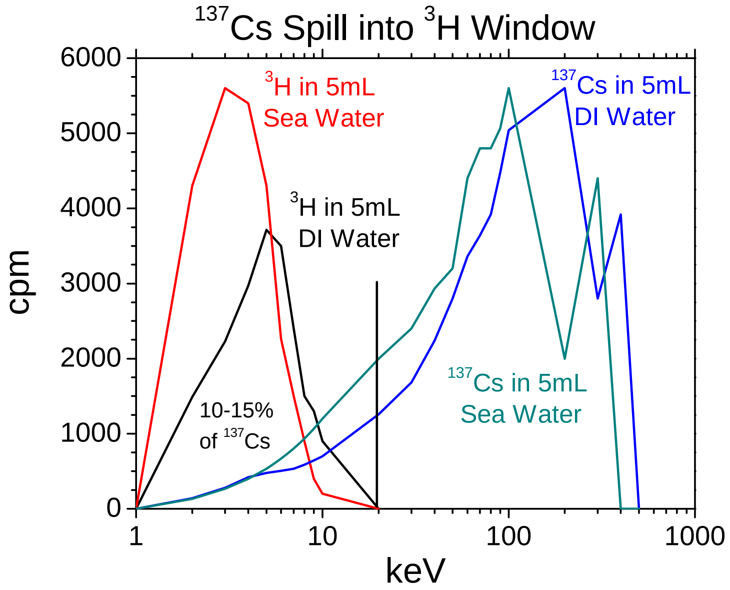
Aliquot 5-10mL of purified sample
to 20mL glass LSC vial.

Add appropriate amount of liquid
scintillation cocktail.

Mix samples and cocktail.
Dark adapt samples for 1-2 hours
before counting by liquid scintillation.







References

1) Eichrom Method H3W02. “Tritium in water,” http://www.eichrom.com/eichrom/radiochem/methods/eichrom/

eichrom®

AN-1610-20

Measurement of $^{59/63}$Ni in Water

Summary of Method Nickel-59/63 is separated and measured from up to 500mL aliquots of water. Samples are preconcentrated by evaporation or ferric hydroxide precipitation, dissolved in 1M HCl, buffered with ammonium citrate and adjusted to pH 8-9 with ammonium hydroxide. Citrate complexes Fe(III), preventing precipitation at pH 8-9. Nickel is loaded onto 2mL cartridges of Nickel Resin. Yields can be improved by adding a 2mL cartridge of prefilter resin below the Nickel cartridge to minimize losses of the Ni-DMG complex. Nickel is recovered in 3M HNO_3 and measured by liquid scintillation counting. Chemical recovery of nickel is determined by ICP-AES measurement of 1-2mg of stable nickel carrier.

Reagents

Nickel Resin Cartridges (Eichrom NI-R50-S)
Prefilter Resin Cartridges (Eichrom PF-R50-L)
Anion Exchange Resin Cartridges (Eichrom A8-R50-M-Cl)*
Deionized Water
Ammonium Citrate
Ammonium Hydroxide
Sodium Hydroxide
HCl
HNO_3
Iron(III) Carrier (10mg/mL)
Nickel Carrier (10mg/mL)
Cobalt Carrier (10mg/mL)*
Phenolphthalein pH indicator
Liquid Scintillation Cocktail

Equipment

Vacuum Box (Eichrom AR-12-BOX or AR-24-BOX)
Vacuum Box Inner Liner (Eichrom AR-12-LINER or AR-24-LINER)
Yellow Outer Tips (Eichrom AR-1000-OT)
Inner Support Tube (Eichrom AR-1000-TUBE-PE)
Cartridge Reservoirs (Eichrom AR-200-RV20)
Centrifuge Tubes - 50mL and 250mL
20mL glass liquid scintillation tubes
Liquid scintillation counter
Calibrated pipets and disposable tips
Appropriately Sized Glass Beakers
ICP-AES system for Ni chemical yield measurement
Analytical balance
Vacuum Pump
Centrifuge
Hotplate

Sample Preparation

Up to 500mL of water sample in glass beaker.

↓

Add 1-2 mg of Ni carrier*

↓

Evaporate to dryness or proceed to ferric hydroxide precipitation steps below.

Ferric Hydroxide Precipitation

Add 2mg of Fe(III) carrier and pH indicator

↓

Heat sample to 80^{o}C

↓

Adjust to pH 8-9 with NaOH.

↓

Mix sample and allow to cool to room temperature.

↓

Allow ppt to settle and decant supernate to <200mL.

↓

Transfer to 250mL centrifuge tube. Rinse beaker with water to ensure complete transfer of ppt.

↓

Centrifuge 10min. Decant supernate to waste.

*1mg of Co and carrier may also be added to improve decontamination from cobalt isotopes. For samples with very high $^{58/60}$Co content, additional separation of cobalt on anion exchange resin may be required. (See Next Page)

*Optional Co/Fe Separation on Anion Exchange Resin

1) Following addition of Ni, Fe, Co carriers and sample evaporation or preconcentration be $Fe(OH)_3$ ppt.
2) Dissolve sample residue or $Fe(OH)_3$ ppt in 10mL 10M HCl.
3) Precondition 2mL anion exchange cartridge with 5mL 10M HCl.
4) Load sample in 10M HCl on anion exchange resin (1mL/min. Fe/Co retained). Collect Ni eluate in glass beaker.
5) Rinse cartridge with 10mL 10M HCl. Collect Ni eluate in glass beaker.
6) Carefully evaporate eluate from steps 4-5 to dryness.

Load Solution Preparation

1) Dissolve ppt/residue in 5-10mL 1M HCl.
2) Add 1-2mL of 1M ammonium citrate.
3) Add pH indicator.
4) Adjust to pH 8-9 with ammonium hydroxide.
5) If ppt. forms, add additional ammonium citrate.

Nickel Separation

1) Set up vacuum box with Nickel cartridges.***
2) Precondition with 5mL 0.2M ammonium citrate.
3) Load samples on Nickel/Prefilter Resin.
4) Rinse Nickel/Prefilter Resin with 20mL 0.2M ammonium citrate.
5) Strip Ni with 10-15mL of 3M HNO_3.
6) Take aliquots for ICP-AES and Liquid Scintillation.

***optional: prefilter cartridges below Ni to improve yield.

References

1) Eichrom Method NIW01VBS. “Nickel-59/63 in water,” http://www.eichrom.com/eichrom/radiochem/methods/eichrom/

eichrom®

AN-1611-10

Measurement of ^{55}Fe in Water (TEVA Separation)

Summary of Method ^{55}Fe is separated and measured from up to 1L aliquots of water. Samples are preconcentrated by evaporation or ferric hydroxide precipitation, dissolved in 4M HCl and loaded onto 2mL cartridges of TEVA Resin. Hold back carriers, 2mg each of Zn, Mn, Cs, Nb, Zr, and Co are added to improve separation from radionuclides of these elements. An iron phosphate precipitate at pH 2.8-3.2 is used to prepare samples for liquid scintillation counting and remove remaining traces of Zn, which can co-elute with iron from TEVA resin. Chemical recovery of iron is determined by ICP-AES measurement of 5mg of stable iron carrier. ^{55}Fe may also be determined using TRU resin, AN-1612 from nitrate media. AN-1612 allows ^{55}Fe incorporation into standard TEVA-TRU actinide separations methods, but is limited to 2mg Fe per sample for a 2mL TRU resin cartridge.

Reagents

TEVA Resin Cartridges (Eichrom TE-R50-S)
Deionized Water
Sodium Hydroxide
HCl
HNO_3
H_3PO_4
LSC Cocktail
Fe, Zn, Mn, Cs, Nb, Zr, Co carriers (10mg/mL)
Phenolphthalein pH indicator
^{55}Fe standard
Nitromethane or other LSC quench agent

Equipment

Vacuum Box (Eichrom AR-12-BOX or AR-24-BOX)
Vacuum Box Inner Liner (Eichrom AR-12-LINER or AR-24-LINER)
Yellow Outer Tips (Eichrom AR-1000-OT)
Inner Support Tube (Eichrom AR-1000-TUBE-PE)
Cartridge Reservoirs (Eichrom AR-200-RV20)
Centrifuge Tubes - 50mL and 250mL
20mL glass liquid scintillation tubes
Liquid scintillation counter
Calibrated pipets and disposable tips
Appropriately Sized Glass Beakers
ICP-AES system for Fe chemical yield measurement
Analytical balance
Vacuum Pump
Centrifuge
Hotplate

Sample Preparation

Up to 1L of water sample in glass beaker.

↓

Add 5 mg Fe, 2mg Zn, Mn, Cs, Nb, Zr, Co

↓

Evaporate to dryness or proceed to ferric hydroxide precipitation steps below.

Ferric Hydroxide Precipitation

Add pH indicator

↓

Heat sample to 80^{o}C

↓

Adjust to pH 8-9 with NaOH.

↓

Mix sample and allow to cool to room temperature.

↓

Allow ppt to settle and decant supernate to <200mL.

↓

Transfer to 250mL centrifuge tube. Rinse beaker with water to ensure complete transfer of ppt.

↓

Centrifuge 10min. Decant supernate to waste.

↓

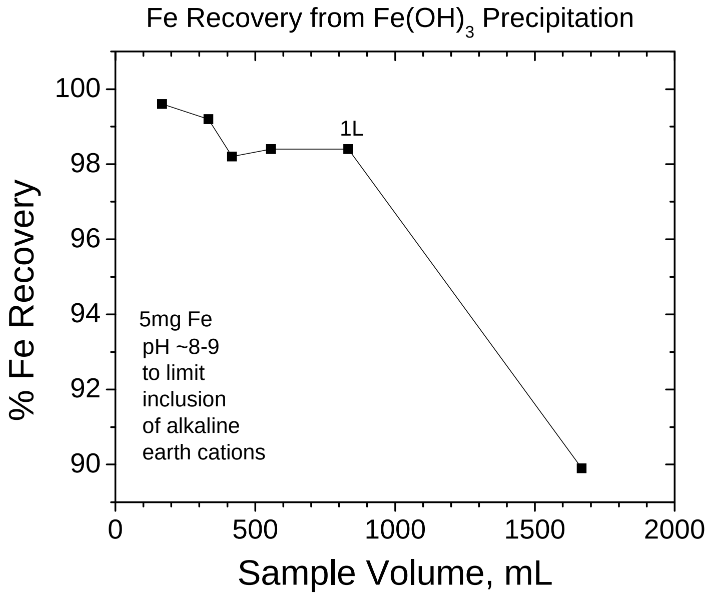
Rinse ppt w/ 50mL Water. Centrifuge. Decant supernate.

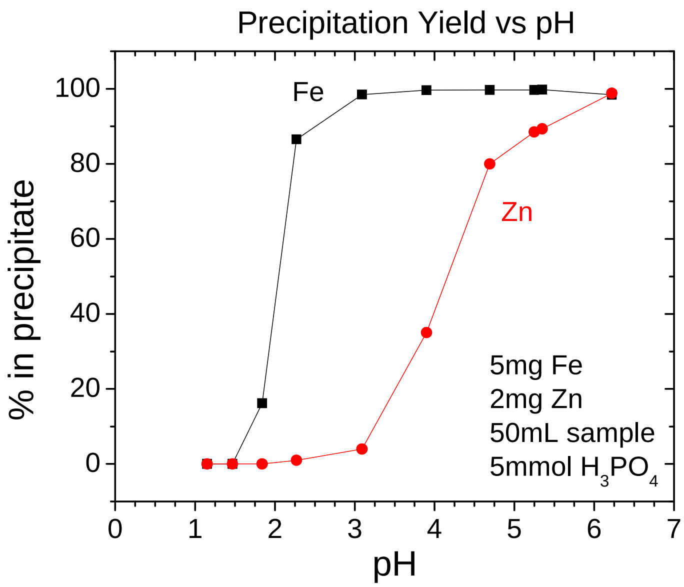
↓

Dissolve ppt/residue in 20mL 4M HCl.

Iron Separation

1) Set up vacuum box with TEVA cartridges.
2) Precondition with 5mL 4M HCl.
3) Load samples on TEVA Resin.
4) Rinse tube with 5mL 4M HCl. Add to TEVA.
5) Rinse TEVA with 10mL 4M HCl.
6) Strip Fe from TEVA with 20mL 0.1M HNO_3.
7) Add 5mL 1M H_3PO_4. Mix.
8) Adjust to pH 2.8-3.2 with NaOH/H_3PO_4. Mix.
9) Centrifuge. Decant Supernate.
10) Wash ppt with 50mL H_2O. Centrifuge. Decant Supernate.
11) Dissolve ppt with minimal 6M HCl.
12) Transfer to 10mL volumetric flask. Dilute to 10mL.
13) Take 0.1-0.2 mL, dilute to 10mL for ICP-AES Fe yield.
14) Transfer balance of sample to 20mL glass LSC vial.
15) Add 6 drops H_3PO4. Evap. on hotplate to ~0.5mL.
16) Add 1mL H_2O. Cool. Add 15mL LSC cocktail. Mix.


Fe Recovery from $Fe(OH)_3$ Precipitation


Precipitation Yield vs pH

Method Performance

Method	Replicate	%Rec 2mg Fe tracer	Fe-55 raw %rec	Fe-55 Tracer corrected	Bias	Impurity*
TEVA	1	95.8	89.2	93.1	-6.9	<0.5%
	2	94.4	89.7	95.0	5.0	
	3	97.6	87.2	89.4	10.6	
	4	95.3	88.2	92.6	7.4	
	5	83.9	79.8	95.1	4.9	
	6	89.1	89.6	100.5	-0.5	
	7	80.6	86.4	107.2	-7.2	
	AVG	91.0	87.2	96.1		
	SD	6.6	3.5	5.9		

References

1) ASTM Method D4922. “Standard Test Method for Determination of Radioactive Iron in Water.”

AN-1612-10

Measurement of ^{55}Fe in Water (TRU Separation)

Summary of Method ^{55}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. ^{55}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.

Reagents

TRU Resin Cartridges (Eichrom TE-R50-S)
Deionized Water
Sodium Hydroxide
HCl
HNO_3
H_3PO_4
LSC Cocktail
Fe, Zn, Mn, Cs, Nb, Zr, Co carriers (10mg/mL)
Phenolphthalein pH indicator
^{55}Fe standard
Nitromethane or other LSC quench agent

Equipment

Vacuum Box (Eichrom AR-12-BOX or AR-24-BOX)
Vacuum Box Inner Liner (Eichrom AR-12-LINER or AR-24-LINER)
Yellow Outer Tips (Eichrom AR-1000-OT)
Inner Support Tube (Eichrom AR-1000-TUBE-PE)
Cartridge Reservoirs (Eichrom AR-200-RV20)
Centrifuge Tubes - 50mL and 250mL
20mL glass liquid scintillation tubes
Liquid scintillation counter
Calibrated pipets and disposable tips
Appropriately Sized Glass Beakers
ICP-AES system for Fe chemical yield measurement
Analytical balance
Vacuum Pump
Centrifuge
Hotplate

Sample Preparation

Up to 1L of water sample in glass beaker.

Add 2mg Fe. 1mg Zn, Mn, Cs, Co. 0.1mg Nb, Zr .

Evaporate to dryness or proceed to $Fe(OH)_3$ ppt steps below.

Ferric Hydroxide Precipitation

Add pH indicator

Heat sample to 80°C

Adjust to pH 8-9 with NaOH.

Mix sample and allow to cool to room temperature.

Allow ppt to settle and decant supernate to <200mL.

Transfer to 250mL centrifuge tube. Rinse beaker with water to ensure complete transfer of ppt.

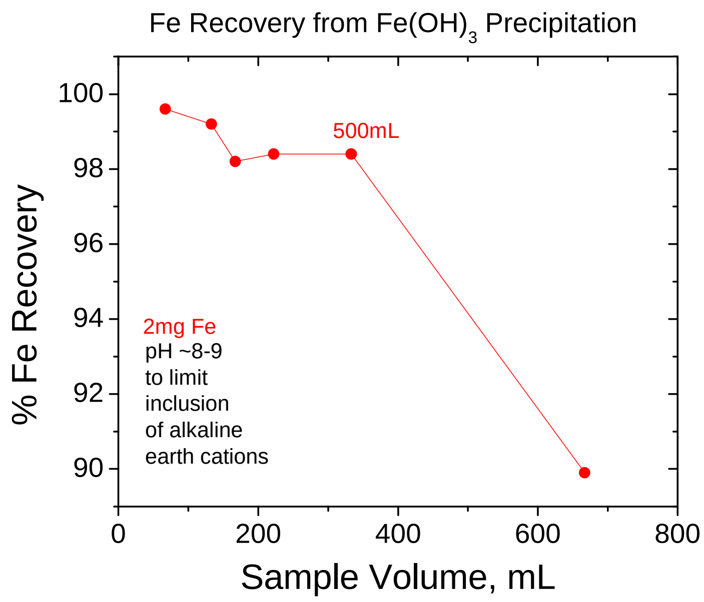
Centrifuge 10min. Decant supernate to waste.

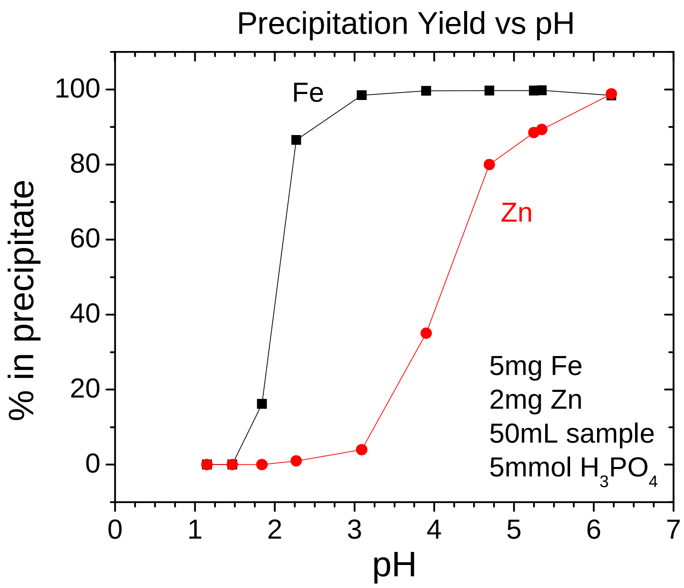
Rinse ppt w/ 50mL Water. Centrifuge. Decant supernate.

Dissolve ppt/residue in 10mL 8M HNO_3.

Iron Separation

1) Set up vacuum box with TRU cartridges.
2) Precondition with 5mL 8M HNO_3.
3) Load samples on TRU Resin.
4) Rinse tube with 5mL 8M HNO_3. Add to TRU.
5) Rinse TRU with 10mL 8M HNO_3.
6) Strip Fe from TRU with 15mL 2M HNO_3.
7) Add 5mL 1M H_3PO_4. Mix.
8) Adjust to pH 2.8-3.2 with NaOH. Mix.
9) Centrifuge. Decant Supernate.
10) Wash ppt with 50mL H_2O. Centrifuge. Decant Supernate.
11) Dissolve ppt with minimal 6M HCl.
12) Transfer to 10mL volumetric flask. Dilute to 10mL.
13) Take 0.1-0.2 mL, dilute to 10mL for ICP-AES Fe yield.
14) Transfer balance of sample to 20mL glass LSC vial.
15) Add 6 drops H_3PO4. Evap. on hotplate to ~0.5mL.
16) Add 1mL H_2O. Cool. Add 15mL LSC cocktail. Mix.


Fe Recovery from $Fe(OH)_3$ Precipitation


Precipitation Yield vs pH

Method Performance

Method	Replicate	%Rec 2mg Fe tracer	Fe-55 raw %rec	Fe-55 Tracer corrected	Bias	Impurity*
TRU	1	90.6	93.1	102.8	2.8	<0.5%
	2	90.0	92.3	102.5	2.5	
	3	94.8	92.4	97.5	-2.5	
	4	89.5	94.0	105.0	5.0	
	5	95.8	94.3	98.5	-1.5	
	6	95.8	92.8	96.9	-3.1	
	AVG	92.8	93.2	100.5		
	SD	3.0	0.8	3.3		

References

1) Eichrom Method FEW01VBS. "Iron-55 in water," http://www.eichrom.com/eichrom/radiochem/methods/eichrom/

eichrom

AN-1613-10

^{68}Ga Generator

Summary of Method ^{68}Ga is a positron emitting radionuclide which has garnered interest for use in positron emission tomography (PET). ^{68}Ga ($t_{1/2}$ = 68 min) can be readily isolated from its parent ^{68}Ge ($t_{1/2}$ = 271 days), which is produced by cyclotron irradiation of gallium or zinc target material. Classic ^{68}Ga generators consist of ^{68}Ge adsorbed onto an inorganic exchanger, such as Al_2O_3, SnO_2 or TiO_2. The ^{68}Ga is then periodically eluted with 0.1-1.0M HCl or dilute EDTA. These generators are simple and robust, yielding 60-80% of ^{68}Ga with minimal ^{68}Ge breakthrough over many elutions. However, the classic generator can be limited by the relatively large volume of solution needed to elute the ^{68}Ga and by metal ion impurities arising from the inorganic substrate. An alternative generator system has been developed, in which the ^{68}Ge source material is stored in dilute HCl. ^{68}Ga is then selectively retained on cation exchange resin, while the ^{68}Ge is remains in solution for future use. A small amount of rinsing of the cation exchange column, completes the ^{68}Ge source recovery. ^{68}Ga is then stripped from the cation exchange resin using a small volume of 4M HCl and adsorbed on a second cartridge of UTEVA resin. A small volume of rinse with 4M HCl provides additional decontamination from ^{68}Ge, and ^{68}Ga is recovered in a small volume of dilute HCl (0.05-0.5M HCl). The chemistry is robust and scalable. The separation has been demonstrated using 0.5 - 2mL columns/cartridges. Typical decay corrected yields of ^{68}Ga are 95 ± 1% in 2-5mL of 0.1M HCl, with $<10^{-7}\%$ ^{68}Ge impurity. Stable metal ion impurities are typically in the low parts per billion range. Operation of the generator has also been demonstrated with the Northstar Medical Radioistope automated generator system.

Reagents

UTEVA Resin Cartridges (Eichrom UT-R50-S)

Cation Exchange 2mL Cartridges (Eichrom C8-R50-H)

^{68}Ge Source* Deionized Water

HCl

*Germanium chloride is relatively volatile and can be spread through the air. Care should be taken to minimize contamination of personnel and work spaces. Use of sealed systems for steps during separation is recommended.

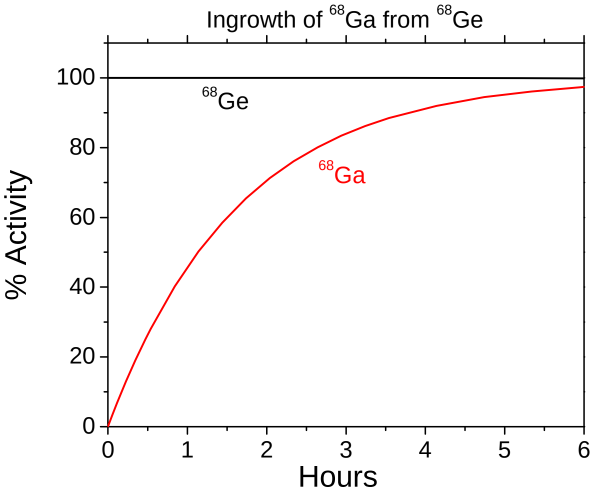
Equipment

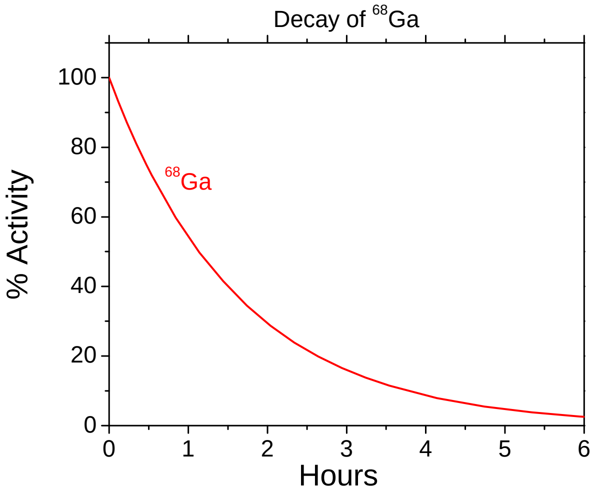
Glass vials for storage of ^{68}Ge source.

Glass or plastic vials/bottles for collection ^{68}Ga product and waste.

5, 10 or 20mL plastic luer lock syringes

Gamma spectroscopy system for measurement of ^{68}Ga. (The electron capture of ^{68}Ge can be measured by liquid scintillation or ^{68}Ge can be determined after decay/ingrowth of ^{68}Ga using the 511keV emission following positron annihilation.)





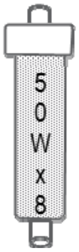
^{68}Ga/^{68}Ge Separation*

(1) Clean 2mL 50Wx8 cartridge with:

-20mL DI water

-20mL 4M HCl

-20mL 0.5M HCl



(2) Load ^{68}Ge/^{68}Ga source in 10-20mL 0.5M HCl. ^{68}Ga is retained.

(3) Rinse cartridge with 1mL 0.5M HCl. Collect in ^{68}Ge source vessel. Push remaining fluid to source vessel with air.

(4) Seal ^{68}Ge source vessel and set aside for future use.

(5) Rinse cartridge with 20mL 0.5M HCl. Dispose as waste.

(6) Precondition 2mL UTEVA cartridge with 5mL 4M HCl.

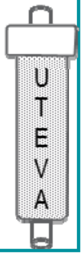
(7) Place 2mL UTEVA cartridge below 2mL 50Wx8 cartridge.

(8) Strip ^{68}Ga from 50Wx8 onto UTEVA with 20mL 4M HCl. Dispose of eluate as waste.

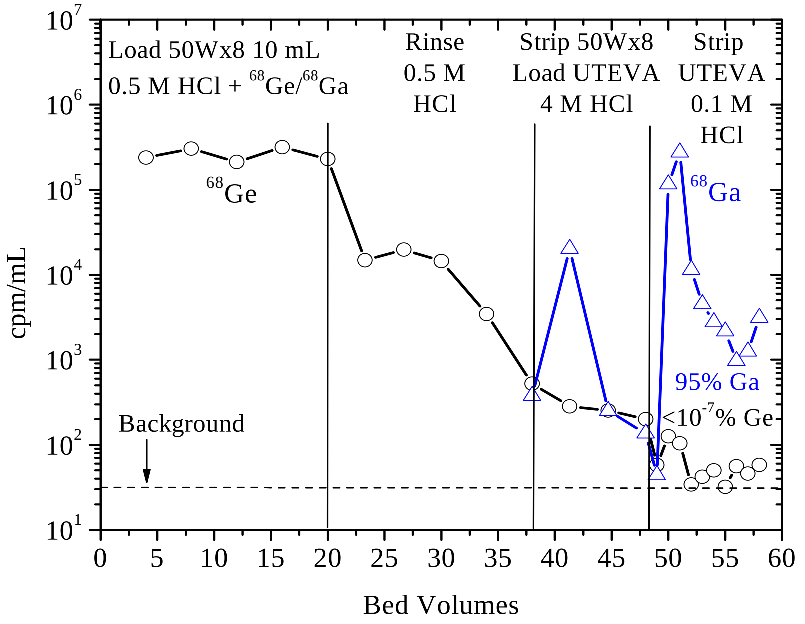
(9) Remove 50Wx8 cartridge.

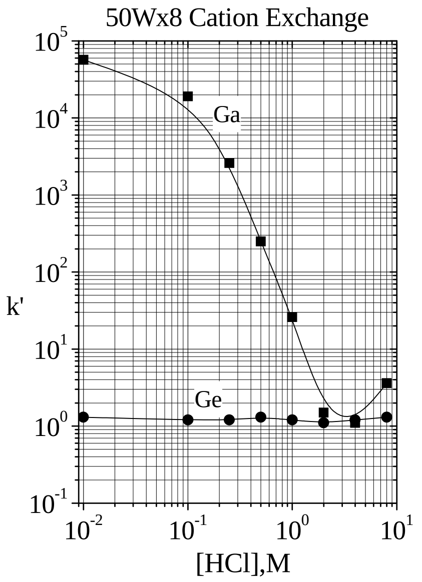
(10) Rinse UTEVA cartridge with 20mL 4M HCl.

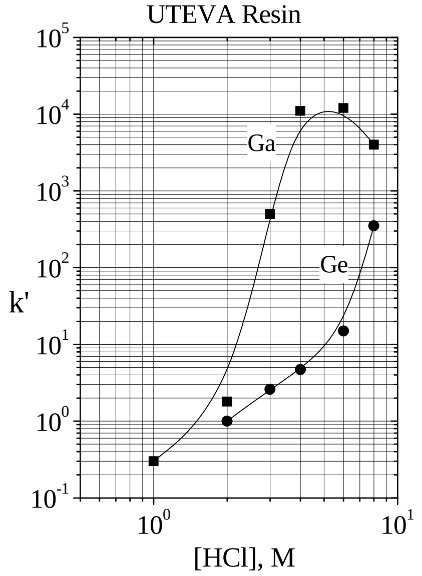
(11) Strip ^{68}Ga with 5-10mL 0.1M HCl.



*The separation may also be performed using 0.5mL or 1mL columns/ cartridges and proportionally scaled eluate volumes to improve method speed and reduce losses from ^{68}Ga decay during separation.







References

1) McAlister and Horwitz, “Automated Two Column Generator Systems for Medical Radionuclides,” *Applied Radiation and Isotopes*, 67:1985-1991 (2009).

AN-1614-11

^{225}Ac/^{225}Ra Generator

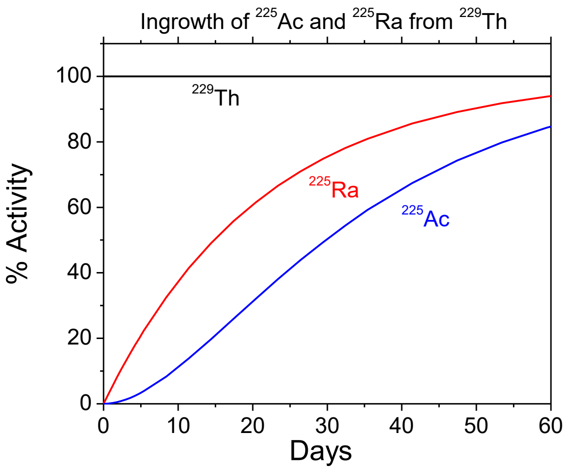
Summary of Method A method for the preparation of ^{225}Ac ($t_{1/2}$ = 10 days) and ^{225}Ra ($t_{1/2}$ = 14.8 days) from ^{229}Th ($t_{1/2}$ = 7340 years) source material is presented. The method employs 2mL cartridges of UTEVA and DGA resins to obtain high purity ^{225}Ac in small volumes of eluate while preserving valuable ^{229}Th source material. The method is meant for ^{225}Ac tracer production from ^{229}Th containing 5-10mg or less of total Th. For separations from larger masses of Th see the Eichrom website bibliography for other options (Recent Advances in the Recovery and Purification of Actinium Isotopes, Horwitz and McAlister, National Meeting of the American Chemical Society, 2009). The source material, containing ^{229}Th, ^{225}Ac, ^{225}Ra and other daughter nuclides in 4M HNO_3, is loaded onto stacked 2mL cartridges of UTEVA and DGA resins. ^{229}Th is retained on UTEVA, while ^{225}Ac is retained on DGA and ^{225}Ra passes through both cartridges. ^{225}Ra can be saved for use as a radiotracer or as an additional source of ^{225}Ac, following a suitable ingrowth period. ^{225}Ac is recovered from DGA with a small volume of 2.0M HCl. The ^{229}Th source is recovered from UTEVA with a small volume of 0.5M HCl. Following a suitable ingrowth period, the ^{229}Th can be acidified to 4M HNO_3 and used to produce additional ^{225}Ac and ^{225}Ra. The ^{229}Th is preserved nearly indefinitely and continuously purified from chemical and radiologic impurities run to run.

