

New QC Procedure for Eichrom Resins: How We're Improving

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Eichrom Technologies, Inc.

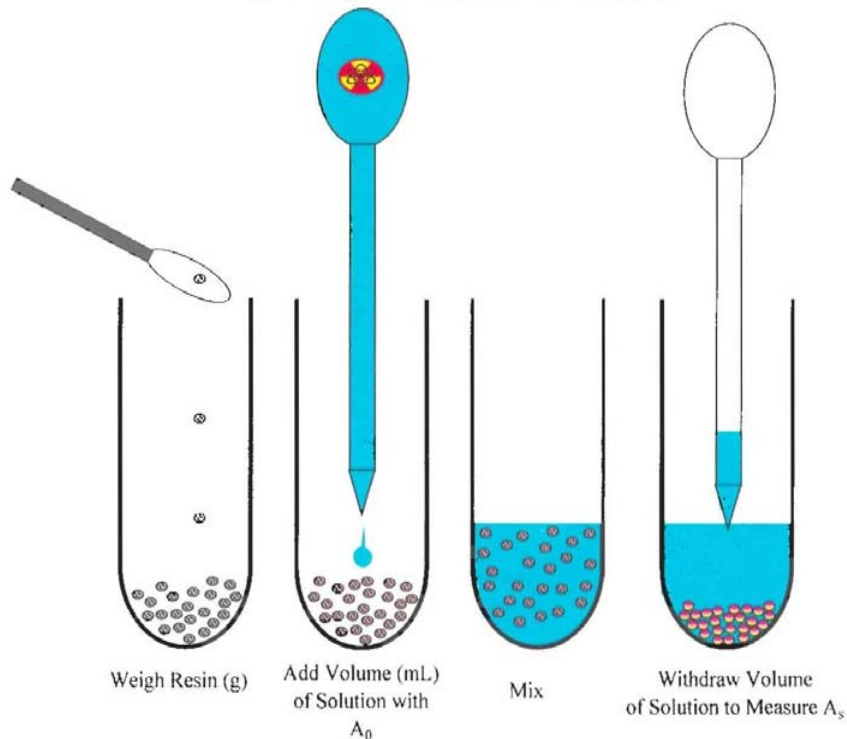
Eichrom's Quality Goals

as part of our ISO 9001:2000 certification

1. Ship >98% of orders (non-specials) next business day
 - Has been achieved since implementation in 2003
 - 99.0% achieved (YTD)
2. Customer satisfaction rating >4 (5 is max.)
 - >4.50 for the past 3 years
3. Implement 1 or more improvements quarterly (2005 highlights)
 - Completed cartridge cap mold modifications
 - New column frit with non-ionic surfactant obtained
 - Improved vacuum box liner design with single piece mold
 - Installed new equipment for resin manufacturing

Since the formation of Eichrom, an essential feature of our quality control system was the measurement of D_w for each batch of new resin manufactured.

Dry Weight Distribution Ratio



$$D_w = \frac{A_0 - A_s}{w(g)} \bigg/ \frac{A_s}{v(\text{mL})}$$

D_w was measured under conditions in which the analyte is strongly retained by the resin and weakly retained by the resin.

What do these types of measurements tell you?

1. Did you use the correct extractant?
2. Did you use the correct quantity of extractant?

What D_w does not tell you.

- How completely will an analyte be retained by the column? In other words, how much breakthrough will occur?
- When exactly will an analyte elute from the column?
- How efficiently will an analyte elute from the column? (% recovery)
- How much cross contamination will occur between two or more analytes?

1) Support Resin

a) Flow rate test

- gravity flow rate specs for A established

- specs for S grade using vacuum are under development

b) Elzone analysis

- correlation of particle size distribution to flow rate being evaluated

2) Extractants

a) solvent extraction test

- helps to determine purity of extractant
- CMPO and TBP done
- Aliquat 336 in development

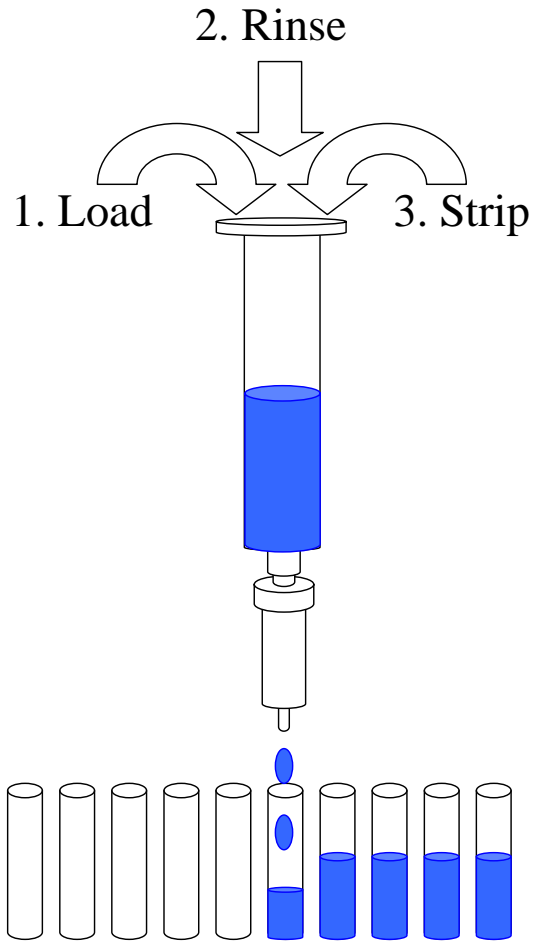
b) LC/MS analysis

- helps to determine the types of impurities present
- 18-crown-6 and DGA complete