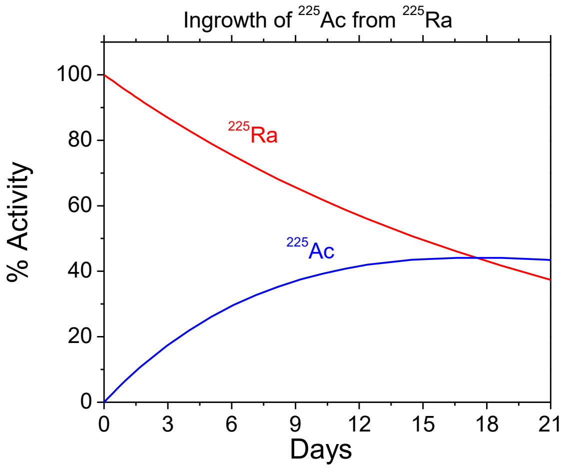
Reagents

UTEVA Resin Cartridges (Eichrom UT-R50-S)
DGA Resin Cartridges (Eichrom DN-R50-S)
^{229}Th Source
Deionized Water
HCl
HNO_3

Equipment

Glass vials for storage of ^{229}Th source.
Glass or plastic vials/bottles for collection of ^{225}Ac. ^{225}Ra and waste.
5, 10 or 20mL plastic luer lock syringes
Gamma spectrometry system and/or alpha spectrometry for measurement of ^{225}Ac(^{221}Fr), ^{225}Ra and ^{229}Th.





^{225}Ac/^{225}Ra/^{229}Th Separation

(1) Precondition stacked 2mL cartridges of UTEVA and DGA with 10mL 4M HNO_3.

(2) Acidify ^{229}Th eluate from previous separation with 5mL HNO_3. (If new ^{229}Th source, dilute to 20mL with 4M HNO_3.)

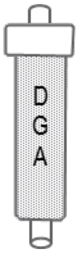
(3) Load ^{229}Th and daughters in 20mL 4M HNO_3. Collect and save eluate containing ^{225}Ra.*

(4) Rinse UTEVA/DGA with 10mL 4M HNO_3. Collect ^{225}Ra.*

(5) Separate UTEVA and DGA cartridges.

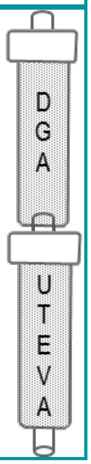
(6) Rinse DGA with 10mL 8M HCl.

(7) Strip ^{225}Ac with 10mL 2M HCl. (Traces of ^{229}Th that may have broken through UTEVA will be retained on DGA.)

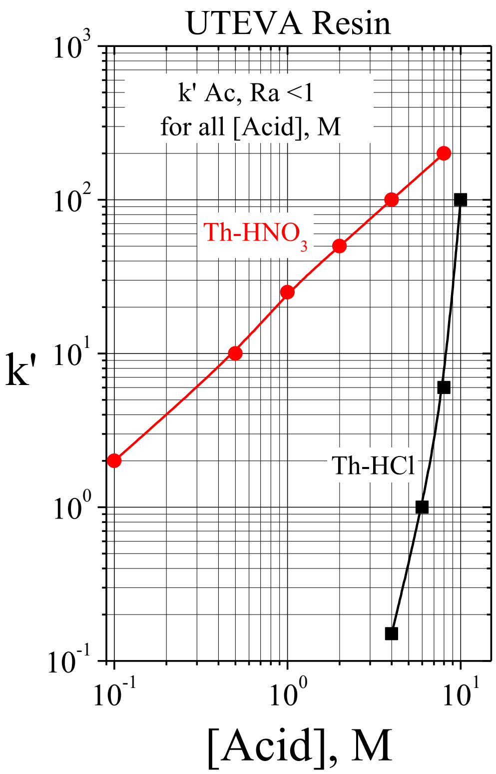


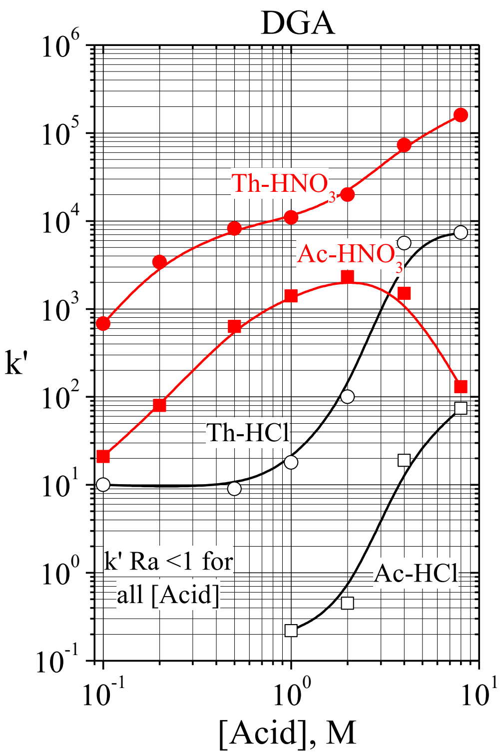
(8) Place DGA (from which ^{225}Ac has been stripped) above the UTEVA cartridge.

(9) Strip ^{229}Th from DGA-UTEVA cartridges with 15mL 0.5M HCl. Save ^{229}Th for future use.



*^{225}Ra can used directly as a tracer or as a source of additional ^{225}Ra.





References

1) McAlister and Horwitz, “Chromatographic Generator Systems for the actinides and natural decay series elements,” *Radiochimica Acta*, 99:1-9 (2011).

eichrom

AN-1615-11

^{90}Y Generator

Summary of Method A method for the preparation of ^{90}Y ($t_{1/2}$ = 64.1 hours) from ^{90}Sr ($t_{1/2}$ = 28.6 years) source material is presented. The method employs 2mL cartridges of Sr and DGA resins to obtain high purity ^{90}Y in small volumes of eluate while preserving valuable ^{90}Sr source material. The source material, containing ^{90}Sr/^{90}Y, in 4M HNO_3, is loaded onto stacked 2mL cartridges of Sr and DGA resins. ^{90}Sr is retained on Sr Resin, while ^{90}Y is retained on DGA. The ^{90}Sr source is recovered from Sr Resin with a small volume of 0.1M HCl. Following a suitable ingrowth period, the ^{90}Sr can be acidified to 4M HNO_3 and used to produce additional ^{90}Y. The ^{90}Sr is preserved nearly indefinitely and continuously purified from chemical and radiologic impurities run to run. ^{90}Y is recovered from DGA resin with 0.1M HCl. For applications where ^{90}Y must be recovered in minimal volumes, DGA, Branched may be used in place of DGA, Normal.

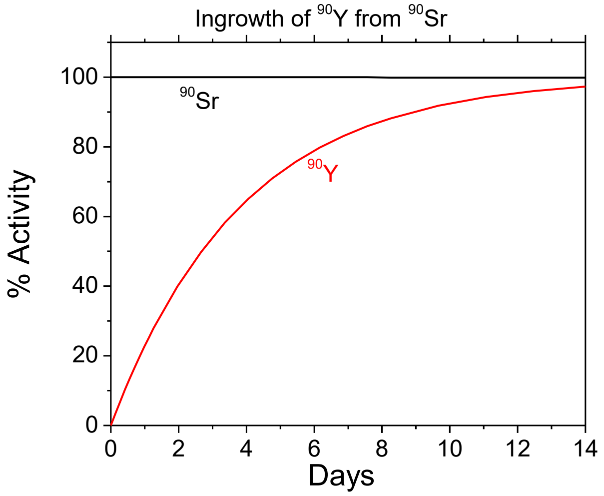
Reagents

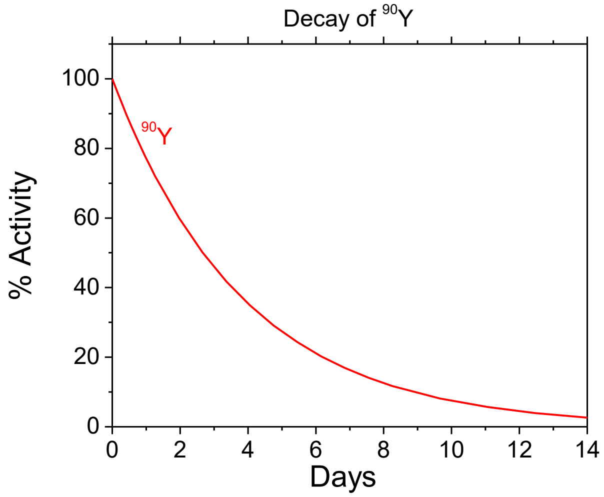
Sr Resin Cartridges (Eichrom SR-R50-S)
DGA, Normal Resin Cartridges (Eichrom DN-R50-S) or
DGA, Branched Resin Cartridges (Eichrom DB-R50-S)
Liquid Scintillation Cocktail
^{90}Sr Source
Deionized Water
HCl
HNO_3

Equipment

Glass vials for storage of ^{90}Sr source.
Glass or plastic vials/bottles for collection of ^{90}Y and waste.
5, 10 or 20mL plastic luer lock syringes
Liquid Scintillation system for measurement of ^{90}Sr and ^{90}Y.*

*^{90}Y may also be measured by Cerenkov counting without the addition of scintillation cocktail.



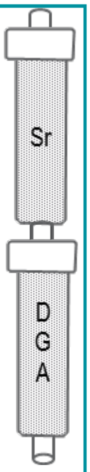


^{90}Sr/^{90} Separation

(1) Precondition stacked 2mL cartridges of Sr and DGA Resins with 10mL 4M HNO_3.

(2) Acidify ^{90}Sr eluate from previous separation with 5mL conc. HNO_3. (If new ^{90}Sr source, dilute to 20mL with 4M HNO_3.)*

(3) Load ^{90}Sr and ^{90}Y in 20mL 4M HNO_3.



(4) Rinse Sr/DGA with 5mL 4M HNO_3.

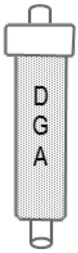
(5) Separate Sr and DGA cartridges.

(6) Place DGA cartridge above Sr resin cartridge.

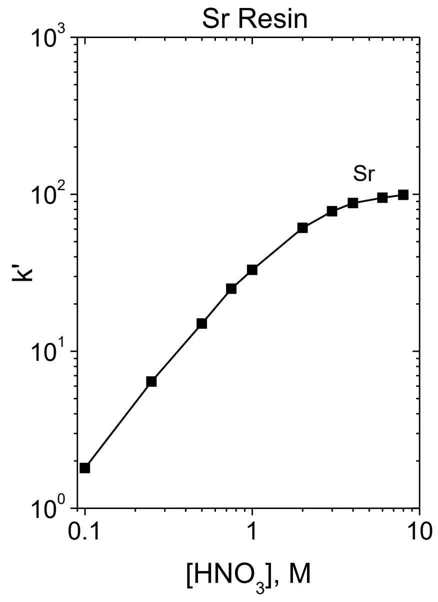
(7) Strip ^{90}Sr from DGA/Sr resin cartridges with 15mL 2M HCl. Save ^{90}Sr for future use.

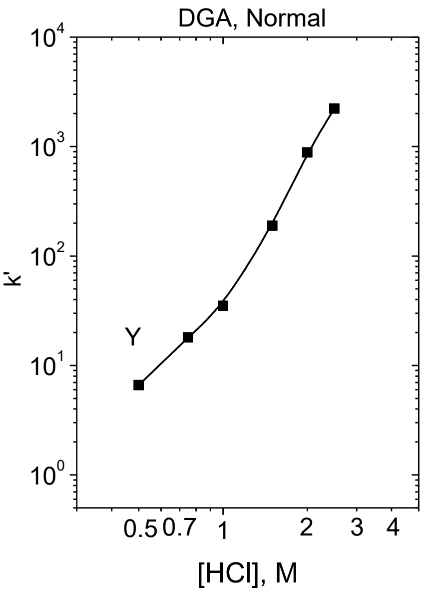
(8) Remove Sr resin cartridge.

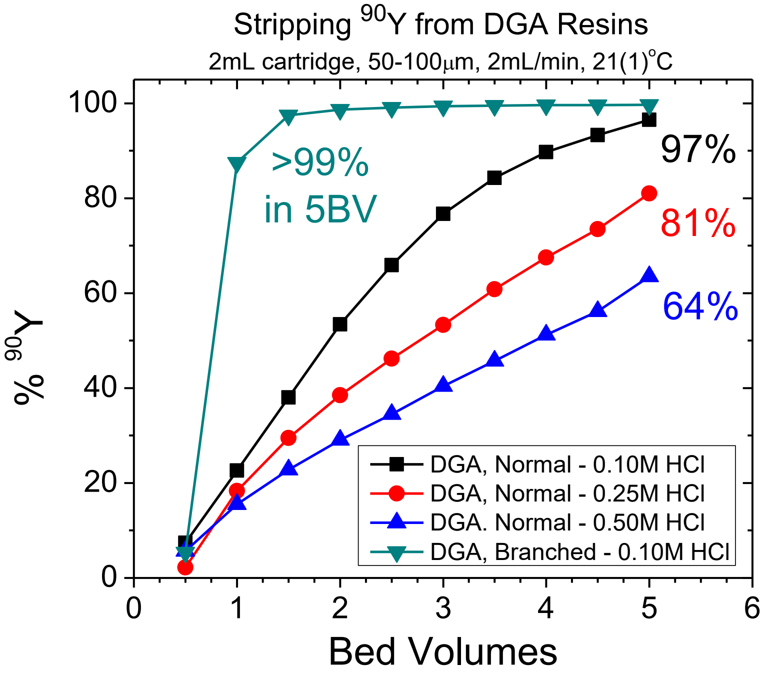
(9) Strip ^{90}Y with 10mL 0.1M HCl.



*Adding 1mg of stable Sr to the ^{90}Sr source can help improve ^{90}Sr recovery from Sr Resin (do only once, not each time).







References

1) McAlister and Horwitz, “Chromatographic Generator Systems for the actinides and natural decay series elements,” *Radiochimica Acta*, 99:1-9 (2011).

^{210}Po/^{210}Bi Generator

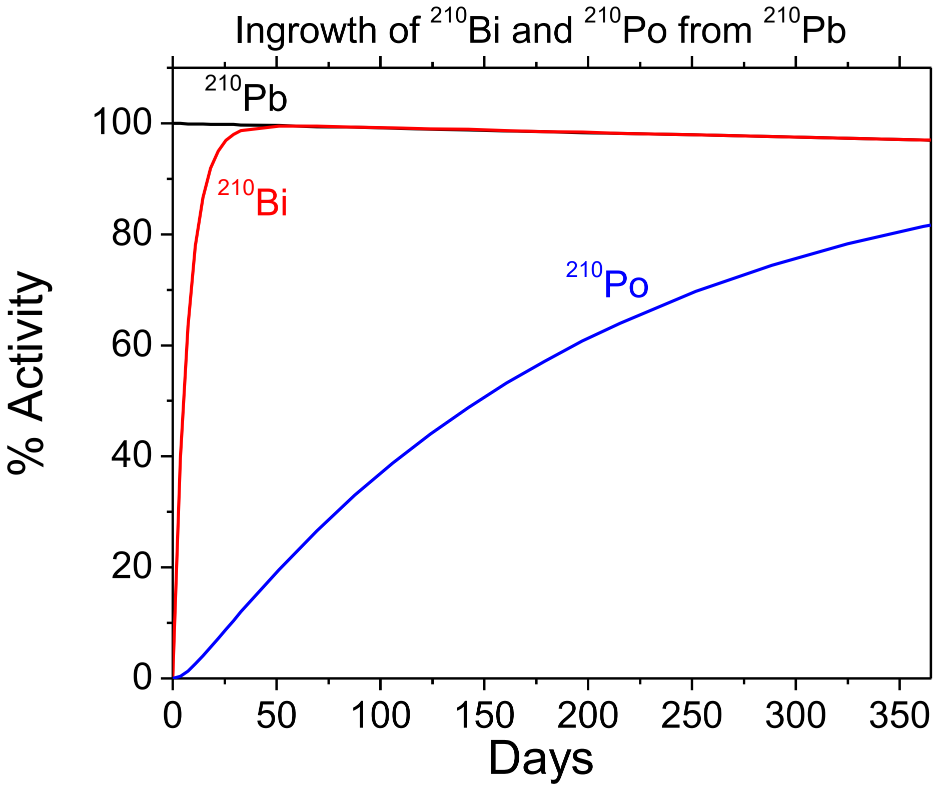
Summary of Method A method for the preparation of ^{210}Po ($t_{1/2}$ = 138.4 days) and ^{210}Bi ($t_{1/2}$ = 5.013 days) from ^{210}Pb ($t_{1/2}$ = 22.26 years) source material is presented. The method employs 2mL cartridges of UTEVA and Sr resins to obtain high purity ^{210}Po and ^{210}Bi in small volumes of eluate while preserving valuable ^{210}Pb source material. The source material, containing ^{210}Pb/^{210}Bi/^{210}Bi in 2.67M HCl, is loaded onto stacked 2mL cartridges of UTEVA and Sr resins. ^{210}Po is retained on UTEVA Resin, while ^{210}Pb is retained on Sr Resin and ^{210}Bi is not retained. The ^{210}Pb source is recovered from Sr Resin with a small volume of 8M HCl. Following a suitable ingrowth period, the ^{210}Pb can be diluted to 2.67M HCl and used to produce additional ^{210}Po and ^{210}Bi. The ^{210}Pb is preserved nearly indefinitely and continuously purified from chemical and radiologic impurities run to run. ^{210}Po is recovered from UTEVA resin with 6M $HN0_3$.

Reagents

Sr Resin Cartridges (Eichrom SR-R50-S)
UTEVA Cartridges (Eichrom UT-R50-S)
Liquid Scintillation Cocktail
^{210}Pb Source
Deionized Water
HCl
HNO_3

Equipment

Glass vials for storage of ^{210}Pb source.
Glass or plastic vials/bottles for collection of ^{210}Po, ^{210}Bi and waste.
10, 20 or 30mL plastic luer lock syringes
Liquid Scintillation System for measurement of ^{210}Bi and ^{210}Po .
Gamma Spectrometry System for measurement of ^{210}Pb.



^{210}Pb/^{210}Bi/^{210}Po Separation

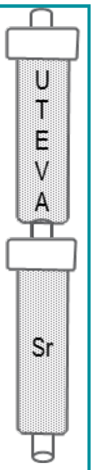
(1) Precondition stacked 2mL cartridges of UTEVA and Sr Resins with 10mL 2M HCl.

(2) Dilute ^{210}Pb eluate from previous separation with 20mL DI H_2O. (If new ^{210}Pb source, dilute to 20mL with 2M HCl.)*

(3) Load ^{210}Pb/^{210}Bi/^{210}Po in 30mL 2.67M HCl. (20mL 2M HCl). Collect ^{210}Bi.

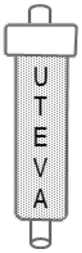
(4) Rinse UTEVA/Sr with 10mL 2M HCl. Collect ^{210}Bi.

(5) Elute 10mL 8M HCl through UTEVA/Sr, collecting ^{210}Pb Source material. Save ^{210}Pb for future use.

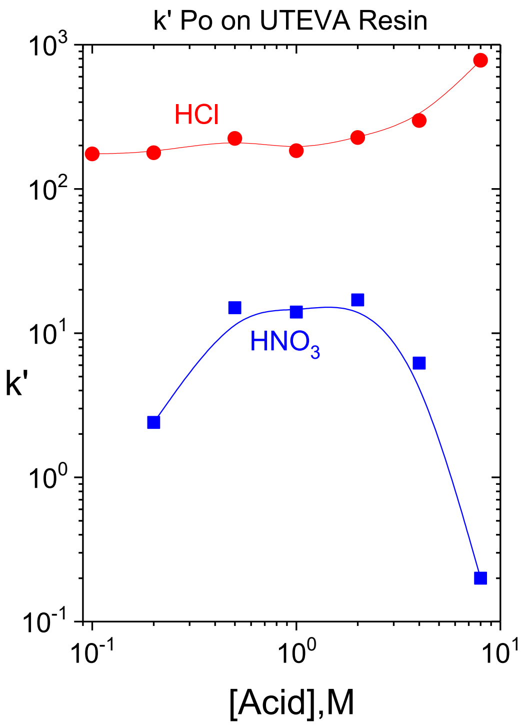


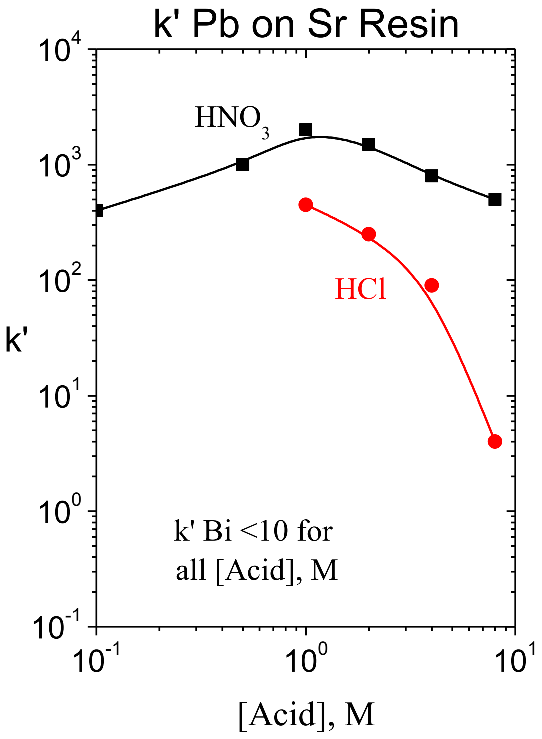
(6) Separate UTEVA/Sr.

(7) Strip Po from UTEVA with 10mL 6M HNO_3.



*Adding 1mg of stable Pb to the ^{210}Pb source can help improve ^{210}Pb recovery from Sr Resin (do only once, not each time).


k' Po on UTEVA Resin


k' Pb on Sr Resin

References

1) McAlister and Horwitz, “Chromatographic Generator Systems for the actinides and natural decay series elements,” *Radiochimica Acta*, 99:1-9 (2011).

eichrom®

AN-1616b-11

^{210}Po Generator

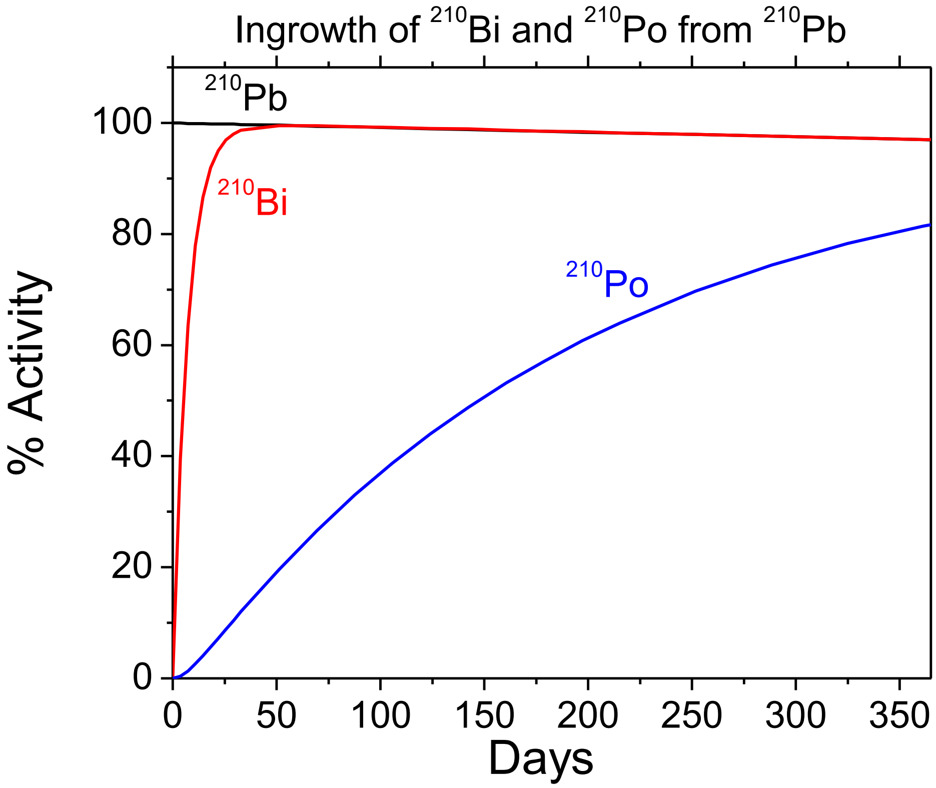
Summary of Method A method for the preparation of ^{210}Po ($t_{1/2}$ = 138.4 days) from ^{210}Pb ($t_{1/2}$ = 22.26 years) source material is presented. The method employs 2mL cartridges of DGA and Sr resins to obtain high purity ^{210}Po in small volumes of eluate while preserving valuable ^{210}Pb source material. The source material, containing ^{210}Pb/^{210}Bi/^{210}Bi in 2.67M HCl, is loaded onto stacked 2mL cartridges of DGA and Sr resins. ^{210}Po and ^{210}Bi are retained on DGA Resin, while ^{210}Pb is retained on Sr Resin. The ^{210}Pb source is recovered from Sr Resin with a small volume of 8M HCl. Following a suitable ingrowth period, the ^{210}Pb can be diluted to 2.67M HCl and used to produce additional ^{210}Po. The ^{210}Pb is preserved nearly indefinitely and continuously purified from chemical and radiologic impurities run to run. ^{210}Po is recovered from DGA resin with 0.05M $HN0_{3,}$ but should be acidified to 1M HNO_3 to prevent loss of Po to glass vials. The ^{210}Bi will remain on the DGA resin during the Po elution, and can be recoverd with 10mL of 0.05M ammonium bioxalate. The DGA/Sr Resin chemistry is an improvement over the UTEVA/Sr Resin chemistry previously described (AN-1616a), which required 6M HNO_3 to recover the ^{210}Po.

Reagents

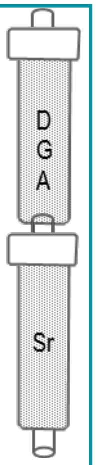
Sr Resin Cartridges (Eichrom SR-R50-S)
DGA, Normal Cartridges (Eichrom DN-R50-S)
Liquid Scintillation Cocktail
^{210}Pb Source
Deionized Water
HCl
HNO_3

Equipment

Glass vials for storage of ^{210}Pb source.
Glass or plastic vials/bottles for collection of ^{210}Po, ^{210}Bi and waste.
10, 20 or 30mL plastic luer lock syringes
Liquid Scintillation System for measurement of ^{210}Bi and ^{210}Po .
Gamma Spectrometry System for measurement of ^{210}Pb.



^{210}Pb/^{210}Bi/^{210}Po Separation



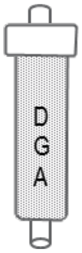
(1) Precondition stacked 2mL cartridges of DGA and Sr Resins with 10mL 2M HCl.

(2) Dilute ^{210}Pb eluate from previous separation with 20mL DI H_2O. (If new ^{210}Pb source, dilute to 20mL with 2M HCl.)*

(3) Load ^{210}Pb/^{210}Bi/^{210}Po in 30mL 2.67M HCl. (20mL 2M HCl).

(4) Rinse DGA/Sr with 10mL 2M HCl.

(5) Elute 10mL 8M HCl through DGA/Sr, collecting ^{210}Pb Source material. Save ^{210}Pb for future use.



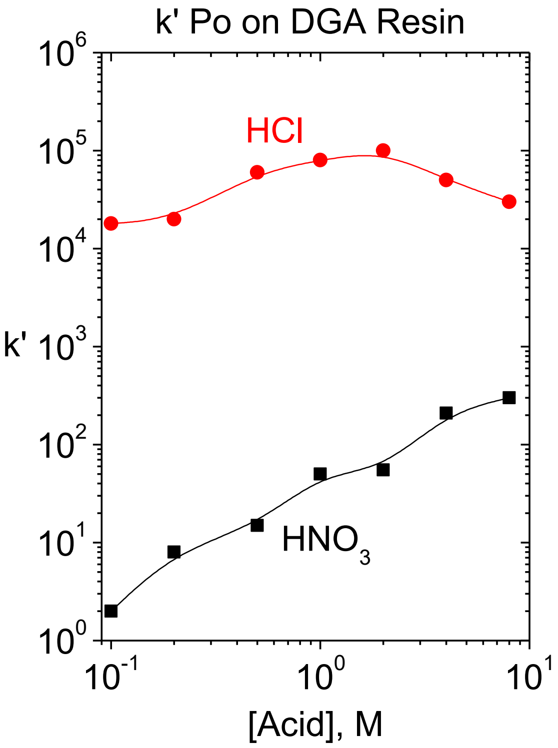
(6) Separate DGA/Sr.

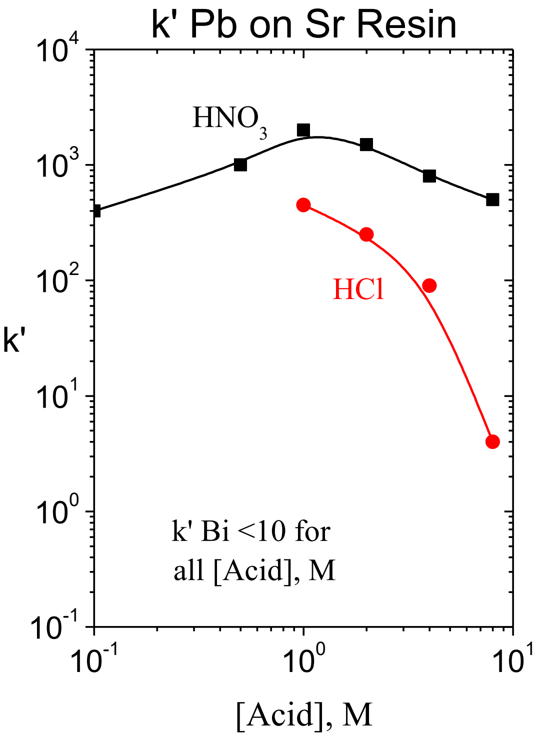
(7) Rinse DGA with 10mL 8M HNO_3. Discard as waste.

(8) Strip ^{210}Po from DGA with 15mL 0.05M HNO_3.*

*Acidification of the ^{210}Pb to 1M HNO_3 is recommended to prevent hydrolysis and loss of the ^{210}Po to the storage vial.

*Adding 1mg of stable Pb to the ^{210}Pb source can help improve ^{210}Pb recovery from Sr Resin (do only once, not each time).





References

1) McAlister and Horwitz, "Chromatographic Generator Systems for the actinides and natural decay series elements," *Radiochimica Acta*, 99:1-9 (2011).

AN-1617-10

$^{227}Th/^{223}Ra$ Generator

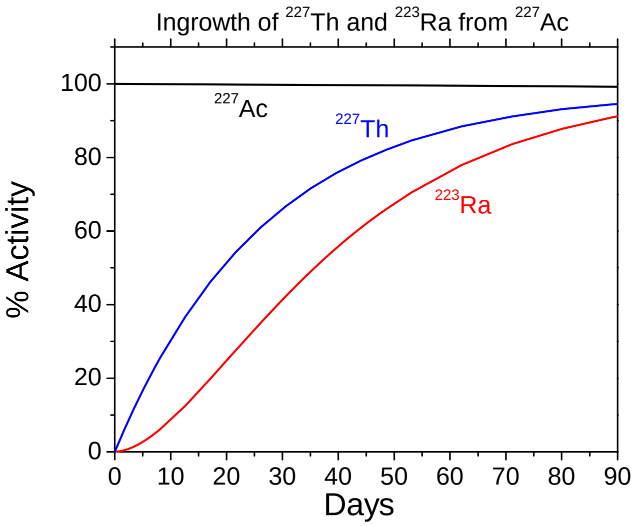
Summary of Method A method for the preparation of ^{227}Th ($t_{1/2}$ = 18.72 days) and ^{223}Ra ($t_{1/2}$ = 11.43 days) from ^{227}Ac ($t_{1/2}$ = 21.77 years) source material is presented. The method employs 2mL cartridges of UTEVA and DGA resins to obtain high purity ^{227}Th and ^{223}Ra in small volumes of eluate while preserving valuable ^{227}Ac source material. The source material, containing $^{227}Ac/^{227}Th/^{223}Ra$ in 4M HNO_3, is loaded onto stacked 2mL cartridges of UTEVA and DGA resins. ^{227}Th is retained on UTEVA Resin, while ^{227}Ac is retained on DGA Resin and ^{223}Ra is not retained. The ^{227}Ac source is recovered from DGA Resin with a small volume of 0.1M HCl. Following a suitable ingrowth period, the ^{227}Ac can be acidified to 4M HNO_3 and used to produce additional ^{227}Th and ^{223}Ra. The ^{227}Ac is preserved nearly indefinitely and continuously purified from chemical and radiologic impurities run to run. ^{227}Th is recovered from UTEVA resin with 0.5M HCl.

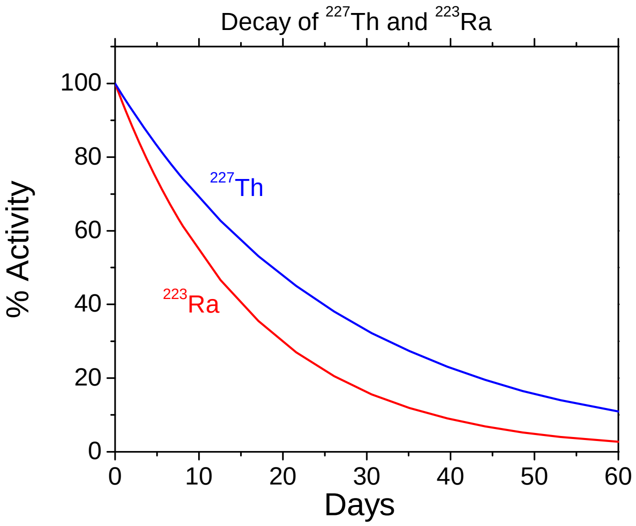
Reagents

UTEVA Cartridges (Eichrom UT-R50-S)
DGA, Normal Cartridges (Eichrom DN-R50-S)
^{227}Ac Source
Deionized Water
HCl
HNO_3

Equipment

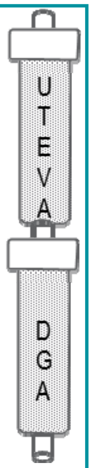
Glass vials for storage of ^{227}Ac source.
Glass or plastic vials/bottles for collection of ^{223}Ra, ^{227}Th and waste.
10, 20 or 30mL plastic luer lock syringes
Gamma Spectrometry System for measurement of ^{227}Th and ^{223}Ra.





^{223}Ra/^{227}Th/^{227}Ac Separation

(1) Precondition stacked 2mL cartridges of UTEVA and DGA Resins with 10mL 4M HNO_3.



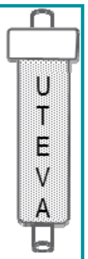
(2) Acidify ^{227}Ac eluate from previous separation with 5mL conc. HNO_3. (If new ^{227}Ac source, dilute to 20mL with 4M HNO_3.)

(3) Load ^{227}Ac/^{227}Th/^{223}Ra in 20mL 4M HNO_3. Collect ^{223}Ra.

(4) Rinse UTEVA/DGA with 10mL 3M HNO_3. Collect ^{223}Ra.

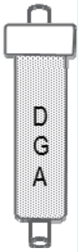
(5) Separate UTEVA/DGA.

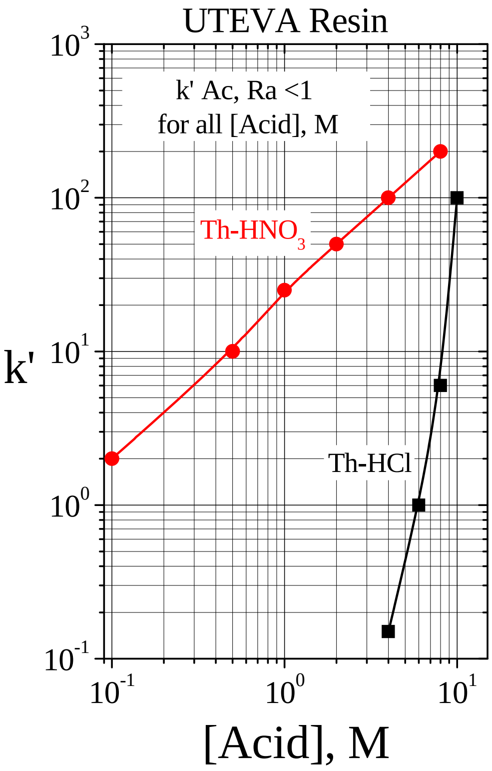
(6) Rinse UTEVA with 10mL 4M HNO_3. Discard as waste.

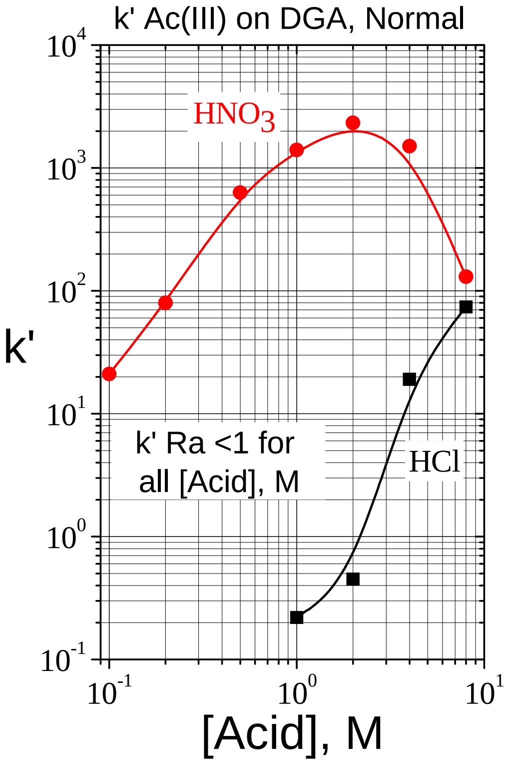


(7) Strip ^{227}Th from UTEVA with 10mL 0.5M HCl. ^{227}Th may be recovered in higher yield in less volume by stripping in opposite direction of load.

(8) Strip ^{227}Ac from DGA with 15mL 0.5M HCl. Save ^{227}Ac for future use.







References

1) McAlister and Horwitz, “Chromatographic Generator Systems for the actinides and natural decay series elements,” *Radiochimica Acta*, 99:1-9 (2011).

AN-1618-10

^{228}Th/^{231}Th Generator

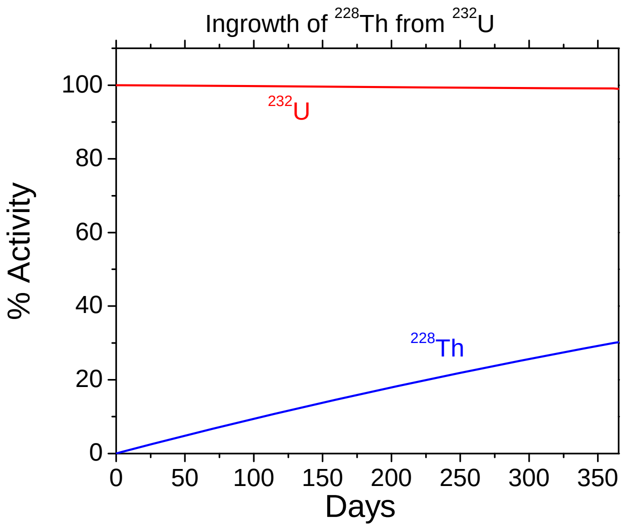
Summary of Method A method for the preparation of ^{228}Th ($t_{1/2}$ = 1.913 years) from ^{232}U ($t_{1/2}$ = 72 years) source material or ^{231}Th ($t_{1/2}$ = 25.52 hours) from ^{235}U ($t_{1/2}$ = 7.04E8 years) is presented. The method employs 2mL cartridges of TEVA and UTEVA resins to obtain high purity ^{228}Th or ^{231}Th in small volumes of eluate while preserving valuable ^{232}U or ^{235}U source material. The source material in 4M HNO_3, is loaded onto stacked 2mL cartridges of TEVA and UTEVA resins. ^{228}Th or ^{231}Th is retained on TEVA Resin, while ^{232}U or ^{235}U is retained on UTEVA Resin. The ^{232}U or ^{235}U source is recovered from UTEVA Resin with a small volume of 1M HCl. Following a suitable ingrowth period, the ^{232}U or ^{235}U can be acidified to 4M HNO_3 and used to produce additional ^{228}Th or ^{231}Th. The ^{232}U or ^{235}U is preserved nearly indefinitely and continuously purified from chemical and radiologic impurities run to run. ^{228}Th or ^{231}Th is recovered from TEVA

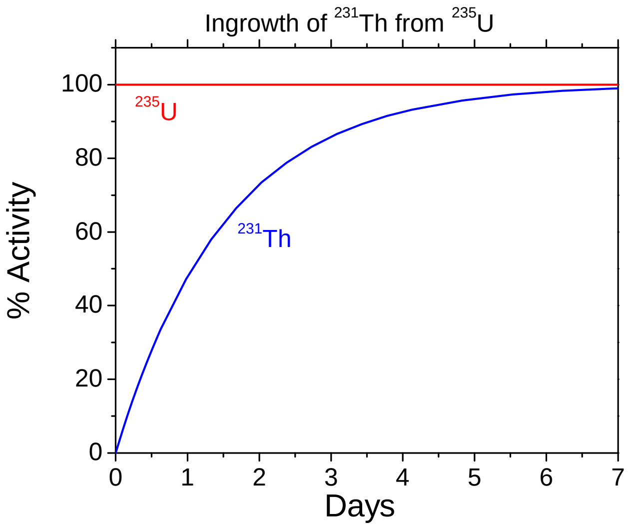
Reagents

UTEVA Cartridges (Eichrom UT-R50-S)
TEVA Cartridges (Eichrom TE-R50-S)
^{232}U or ^{235}U Source
Deionized Water
HCl
HNO_3

Equipment

Glass vials for storage of ^{232}U or ^{235}U source.
Glass or plastic vials/bottles for collection of ^{228}Th or ^{231}Th and waste.
10, 20 or 30mL plastic luer lock syringes
Gamma Spectrometry System and/or Alpha Spectrometry System for measurement of ^{228}Th and ^{232}U or ^{231}Th and ^{235}U.





^{228}Th/^{232}U or ^{231}Th/^{235}U Separation

(1) Precondition stacked 2mL cartridges of TEVA and UTEVA Resins with 10mL 4M HNO_3.

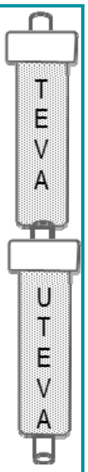
(2) Acidify ^{232}U or ^{235}U eluate from previous separation with 5mL conc. HNO_3. (If new ^{232}U or ^{235}U source, dilute to 20mL with 4M HNO_3.)

(3) Load ^{228}Th/^{232}U or ^{231}Th/^{235}U in 20mL 4M HNO_3

(4) Rinse TEVA/UTEVA with 10mL 4M HNO_3.

(5) Discard syringe used for load/first rinse. Replace with clean syringe.

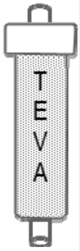
(6) Rinse TEVA/UTEVA with 10mL 4M HNO_3.



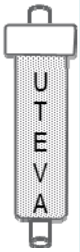
(7) Separate TEVA/UTEVA.

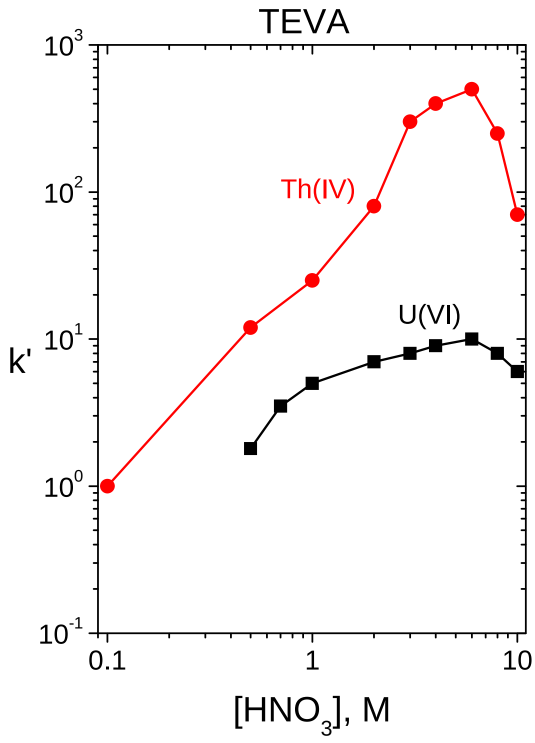
(8) Rinse TEVA with 20mL 4M HNO_3.

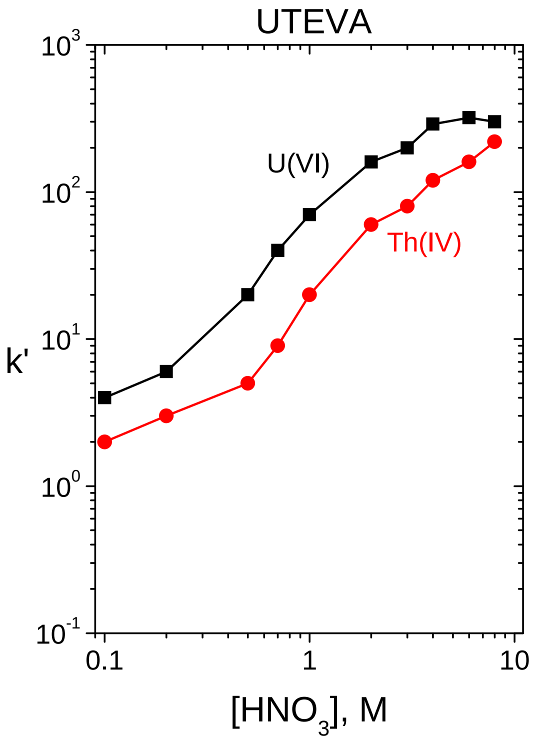
(9) Strip ^{228}Th or ^{231}Th with 10mL 0.5M HCl.



(10) Strip ^{232}U or ^{235}U from UTEVA with 15mL 1M HCl. Save ^{232}U or ^{235}U for future use.







References

1) McAlister and Horwitz, "Chromatographic Generator Systems for the actinides and natural decay series elements," *Radiochimica Acta*, 99:1-9 (2011).

AN-1619-10

^{239}Np Generator

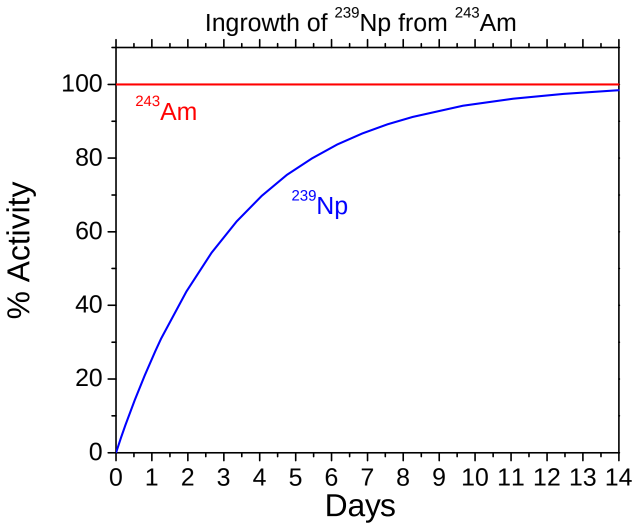
Summary of Method A method for the preparation of ^{239}Np ($t_{1/2}$ = 2.355 days) from ^{243}Am ($t_{1/2}$ = 7380 years) source material is presented. The method employs 2mL cartridges of UTEVA and DGA resins to obtain high purity ^{239}Np in small volumes of eluate, while preserving valuable ^{243}Am material. The source material is adjusted to 4M HNO_3, treated with iron, sulfamic acid and ascorbic acid to fix the Np(IV) oxidation state, and loaded onto stacked 2mL cartridges of UTEVA and DGA resins. ^{239}Np is retained on UTEVA Resin, while ^{243}Am is retained on DGA Resin. The ^{243}Am source is recovered from DGA Resin with a small volume of 0.5M HCl. Following a suitable ingrowth period, the ^{243}Am can be acidified to 4M HNO_3 and used to produce additional ^{239}Np. The ^{243}Am is preserved nearly indefinitely and continuously purified from chemical and radiologic impurities run to run. ^{239}Np is recovered from UTEVA resin with 0.5M HCl.

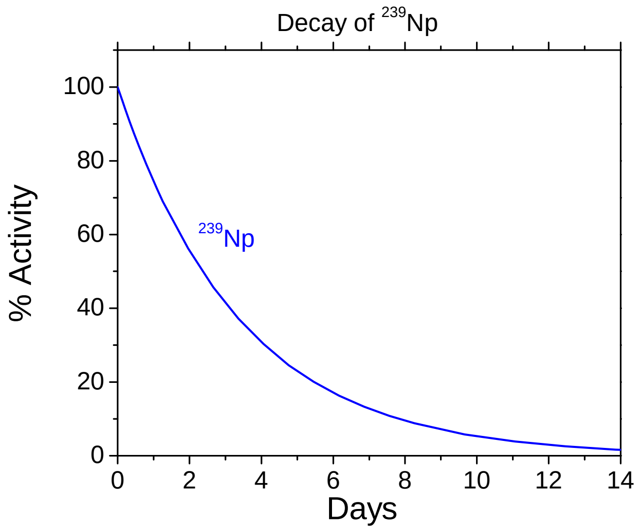
Reagents

UTEVA Cartridges (Eichrom UT-R50-S)
DGA Cartridges (Eichrom DN-R50-S)
^{243}Am Source
Deionized Water
HCl
HNO_3
Sulfamic Acid
Fe carrier (10mg/mL)
Ascorbic Acid

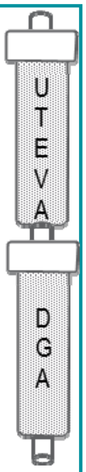
Equipment

Glass vials for storage of ^{243}Am.
Glass or plastic vials/bottles for collection of ^{239}Np and waste.
10, 20 or 30mL plastic luer lock syringes
Gamma Spectrometry System for measurement of ^{239}Np and ^{243}Am.





^{228}Th/^{232}U or ^{231}Th/^{235}U Separation



(1) Precondition stacked 2mL cartridges of UTEVA and DGA Resins with 10mL 4M HNO_3.

(2) Acidify ^{243}Am eluate from previous separation with 5mL conc. HNO_3. (If new ^{243}Am source, dilute to 20mL with 4M HNO_3.)

(3) Add 0.1mL of 10mg/mL Fe carrier and 1mL 1.5M sulfamic acid. Mix.

(4) Add 1mL 1M ascorbic acid. Mix. Wait 10-20 min.

(5) Load ^{239}Np/^{243}Am on UTEVA/DGA.

(6) Rinse UTEVA/DGA with 10mL 4M HNO_3.

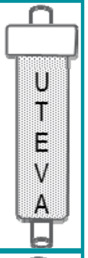
(7) Remove syringe used for load/ rinse and discard.

(8) Add clean syringe.

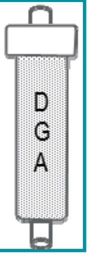
(9) Rinse UTEVA/DGA with 10mL 4M HNO_3.

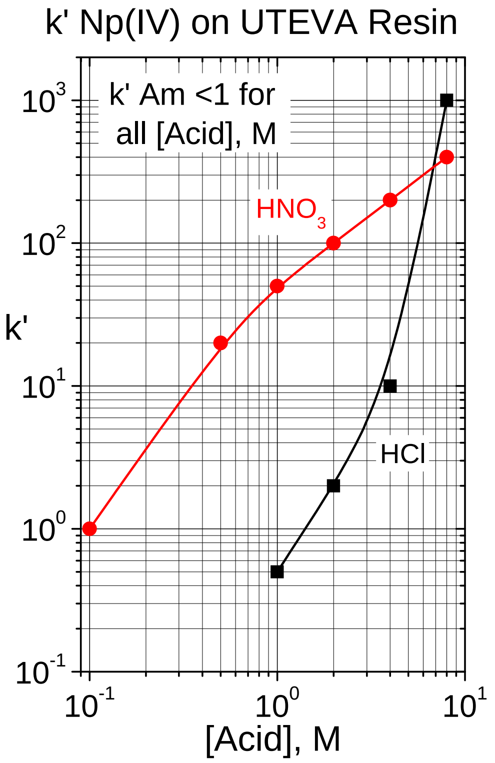
(10) Separate UTEVA/DGA.

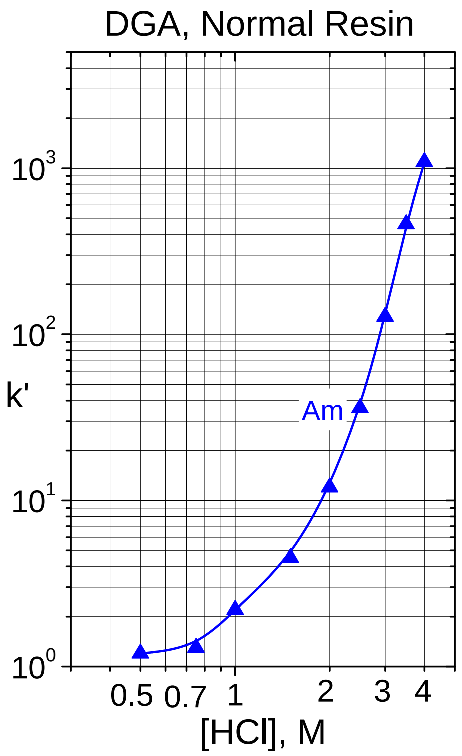
(11) Strip ^{239}Np from UTEVA with 10mL 0.5M HCl. Recovery of ^{239}Np can be improved by stripping in opposite direction of load.



(12) Strip ^{243}Am from DGA with 13mL 0.5M HCl. Save ^{243}Am for future use.




k' Np(IV) on UTEVA Resin


DGA, Normal Resin

References

1) McAlister and Horwitz, “Chromatographic Generator Systems for the actinides and natural decay series elements,” *Radiochimica Acta*, 99:1-9 (2011).

eichrom®

AN-1620-10

$^{224}Ra/^{212}Pb$ Generator

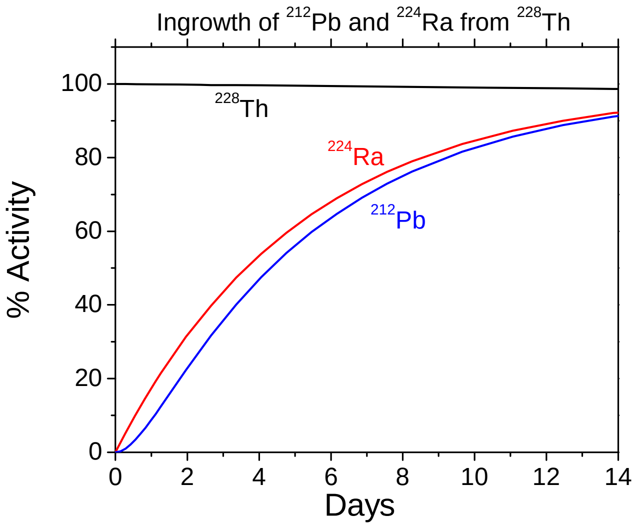
Summary of Method A method for the preparation of ^{224}Ra ($t_{1/2}$ = 3.62 days) and ^{212}Pb ($t_{1/2}$ = 10.64 hours) from ^{228}Th ($t_{1/2}$ = 1.913 years) source material is presented. The method employs 2mL cartridges of UTEVA and Sr resins to obtain high purity ^{224}Ra and ^{212}Pb in small volumes of eluate, while preserving valuable ^{228}Th material. The source material is adjusted to 4M HNO_3 and loaded onto stacked 2mL cartridges of UTEVA and Sr resins. ^{228}Th is retained on UTEVA Resin, while ^{212}Pb is retained on Sr Resin and ^{224}Ra is unretained. The ^{228}Th source is recovered from UTEVA Resin with a small volume of 0.5M HCl. Following a suitable ingrowth period, the ^{228}Th can be acidified to 4M HNO_3 and used to produce additional ^{224}Ra and ^{212}Pb. The ^{228}Th is preserved nearly completely and continuously purified from chemical and radiologic impurities run to run, allowing repeated use until radioactive decay depletes the ^{228}Th activity. ^{212}Pb may recovered from Sr resin with a variety of reagents, including 6-8M HCl, citrate, tartrate, acetate and bioxalate.

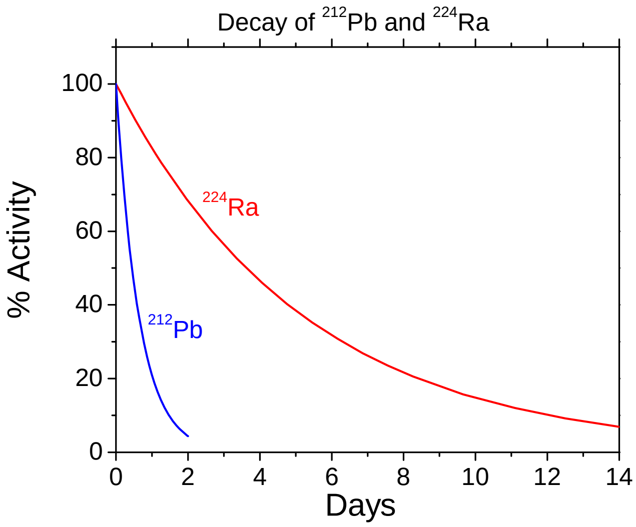
Reagents

UTEVA Cartridges (Eichrom UT-R50-S)
Sr Resin Cartridges (Eichrom SR-R50-S)
^{228}Th Source
Deionized Water
HCl
HNO_3
<u>Option for ^{224}Ra only:</u>
LN Resin cartridges (Eichrom LN-R50-S)

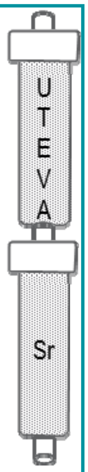
Equipment

Glass vials for storage of ^{228}Th source.
Glass or plastic vials/bottles for collection of ^{224}Ra, ^{212}Pb and waste.
10, 20 or 30mL plastic luer lock syringes
Gamma Spectrometry System of alternative for measurement of ^{228}Th, ^{224}Ra, and ^{212}Pb.





^{212}Pb/^{224}Ra/^{228}Th Separation*



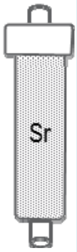
(1) Precondition stacked 2mL cartridges of UTEVA and Sr Resins with 10mL 4M HNO_3.

(2) Acidify ^{228}Th eluate from previous separation with 5mL conc. HNO_3. (If new ^{228}Th source, dilute to 20mL with 4M HNO_3.)

(3) Load ^{212}Pb/^{224}Ra/^{228}Th on UTEVA/Sr Resin. Collect ^{224}Ra.

(4) Rinse UTEVA/Sr Resin with 10mL 4M HNO_3. Collect ^{224}Ra.

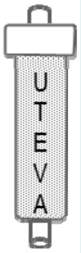
(5) Separate UTEVA/Sr Resin cartridges.



(6) Rinse Sr Resin with 10mL 0.1M HNO_3.

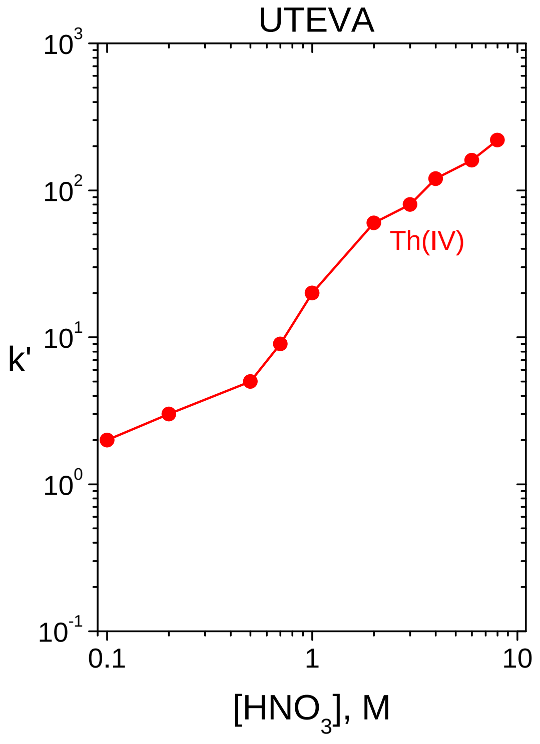
(7) Strip ^{212}Pb from Sr Resin with one of the following:

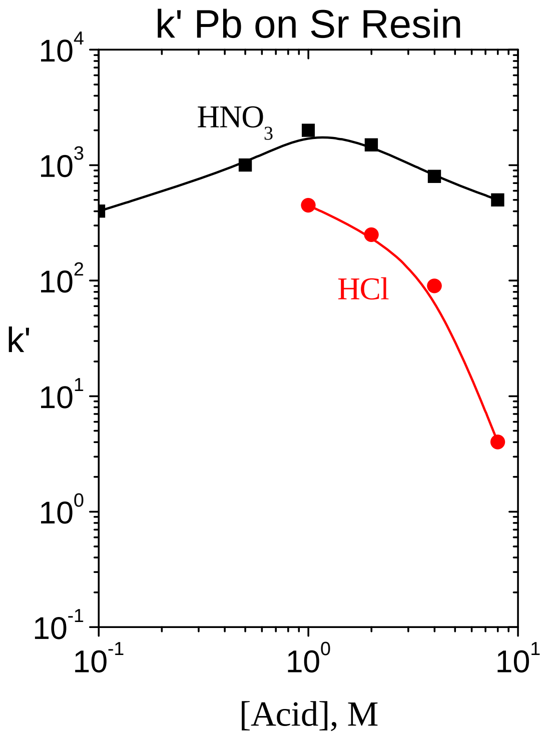
-15mL 6M HCl
-10mL 8M HCl
-10mL 0.05 ammonium citrate
-10mL 0.05 ammonium tartrate
-10mL 0.05 ammonium acetate
-10mL 0.05 ammonium bioxalate



(8) Strip ^{228}Th from UTEVA with 15mL 0.5M HCl. Recovery of ^{228}Th can be improved by stripping in opposite direction of load. Save ^{228}Th for future use.

*If only ^{224}Ra is desired, a simplified generator can be made by loading ^{228}Th onto a 2mL cartridge of LN resin from 0.1M HNO_3. ^{224}Ra can then be periodically milked using 5-10mL of 0.1M HNO_3 or HCl.





References

1) McAlister and Horwitz, “Chromatographic Generator Systems for the actinides and natural decay series elements,” *Radiochimica Acta*, 99:1-9 (2011).

AN-1621-10

^{234}Th Generator

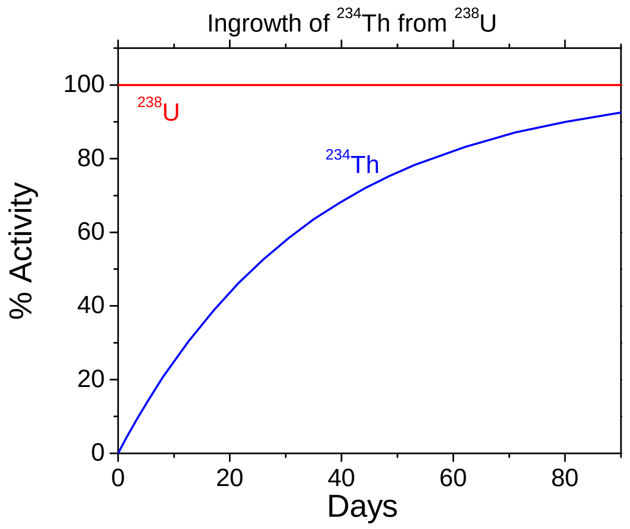
Summary of Method A method for the preparation of ^{234}Th ($t_{1/2}$ = 24.1 days) from natural or depleted Uranium ($t_{1/2}$ = 4.47E9 years) source material is presented. The method utilizes extraction chromatography with a column of DGA, Normal resin and 2mL cartridges of DGA and UTEVA resins to obtain high purity ^{234}Th in small volumes of eluate, while preserving ^{238}U material. The source material is adjusted to 2M HNO_3 and loaded onto a column of DGA, Normal resin. ^{234}Th is retained on DGA Resin from up to 0.2M uranium, while uranium is unretained. The uranium source is recovered and, following a suitable ingrowth period, can be used to produce additional ^{234}Th. ^{234}Th is stripped from the DGA resin column and further purified using 2mL cartridges of DGA and TEVA resins.

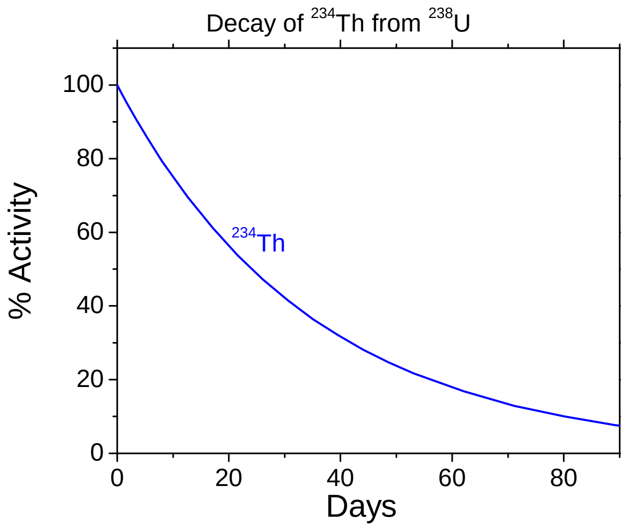
Reagents

TEVA Cartridges (Eichrom TE-R50-S)
DGA Cartridges (Eichrom DN-R50-S)
DGA, Normal Resin (Eichrom DN-B25-A)
Natural or Depleted U Source
Deionized Water
Oxalic Acid
Ammonium Oxalate
HCl
HNO_3

Equipment

Glass/Plastic bottles for storage of Uranium source.
Glass or plastic vials/bottles for collection of ^{234}Th and waste.
10, 20 or 30mL plastic luer lock syringes.
Gamma Spectrometry System or alternative for measurement of ^{234}Th.
ICP-AES or alternative for measurement of U.
1.9cm i.d. glass or plastic column, minimum 15cm height, with 250mL-1L reservoir.
Glass wool or frit material for top bed support.
Peristaltic pump or alternative to increase flow rate.





^{234}Th Separation (50g U/17µCi ^{234}Th)*

(1) Slurry pack a column of 12g DGA, Normal resin (100-150um), 1.9cm i.d. x 12.5cm height.**

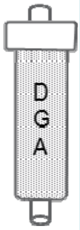
(2) Place top bed support on column. Rinse column with 2-3 bed volumes of 0.1M HNO_3. Store column in 0.1M HNO_3 between uses.

(3) Precondition column with 2 bed volumes of 2M HNO_3.

(4) Load 50g U source in 1L 2M HNO_3 at 10-20mL/min. Discard first 20mL of eluate. Collect remaining eluate containing U source material. Save for future ^{234}Th production.

(5) Rinse DGA column with 200mL of 1M HNO_3. First 20mL may be collected and added to source.

(6) Strip ^{234}Th from DGA with 200mL of 0.05M HNO_3-0.05M Oxalic Acid.

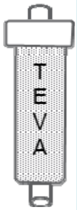


(7) Add 29mL of conc. HNO_3 to acidify eluate to 2M HNO_3.

(8) Load onto 2mL cartridge of DGA Resin.

(9) Rinse DGA with 25mL 0.5M HNO_3.

(10) Strip ^{234}Th from DGA with 20mL 0.1M ammonium bioxalate.



(11) Acidify with 8mL conc. HNO_3.

(12) Precondition 2mL TEVA cartridge with 5mL 4M HNO_3.

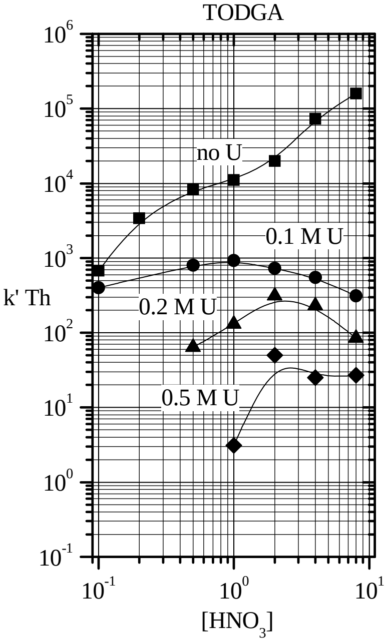
(13) Load onto TEVA.

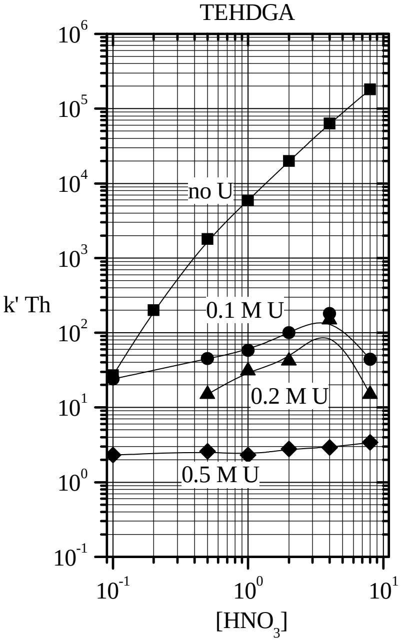
(14) Rinse TEVA w/ 25mL 4M HNO_3.

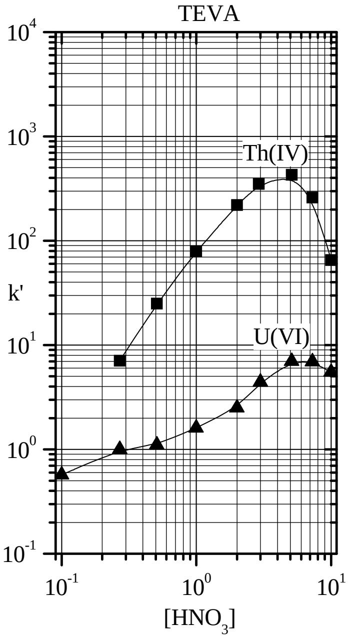
(15) Strip ^{234}Th w/ 15mL 0.5M HCl.

*Separation is scalable. Simply adjust volumes of the initial DGA column and load solution to accommodate other source sizes.

**DGA resin can be difficult to wet. Slurry the resin in 2x its volume of 1.0-1.5M HNO_3 by gently swirling for 2-3 minutes (avoid vigorous shaking as this can incorporate air bubbles and cause resin to float). Centrifuge resin slurry for 5-10 minutes. Repeat until most of the resin sinks to the bottom of tube. Repeat swirling/centrifugation, if needed. Use only well wetted resin to pack the column (omit floating resin). The column may be reused many times if stored in dilute acid between uses.







References

1) E. P. Horwitz and D. R. McAlister, "The recovery of trace thorium from large quantities of uranium," *Solv. Extr. Ion Exch.*, 27, 474-488, (2009).

Separation of ^{89}Zr From Y Target

AN-1622-10

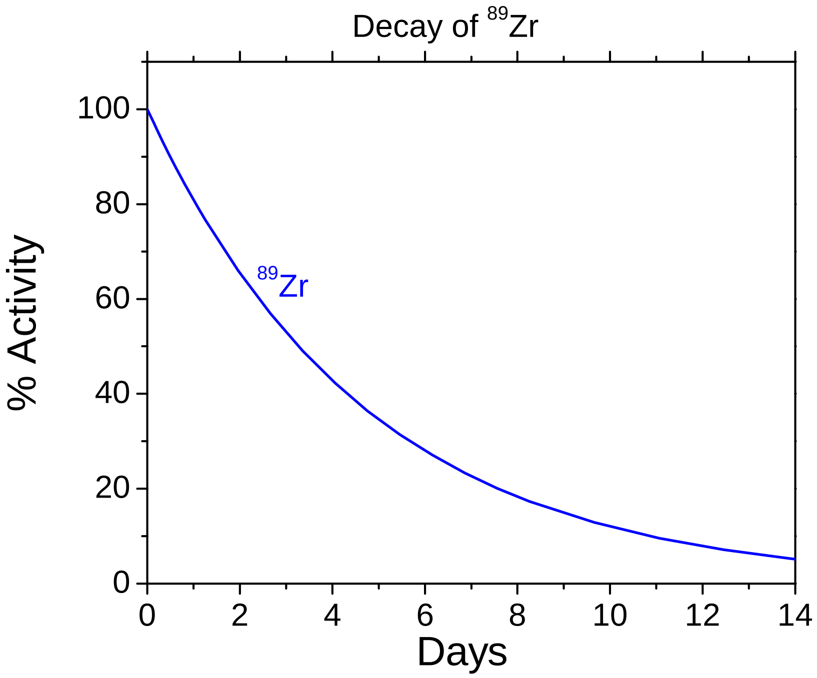
Summary of Method A method for the separation of ^{89}Zr ($t_{1/2}$ = 78.43 hours) from yttrium target material is presented. The method employs 2mL cartridges of LN3 and Anion Exchange resins to obtain high purity ^{89}Zr in small volumes of eluate, while providing high separation factors from chemical and radiologic impurities. The primary separation of ^{89}Zr from the dissolved yttrium target can be performed in 2-8M HNO_3 or HCl using LN3 resin. ^{89}Zr is retained while yttrium passes through LN3. ^{89}Zr is recovered from LN3 with a small volume of 0.05M HCl-oxalic acid and directly loaded onto a 2mL cartridge of Anion Exchange resin. ^{89}Zr is retained while additional decontamination from yttrium and niobium is achieved. ^{89}Zr is then recovered in a small volume of 2-4M HCl. Average yield of Zr, separated from 500mg Y, was >90%, with >10^6 separation factor from Y and Nb.

Reagents

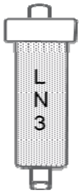
LN3 Cartridges (Eichrom L3-R50-S)
Anion Exchange Cartridges (Eichrom A8-R50-M-Cl)
Deionized Water
Oxalic Acid
Ammonium Oxalate
HCl
HNO_3

Equipment

Glass or plastic vials/bottles for collection of ^{89}Zr and waste.
30mL and 60mL plastic luer lock syringes.
Gamma Spectrometry System or alternative for measurement of ^{89}Zr.
ICP-AES or alternative for measurement of Y.



^{89}Zr Separation



(1) Dissolve yttrium target. Adjust to 50-100mL of 2-8M HCl or HNO_3.

(2) Precondition 2mL LN3 cartridge with 10mL 2-8M HCl or HNO_3.

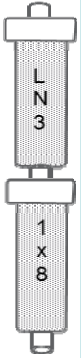
(3) Load sample onto LN3 resin at 2-3mL/min.

(4) Rinse LN3 with 10mL 2M HCl.

(5) Replace syringe or reservoir with clean syringe or reservoir.

(6) Rinse LN3 with 40mL 2M HCl.

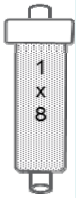
(7) Precondition 2mL anion exchange cartridge with 10mL 0.05M HCl-0.05 oxalic acid.



(8) Place anion exchange cartridge below LN3 cartridge.

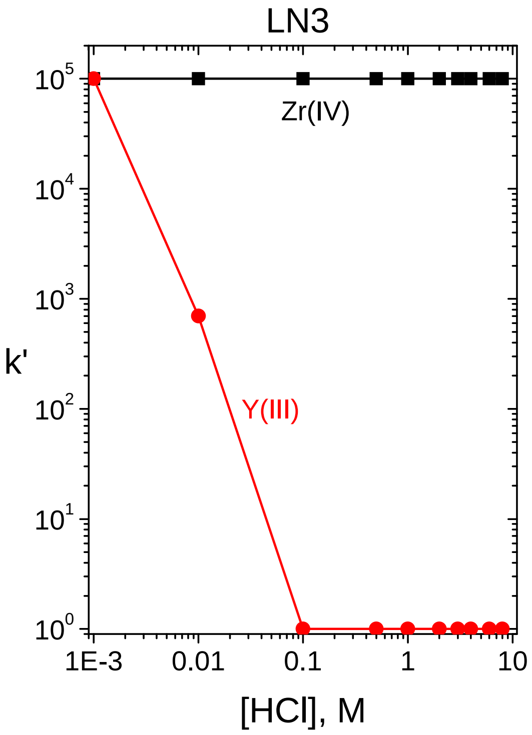
(9) Strip ^{89}Zr from LN3 and load onto anion exchange with 25mL 0.05M HCl-0.05M oxalic acid.

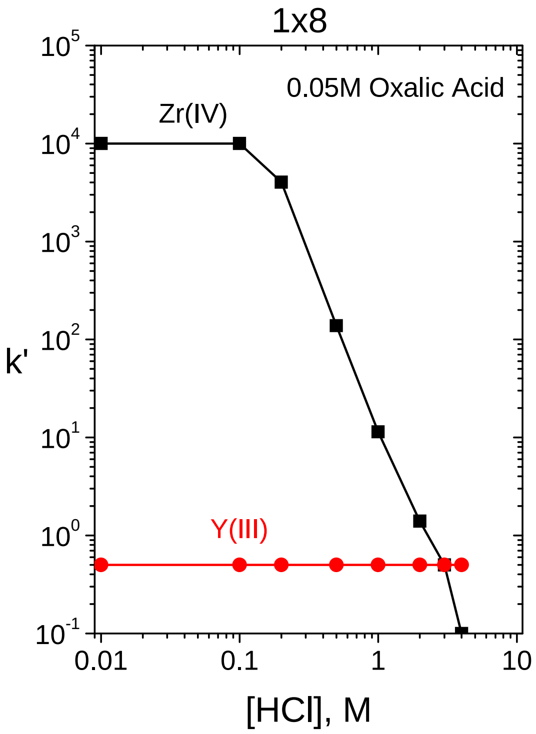
(10) Separate LN3 and anion exchange cartridges.



(11) Rinse anion exchange cartridge with 10mL 37% HCl.

(12) Strip ^{89}Zr with 10mL 2-4M HCl.





References

1) E. P. Horwitz and D. R. McAlister, Unpublished data (2015 and 2016)

Separation of ^{86}Y From Sr Target

AN-1623-10

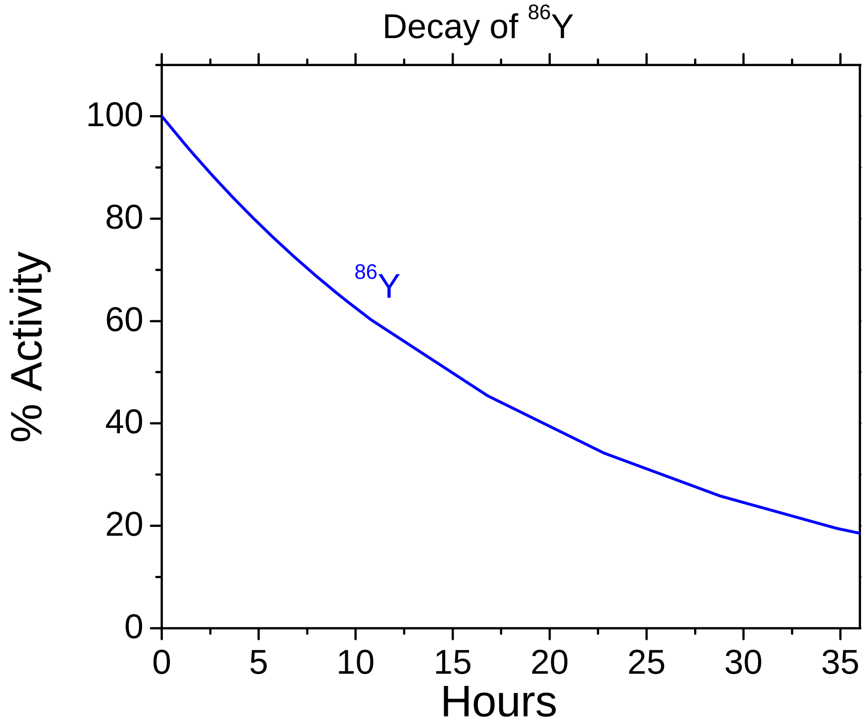
Summary of Method A method for the separation of ^{86}Y ($t_{1/2}$ = 14.74 hours) from strontium target material is presented. The method employs 2mL cartridges of DGA and LN resins to obtain high purity ^{86}Y in small volumes of eluate, while providing high separation factors from chemical and radiologic impurities. The primary separation of ^{86}Y from the dissolved yttrium target can be performed in 8M HNO_3 or HCl using DGA resin. ^{86}Y is retained while strontium passes through DGA. ^{86}Y is recovered from DGA with a small volume of 0.25M HCl and directly loaded onto a 2mL cartridge of LN resin. ^{86}Y is retained while additional decontamination from strontium is achieved. ^{86}Y is then stripped from LN resin onto a second 2mL cartridge of DGA resin using 8M HCl. ^{86}Y is then eluted from DGA using 10mL 0.1M HCl. DGA, Branched is used to allow stripping of ^{86}Y in a minimal volume of 0.1M HCl. Average yield of Y separation from 500mg of Sr was >95% with >10^{10} separation factor from Sr.

Reagents

DGA, Branched Cartridges (Eichrom DB-R50-S)
LN Resin Cartridges (Eichrom LN-R50-S)
Deionized Water
HCl
HNO_3

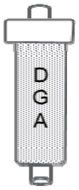
Equipment

Glass or plastic vials/bottles for collection of ^{89}Zr and waste.
30mL and 60mL plastic luer lock syringes.
Gamma Spectrometry System or alternative for measurement of ^{86}Y.
ICP-AES or alternative for measurement of Sr.



^{86}Y Separation Using DGA and LN Resin

(1) Dissolve strontium target. Adjust to 50-100mL of 8M HCl or HNO_3.



(2) Precondition 2mL DGA cartridge with 10mL 8M HCl or HNO_3.

(3) Load sample onto DGA at 4-5 mL/min.

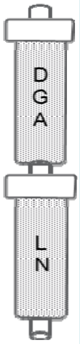
(4) Rinse DGA with 25mL 8M HNO_3.

(5) Rinse DGA with 25mL 1M HNO_3.

(6) Replace syringe or reservoir with clean syringe or reservoir.

(7) Precondition 2mL LN resin cartridge with 10mL 0.25M HCl.

(8) Place LN resin cartridge below DGA cartridge.



(8) Strip ^{86}Y from DGA and load onto LN with 25mL 0.25M HCl.

(9) Separate DGA and LN cartridges.

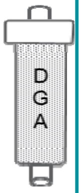
(10) Rinse LN resin cartridge with 25mL 0.5M HCl.

(11) Precondition 2mL DGA resin cartridge with 10mL 8M HCl.

(12) Place DGA resin cartridge below LN cartridge.

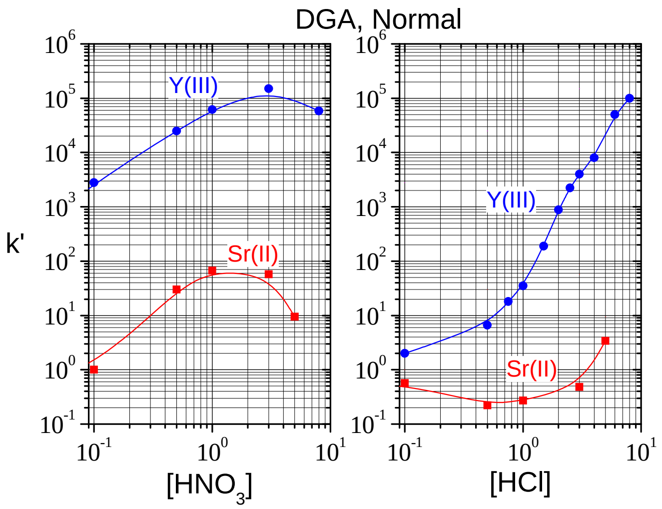
(13) Strip ^{86}Y from LN and load onto DGA with 25mL 8M HCl.

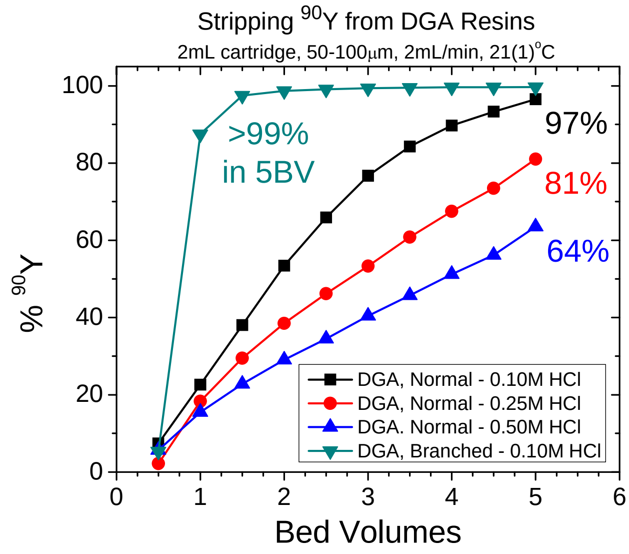
(14) Separate LN and DGA cartridges.



(15) Rinse DGA with 25mL 5M HCl.

(16) Strip ^{86}Y with 5-10mL 0.1M HCl.





References

1) E. P. Horwitz and D. R. McAlister, Unpublished data (2015 and 2016).

AN-1624-11

Options for ^{89}Sr and ^{90}Sr Determination

Summary There are many methods (Table 1) available for the measurement of radiostrontium (Table 2) from environmental, building materials, or biological samples. Typically, analysts are interested in the measurement of the fission products ^{89}Sr (decays to stable ^{89}Y) and ^{90}Sr (decays to β^- emitting ^{90}Y). Stable strontium or ^{85}Sr may also be used as a chemical yield tracer. Methods typically begin with a concentration or matrix removal step, followed by the separation of strontium from interfering nuclides using Sr Resin (Figure 1A). Methods may also incorporate steps to discriminate between ^{89}Sr and ^{90}Sr, including multiple counts, nuclide selective counting techniques and ingrowth and secondary separations of ^{90}Y (daughter of ^{90}Sr). Measurement instrumentation includes low background gas flow proportional counting (GFPC), liquid scintillation (LSC), Cerenkov counting, and inductively coupled plasma-mass spectrometry (ICP-MS).

This application note will offer guidance in choosing an appropriate method for the determination of radiostrontium, taking into account process knowledge, available measurement equipment and data quality objectives (required detection limits, urgency of measurement, age of sample, and need for $^{89/90}$Sr discrimination). The measurement methods are meant be to used in concert with the appropriate sample preparation method for the matrix being analyzed. For a more comprehensive treatment of sample preparation methods, see the application notes available at http://www.eichrom.com/eichrom/appnotes/applications/index.aspx. A detailed discussion of interferences for the various measurement techniques can also be found in references [2] and [5].

Table 1. Summary of $^{89/90}$Sr Method Options

Method	Primary Separation	Primary Measurement	Secondary Separation	Secondary Measurement	Sr Yield Monitor	Ref.
ICP-MS	1A	^{90}Sr by ICP-MS	None	None	Stable Sr	1
Total $^{89/90}$Sr	1A	$^{89/90}$Sr by GFPC/LSC	None	None	Stable Sr	4,5
^{90}Y Direct	1C	^{90}Y by GFPC/LSC/Cerenkov	None	None	Stable Sr	6
Two Count ^{89}Sr and ^{90}Sr	1A	$^{89/90}$Sr by GFPC/LSC	None	$^{89/90}$Sr by GFPC/LSC*	Stable Sr	2
Rapid ^{89}Sr and ^{90}Sr	1A	^{89}Sr by Cerenkov	None	$^{89/90}$Sr by LSC	Stable Sr	5
Cerenkov ^{89}Sr and ^{90}Sr(^{90}Y)	1A	^{89}Sr by Cerenkov	1B or 1C	^{90}Y by Cerenkov	Stable Sr/^{85}Sr	3
Gas Flow Porportional	1A	$^{89/90}$Sr by GFPC	1B or 1C	^{90}Y by GFPC	Stable Sr/^{85}Sr	4

*^{89}Sr/^{90}Sr descrimination by solving equations for ^{89}Sr decay and ^{90}Y ingrowth during time between 2 measurements

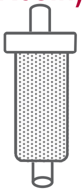
Table 2. Properties of Selected Nuclides

		Decay		Detector Suitable for Measurement				
Nuclide	Half-Life	Mode	Energy	GFPC	LSC	Cerenkov	MS/AES	Gamma
Stabel Sr	^{84}Sr (0.56%), ^{86}Sr(9.86%), ^{87}Sr (7.0%), ^{88}Sr (82.58%)			No	No	No	Yes	No
^{85}Sr	64.849 days	ε/γ	γ = 514 keV (96%)	No	Yes	No	No	Yes
^{89}Sr	50.563 days	β^-	β_{max} = 1500 keV β_{mean} = 587 keV	Yes	Yes	Yes	No	No
^{90}Sr	28.79 years	β^-	β_{max} = 546 keV β_{mean} = 196 keV	Yes	Yes	No	Yes	No
^{90}Y	64 hours	β^-	β_{max} = 2280 keV β_{mean} = 934 keV	Yes	Yes	Yes	No	No

Figure 1. Strontium Separation Options

A. Sr separation (Sr Resin)

Sr Resin
2-4 mL in cartridges



(1) Precondition Sr Resin with 3 bed volumes 8M HNO_3.

(2) Load sample at 1-2 mL/min.

(3) Rinse sample tube with 5 mL 8M HNO_3.

(4) Add tube rinse to Sr Resin. Elute at 1-2 mL/min.

(5) Rinse Sr Resin sequentially with*
- 3 bed volumes 8M HNO_3.
- 3 bed volumes 3M HNO_3 - 0.05M oxalic acid.
- 3 bed volumes 8M HNO_3.

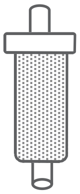
(6) Dispose of (1) to (5) as waste.

(7) Strip Sr with 5 bed volumes 0.05M HNO_3 at 1 mL/min.

*Rinse solutions and volumes listed are general guidelines. See detailed app. notes for recommended rinsing for each matrix.

B. Sr/^{90}Y Separation (Sr Resin)

Sr Resin
2 mL cartridge



(1) Dissolve Sr Residue from planchet used for total $^{89/90}$Sr measurement with 5 mL 8M HNO_3. (Or acidify Cerenkov sample to 8M HNO_3.)

(2) Using transfer pipet, transfer dissolved residue to 15mL c-tube.

(3) Rinse planchet with 5 mL 8M HNO_3. Add to 15 mL c-tube.

(4) Precondition 2 mL Sr Resin cartridge with 5 mL 8M HNO_3.

(5) Load sample at 1-2 mL/min, collecting eluate (^{90}Y-fraction).

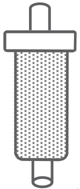
(6) Rinse c-tube with 5 mL 8M HNO_3.

(7) Add tube rinse to column. Elute, collecting eluate (^{90}Y fraction).

Method appropriate for high ^{90}Sr/^{89}Sr ratios or ^{90}Sr confirmation. Potential for 1-3% Sr break-through, limits application for high ^{89}Sr/^{90}Sr ratios.

C. Sr/^{90}Y Separation (DGA Resin)

DGA Resin, Normal
2 mL cartridge



(1) Dissolve Sr Residue from planchet used for total $^{89/90}$Sr measurement with 5 mL 8M HNO_3. (Or acidify Cerenkov sample to 8M HNO_3.)

(2) Transfer dissolved residue to 15mL c-tube.

(3) Rinse planchet with 5 mL 8M HNO_3. Add to 15 mL c-tube.

(4) Precondition 2 mL DGA Resin cartridge with 5 mL 8M HNO_3.

(5) Load sample at 1-2 mL/min.

(6) Rinse c-tube with 5 mL 8M HNO_3.

(7) Add tube rinse to cartridge.

(8) Rinse DGA with 15 mL 0.1M HNO_3.

(9) Save (4) to (8) as Sr fraction.

(10) Strip ^{90}Y with 10 mL 0.1M HCl.

Preferred method for high ^{89}Sr/^{90}Sr ratios. Additional rinses may be required for direct determination of ^{90}Sr via ^{90}Y, see AN-1414.

ICP-MS (^{90}Sr only) ^{90}Sr may be determined by inductively coupled plasma-mass spectrometry (ICP-MS) following separation on Sr Resin (Figure 1A). However, due to the relatively short half-life of ^{90}Sr, **application to low level environmental analyses will be limited** by the available mass spectrometry technology and concentration chemistries. Recent publications suggest detection limits of ~1Bq/g are possible. However the achievable detection limit will depend on several factors, including sample type, sample size, and the age and type of MS instrument available [1]. **Radiometric techniques will allow lower detection limits** for ^{90}Sr and the ability to also determine ^{89}Sr.

Accurate measurement of strontium chemical yield as ^{88}Sr by ICP-MS has very little chance for impact by **isobaric interferences** (^{87}Rb-H^{+}, which should be effectively removed by Sr Resin Separation). ^{90}Zr is the only significant isobaric interference for ^{90}Sr measurement. Separation chemistries should be tailored to ensure complete removal of zirconium (rinsing with 3M HNO_3-0.05M oxalic acid for Sr Resin separations). Determination of ^{90}Sr by ICP-MS **greatly simplifies the calculation of ^{90}Sr activity**, which can be complicated in radiometric detection techniques by the ingrowth of ^{90}Y and the decay of ^{89}Sr.

ICP-MS and **ICP-AES** (atomic emission spectrometry) are also very effective tools for the determination of **strontium chemical yield** when using radiometric detection methods for ^{89}Sr and ^{90}Sr. ICP-MS and ICP-AES will often give more precise chemical yield data than gravimetric techniques, while also allowing the use of **less Sr carrier** (<1 mg vs 4-10 mg stable Sr). Many environmental samples (soils, brines, sea water and some ground waters) may contain significant levels of stable strontium. Pre-anlysis of these samples by ICP-MS or ICP-AES for **native Sr** content may be necessary to adjust the amount of stable Sr yield monitor added and to ensure accurate measurement of the Sr yield throughout the separation process.

Total $^{89/90}$Sr In instances where **^{90}Sr is the only radiostrontium isotope** likely to be present or where total ^{89}Sr + ^{90}Sr determination is desired, radiostrontium may be determined by liquid scintillation counting or gas flow proportional counting following separation on Sr Resin (Figure 1A). **Confirmation of ^{90}Sr** activity will require **discrimination from ^{89}Sr** through two count methods and calculations [2], ingrowth and separation of ^{90}Y (Figure 1B or 1C) [4,5] or selective ^{89}Sr/^{90}Y measurement(s) using Cerenkov counting [3].

^{90}Sr by Direct Recovery of ^{90}Y In the analysis of debris from decommissioning of older facilities or other instances where **^{90}Sr is likely to be present in the absence of short-lived fission products** (^{91}Y, ^{89}Sr, etc.) and where **^{90}Y is in equilibrium with ^{90}Sr**, ^{90}Sr may be determined by liquid scintillation, gas flow proportional counting, or Cerenkov counting following the direct **separation of ^{90}Y on DGA Resin** (Figure 1C) [6]. The **very high retention of ^{90}Y** on DGA resin allows recovery of ^{90}Y from **difficult matrices, large sample volumes, and/or samples with high native Sr content** that may prove problematic using the isolation of Sr on Sr Resin. However, the presence of fresh fission products, such as **^{91}Y and rare earth radionuclides**, will cause **significant positive bias** in the ^{90}Sr determination performed using this method. Stable yttrium carrier yield can be measured by ICP-MS or ICP-AES.

Two Count Method Total $^{89/90}$Sr can be measured in an **initial count** using GFPC or LSC following separation on Sr Resin (Figure 1A). Following a period of **ingrowth for ^{90}Y**, the samples can be **counted again**, and individual ^{89}Sr and ^{90}Sr activities calculated by **solving a series of equations** related to the decay of ^{89}Sr and the ingrowth of ^{90}Y (two count method) [2]. Ideally, the **initial count** is performed **as quickly as possible** following the separation on Sr Resin to minimize ^{90}Y ingrowth. The **second count** is ideally performed **after 1-2 weeks of ^{90}Y ingrowth**. Computational methods for resolving the ^{89}Sr and ^{90}Sr activities are outlined in Appendix B of reference [2].

Cerenkov vs LSC The beta emission of **^{90}Sr is below the threshold for efficient measurement by Cerenkov counting** (LSC without addition of cocktail). **^{89}Sr may be determined by Cerenkov counting** immediately following separation of radiostrontium on Sr Resin (Figure 1A). **^{90}Y will also be efficiently measured by Cerenkov counting**.

Counting of ^{89}Sr by Cerenkov counting is **less efficient than by liquid scintillation** (LSC). However, the lower counting efficiency is partially offset by the **lower background** observed in Cerenkov counting. Furthermore, Cerenkov counting **eliminates interferences** from many low energy beta emitters that may cause bias in measurements made by LSC or GFPC. Additionally, Cerenkov counting **does not require the addition of cocktail**, offering cost savings in waste disposal and reagent costs. Cerenkov counting **does not suffer from quenching** which can occur in liquid scintillation.

Counting ^{89}Sr by Cerenkov or total $^{89/90}$Sr by GFPC as a first measurement leaves open the option to separate ^{90}Y from the strontium fraction for further confirmation of ^{90}Sr or discrimination of ^{89}Sr/^{90}Sr. Counting the strontium fraction by LSC limits any further separation on the sample due to the addition of the LSC cocktail, leaving the two count method as the only viable ^{89}Sr/^{90}Sr discrimination option after LSC measurements of $^{89/90}$Sr, unless the sample is split prior to LSC.

^{89}Sr by Cerenkov, $^{89/90}$Sr by LSC Counting (Rapid $^{89/90}$Sr) ^{89}Sr is determined by Cerenkov counting immediately following separation of radiostrontium on Sr Resin (Figure 1A) [3]. After the addition of LSC cocktail, ^{90}Sr may then be determined by measuring total radiostrontium (^{89}Sr + ^{90}Sr) by standard liquid scintillation counting and calculating the difference (total radiostrontium - ^{89}Sr), taking into account ingrowth of ^{90}Y and decay of ^{89}Sr during counting.

This approach **may not be appropriate for samples that require long count times** to meet data quality objectives or for samples with low ratios of ^{89}Sr/^{90}Sr, due to interference from ^{90}Y ingrowth. However, this method can be used very effectively to **rapidly determine ^{89}Sr and ^{90}Sr** in samples containing relatively high amounts of radiostrontium in the **immediate aftermath of a release of fresh fission products**.

When utilizing Cerenkov and/or liquid scintillation counting, chemical yield of Sr throughout the separation process is most effectively measured via stable strontium carrier by taking a small aliquot of the separated strontium fraction for analysis by ICP-MS or ICP-AES. Recovery by ^{85}Sr gamma emission is not recommended, due to interference with the measurement of total radiostrontium by liquid scintillation counting.

DGA vs Sr Resin for ^{90}Y Separation For samples with expected high ratios of ^{89}Sr/^{90}Sr, using DGA to selectively retain ^{90}Y (Figure 1C) is recommended, as the Sr can be effectively rinsed from the column, while ^{90}Y recovery remains strongly adsorbed (k' Y on DGA >>1000). In high ^{89}Sr/^{90}Sr samples, small breakthrough of strontium from separation on Sr Resin (Figure 1B) into the ^{90}Y fraction (k' Sr only ~100 on Sr Resin) can lead to high bias of the ^{90}Sr measurement and a corresponding low bias of the ^{89}Sr.

^{89}Sr by Cerenkov, ^{90}Y by Cerenkov Following Ingrowth and Separation ^{89}Sr is measured by Cerenkov counting following separation of strontium on Sr Resin (Figure 1A). ^{90}Sr may then be determined by waiting 1-14 days for ^{90}Y ingrowth, acidifying the Cerenkov counted sample with HNO_3, separating the ^{90}Y using Sr Resin (Figure 1B) or DGA Resin (Figure 1C), and measuring ^{90}Y by Cerenkov counting.

This method is **not as rapid as the Cerenkov/LSC rapid $^{89/90}$Sr method**, due to the ^{90}Y ingrowth period. However, due to the separation of pure ^{90}Y from strontium and other beta emitting nuclides with high decontamination factors, this method **may provide more accurate and sensitive measurements** for samples with **high ^{89}Sr/^{90}Sr or ^{90}Sr/^{89}Sr ratios**. The chemical yield of Sr may be measured via stable strontium carrier by taking a small aliquot of the separated strontium fraction for analysis by ICP-MS or ICP-AES or by ^{85}Sr gamma emission. Yttrium yields may also be tracked using stable Y and measurement by ICP-MS or ICP-AES.

$^{89/90}$Sr by GFPC, ^{90}Y by GFPC Following Ingrowth and Separation Total $^{89/90}$Sr can be measured effectively using low background gas flow proportional counting (GFPC) following separation of strontium on Sr Resin (Figure 1A) [4,5]. Prior to measurement by GFPC, purified strontium fractions are dried onto cupped planchets or precipitated as carbonates or phosphates and collected on filter paper (and dried). Immediate counts of the purified strontium fractions yield the total radiostrontium (^{89}Sr + ^{90}Sr). Following the initial count for total radiostrontium and a period of ^{90}Y ingrowth, the dried sample can be dissolved from the planchet or filter using HNO_3, and additional separations performed to isolate ^{90}Y from the strontium fraction using Sr Resin (Figure 1B) or DGA Resin (Figure 1C). ^{90}Y can then be measured by GFPC after evaporation on a stainless steel planchet or collection of an YF_3 precipitate on a filter. ^{90}Sr activity can be calculated using the measured ^{90}Y activity and the period of ingrowth from the initial separation. ^{89}Sr activity can be calculating by difference (total radiostrontium - ^{90}Sr).

When using GFPC, strontium chemical yield may be determined via stable strontium using ICP-MS, ICP-AES or gravimetric methods. Additionally, multiple drawer GFPC systems allow for the **simultaneous counting** of multiple samples, an option which is normally not available for Cerenkov or LSC.

References

1) Ohno, T., Hirono, M., Sakata, S., “Determination of Strontium 90 in environmental samples by triple quadrapole ICP-MS and its application to Fukashima soil samples.” *J. Anal. At. Spectrom.* 33, 1081-1085 (2018).

2) EPA 402-R-10-001d, “Rapid Radiochemical Method for Total Radiostrontium (Sr-90) In Water for Environmental Remediation Following Homeland Security Events,” October 2011.

3) Banavali, A. D. et al. "Strontium-89, 90 Analysis by Eichrom Column Chemistry and Cerenkov Counting". 38th Annual Conference on Bioassay Analytical and Environmental Radiochemistry. Santa Fe, NM. November 1992.

4) ASTM D5811-08, “Standard Test Method for Strontium-90 in Water.”

5) IAEA Analytical Quality in Nuclear Applications Series No. 27. “Rapid Simultaneous Determination fo 89Sr and 90Sr in Milk: A Procedure Using Cerenkov and Scintillation Counting,” IAEA/AQ/27 (2013). https://www-pub.iaea.org/MTCD/Publications/PDF/IAEA-AQ-27_web.pdf

6) Maxwell, S.L.; Culligan, B.K.; Hutchinson, J.B.; Utsey, R.C.; McAlister, D.R.; “Rapid Determination of ^{90}Sr in Seawater Samples,” *J. Radional. Nucl. Chem.*, 303, 709-717 (2015).

eichrom®

AN-1701-10

Measurement of ^{36}Cl and ^{129}I in Water

Summary of Method Chlorine-36 and Iodine-129 are separated and measured from up to 500mL aliquots of water. Samples are adjusted to 0.1-1.0M H_2SO_4 and 0.1M $SnSO_4$ is added to ensure reduction of any oxidized species to chloride (Cl^-) and Iodide (I^-). The CL resin is prepared by loading with Ag^+ cations which will facilitate the retention of Cl^- and I^-. The sample is loaded onto the CL Resin, the column is rinsed with deionized water, and Cl^- is recovered using 0.1M ammonium thiocyanate (NH_4SCN). If iodine determination is also required, the resin is the rinsed with 0.1% NaOH and then I^- is recovered using 0.35M sodium sulfide (Na_2S). ^{36}Cl ($t_{1/2}$ = 3.01E5 years, β^{-}_{mean} = 251keV, β^{-}_{max} = 709.55keV, abundance = 98.1%) and ^{129}I ($t_{1/2}$ = 1.57E7 years, β^{-}_{mean} = 40.03keV, β^{-}_{max} = 149.3keV, abundance = 100%) can then be measured using liquid scintillation counting.

Reagents

Cl Resin Bulk (CL-B50-A or CL-B50-S)*

-or-

Cl Resin Prepacked 2mL columns (CL-C50-A)

Deionized Water

NaOH

H_2SO_4

$SnSO_4$

Na_2S

NH_4SCN

$AgNO_3$ (or Solution of 10mg/mL Ag^+)

Liquid Scintillation Cocktail

*If packing own columns for gravity flow, then use CL-B50-A. If using vacuum assisted flow, then use CL-B50-S.

Equipment

Centrifuge tubes - 50mL

Liquid scintillation vials, 20mL glass

Liquid scintillation counter

Calibrated pipets and disposable tips

Appropriately sized glass beakers and flasks

Analytical balance

Filter funnel and paper

Optional: Empty columns and frits for packing own columns

Sample Preparation

Up to 500mL of water sample in glass beaker.

↓

Adjust to 0.1-1.0M H_2SO_4.

↓

Add 1 mL of 0.1M $SnSO_4$ per 50mL of sample.

↓

Mix sample well.

CL Resin Preparation (Batch Mode)

Reagent amounts may be adjusted proportionally to as needed.

Weigh 10 grams CL Resin into a 250mL flask.

↓

Dissolve 0.65 grams $AgNO_3$ in 100mL 1M H_2SO_4.

↓

Add Ag/H_2SO_4 solution to resin and Mix for 2 hrs.

↓

Filter CL Resin and rinse twice with 1M H_2SO_4.

↓

Slurry resin in 100mL 0.1M H_2SO_4 and pack into the appropriate sized column.

CL Resin Preparation (Column)

Reagent amounts may be adjusted

Slurry resin in 100mL 0.1M H_2SO_4 and pack into the appropriate sized column or obtain prepacked 2mL column of CL Resin.

↓

Rinse 2mL column with 10mL 1M H_2SO_4.

↓

Dissolve 0.65 grams $AgNO_3$ in 100mL 1M H_2SO_4.
Load 2 mL of solution onto each 2mL column.
Wait 2hr for Ag to be completely absorbed.

↓

Rinse 2mL column with 10mL 1M H_2SO_4.

Chlorine/Iodine Separation

1) Load Sample onto 2mL CL Resin column at 2mL/min.
2) Rinse 2mL CL Resin column with 10mL 0.1M H_2SO_4.
3) Rinse 2mL CL Resin column with 10mL deionized water.
4) Strip 2mL CL Resin into 20mL LSC vial with 5mL 0.1M NH_4SCN to recover chloride.
5) Rinse 2mL CL Resin column with 10mL 0.1% NaOH.
6) Strip 2mL CL Resin column into a 2mL LSC vial with 5mL 0.35M Na_2S.
7) Add 15 LSC cocktail to each LSC vial.
8) Count ^{36}Cl and ^{129}I samples by LSC.

Decontamination Factors (DF) for Chloride and Iodide Frations		
Analyte	**DF Cl- fraction**	**DF I- fraction**
Ba	>1000	>600
Cd	>6900	>7700
Cu	>210	>190
Mn	>210	>370
Ni	>170	>320
Pb	>300	>720
U	>1900	>200
^{60}Co	>320	>320
^{137}Cs	>150	>150
^{90}Sr/^{90}Y	>180	>160
^{36}Cl	N/A	>160
^{129}I	>420	N/A

Retention of Cl and I on CL Resin from 1M H_2SO_4	
Analyte	**Dw**
^{36}Cl	1600
^{129}I	1980

Chloride capcity: 4 mg / 2mL column
Iodide capcity: 15 mg / 2mL column

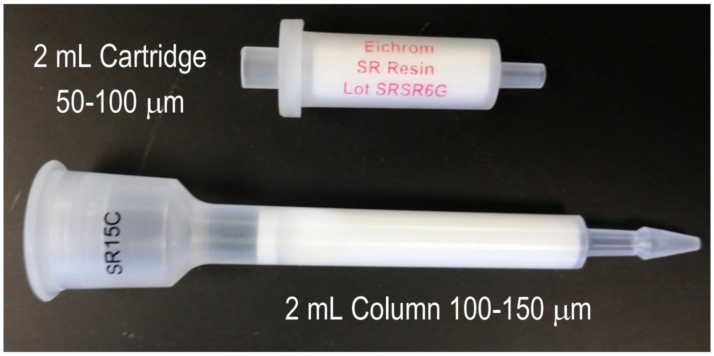
References

1) A. Zulauf, S. Happel, M.B. Mokili, A. Bombard, H. Jungclas, “Characterization of an extraction chromatographic resin for the separation and determination of 36Cl and 129I.” J. Radioanal. Nucl. Chem. 286(2), 539-546 (2010).

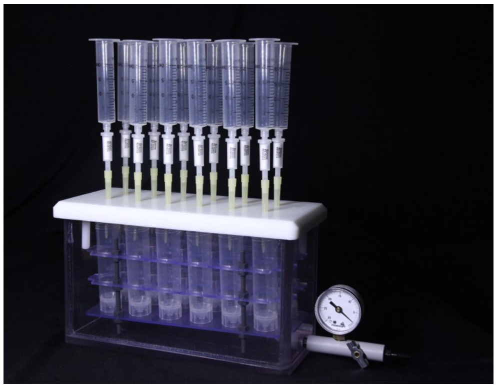
AN-1702-10

Converting Methods from Gravity Columns to Cartridges

Summary Performing separations using Eichrom 2 mL pre-packed cartridges on a vacuum box system offers many advantages over gravity flow columns, including faster flow rates, improved chromatographic resolution, and the ability to stack multiple cartridges and measure multiple analytes from one sample aliquot. Converting separations methods from gravity flow columns to Eichrom cartridges is normally simple:



1) Obtain a 12-Hole or 24-Hole vacuum box, vacuum pump and tubing.



2) Add cartridges appropriate for the separation.

3) Run separation procedure using the same solutions and volumes as the column method, adjusting the vacuum to achieve the optimal flow rate:

- 1 to 2 mL/min for sample load and rinses.
- 1 mL/min for stripping steps.

4) Drawing air through the cartridges for a short time (<5-10 minutes) between elution steps will not adversely impact the separation. Some samples will run faster than others.

5) Sample aliquots may be collected in individual 50 mL centrifuge tubes or collectively in the vacuum box liner.

6) Change cartridge reservoirs and inner and outer tips prior to elution of each analyte to improve purity.

7) The Eichrom vacuum box can also be used for CeF_3 microprecipitation source preparation using resolve filters.

Table 1. TEVA Resin Performance (2006-2017)*

Parameter	2 mL Cartridge 50-100 µm	2 mL Column 100-150 µm
^{230}Th % yield	99.3 ± 0.5	99.1 ± 0.7
^{239}Pu % yield	98.3 ± 1.6	98.1 ± 1.5
^{239}Pu % impurity in Th	0.2 ± 0.1	0.2 ± 0.1
^{230}Th % impurity in Pu	0.3 ± 0.2	0.3 ± 0.2

*Eichrom Quality Method QA-0213

Table 2. TRU Resin Performance (2006-2017)*

Parameter	2 mL Cartridge 50-100 µm	2 mL Column 100-150 µm
^{241}Am % yield	99.4 ± 0.4	99.4 ± 0.6
^{239}Pu % yield	97.2 ± 0.6	97.7 ± 0.7
^{239}Pu % impurity in Am	0.3 ± 0.2	0.3 ± 0.1
^{241}Am % impurity in Pu	0.3 ± 0.2	0.4 ± 0.2

*Eichrom Quality Method QA-0212

Vacuum Box -12 Hole (AR-12-BOX)

Includes:

- Rack for 50mL c-tubes (AR-12-RACK)
- Vacuum Gauge (AR-01-GAUGE-PVC)
- Vacuum Box Lid (AR-12-LID)
- White Inner Support Tubes (25)
- Yellow Outer Tips (25)
- Vacuum Box Manifold Plugs (50)
- Cartridge Reservoir, 10 mL (25)

Optional:

- Inner Liner (AR-12-LINER)
- Top Support (AR-12-TS)

Vacuum Box –24 Hole (AR-24-BOX)

Includes:

- Rack for 50mL c-tubes (AR-24-RACK)
- Vacuum Gauge (AR-01-GAUGE-PVC)
- Vacuum Box Lid (AR-24-LID)
- White Inner Support Tubes (50)
- Yellow Outer Tips (50)
- Vacuum Box Manifold Plugs (50)
- Cartridge Reservoir, 10 mL (25)

Optional:

- Inner Liner (AR-24-LINER)
- Top Support (AR-24-TS)

Additional Equipment

- Vacuum Pump (Fisher no. 01-092-25 or equivalent)
- Tubing Tygon 1/4 in. I.D., 7/16 in. O.D.
  (Fisher no. 14-169-1K, or equivalent)
- White Inner Support Tubes (AR-1000-TUBE-PE)
- Yellow Outer Tips (AR-1000-OT)
- Stopcock, Polycarbonate (12) (AR-12-PC)
- 10 mL Cartridge Reservoir (200) (AR-200-RV10)
- 20 mL Cartridge Reservoir (200) (AR-200-RV20)

Table 3. UTEVA Resin Performance (2006-2017)*

Parameter	2 mL Cartridge 50-100 µm	2 mL Column 100-150 µm
^{230}Th % yield	99 ± 3	99 ± 3
^{233}U % yield	99 ± 4	99 ± 4
^{233}U % impurity in Th	0.2 ± 0.1	0.1 ± 0.1
^{230}Th % impurity in U	0.1 ± 0.1	0.1 ± 0.1

*Eichrom Quality Method QA-0214

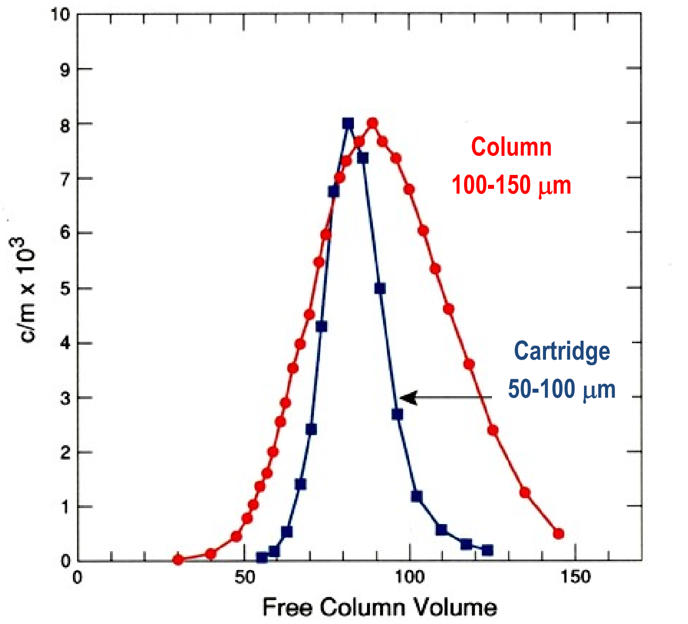
Table 4. Sr Resin Performance (2006-2017)*

Parameter	2 mL Cartridge 50-100 µm	2 mL Column 100-150 µm
Sr % yield	93 ± 3	95 ± 3
Ba % impurity in Sr	0.2 ± 0.1	0.2 ± 0.1
Ca % impurity in Sr	< 0.02	< 0.02
Y % impurity in Sr	< 0.01	< 0.01

*Eichrom Quality Method QA-0215

Comparison of Elution Curves for Sr^{2+} for Two Particle sizes of Sr Resin

Elutrient 3.2 M HNO$_3$, 23-24° C

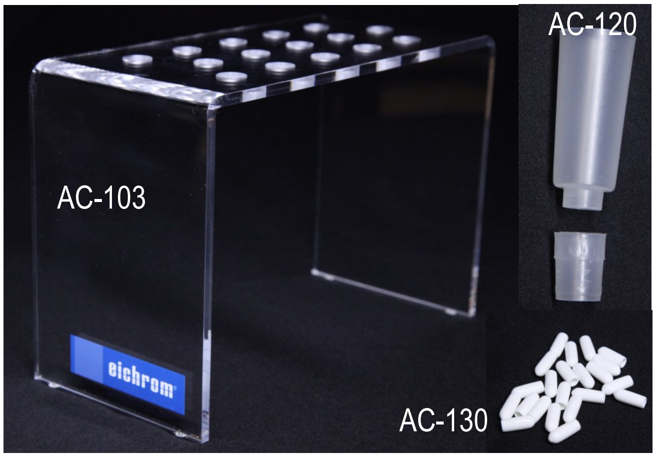


AN-1703-10

Packing Eichrom 2 mL Columns

Summary Eichrom offers empty 2 mL columns and bulk resin for customers who wish to pack their own columns. This application note will offer advice on slurry packing columns that will exhibit favorable flow conditions and efficient separations. Some hydrophobic, difficult to wet resins may require additional treatment prior to slurry packing or may be dry packed.

Eichrom snap tip 2 mL Columns (AC-141-AL) come with the bottom frit inserted. Top frits and column top caps are also included.



2 mL Column Racks (AC-103), Column Tip Closures (AC-130), and 2-piece 25 mL Extension Funnels (AC-120) are also available.

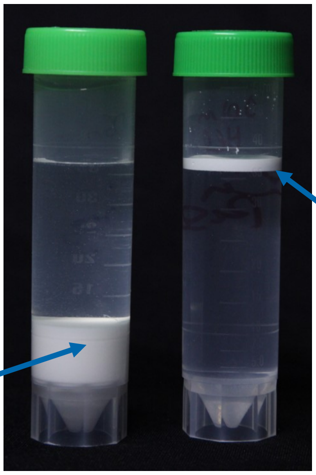
The first step in slurry packing columns is to wet the resin by mixing with an appropriate aqueous phase. For most Eichrom resins, a solution of 0.05M HNO_3 is ideal for column packing. Add a volume of resin sufficient to pack your columns to 3-5x that volume of 0.05M HNO_3. Mix the resin by vortexing, swirling or gently tumbling. Avoid vigorous shaking, which can lead to air bubbles that can degrade column flow and separation efficiency. If a portion of the resin floats on the surface of the 0.05M HNO_3, centrifuge the sample. Repeat mixing and centrifuging as necessary to achieve a well wetted resin with minimal amount of floating material.

Some more **hydrophobic resins will not wet well in 0.05M HNO_3**. Table 1 lists some difficult to wet resins and **alternative matrices to facilitate wetting**. The resins in Table 1 can be wetted by **replacing the 0.05M HNO_3 with the alternative slurry matrix** and following the steps above. Once DGA, LN2 and LN3 have been wetted with the alternative slurry matrix, centrifuge and decant the aqueous phase and replace with 0.05M HNO_3 for storage and column packing.

Table 1. Slurry Matrices for Difficult to Wet Resins

Resin	Slurry Matrix
Prefilter	0.05M HNO_3
Ni Resin	0.15M Ammonium Citrate
DGA, Normal	2M HNO_3
DGA, Branched	2M HNO_3
Cu Resin*	0.05 - 2M HCl
LN2	1M HNO_3
LN3	2M HNO_3

*Cu Resin will float on the surface of the solution even when wetted.



Wetted resin, ideal for slurry packing columns.

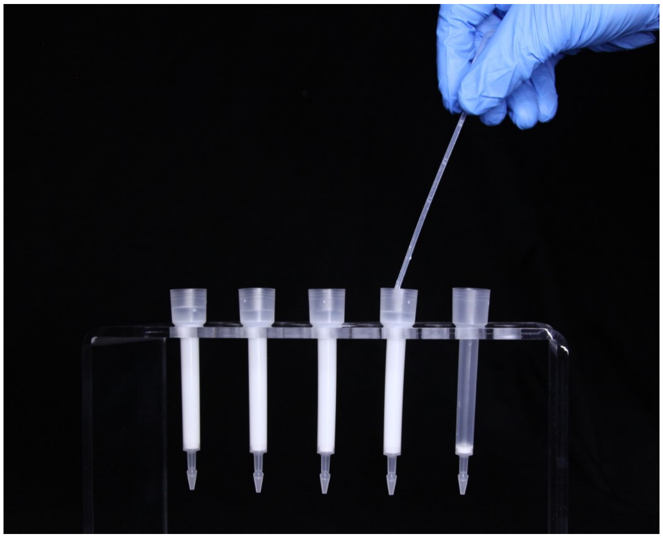
Floating or poorly wetted resin, difficult to slurry pack into columns.

The packing method is written assuming a 0.05M HNO_3 slurry matrix. For Ni Resin and Cu Resin, replace 0.05M HNO_3 with the appropriate alternative. Pre-packed Eichrom 2 mL columns contain 1.6 mL of resin. This method was written to replicate this fill volume.

As the resin is wetting in the appropriate matrix, add empty 2 mL columns to a column rack or other support. Add a small volume of 0.05M HNO_3 to soak the bottom frit and remove air bubbles. Soak until no can be seen escaping from the frits.

Add enough top frits for each column to a centrifuge tube with a small volume of 0.05M HNO_3. Soak the frits to remove air bubbles. Swirl or vortex to mix, but avoid vigorous shaking.

Decant the 0.05M HNO_3 from the empty columns. Mix the slurry of resin and 0.05M HNO_3 to suspend the resin. Add the resin slurry to each column until the reservoir above the column is ~half full. Allow the resin to settle (~1 hr).



Full 2 mL columns should have a bed height of 4.1 ± 0.2 cm. Add additional slurry to meet this height or remove excess resin using a plastic transfer pipet. Leave enough 0.05M HNO_3 above the packed bed to fill the column and a portion of the reservoir.

Place a pre-soaked frit into each column. Using a glass or plastic stir rod, push the frit to the top of the packed bed. Decant the 0.05M HNO_3 above the top frit and rinse away any residual resin from above the top frit using 0.05M HNO_3.

If storing the columns for future use, fill the reservoir above the top frit ~half full with 0.05M HNO_3 and place a top cap on each column. If using the column immediately, snap off the bottom tip, allow any excess 0.05M HNO_3 to drain, and begin the column preconditioning step.

Dry Packing Columns Some difficult to wet resins can also be dry-packed into columns:

1) Place 2 mL columns with bottom frits in column rack.
2) Weigh 0.65 ± 0.05 g of dry resin into each column.
3) Tap to settle the resin.
4) Place a top frit on each column and push the frit to the top of the resin bed.
5) Rinse away any excess resin above the top frit.
6) Add preconditioning solution to the column reservoir. Over pressure or vacuum may be required to initiate column flow.
7) Allow solution to drain through column.

AN-1801-10

Rapid Determination of $^{89/90}$Sr in Steel Samples

Summary of Method Strontium is separated and measured from 1-2 gram steel samples. Samples are digested with concentrated nitric, hydrochloric, and hydrofluoric acids. The digestate is evaporated to dryness, the residue dissolved in HNO_3/H_3BO_3, and a calcium fluoride precipitate is used to concentrate the strontium and remove matrix. An optional NaOH fusion may also be performed, post sample digestion, to dissolve concrete or stone included in the sample. Strontium is separated from matrix impurities and potentially interfering radionuclides in the sample using stacked 2 mL and 1 mL cartridges of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter. Average chemical recovery of strontium, determined by gravimetric yield of stable strontium carrier, was 90–94%. Measured values of ^{90}Sr agreed to within 3% of reference values for 60 minute count times. The minimum detectable activity for ^{90}Sr in 2 g samples with 60 minute count times was 0.56 Bq/g. A single operator can prepare batches of 12 samples for the measurement of ^{90}Sr in less than 8 hours.

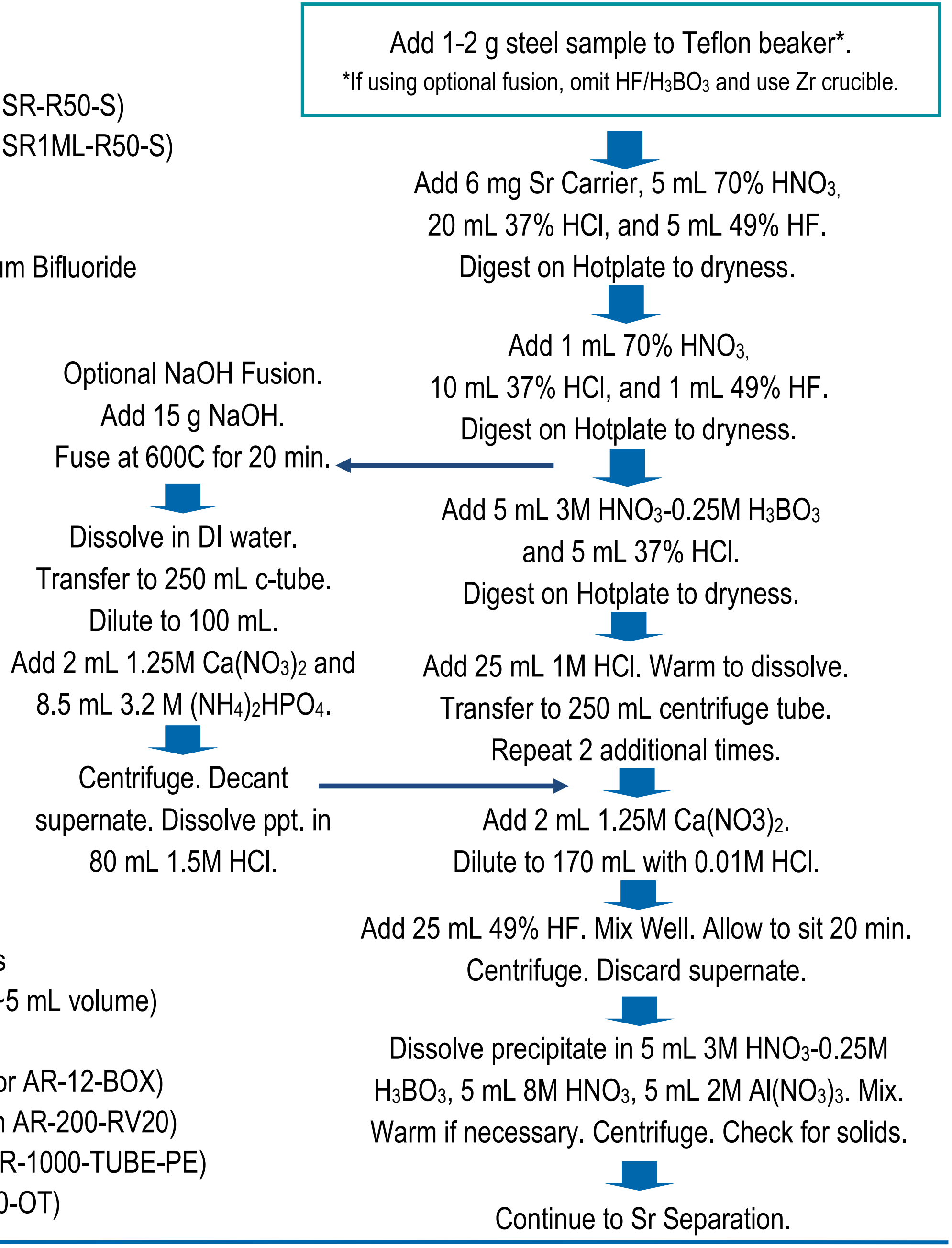
Reagents

Sr Resin, 2 mL Cartridges (Eichrom SR-R50-S)
Sr Resin, 1 mL Cartridges (Eichrom SR1ML-R50-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Ammonium Bifluoride
Deionized Water
1.25M $Ca(NO_3)_2$
Strontium Carrier (10 mg/mL)
2M $Al(NO_3)_3$
Sr-90 standard
Oxalic acid
Boric acid
3.2M $(NH_4)_2HPO_4$*
Sodium Hydroxide*

Equipment

Vacuum Pump
Centrifuge
Muffle Furnace*
Hot Plate
Analytical Balance
Teflon Beakers (Zr Crucibles*)
50 mL and 250 mL Centrifuge Tubes
Cupped Stainless Steel Planchets (~5 mL volume)
Gas Flow Proportional Counter
Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Cartridge Reservoir, 20 mL (Eichrom AR-200-RV20)
Inner Support Tubes-PE (Eichrom AR-1000-TUBE-PE)
Yellow Outer Tips (Eichrom AR-1000-OT)

Figure 1. Sample Preparation



Add 1-2 g steel sample to Teflon beaker*.
*If using optional fusion, omit HF/H_3BO_3 and use Zr crucible.

Add 6 mg Sr Carrier, 5 mL 70% HNO_3, 20 mL 37% HCl, and 5 mL 49% HF. Digest on Hotplate to dryness.

Add 1 mL 70% HNO_3, 10 mL 37% HCl, and 1 mL 49% HF. Digest on Hotplate to dryness.

Optional NaOH Fusion. Add 15 g NaOH. Fuse at 600C for 20 min.

Dissolve in DI water. Transfer to 250 mL c-tube. Dilute to 100 mL. Add 2 mL 1.25M $Ca(NO_3)_2$ and 8.5 mL 3.2 M $(NH_4)_2HPO_4$.

Centrifuge. Decant supernate. Dissolve ppt. in 80 mL 1.5M HCl.

Add 5 mL 3M HNO_3-0.25M H_3BO_3 and 5 mL 37% HCl. Digest on Hotplate to dryness.

Add 25 mL 1M HCl. Warm to dissolve. Transfer to 250 mL centrifuge tube. Repeat 2 additional times.

Add 2 mL 1.25M Ca(NO3)2. Dilute to 170 mL with 0.01M HCl.

Add 25 mL 49% HF. Mix Well. Allow to sit 20 min. Centrifuge. Discard supernate.

Dissolve precipitate in 5 mL 3M HNO_3-0.25M H_3BO_3, 5 mL 8M HNO_3, 5 mL 2M $Al(NO_3)_3$. Mix. Warm if necessary. Centrifuge. Check for solids.

Continue to Sr Separation.

Figure 2. Strontium Separation and Measurement

(1) Precondition Sr Resin with 10 mL 8M HNO_3.

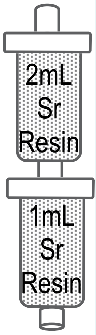
(2) Load sample.

(3) Rinse sample tube with 5 mL 8M HNO_3.

(4) Add tube rinse to Sr Resin.

(5) Rinse Sr Resin sequentially with:

- 15 mL 8M HNO_3
- 10 mL 3M HNO_3 - 0.05 oxalic acid
- 10 mL 8M HNO_3



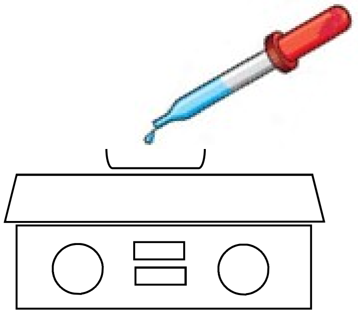
(6) Dispose of (1) to (5) as waste.

(7) Strip Sr with 15 mL 0.05M HNO_3.

Gas Flow Proportional Counting*:

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.

(9) Rinse Sr sample vials with 2 mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.

(11) Measure radiostrontium in samples on low background gas flow proportional counter.

*(Options for $^{89/90}$Sr Discrimination)

(a) Sr fraction from step (7) can be transferred to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (no LSC cocktail). $^{89/90}$Sr can then be measured by adding liquid scintillation cocktail.

(b) Sr fraction from step (10) can be dissolved in 10 mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

*Additional discussion of $^{89/90}$Sr separation and measurement options can be found in Eichrom Application Note AN-1624-10.

Method Performance for 2 g Steel Samples

Details	Sample replicates	Reference (mBq/sample)	Measured (mBq/sample)	Average % Diff.	Sr Carrier % Yield
90Sr	10	1.415	1.41 ± 0.04	2.6	90.1 ± 2.4
89Sr+90Sr	8	3.816	3.97 ± 0.09	4.1	94.1 ± 2.8

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchison, Robin. C. Utsey, Ralf Sudowe, Daniel R. McAlister, "Rapid method to determine $^{89/90}$Sr in steel samples," *J. Radioanal. Nucl. Chem., 314(1), 439-450* (2017).

eichrom®

AN-1802-10

Rapid Determination of Pu in Steel Samples

Summary of Method Plutonium is separated and measured from 1-2 gram steel samples. Samples are digested with concentrated nitric, hydrochloric, and hydrofluoric acids. The digestate is evaporated to dryness, the residue dissolved in HNO_3/H_3BO_3, and a CaF_2/LaF_3 precipitate is used to concentrate the Pu and remove matrix. An optional NaOH fusion may also be performed, post sample digestion, to dissolve concrete or stone included in the sample and deal more rigorously with refractory Pu. Plutonium is separated from matrix impurities and potentially interfering radionuclides in the sample using 2 mL cartridges of Eichrom TEVA Resin. Plutonium is measured by alpha spectrometry following rare earth fluoride microprecipitation onto Eichrom Resolve filters. The chemical recovery of Pu, determined by ^{242}Pu tracer, was 90–99%. Measured values of Pu typically agreed to within 7-8% of reference values for 16 hour count times. The minimum detectable activity for Pu in 2 g samples with 16 hour count times was 0.25 mBq/g. A single operator can prepare batches of 12 samples for the measurement of Pu in less than 8 hours.

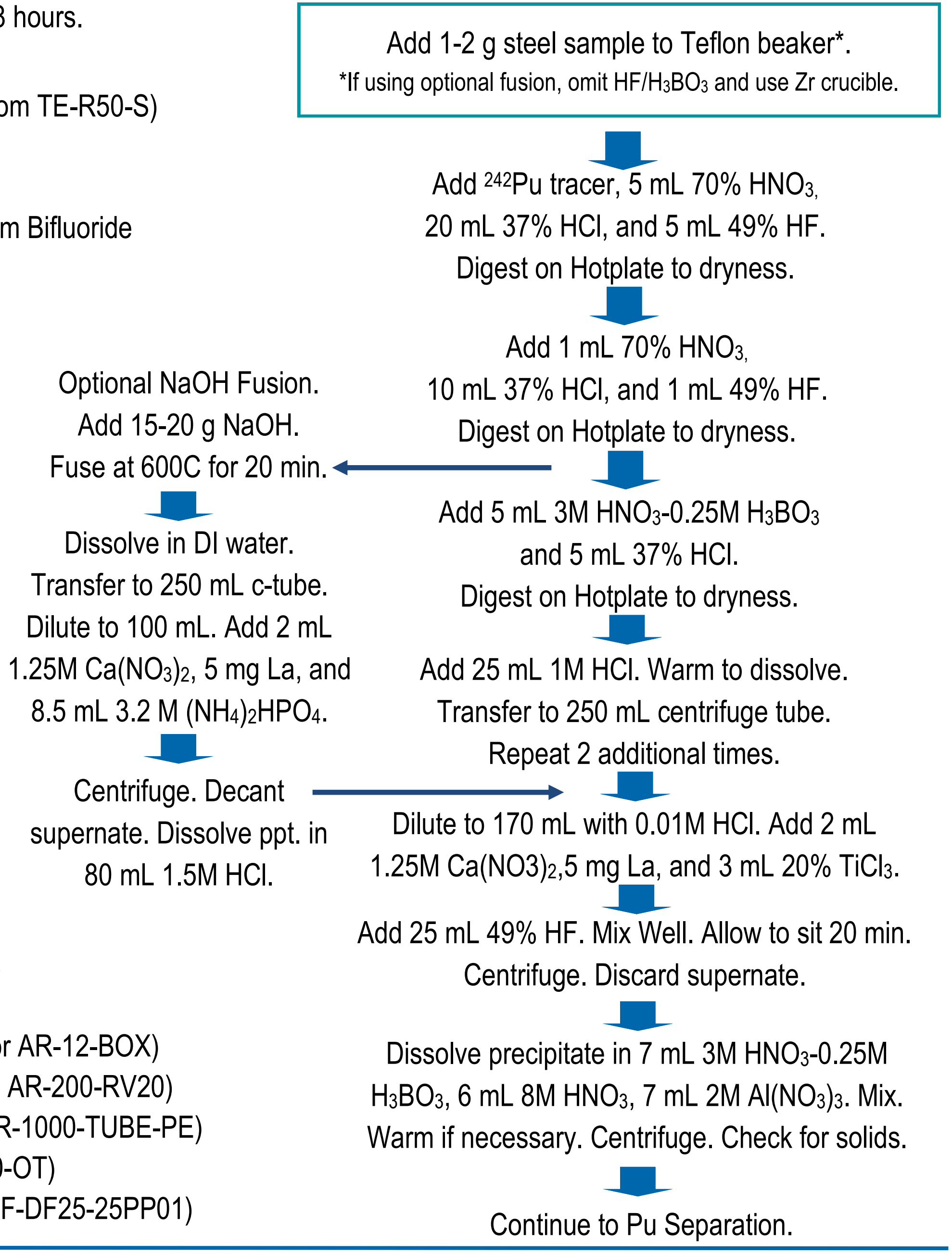
Reagents

TEVA Resin, 2 mL Cartridges (Eichrom TE-R50-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrofluoric Acid (49%) or Ammonium Bifluoride
Lanthanum Carrier (10 mg/mL)
Cerium Carrier (10 mg/mL)
Deionized Water 1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$ ^{242}Pu Tracer
Boric acid $NaNO_2$
Ascorbic Acid 30% H_2O_2
10-20% (w:w) $TiCl_3$ in HCl
3.2M $(NH_4)_2HPO_4$*
Sodium Hydroxide*

Equipment

Vacuum Pump
Centrifuge
Muffle Furnace*
Hot Plate
Analytical Balance
Teflon Beakers (Zr Crucibles*)
50 mL and 250 mL Centrifuge Tubes
Alpha Spectrometry System
Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Cartridge Reservoir, 20 mL (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)

Figure 1. Sample Preparation



Add 1-2 g steel sample to Teflon beaker*.
*If using optional fusion, omit HF/H_3BO_3 and use Zr crucible.

Add ^{242}Pu tracer, 5 mL 70% HNO_3, 20 mL 37% HCl, and 5 mL 49% HF. Digest on Hotplate to dryness.

Add 1 mL 70% HNO_3, 10 mL 37% HCl, and 1 mL 49% HF. Digest on Hotplate to dryness.

Optional NaOH Fusion. Add 15-20 g NaOH. Fuse at 600C for 20 min.

Dissolve in DI water. Transfer to 250 mL c-tube. Dilute to 100 mL. Add 2 mL 1.25M $Ca(NO_3)_2$, 5 mg La, and 8.5 mL 3.2 M $(NH_4)_2HPO_4$.

Centrifuge. Decant supernate. Dissolve ppt. in 80 mL 1.5M HCl.

Add 5 mL 3M HNO_3-0.25M H_3BO_3 and 5 mL 37% HCl. Digest on Hotplate to dryness.

Add 25 mL 1M HCl. Warm to dissolve. Transfer to 250 mL centrifuge tube. Repeat 2 additional times.

Dilute to 170 mL with 0.01M HCl. Add 2 mL 1.25M $Ca(NO3)_2$,5 mg La, and 3 mL 20% $TiCl_3$.

Add 25 mL 49% HF. Mix Well. Allow to sit 20 min. Centrifuge. Discard supernate.

Dissolve precipitate in 7 mL 3M HNO_3-0.25M H_3BO_3, 6 mL 8M HNO_3, 7 mL 2M $Al(NO_3)_3$. Mix. Warm if necessary. Centrifuge. Check for solids.

Continue to Pu Separation.

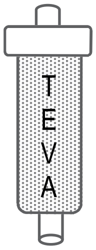
Figure 2. Load Solution Preparation and Plutonium Separation

(1) Ensure samples are cooled to room temperature.

(2) Add 1.25 mL of 1.5M ascorbic acid. Mix well. Wait 5-10 minutes.

(3) Add 1 mL of 3M $NaNO_2$. Mix well. Wait 5-10 minutes.

(4) Precondition 2 mL TEVA Resin with 5 mL 3M HNO_3.



(6) Load sample.

(7) Rinse sample tube with 5 mL 8M HNO_3 + 50 μL 30% H_2O_2.

(8) Add tube rinse to TEVA Resin.

(9) Rinse TEVA Resin sequentially with:
- 15 mL 3M HNO_3 (U decon.)
- 20 mL 9M HCl (Th)
- 5 mL 3M HNO_3

(10) Dispose of (4) to (9) as waste.

(11) Strip Pu with 15 mL 0.1M HCl-0.05M HF-0.01M $TiCl_3$.*

*If preparing alpha sources by electrodeposition, replace $TiCl_3$ with Rongalite or hydroxylamine.

(12) Add 50 ug Ce carrier and 0.5 mL 30% H_2O_2 to all samples. Mix well.

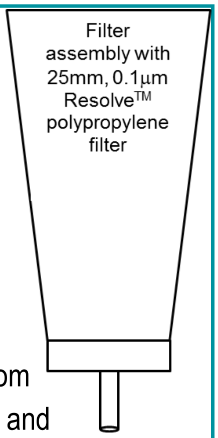
(13) Add 1 mL 49% HF. Mix well. Wait 15-20 minutes.

(14) Set up Resolve® Filter Funnel on vacuum box.

(15) Wet filter with 3 mL 80% ethanol followed by 3 mL DI water.

(16) Filter sample.

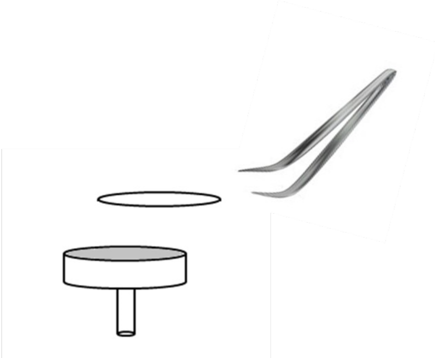
(17) Rinse sample tube with 5 mL DI water and add to filter.



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

(19) Draw vacuum until filter is dry.

(20) Remove filter from funnel assembly and mount filter on stainless steel planchet with 2-sided tape.



(21) Dry filter under heat lamp for 3-5 minutes.

(22) Measure actinides by alpha spectrometry.

Method Performance for Pu in Steel Samples

Details	Sample replicates	Reference (mBq/sample)	Measured (mBq/sample)	Average % Diff.	242Pu tracer % Yield
238Pu in 2 g Steel	5	37.0	37.7 ± 1.6	4.2	89.3 ± 2.3
239Pu in 2 g Steel	5	24.5	24.4 ± 1.6	6.6	96.5 ± 3.4
239Pu (refractory) in 2 g Steel	5	24.5	23.4 ± 0.9	3.8	98.9 ± 6.6
239 Pu in 5 g Steel	4	37.0	38.3 ± 1.0	2.6	92 ± 14

References

1) Sherrod L. Maxwell, Brian K. Culligan, Jay B. Hutchison, Robin. C. Utsey, Ralf Sudowe, Daniel R. McAlister, "Rapid method to determine plutonium isotopes in steel samples," *J. Radioanal. Nucl. Chem., 314(2), 1103-1111* (2017).

AN-1803-10

Rapid Determination of ^{226}Ra in Steel Samples

Summary of Method ^{226}Ra is isolated from 1 gram steel samples. Samples are digested with concentrated nitric, hydrochloric, and hydrofluoric acids. The digestate is evaporated to dryness, the residue dissolved in HNO_3/H_3BO_3 and calcium fluoride precipitate is used to concentrate the radium and remove matrix. Radium is separated from matrix impurities and potentially interfering radionuclides in the sample using cation exchange and DGA Resin. Radium is measured by alpha spectrometry following barium sulfate microprecipitation onto Eichrom Resolve Filters. The chemical recovery, determined by ^{133}Ba tracer, was 89–95%. Measured values of ^{226}Ra agreed to within 5% of reference values for 16 hour count times. The minimum detectable activity for ^{226}Ra in 1 g samples with 16 hour count times was 0.5 mBq/g. A single operator can prepare batches of 12 samples for the measurement of ^{226}Ra in less than 8 hours.

Reagents

Cation Exchange Resin (Eichrom C8-B500-F-H)
DGA Resin, Normal 2 mL Cartridges (Eichrom DN-R50-S)
Ammonium Hydroxide (Listed as 28% NH_3 or 56% NH_4OH)
Nitric Acid (70%)
Deionized Water
^{133}Ba Tracer
1.25M $Ca(NO_3)_2$
Barium Carrier (1 mg/mL)
Isopropyl Alcohol
Ammonium Sulfate
Denatured Ethanol
Hydrochloric Acid (37%)
Hydrogen Peroxide (30%)

Equipment

Plastic Chromatography Column (Eichrom AC-50E-5M)
Column Extension Funnel (Eichrom AC-20X-20M)
Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Cartridge Reservoir, 20 mL (Eichrom AR-200-RV20)
Inner Support Tubes-PE (Eichrom AR-1000-TUBE-PE)
Yellow Outer Tips (Eichrom AR-1000-OT)
Resolve Filter in Disposable Funnel (Eichrom RF-DF-25-25PP01)
50 mL Centrifuge Tubes
Centrifuge
Hotplate
150 mL Glass beakers
Vacuum Pump
Heat Lamp
Stainless Steel Planchets with adhesive tape
Alpha Spectrometry System
Gamma Spectrometry System (^{133}Ba tracer)

Figure 1. Sample Preparation

Add 1 g steel sample to Teflon beaker.

↓

Add ^{133}Ba tracer, 5 mL 70% HNO_3,
20 mL 37% HCl, and 5 mL 49% HF.
Digest on Hotplate to dryness.

↓

Add 5 mL 70% HNO_3,
10 mL 37% HCl, and 1 mL 49% HF.
Digest on Hotplate to dryness.

↓

Add 5 mL 3M HNO_3-0.25M H_3BO_3
and 5 mL 37% HCl.
Digest on Hotplate to dryness.

↓

Add 10 mL 0.25M HCl. Warm to dissolve.
Transfer to 50 mL centrifuge tube.
Repeat 2 additional times.

↓

Add 2 mL 1.25M $Ca(NO_3)_2$ and 6 mL 49% HF.

↓

Mix Well. Allow to sit 20 min.
Centrifuge. Discard supernate.

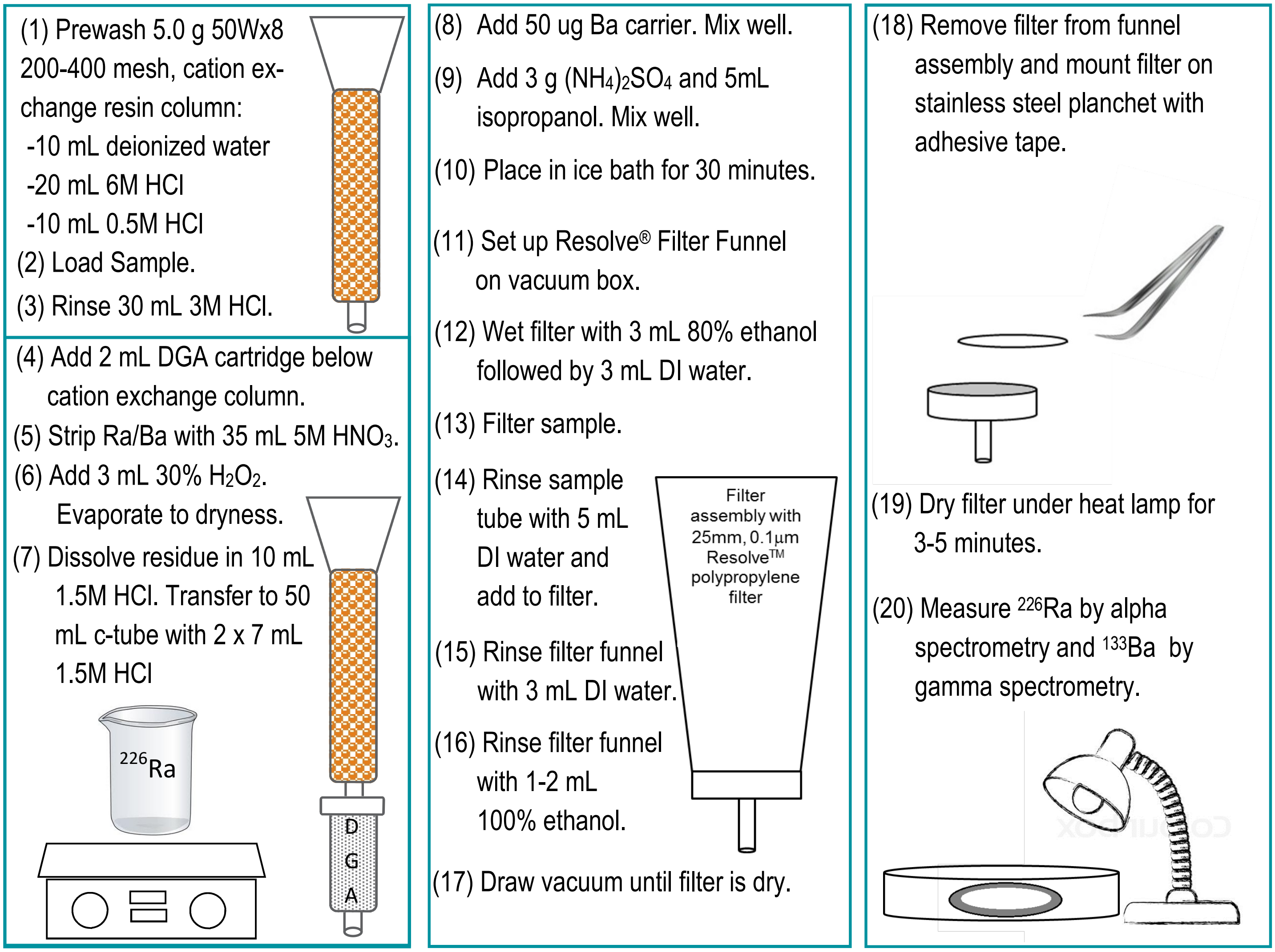
↓

Dissolve precipitate in 10 mL 1M HCl
-0.25M H_3BO_3 and 10 mL 1M HCl.
Mix. Warm if necessary. Centrifuge.
Check for solids.

↓

Continue to Ra Separation.

Figure 2. Column Purification and Alpha Source Preparation



(1) Prewash 5.0 g 50Wx8 200-400 mesh, cation exchange resin column:
-10 mL deionized water
-20 mL 6M HCl
-10 mL 0.5M HCl
(2) Load Sample.
(3) Rinse 30 mL 3M HCl.

(4) Add 2 mL DGA cartridge below cation exchange column.
(5) Strip Ra/Ba with 35 mL 5M HNO_3.
(6) Add 3 mL 30% H_2O_2. Evaporate to dryness.
(7) Dissolve residue in 10 mL 1.5M HCl. Transfer to 50 mL c-tube with 2 x 7 mL 1.5M HCl

(8) Add 50 ug Ba carrier. Mix well.
(9) Add 3 g $(NH_4)_2SO_4$ and 5mL isopropanol. Mix well.
(10) Place in ice bath for 30 minutes.
(11) Set up Resolve® Filter Funnel on vacuum box.
(12) Wet filter with 3 mL 80% ethanol followed by 3 mL DI water.
(13) Filter sample.
(14) Rinse sample tube with 5 mL DI water and add to filter.
(15) Rinse filter funnel with 3 mL DI water.
(16) Rinse filter funnel with 1-2 mL 100% ethanol.
(17) Draw vacuum until filter is dry.

(18) Remove filter from funnel assembly and mount filter on stainless steel planchet with adhesive tape.
(19) Dry filter under heat lamp for 3-5 minutes.
(20) Measure ^{226}Ra by alpha spectrometry and ^{133}Ba by gamma spectrometry.

Method Performance for 1 gram Steel Samples

Sample replicates	Reference (mBq/sample)	Measured (mBq/sample)	Average % Diff.	133Ba tracer % Yield
5	36.8	36.5 ± 0.8	1.9	95.4 ± 5.9
5	73.7	74.9 ± 3.1	3.7	88.8 ± 1.8
5	184	183 ± 5	1.9	90 ± 13

References

1) Sherrod L. Maxwell, Brian K. Culligan, Robin C. Utsey and Daniel R. McAlister, “Rapid Method to Determine ^{226}Ra in Steel Samples,” *J. Radioanal. Nucl. Chem.*, *314*(2), 1417-1423 (2017).

AN-1804-10

Rapid Determination of Pu/Np and Am/Cm in Granite

Summary of Method Pu/Np and Am/Cm are separated and measured from 1 gram samples of granite. Samples are finely ground and fused in a zirconium crucible for 15 minutes at 600°C with 15 grams of NaOH. The fusion cake is dissolved in water, and actinides are concentrated and separated from the matrix using a calcium phosphate precipitate enhanced with iron. A secondary precipitation with calcium fluoride removes additional matrix (including silicates) and decreases the volume of precipitate. The calcium fluoride precipitate is dissolved with nitric acid-boric acid-aluminum nitrate to form the load solution. Analytes are separated from remaining matrix and potentially interfering radionuclides using stacked 2 mL TEVA and DGA Resin cartridges. Actinides are measured by alpha spectrometry after CeF_3 microprecipitation onto Resolve$^{(R)}$ Filters. An additional separation of Am/Cm from rare earth elements using TEVA resin and ammonium thiocyanate may be required for samples with significant rare earth content. The rugged sample preparation technique enables high tracer recovery and excellent analytical results, even when refractory materials are present.

Reagents

TEVA Resin, 2 mL Cartridges (Eichrom TE-R50-S)
DGA Resin, 2 mL Cartridges (Eichrom DN-R50-S)
Lanthanum and Cerium Carriers (10 mg/mL)
Iron Carrier (50 mg/mL Fe, as ferric nitrate)
^{242}Pu (or ^{236}Pu if Np is measured) tracer
^{243}Am tracer
Ammonium Thyiocyanate
30% H_2O_2
Hydrochloric Acid (37%)
1.25M $Ca(NO_3)_2$
2M $Al(NO_3)_3$
Sodium Hydroxide
$NaNO_2$
10-20% $TiCl_3$
HF(49%)
Nitric Acid (70%)
Deionized Water
3.2M $(NH_4)_2HPO_4$
Boric Acid
Ascorbic Acid

Equipment

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Cartridge Reservoir, 20 mL (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)
50 mL and 250 mL Centrifuge Tubes
250 mL Zirconium crucibles with zirconium lids
Alpha Spectrometry System
Stainless Steel planchets with two sided tape
Centrifuge
Muffle Furnace
Hot Plate/Heat Lamp
Analytical Balance
Vacuum Pump

Figure 1. Sample Preparation

1 g finely ground sample in zirconium crucible
Add ^{242}Pu or ^{236}Pu and ^{243}Am tracers.

Heat samples to dryness on hot plate.

Add 15 g of NaOH.
Cover crucibles with zirconium lids.
Fuse at 600°C for 15-20 minutes.

Carefully remove samples from furnace and cool in fume hood. Add 25-50 mL of water and heat on hot plate to dissolve fusion cake.

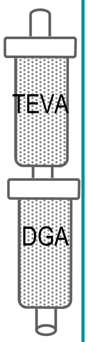
Transfer to a 250 mL centrifuge tube. Rinse crucible with water. Dilute to 180 mL with water.

Cool to room temperature. Add 125 mg Fe, 4 mg La, and 50 mg Ca. Mix. Add 5 mL 3.2M $(NH_4)_2HPO_4$. Mix. Add 4 mL 20% $TiCl_3$. Mix. Centrifuge 10 min. Decant supernate.

Dissolve precipitate in 80 mL 1.5M HCl. Dilute to 170 mL. Add 2 mL 20% $TiCl_3$, 25 mg Ca, and 20 mL 49% HF. Mix. Cool in ice bath 10 min. Centrifuge 10min. Decant supernate.

Dissolve precipitate in 7 mL 3M HNO_3-0.25M H_3BO_3, 6 mL 7M HNO_3, and 7 mL 2M $Al(NO_3)_3$.
Adjust valence with 1 mg Fe, 1.25 mL 1M ascorbic acid. Mix. Wait 5-10 min. Add 1 mL 3.5M $NaNO_2$ and 1.5 mL 70% HNO_3.

Figure 2. Actinide Separation on TEVA-DGA and Source Preparation



(1) Precondition stacked 2 mL TEVA and DGA cartridges with 10 mL 3M HNO_3.

(2) Load sample solution.

(3) Rinse sample tube with 5 mL 6M HNO_3. Add tube rinse to cartridges.*

(4) Rinse cartridges with 10 mL 3M HNO_3.

(5) Separate TEVA and DGA cartridges.

(6) Rinse TEVA cartridge with:

- -15 mL 3M HNO_3 (U decon.)
- -20 mL 9M HCl (Th)
- -5 mL 3M HNO_3

(7) Strip Pu (and Np) from TEVA cartridge with 20 mL 0.1M HCl-0.05MHF-0.01M $TiCl_3$.

(8) Rinse DGA cartridge with:

- -10 mL 3M HCl
- -3 mL 1M HNO_3
- -20 mL 0.1M HNO_3 (U decon.)
- -10 mL 0.05M HNO_3
- -20 mL 3M HNO_3-0.25M HF (Th)
- -5 mL 4M HCl

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

Optional Am/Cm rare earth separation.

(10) Add 2 mL 70% HNO_3 + 50 uL 10% H_2SO_4 to Am/Cm. Evaporate to dryness.

(11) Ash to dryness with 3 mL 70% HNO_3 + 2 mL 30% H_2O_2.

(12) Dissolve Am/Cm in 5 mL 4M NH_4SCN-0.1M Formic acid.

(13) Precondition 2 mL TEVA with 5 mL 4M NH_4SCN-0.1M Formic acid.

(14) Load Am/Cm on TEVA.

(15) Rinse Am/Cm beaker with 5 mL 4M NH_4SCN-0.1M Formic acid. Add to TEVA.

(16) Rinse TEVA w/ 10 mL 1.5M NH_4SCN-0.1M Formic acid.

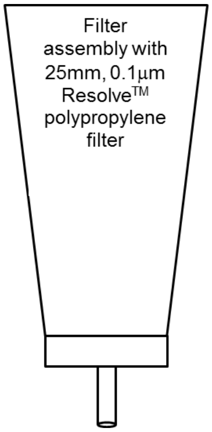
(17) Strip Am/Cm from TEVA with 20 mL 1M HCl.

(18) Add 0.5 mL 30% H_2O_2 to Pu, and 0.2 mL 30% H_2O_2 to Am/Cm samples.

(19) Add 50ug Ce to Pu and Am/Cm samples. Mix well. Add 1 mL 49% HF. Mix well. Wait 15-20 minutes.

(20) Set up Resolve® Filter Funnel on vacuum box.

(21) Wet filter with 3 mL 80% ethanol followed by 3 mL DI water.



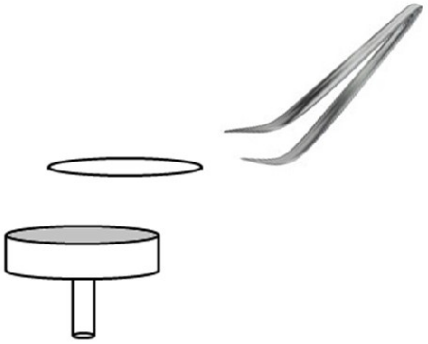
(22) Filter sample.

(23) Rinse sample tube with 5 mL DI water. Add to filter.

(24) Rinse funnel with 3 mL DI water and 2 mL 100 ethanol.

(25) Draw vacuum until filter is dry.

(26) Remove filter from funnel. Mount filter on stainless steel planchet with 2-sided tape.



(27) Dry filter under heat lamp for 3-5 minutes.

(28) Measure actinides by alpha spectrometry.

*Adding 50uL 30% H_2O_2 to the tube rinse can improve Uranium recoveries and decontamination in Pu(Np) fractions.

Method Performance for 1 gram Granite Samples

Analyte	Sample replicates	Reference (mBq/g)	Measured (mBq/g)	Average % Diff.	Tracer % Yield
^{239}Pu	8	29.4	29.2 ± 1.4	4.3	92.1 ± 5.5
^{239}Pu	6	21.2	20.1 ± 1.2	5.4	97.2 ± 5.8
^{238}Pu	6	25.2	25.0 ± 2.2	6.9	97.2 ± 5.8
^{237}Np	6	37.0	37.1 ± 1.7	3.5	97.2 ± 5.8
^{241}Am	4	37.0	37.7 ± 3.3	7.0	90.7 ± 5.1
^{244}Cm	4	33.1	34.4 ± 2.0	5.2	90.7 ± 5.1

References

1) Maxwell, S.L. Culligan, B. Hutchinson, J.B. Sudowe, R. McAlister, D.R. “Rapid Method to Determine Pu, Np, Am/Cm in Granite Samples,” *J. Radioanal. Nucl. Chem.* 140, 102-108 (2018).

eichrom®

AN-1805-10

Alpha Spectrometry Source Preparation: Rare Earth Fluoride Microprecipitation

Summary of Method Rare earth fluoride microprecipitation is an alternative to electrodeposition for alpha spectrometry source preparation, which provides adequate alpha peak resolution for most analytical applications, while greatly reducing the time for sample preparation. Alpha spectrometry sources can often be prepared directly from the eluate used to recover the actinide fraction from the chromatographic column used to separate the actinides from the sample matrix and potentially interfering nuclides. The direct precipitation eliminates the numerous evaporation and digestion steps normally required for electrodeposition, reducing the alpha spectrometry source preparation time from 3-8 hours to 30-60 minutes and eliminating the emission of corrosive acid fumes through the laboratory fume hood vents.

Lanthanum, cerium or neodymium carrier and hydrofluoric acid are normally used to produce the rare earth fluoride precipitate. Ammonium bifluoride may be used instead of HF. Additionally, for laboratories which are restricted from the use of fluoride, $Ce(OH)_4$ precipitation (AN-1807) may be a suitable alternative.

Rare earth fluoride precipitates will nearly quantitatively carry trivalent and tetravalent actinides, while rejecting pentavalent and hexavalent actinides. Therefore, the addition of $TiCl_3$ is required to reduce U(VI) to U(IV) to prepare uranium samples. Samples of the other actinides may be further purified from U during the rare earth fluoride precipitation by the addition of H_2O_2, which will ensure U(VI) that will not be carried on rare earth fluorides.

Eichrom's Resolve Filters (RF-DF25-25PP01) are manufactured specifically for alpha spectrometry source preparation. The manufacture and quality control procedures ensure a uniform surface for the collection of the rare earth fluoride precipitate, reducing self attenuation of the alpha emissions, which can degrade peak resolution. Other filter membranes may not be suitable for alpha source preparation or may require the addition of substrate to achieve adequate resolution.

Sources prepared by rare earth precipitation typically sit closer to the detector in alpha spectrometry systems than electrodeposition sources. The difference in distance from the source to the detector can lead to a 5-10% higher efficiency for the measurement of microprecipitation sources. Since most laboratories calibrate their alpha spectrometry systems with electrodeposited sources, the efficiency difference must be considered when determining the absolute recovery of the chemical yield tracers.

Rare earth fluoride microprecipitation onto Eichrom Resolve Filters produces alpha spectra which are suitable for most analytical applications. However, electrodeposition may be required for some applications, such as the preparation of calibration sources and the measurement of nuclides with difficult to resolve alpha peaks.

Reagents

Lanthanum, Cerium or Neodymium Carrier (10 mg/mL)
HF(49%)
30% H_2O_2
Deionized Water
Denatured Ethanol
10-20% $TiCl_3$ (for Uranium fractions)

Equipment

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Yellow Outer Tips (Eichrom AR-1000-OT)
Resolve Filters in Funnel (Eichrom RF-DF25-25PP01)
50 mL Centrifuge Tubes
Alpha Spectrometry System
Stainless Steel planchets with two sided tape
Heat Lamp
Vacuum Pump

Figure 1. Rare Earth Fluoride Alpha Spectrometry Source Preparation

Uranium Samples*

(1) Obtain a purified sample of U in a 50 mL centrifuge tube using an appropriate separation method. Samples are typically in 10-20 mL of 1M HCl or 0.1M ammonium bioxalate. Other matrices and volumes should be tested prior to application. Concentrations of >0.1M HNO_3 may interfere with uranium reduction by $TiCl_3$ and lead to poor recoveries in the rare earth fluoride precipitation.

(2) Add 0.25 mL of 20% $TiCl_3$ and 100 µg of La, Ce or Nd carrier. Mix.

(3) Add 1 mL 49% HF. Mix well. Wait 15-20 minutes. Proceed to step (4).

Thorium Samples*

(1) Obtain a purified sample of Th in a 50 mL centrifuge tube using an appropriate separation method. Samples are typically in 10-15 mL of 6-9M HCl.

(2) Add 50 µg of La, Ce or Nd carrier. Dillute to 30 mL with DI H_2O. Mix.

(3) Add 1 mL 49% HF. Mix well. Wait 15-20 minutes. Proceed to step (4).

Pu/Np Samples*

(1) Obtain a purified sample of Pu/Np in a 50 mL centrifuge tube using an appropriate separation method. Samples are typically in 15-20 mL of dilute HCl-HF with a reducing agent.

(2) Add 50 µg of La, Ce or Nd carrier and 0.5 mL 30% H_2O_2. Mix.

(3) Add 1 mL 49% HF. Mix well. Wait 15-20 minutes. Proceed to step (4).

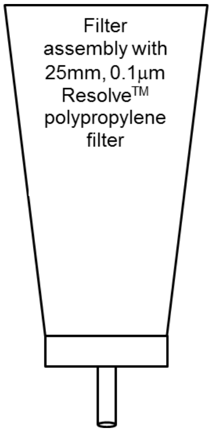
Am/Cm, An(III), and Ln(III) Samples*

(1) Obtain a purified sample of Am/Cm, An(III) or Ln(III) in a 50 mL centrifuge tube using an appropriate separation method. Samples are typically in 15-20 mL of 0.1-4M HCl. Samples with high native rare earth content will require removal of rare earths using TEVA-SCN (AN-1806).

(2) Add 50 µg of La, Ce or Nd carrier and 0.2 mL 30% H_2O_2. Mix.

(3) Add 1 mL 49% HF. Mix well. Wait 15-20 minutes. Proceed to step (4).

(4) Set up Resolve® Filter Funnel on vacuum box.



(5) Wet filter with 3 mL 80% ethanol followed by 3 mL DI water.

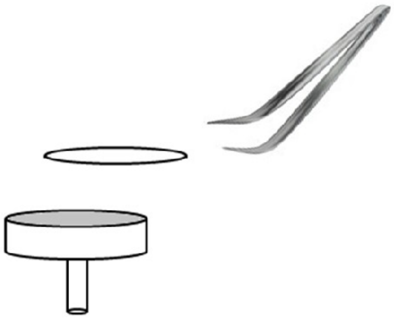
(6) Filter sample.

(7) Rinse sample tube with 5 mL DI water. Add to filter.

(8) Rinse funnel with 3 mL DI water and 2 mL ethanol.

(9) Draw vacuum until filter is dry.

(10) Remove filter from funnel. Mount filter on stainless steel planchet with 2-sided tape.



(11) Dry filter under heat lamp for 3-5 minutes.

(12) Measure actinides by alpha spectrometry.

Typical Performance of CeF_3 Microprecipitation onto Eichrom Resolve Filters

Nuclide	µg Ce	Matrix	Yield	Resolution (FWHM)
^{230}Th	50	30 mL 4.5M HCl	>95%	20-30 keV
$^{238/234}U$	100	20 mL 1M HCl	>95%	30-40 keV
^{239}Pu	50	20 mL 0.1M HCl-0.05MHF-0.01M$TiCl_3$	>95%	30-40 keV
^{241}Am	50	15 mL 4M HCl	>95%	22-28 keV

References

1) Claude W. Sill, "Precipitation of Actinides as Fluorides or Hydroxides for High-Resolution Alpha Spectrometry," Nuclear and Chemical Waste Management, 7, 201-215 (1987).

2) ASTM C1163-14, Standard Practice for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride

eichrom®

AN-1806-10

Actinide/Rare Earth Separation (TEVA-SCN)

Summary of Method Am/Cm or other trivalent actinide(s) are separated from trivalent rare earth cations prior to preparation of rare earth fluoride microprecipitation sources for alpha spectrometry. Some samples (soil, rock, building materials, etc.) may have a high native content of rare earth metal ions, which cannot be adequately separated from the trivalent actinides during the normal analytical scale chromatographic separations used to purify these elements. The mass of the native rare earths can degrade the alpha spectra of the nuclides through mass self-attenuation. For these samples, an additional separation of the actinides from the rare earths using TEVA Resin in the thiocyante (SCN) form will improve the resolution of the alpha spectra.

After purification of the Am/Cm (or other trivalent actinides) on TRU or DGA Resin, the actinide fraction is digested with HNO_3-H_2SO_4, evaporated to dryness, and dissolved in 4M NH_4SCN-0.1M formic acid. The sample is then loaded onto a 2 mL cartridge of TEVA resin, which retains the actinides, while the rare earth elements are not retained. The Am/Cm (or other trivalent actinide) are then recovered in 1M HCl and prepared for alpha spectrometry by rare earth fluoride microprecipitation (AN-1805).

Reagents

TEVA Resin, 2 mL Cartridges (Eichrom TE-R50-S)
Ammonium Thiocyanate (NH_4SCN)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Sulfuric Acid (98%)
Deionized Water
Formic Acid

Equipment

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Cartridge Reservoir, 20 mL (Eichrom AR-200-RV20)
Inner Support Tubes-PE (Eichrom AR-1000-TUBE-PE)
Yellow Outer Tips (Eichrom AR-1000-OT)
50 mL Centrifuge Tubes
Hot Plate
Vacuum Pump

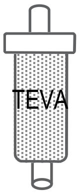
Figure 1. Actinide/Rare Earth Separation on TEVA Resin

(1) Add 2 mL 70% HNO_3 + 50 uL 10% H_2SO_4 to Am/Cm eluate from TRU or DGA Resin separation. Evaporate to dryness.

(2) Ash to dryness with 3 mL 70% HNO_3 + 2 mL 30% H_2O_2.

(3) Dissolve Am/Cm in 5 mL 4M NH_4SCN -0.1M Formic acid.

(4) Precondition 2 mL TEVA cartridge with 5 mL 4M NH_4SCN -0.1M Formic acid.



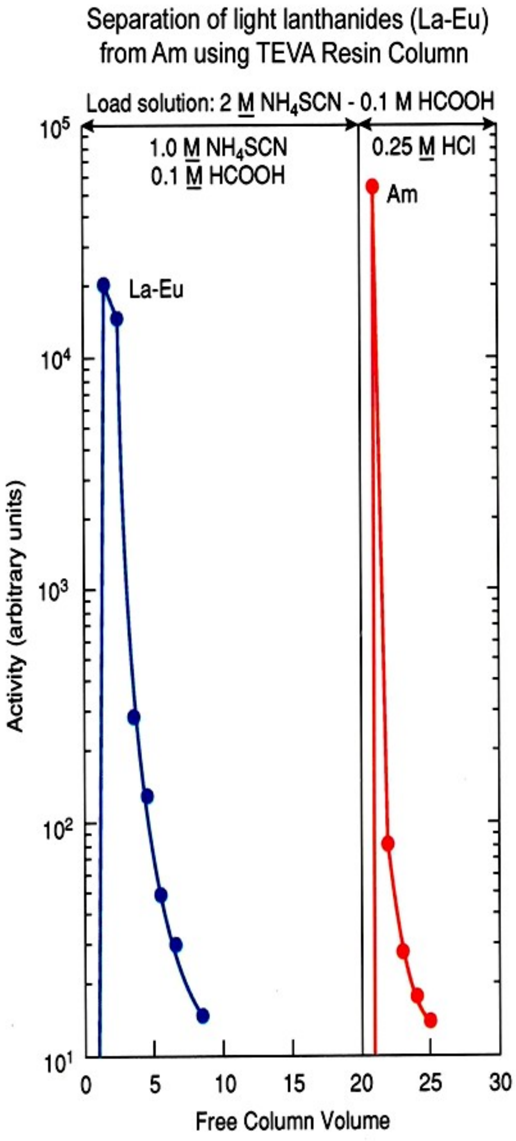
(5) Load Am/Cm from step (3) on TEVA.

(6) Rinse Am/Cm beaker with 5 mL 4M NH_4SCN-0.1M Formic acid. Add to TEVA.

(7) Rinse TEVA w/ 10 mL 1.5M NH_4SCN-0.1M Formic acid.

(8) Strip Am/Cm from TEVA with 20 mL 1M HCl.

(9) Prepare alpha spectrometry source using rare earth fluoride microprecipitation (AN-1805).



References

1) SEPERATION OF AMERICIUM FROM RARE EARTHS, Eichrom Method SPA-03.

eichrom

AN-1807-10

Alpha Spectrometry Source Preparation: Cerium Hydroxide Microprecipitation

Summary of Method Cerium hydroxide microprecipitation is an alternative to rare earth fluoride microprecipitation and electrodeposition for alpha spectrometry source preparation. Microprecipitation provides adequate alpha peak resolution for most analytical applications, while greatly reducing the time for sample preparation relative to electrodeposition. Alpha spectrometry sources can often be prepared directly from the eluate used to recover the actinide fraction from the chromatographic column used to separate the actinides from the sample matrix and potentially interfering nuclides. The direct precipitation eliminates the numerous evaporation and digestion steps normally required for electrodeposition, reducing the alpha spectrometry source preparation time from 3-8 hours to 30-60 minutes and eliminating the emission of corrosive acid fumes through the laboratory fume hood vents.

Cerium hydroxide is an alternative to rare earth fluoride microprecipitation for labs looking to avoid the use of hydrofluoric acid. Cerium hydroxide precipitates will nearly quantitatively carry actinides in all oxidation states. Therefore, the additional U decontamination of Th, Np/Pu and Am/Cm samples achieved by the rare earth fluoride precipitation will not occur using the cerium hydroxide precipitation. The cerium hydroxide precipitate has a yellow color, providing visual confirmation of the collection of the precipitate on the Resolve Filter and easy identification of the surface of the filter containing the precipitate.

Cerium carrier, hydrogen peroxide and a pH indicator are added to each sample fraction from the appropriate separation method. After mixing to distribute the carrier, ammonium hydroxide is added to adjust the pH. The optimal pH and the appropriate pH indicator will depend on the actinide metal ion being collected. U and Th show the highest recovery from pH 5-7, utilizing the bromocresol purple pH indicator. However, U and Th recoveries do not decrease dramatically if the pH is increased to 8-10. Am and Pu/Np recoveries are most consistent utilizing thymol blue, with a color change from pH 8-10. The higher pH range is important to ensure high recoveries of Am. Since Pu and Np are often measured together, with a single ^{236}Pu yield tracer, it is important that their recoveries are very similar. The pH of 8-10 is important to ensure similar recoveries of Pu and Np. At lower or higher pH, Np recovery can diverge significantly from Pu. [2]

Eichrom's Resolve Filters (RF-DF25-25PP01) are manufactured specifically for alpha spectrometry source preparation. The manufacture and quality control procedures ensure a uniform surface for the collection of the rare earth fluoride precipitate, reducing self attenuation of the alpha emissions, which can degrade peak resolution. Other filter membranes may not be suitable for alpha source preparation or may require the addition of substrate to achieve adequate resolution.

Sources prepared by microprecipitation precipitation typically sit closer to the detector in alpha spectrometry systems than electrodeposition sources. The difference in distance from the source to the detector can lead to a 5-10% higher efficiency for the measurement of microprecipitation sources. Since most laboratories calibrate their alpha spectrometry systems with electrodeposited sources, the efficiency difference must be considered when determining the absolute recovery of the chemical yield tracers.

Reagents

Cerium Carrier (10 mg/mL)
30% Hydrogen Peroxide (H_2O_2)
Ammonium Hydroxide (NH_4OH)
Deionized Water
Bromocresol Purple
Denatured Ethanol
Thymol Blue

Equipment

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Yellow Outer Tips (Eichrom AR-1000-OT)
Resolve Filters in Funnel (Eichrom RF-DF25-25PP01)
Stainless Steel planchets with two sided tape
Alpha Spectrometry System
Heat Lamp
50 mL Centrifuge Tubes
Vacuum Pump

Figure 1. Cerium Hydroxide Alpha Spectrometry Source Preparation*

Uranium Samples

(1) Obtain a purified sample of U in a 50 mL centrifuge tube using an appropriate separation method. Samples are typically in 10-20 mL of 1M HCl.

(2) Add 25-50 μg of Ce carrier, 0.2 mL of 30% H_2O_2 and bromocresol purple. Mix.

(3) Adjust to pH 5-7 (blue/purple color). Mix well. Proceed to step (4).

Thorium Samples

(1) Obtain a purified sample of Th in a 50 mL centrifuge tube using an appropriate separation method. Samples are typically in 20 mL of 6-9M HCl.

(2) Add 25-50 μg of Ce carrier, 0.2 mL of 30% H_2O_2 and bromocresol purple. Mix.

(3) Adjust to pH 5-7 (blue/purple color). Mix well. Proceed to step (4).

Pu/Np Samples

(1) Obtain a purified sample of Pu/Np in a 50 mL centrifuge tube using an appropriate separation method. Samples are typically in 15-20 mL of dilute HCl-HF with a reducing agent.

(2) Add 25-50 μg of Ce carrier, 0.2 mL of 30% H_2O_2 and thymol blue. Mix.

(3) Adjust to pH 8-10 (light blue color). Mix well. Proceed to step (4).

Am/Cm, An(III), and Ln(III) Samples

(1) Obtain a purified sample of Am/Cm, An(III) or Ln(III) in a 50 mL centrifuge tube using an appropriate separation method. Samples are typically in 15-20 mL of 0.1-4M HCl. Samples with high native rare earth content will require removal of rare earths using TEVA-SCN (AN-1806).

(2) Add 25-50 μg of Ce carrier, 0.2 mL of 30% H_2O_2 and thymol blue. Mix.

(3) Adjust to pH 8-10 (light blue color). Mix well. Proceed to step (4).

(4) Set up Resolve® Filter Funnel on vacuum box.

(5) Wet filter with 3 mL 80% ethanol followed by 3 mL DI water.

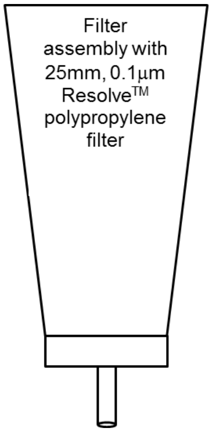
(6) Filter sample.

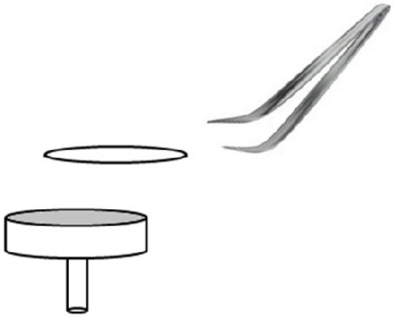
(7) Rinse sample tube with 5 mL DI water. Add to filter.

(8) Rinse funnel with 3 mL DI water and 2 mL ethanol.

(9) Draw vacuum until filter is dry.

(10) Remove filter from funnel. Mount filter on stainless steel planchet with 2-sided tape.





(11) Dry filter under heat lamp for 3-5 minutes.

(12) Measure actinides by alpha spectrometry.

***Results in Table 1 are for typical sample matrix from separation method. Performance with other matrices or volumes should be verified prior to implementation.**

Typical Performance of $Ce(OH)_4$ Microprecipitation onto Eichrom Resolve Filters

Nuclide	pH	μg Ce	Matrix	Yield	Resolution (FWHM)
^{230}Th	5-7	25	20 mL 9M HCl	95-99%	25-35 keV
$^{238/234}$U	5-7	25	20 mL 1M HCl	93-97%	25-35 keV
^{237}Np	8-10	25	20 mL 0.15M HCl-0.05M KF-0.04M Rongalite	89-93%	25-35 keV
^{239}Pu	8-10	25	20 mL 0.15M HCl-0.05M KF-0.04M Rongalite	86-92%	25-35 keV
^{241}Am	8-10	25	15 mL 4M HCl	91-95%	25-35 keV

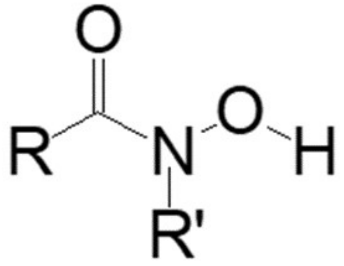
References

1) Claude W. Sill, “Precipitation of Actinides as Fluorides or Hydroxides for High-Resolution Alpha Spectrometry,” Nuclear and Chemical Waste Management, 7, 201-215 (1987).

2) Hiromu Kurosaki, Rebbeca J. Mueller, Susan B. Lambert, Govind R. Rao, “Alternate method of source preparation for alpha spectrometry: no electrodeposition, no hydrofluoric acid,” *J. Radioanal. Nucl. Chem.*, 311, 323-329 (2017).

AN-1808-10

Zirconium Separation on ZR Resin



Hydroxamate Extractant

Summary of Method ZR resin contains a hydroxamate extractant which exhibits a high selectivity for Zr(IV), Ti(IV) and Nb(V) over Y(III), Sc(III) and Fe(III). From 0.01-10M HCl, Zr, Ti and Nb are strongly retained by the ZR resin, while Y and Sc are poorly retained. Fe(III) is strongly retained from 0.01-1M HCl and can be eluted from the ZR resin with 2-3M HCl. Zr can be recovered from the ZR resin with 0.1M oxalic acid, while Ti and Nb elution requires >0.25M oxalic acid.

The unique selectivity of ZR resin makes it a useful material for the separation of emerging PET nuclides from their target materials, such as Zr(IV) from Y(III) and Ti(IV) from Sc(III). The target materials can be dissolved in high concentrations of hydrochloric acid and the dissolved target loaded onto ZR resin. Zr(IV) or Ti(IV) is retained, while the bulk target mass, Y(III) or Sc(III) passes through the ZR resin. Rinsing the ZR with 2-10M HCl completes removal of the target material and any Fe(III) present in the sample. The ZR resin can then be rinsed with more dilute HCl to reduce the residual acidity, Zr(IV) can be stripped using 0.1M oxalic acid, and Ti(IV) can be stripped with 0.25M oxalic acid. Further purification of the Zr(IV) or Ti(IV) can be achieved by loading the Zr(IV) or Ti(IV) onto strong base anion exchange resin from dilute oxalic acid-HCl.

The easily hydrolyzed Zr(IV), Ti(IV) and Nb(V) should be stored in solution containing trace HF or oxalic acid to prevent loss of material to vials or formation of colloidal aggregates.

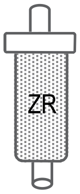
Reagents

ZR Resin 2 mL Cartridges (Eichrom ZR-R10-S)
1 mL Cartridges (Eichrom ZR1-R10-S)
0.3 mL Cartridges (Eichrom ZR0.3-R10-S)
Bulk Resin (Eichrom ZR-B25-S)
Hydrochloric Acid (37%)
Oxalic Acid
Deionized Water
Hydrofluoric Acid (49%) - Optional

Equipment

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Cartridge Reservoir, 20 mL (Eichrom AR-200-RV20)
Inner Support Tubes-PE (Eichrom AR-1000-TUBE-PE)
Yellow Outer Tips (Eichrom AR-1000-OT)
50 mL Centrifuge Tubes
Vacuum Pump

Zirconium Separation on ZR Resin and Anion Exchange



(1) Precondition 2 mL ZR Resin cartridge with 10 mL 6M HCl.

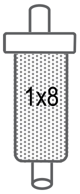
(2) Load 10-100 mL sample in 6M HCl. Zr is retained. Y(III) elutes.

(3) Rinse column with 25 mL 2M HCl. Add 0.1M ascorbic acid to improve Fe decontamination.

(4) Rinse column with 10 mL 2M HCl.

(5) Strip Zr with 15 mL 0.05M oxalic acid.

(6) Precondition 1 mL 1x8 cartridge with 10 mL 0.05M oxalic acid-0.05M HCl.



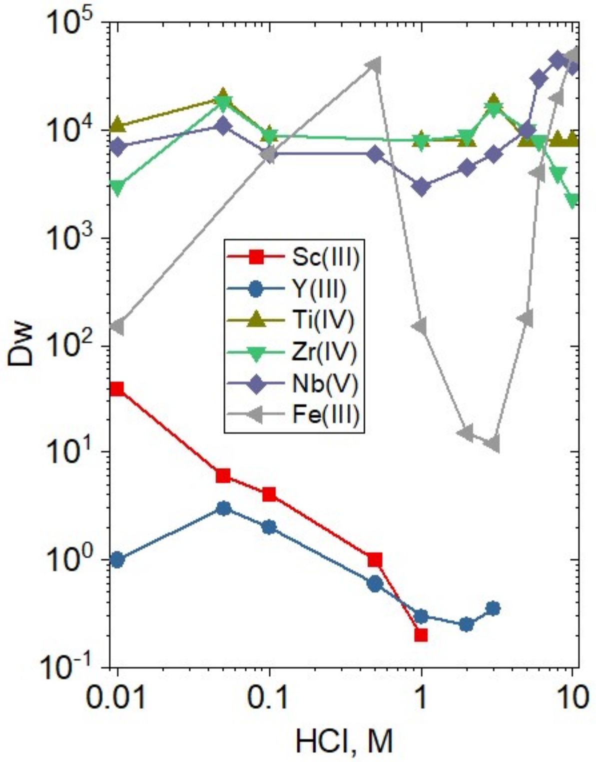
(7) Add 5 mL 0.1M HCl to sample from step (5). Mix.

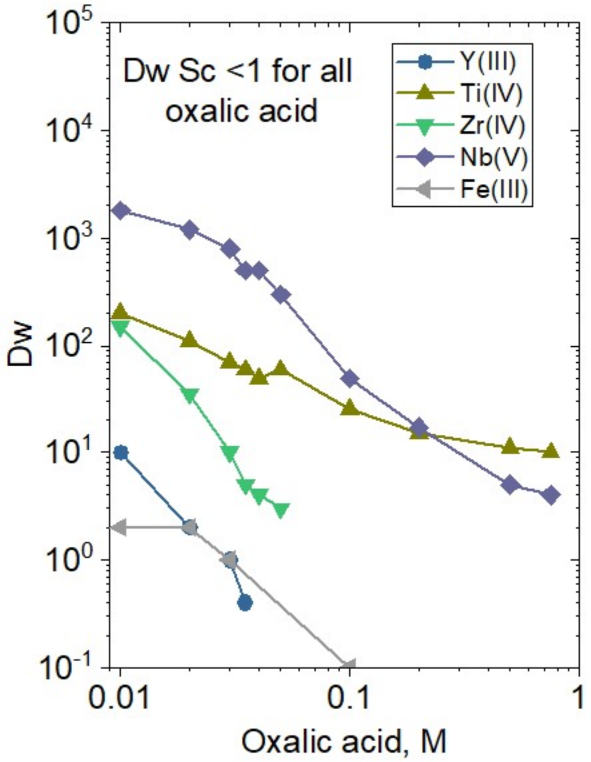
(8) Load onto 1 mL 1x8 cartridge.

(9) Rinse cartridge with 20 mL 0.05M oxalic acid-0.05M HCl.

(10) Rinse cartridge with 5 mL 37% HCl.

(11) Strip ^{89}Zr with 5 mL 2-4M HCl.


Dw on Zr resin vs HCl.


Dw on Zr resin vs oxalic acid.

Dw Sc <1 for all oxalic acid

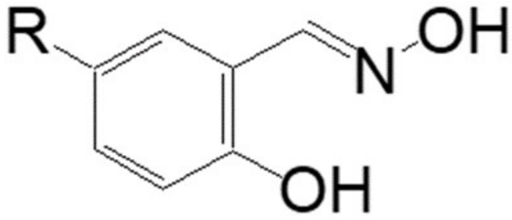
References

1) Dirks, et al., “On the Development and Characterization of a hydroxamate based extraction chromatographic resin,” 61st Radiobioassy and Radiochemical Measurements Conference, October 25-30, 2015, Iowa City, Iowa.

2) Triskem INFOS, No 15, February 2016. http://www.triskem-international.com/scripts/files/59d1f4fc31f796.50370140/tki_15_en_web.pdf

AN-1809-10

Copper Separation on CU Resin



Benzaldoxime extractant

Summary of Method CU Resin contains a benzaldoxime extractant adsorbed on an inert polymeric support. CU resin can be used to separate copper from other transition metals, such as zinc or nickel target material used in the production of Cu-64 and Cu-67. CU resin will selectively retain Cu from pH 2-5 HCl, HNO_3 or H_2SO_4, while Ni(II), Zn(II), Cd(II), Co(II), Fe(II), Fe(III), and Cr(III) are rejected. Cu can then be recovered from the Cu resin using 1-8 M HCl. Additional purification of Cu can be achieved by stripping the Cu resin with 8M HCl through a strong base anion exchange resin (AG1x8). The Cu will be retained on the AG1x8 and can then be recovered in dilute HCl.

The CU is very hydrophobic and can be difficult to wet in dilute acid. Soaking the CU resin in 2M HCl improves the wetting. However, the wetted resin will still float on top of the liquid, making it difficult to slurry pack the CU resin. It is therefore recommended that the CU resin be used in prepacked cartridges or dry packed columns. Wet the columns or cartridges with 5-10 bed volumes of 2M HCl and then precondition the CU resin with dilute acid prior to loading the Cu sample. To initiate flow on the dry packed column or cartridge, a vacuum box, peristaltic pump or luer syringe will be required.

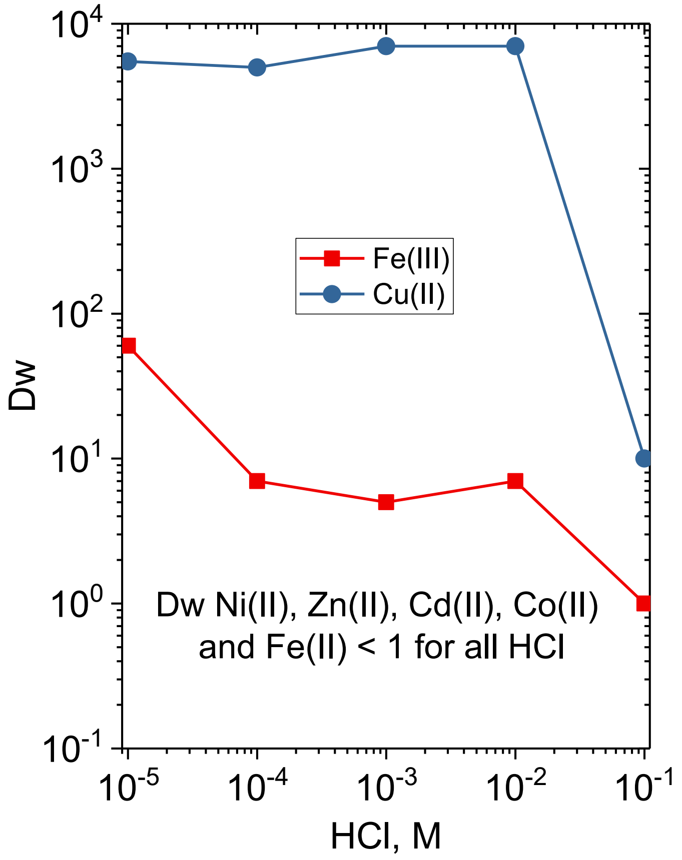
Reagents

CU Resin
  2 mL Cartridges (Eichrom CU-R10-S)
  1 mL Cartridges (Eichrom CU1-R10-S)
  25 g bulk resin, 100-150 μm (Eichrom CU-B25-A)
  25 g bulk resin, 50-100 μm (Eichrom CU-B25-S)
Anion Exchange Resin (Eichrom A8-B500-F-CL)
Hydrochloric Acid (37%)
Ammonium Hydroxide (56%)
Deionized Water

Equipment

Vacuum Box (Eichrom AR-24-BOX or AR-12-BOX)
Cartridge Reservoir, 20 mL (Eichrom AR-200-RV20)
Inner Support Tubes-PE (Eichrom AR-1000-TUBE-PE)
Yellow Outer Tips (Eichrom AR-1000-OT)
50 mL Centrifuge Tubes
Vacuum Pump

Dw on CU Resin vs HCl

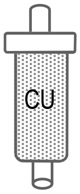


Dw Ni(II), Zn(II), Cd(II), Co(II) and Fe(II) < 1 for all HCl

Figure 1. Cu Separation

(1) Dissolve Cu sample in HCl. Evaporate to dryness. Dissolve in 0.001M HCl. Adjust to pH 2-3 as necessary.*

(2) Wet 1 mL CU resin cartridge with 10 mL 2M HCl.



(3) Precondition CU resin with 10 mL 0.01M HCl.

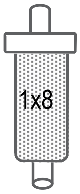
(4) Load sample.

(5) Rinse CU resin with 10 mL 0.01M HCl.

(6) Strip Cu with 2-3 mL 8M HCl.

*Sulfate may also be used and may provide buffering capacity, simplifying the pH adjustment.

(7) Precondition 1 mL cartridge of 1x8 with 5 mL 8M HCl.



(8) Load sample from step (6).

(9) Rinse 1x8 with 2 mL 8M HCl.

(10) Strip Cu with 1-3 mL 0.01M HCl.

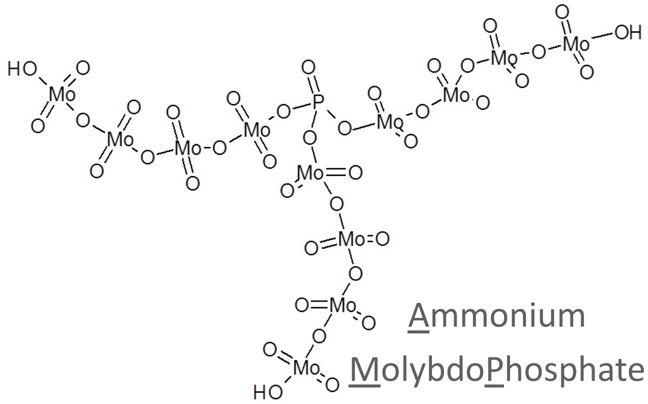
References

1) C. Dirks, B. Scholten, S. Happel, A. Zulauf, A. Bombard, H. Jungclas, “Characterization of a Cu selective resin and its application to the production of 64Cu,” *J. Radioanal, Nucl. Chem.*, 286, 671-674 (2010).

2) Triskem INFOS, No 6, July 2011. http://www.triskem-international.com/scripts/files/59d1f4fc2c2091.54193347/tki6_en_binderonline_1.pdf

AN-1810-10

Cs Separation on AMP-PAN and KNiFC-PAN Resins



AMP-PAN contains an inorganic ion exchange material (ammonium molybdophosphate, AMP) dispersed in an inert polymeric support (polyacrylonitrile, PAN). The AMP has been shown to exhibit high selectivity for Cs from a wide range of solutions, including high acid concentrations and high salt concentrations (sea water). The AMP is imbedded into the PAN to improve the flow characteristics of packed columns. The material exhibits fast kinetics and high radiation stability, with no change in uptake observed for radiation doses of up to 1000 kGy [1]. Recovery of Cs from the AMP-PAN resin requires elution with 10 bed volumes of 5M NH_4Cl or NH4NO3.

AMP-PAN has been used to remove Cs-137 from radioactive acidic waste streams containing high levels of sodium and potassium [2]. Actual waste and waste simulants were loaded onto 1.5 mL columns at 0.7 mL/min. In the first cycle, 0.15% breakthrough of Cs-137 was measured after 1500 mL of feed (99.85% Cs-137 removal). After regenerating the column by eluting Cs-137 with 50 mL of 5M NH_4NO_3, 0.53% breakthrough of Cs-137 was measured after 1250 mL of feed (99.47% removal of Cs-137). Average recovery of Cs-137 in the 5M NH_4NO_3 regeneration cycles was 87%.

AMP-PAN has also been used to recover Cs-137 from sea water samples [3]. 5 mL columns of AMP-PAN were used to process 20 L samples of sea water which had been acidified to pH 1-2. Stable Cs, measured by ICP-MS, was used to trace the chemical recovery during the column separation. Flow rates of 35 mL/min were used. Recovery of Cs was 93.5 ± 5.0%.

Reagents

AMP-PAN Resin
  5 mL Cartridges (HC5-R10-M)
  2 mL Columns (HC-C50-M)
  5 mL Columns (HC5-C20-M)
  8 mL Columns (HC8-C20-M)
  10 mL Columns (HC10-C20-M)
Nitric Acid (70%)
Deionized Water
Ammonium chloride or Ammonium Nitrate

Equipment

Vacuum Box (AR-24-BOX or AR-12-BOX)*
Cartridge Reservoir, 20mL (AR-200-RV20)*
Inner Support Tubes-PE (AR-1000-TUBE-PE)*
Yellow Outer Tips (AR-1000-OT)*
50 mL Centrifuge Tubes
Vacuum Pump*

*Or appropriately sized gravity flow column and accessories (see reverse).

References

1) F. Sebesta, V. Stefula, "Composite ion exchanger with ammonium molybdophosphate and its properties" *J. Radioanal, Nucl. Chem.*, 140(1), 15-21 (1990).

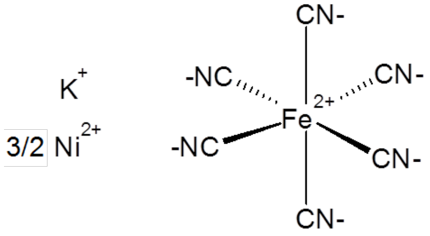
2) Brewer, et al. "AMP-PAN column tests for the removal of Cs-137 from actual and simulated INEEL high-activity wastes," *Czechoslov J Phys*, 49(S1), 959-964 (1999).

3) Pike, et al. "Extaction of cesium in seawater off Japan using AMP-PAN resin and quantification via gamma spectroscopy and inductively coupled plasma mass spectrometry," *J Radioanal Nucl Chem*, 296(1), 369-374 (2012).

4) Triskem INFOS, No 10, July 2013. http://www.triskem-international.com/scripts/files/59d1f4fc2ec7b3.42683976/tki10_binder_en_web.pdf

KNiFC-PAN contains an inorganic ion exchange material (potassium nickel ferrocyanate, KNiFC) dispersed in an inert polymeric support (polyacrylonitrile, PAN). The KNiFC has been shown to exhibit high selectivity for Cs from a wide range of solutions, including sea water and other environmental waters. The KNiFC is imbedded into the PAN to improve the flow characteristics of packed columns.

KNiFC-PAN has been used to remove cesium from sea water samples [5]. 100 L samples of sea water were processed through 25 mL columns of KNiFC-PAN at flow rates of up to 300 mL/min. Stable Cs was added as a yield tracer (measured by ICP-MS). Yields for cesium were 92.9 ± 1.1% for 100 L samples of sea water acidified to pH 1. For 100 L samples of sea water (unacidified), cesium yields were 90.2 ± 2.7%.



Potassium Nickel FerroCyanate (KNiFC)

Reagents

KNiFC-PAN Bulk Resin (NC-B50-M)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Deionized Water

Equipment

Empty Columns
  2 mL snap tip (AC-141-AL)
  2 mL cap tip (AC-100-MT-PP)
  5 mL (AC-50E-5M)
  20 mL (AC-20E-20M)
Column Reservoir
  For 2 mL columns (AC-120-TK)
  250 mL For 5 and 20 mL columns (AC-20X-20M)
Column Rack
  15 hole for 2 mL columns (AC-103)
  12 hole for 5 and 20 mL columns (AC-20M-RACK)
50 mL Centrifuge Tubes

Comparison of AMP-PAN and KNiFC-PAN Resins

Paramater	AMP-PAN	KNiFC-PAN
Cs Capacity	64 mg / g dry resin	256 mg / g dry resin
Density	0.27 g/mL	0.20 g/mL
Recommended sample pH	1 - 2	1 - 7
Sample Types	Waste, Environmental	Environmental
Regeneration	5M NH_4Cl or NH_4NO_3	None

References

5) Kamenik, et al. “Fast concentration of dissolved forms of cesium radioisotopes from large sea water samples,” *J. Radioanal. Nucl. Chem.*, 292(2), 841-846 (2012)

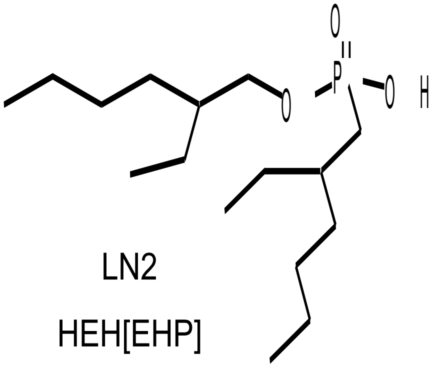
eichrom®

AN-1811-10

Ce Separation from Rare Earth Nitrate Solutions

Summary of Method Cerium is oxidized from Ce(III) to Ce(IV) using sodium bromate and then selectively extracted from rare earth nitrate solutions using a column of LN2 resin. LN2 is an extraction chromatographic resin containing 2-ethyl-1-hexyl(2-ethyl-1-hexyl)phosphonic acid (HEH[EHP] .

Ce can be oxidized to Ce(IV) from solutions of nitric acid and rare earth nitrate using $NaBrO_3$, while the other rare earth metal ions remain in the trivalent oxidation state. The oxidation of Ce(III) to Ce(IV) and the retention on LN2 increases with the concentration of nitrate. The oxidation will not work from chloride solutions. Berkelium (Bk) can also be oxidized to Bk(IV) and separated from other trivalent actinides and rare earths using very similar conditions.

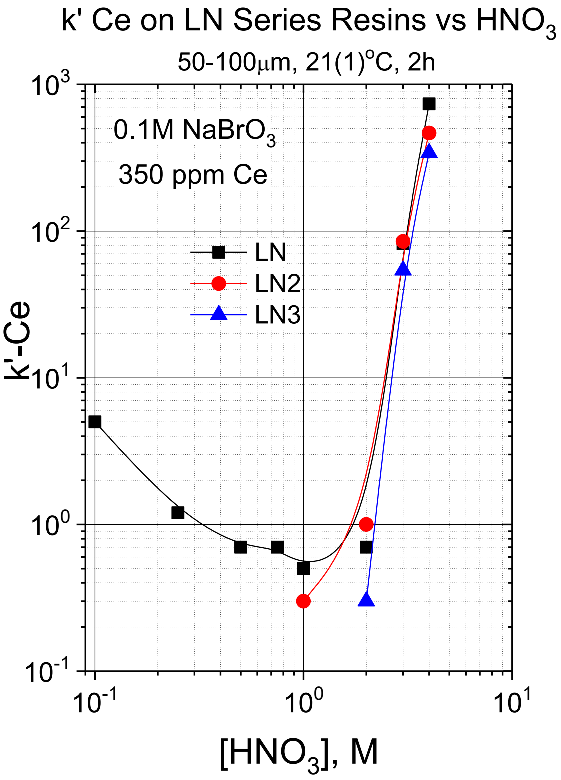

LN2

HEH[EHP]

Once oxidize, the Ce(IV) or Bk(IV) are retained on the LN2 resin from 2-3M HNO_3/Rare Earth Nitrate solutions, while trivalent metal ions are not retained. After rinsing with HNO_3 to remove any residual trivalent metal ions, the Ce or Bk can be recovered from the LN2 by elution with 0.25-0.50M HCl or HNO_3 + reducing agent (H_2O_2, hydroxylamine or ascorbic acid). Removal of Ce from 500 mL 2M HNO_3 + 0.75 M Y/Yb$(NO_3)_3$ was >99.9% using a 10 mL column of LN2 resin[1].

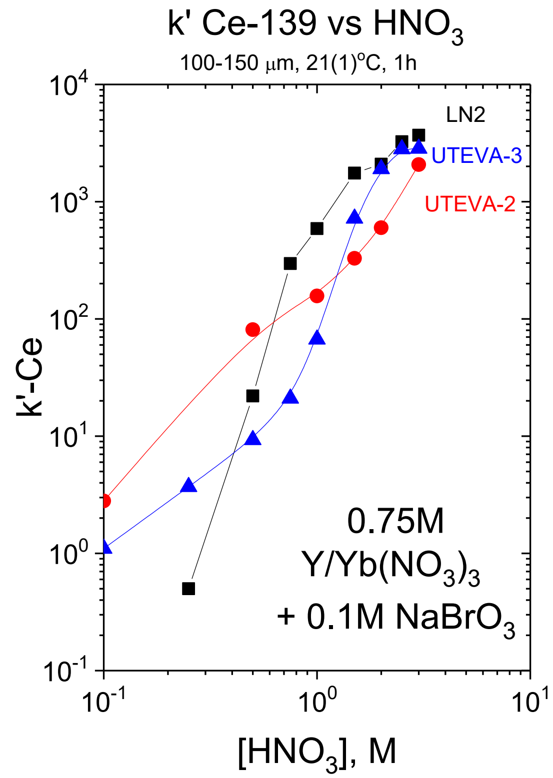
Reagents

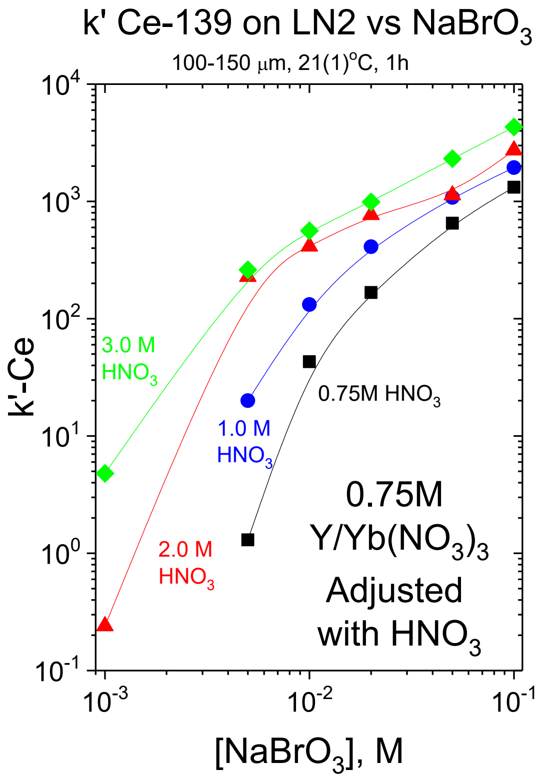
LN2 Bulk Resin (L2-BO1-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Sodium Bromate ($NaBrO_3$)
H2O2 (30%), Hydroxylamine·HCl or Ascorbic Acid
Deionized Water

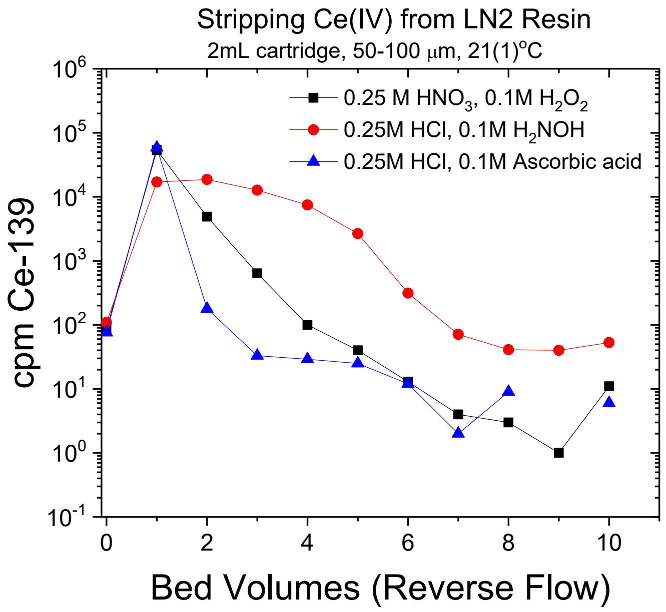


Equipment

Empty Columns
  2 mL snap tip (AC-141-AL)
  2 mL cap tip (AC-100-MT-PP)
  5 mL (AC-50E-5M)
  20 mL (AC-20E-20M)
Column Reservoir
  For 2 mL columns (AC-120-TK)
  250 mL For 5 and 20 mL columns (AC-20X-20M)
Column Rack
  15 hole for 2 mL columns (AC-103)
  12 hole for 5 and 20 mL columns (AC-20M-RACK)
50 mL Centrifuge Tubes





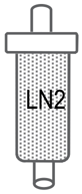


Ce Separation

(1) Adjust rare earth sample to 2-3M HNO_3.

(2) Add enough $NaBrO_3$ to make 0.05-0.10M.

(3) Precondition LN2 resin with 5 bed volumes of 2M HNO_3-0.05M $NaBrO_3$.



(4) Load sample.

(5) Rinse LN2 with 5-10 bed volumes of 2-3M HNO_3-0.05M $NaBrO_3$.

(6) Rinse LN2 with 2 bed volumes of 2-3M HNO_3.

(7) Strip Ce with 5-10 bed volumes of 0.5M HCl + 0.1M reducing agent.

References

1) D.R. McAlister and E.P. Horwitz, unpublished data (2013).

AN-1812-10

Fe Separation from Rare Earth Chlorides

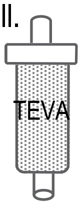
Summary of Method Fe(III) is removed from rare earth chloride solutions by extraction of $[FeCl_4]^-$ on TEVA resin. The anionic ferric chloride complex is strongly retained by the TEVA Resin, while the rare earth chlorides are rejected. Hydrogen peroxide is added to the sample to ensure Fe(III), as Fe(II) is not extracted. The TEVA column can be regenerated by eluting Fe with five bed volumes of 0.1M HNO_3. 99.7% removal of Fe from 500 mL 0.75M YCl_3-1M HCl was achieved on a 10 mL column of TEVA resin (3 mL/min flowrate) [1].

Reagents

TEVA Bulk Resin (TE-B25-S)
Nitric Acid (70%)
Hydrochloric Acid (37%)
Hydrogen Peroxide (30% H_2O_2)
Deionized Water

Tc Separation on WBEC Resin

(1) Add 1-2 mL 30% H_2O_3 per 100 mL of sample to ensure Tc(VII). Adjust to 0.01M HNO_3. Mix well.



(2) Precondition WBEC column with 3 bed volumes of 0.01M HNO_3.

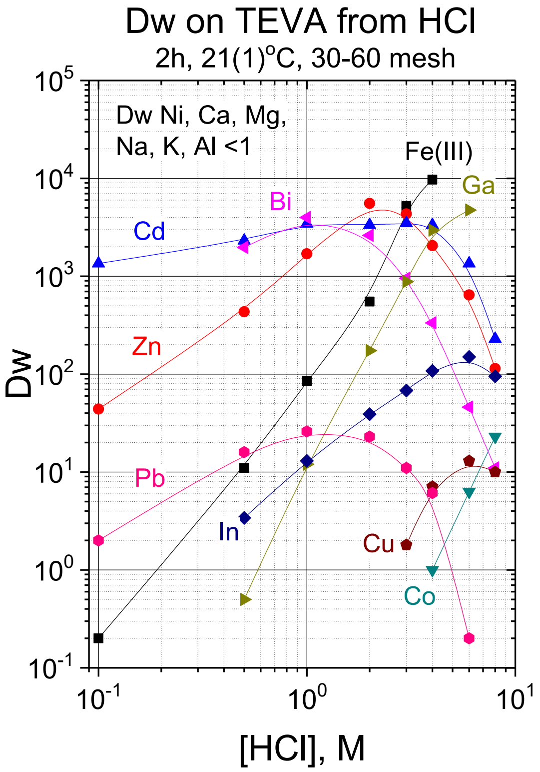
(3) Load Sample.

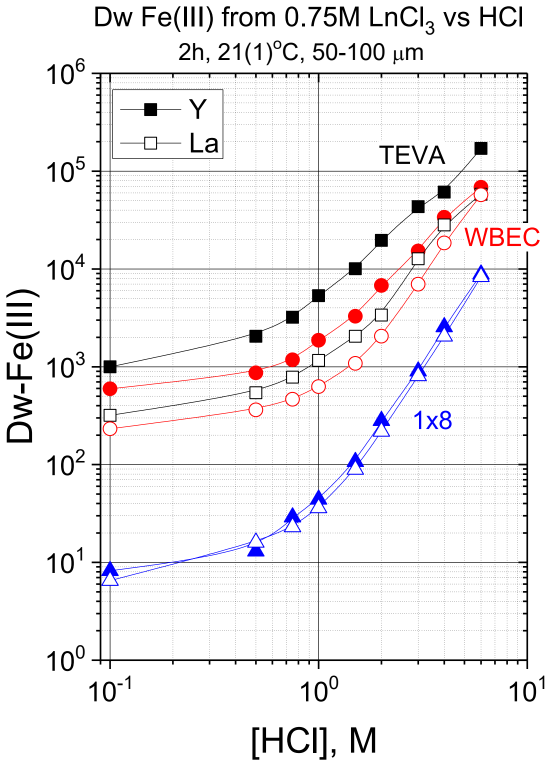
(4) Rinse column with 10 bed volumes of 0.01M HNO_3.

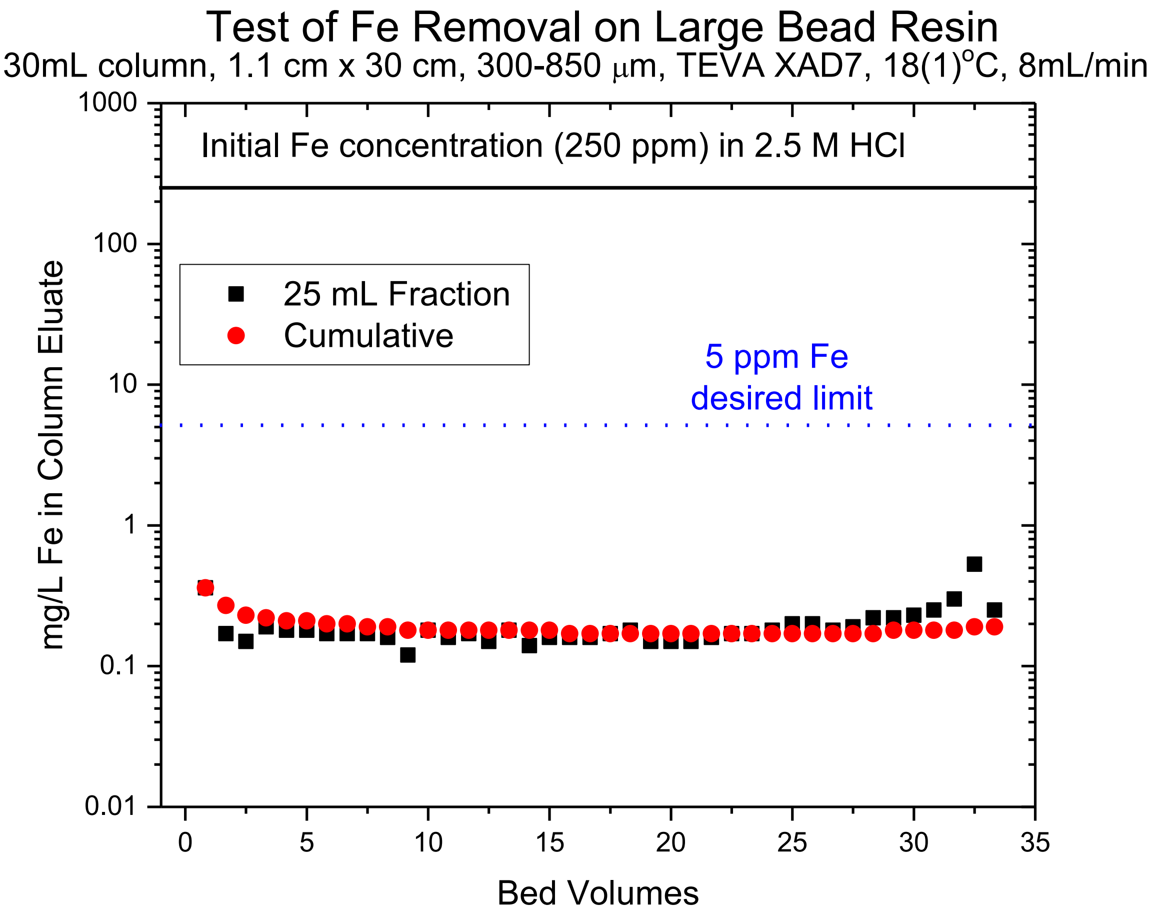
(5) Strip Tc with 5 bed volumes of 1M NH_4OH.

Equipment

- Empty Columns
  - 2 mL snap tip (AC-141-AL)
  - 2 mL cap tip (AC-100-MT-PP)
  - 5 mL (AC-50E-5M)
  - 20 mL (AC-20E-20M)
- Column Reservoir
  - For 2 mL columns (AC-120-TK)
  - 250 mL For 5 and 20 mL columns (AC-20X-20M)
- Column Rack
  - 15 hole for 2 mL columns (AC-103)
  - 12 hole for 5 and 20 mL columns (AC-20M-RACK)
- 50 mL Centrifuge Tubes







Test of Fe Removal on Large Bead Resin

30mL column, 1.1 cm x 30 cm, 300-850 μm, TEVA XAD7, 18(1)^{o}C, 8mL/min

References

1) D.R. McAlister and E.P. Horwitz, unpublished data (2013).

AN-1813-10

Tc Separation on WBEC Resin

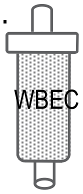
Summary of Method Pertechnetate, Tc(VII), is removed from dilute acid solution with WBEC Resin. The WBEC resin contains a tertiary amine exractant (Alamine 336) on an inert polymeric support. The Alamine 336 acts as an anion exchanger when protonated in dilute acidic media. However, from basic media, the Alamine 336 is deprotanated and no longer acts as an anion exchanger. This behavior allows anions, such as pertechnetate to be efficiently stripped from the WBEC resin using 1M NH_4OH, whereas a quaternary amine, such as Aliquat 336 (TEVA) will continue to act as an anion exchanger from basic media and requires 8-10 M HNO_3 to strip pertechnetate.

Reagents

WBEC Bulk Resin (WB-B25-S)
Nitric Acid (70%)
Hydrogen Peroxide (30% H_2O_2)
Ammonium Hydroxide (56%)

Tc Separation on WBEC Resin

(1) Add 1-2 mL 30% H_2O_2 per 100 mL of sample to ensure Tc(VII). Adjust to 0.01M HNO_3. Mix well.



(2) Precondition WBEC column with 3 bed volumes of 0.01M HNO_3.

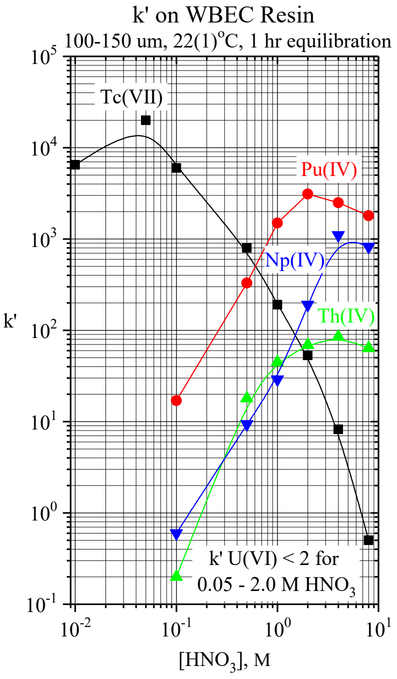
(3) Load Sample.

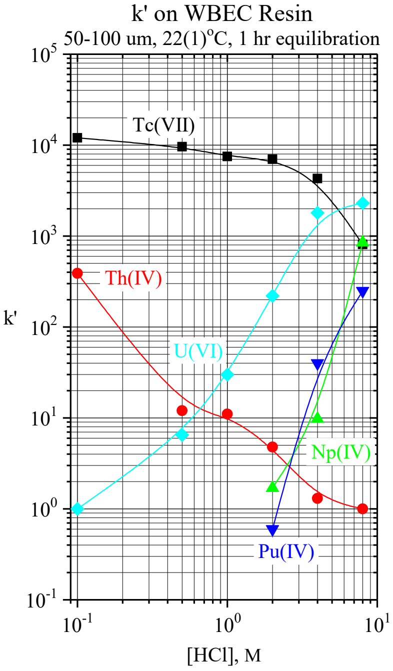
(4) Rinse column with 10 bed volumes of 0.01M HNO_3.

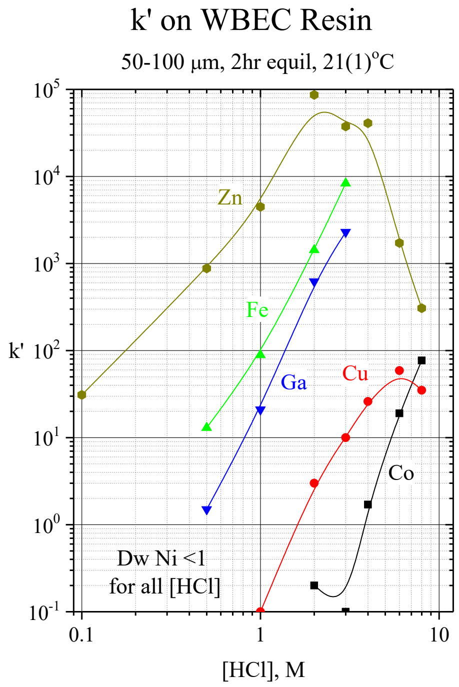
(5) Strip Tc with 5 bed volumes of 1M NH_4OH.

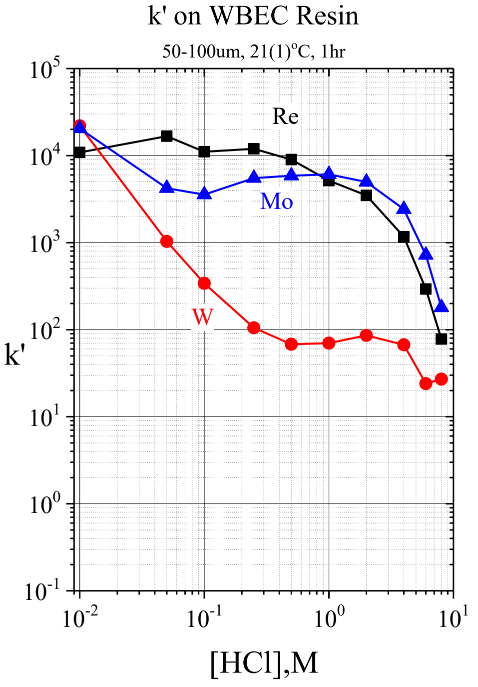
Equipment

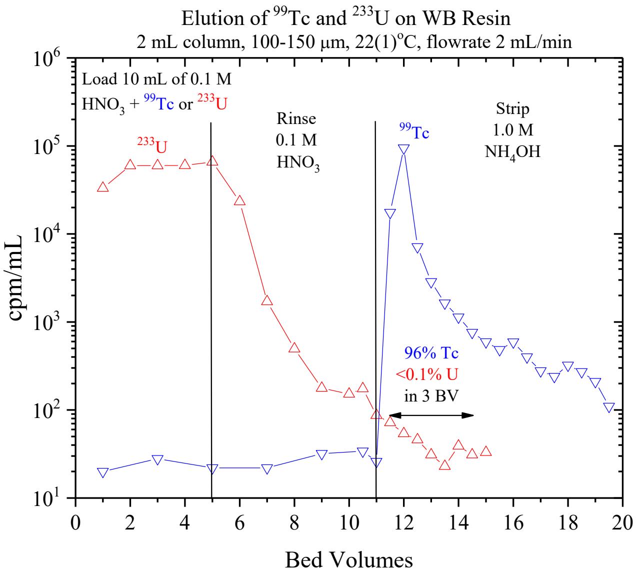
Empty Columns
  2 mL snap tip (AC-141-AL)
  2 mL cap tip (AC-100-MT-PP)
  5 mL (AC-50E-5M)
  20 mL (AC-20E-20M)
Column Reservoir
  For 2 mL columns (AC-120-TK)
  250 mL For 5 and 20 mL columns (AC-20X-20M)
Column Rack
  15 hole for 2 mL columns (AC-103)
  12 hole for 5 and 20 mL columns (AC-20M-RACK)
50 mL Centrifuge Tubes

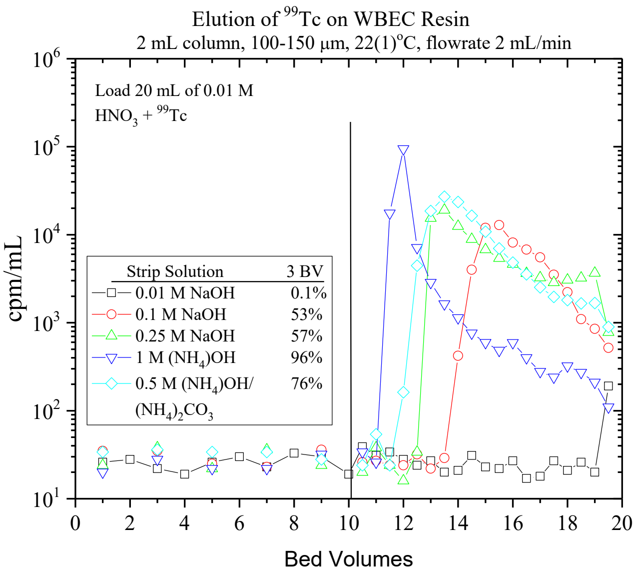












References

G.D. Jarvinen, K.M. Long, G.S. Goff, W.H. Runde, E.J. Mausolf, K.R. Czerwinski, F. Poineau, D.R. McAlister, E.P. Horwitz, “Separation of Pertechnetate from Uranium in a Simulated UREX Processing Solution Using Anion Exchange Extraction Chromatography,” Solv. Extr. Ion Exch., 31, 416-429, (2013).