- 2) Every batch of resin (D_w test)
-ensures correct extractant used

Resin	Dw spec.	Metal Ion
TRU	>60	Eu
TEVA	>200	Th
UTEVA	>100	Th
Sr	>100	Sr
Ln	>700	Eu
RE	>120	Eu
Actinide	>300	Bi

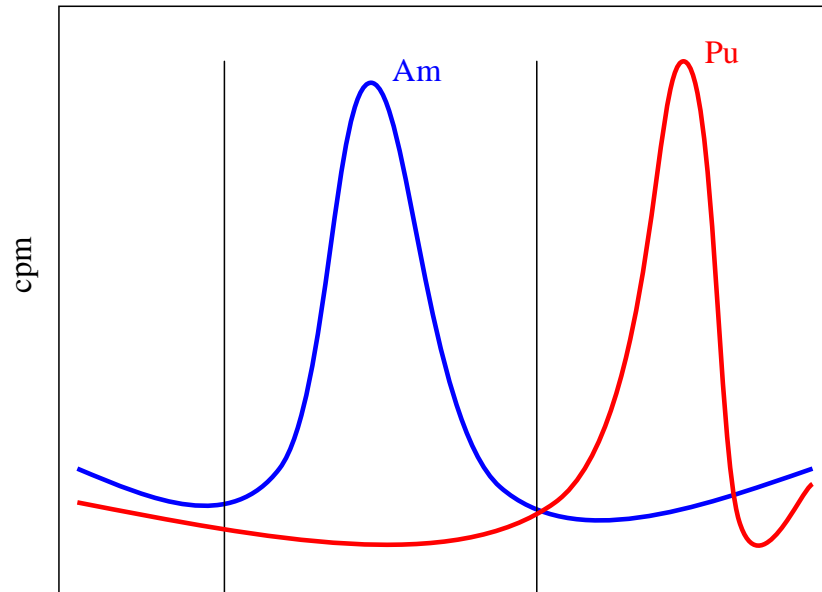
- 3) Resin with new lot of at least one starting material (New column/cartridge QC)
 - a) 1 chromatogram
 - mL by mL samples taken
 - b) 4 vacuum box or gravity tests
 - fraction by fraction samples taken
 - c) More demanding QC protocol results in greater assurance of lot to lot consistency



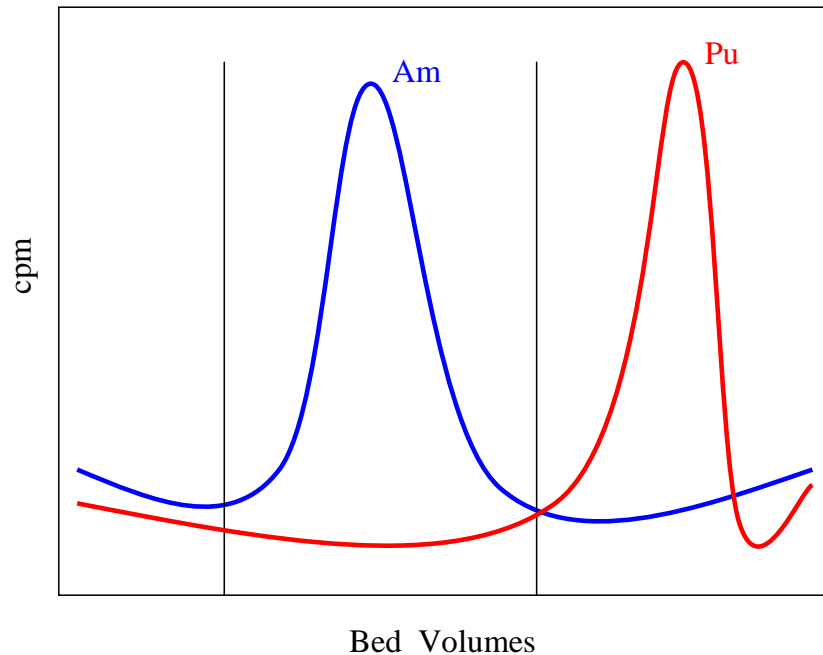
Simplified version of common
Eichrom method performed

Aliquots taken for gamma, LSC or
ICP-AES analysis

cpm or ppm plotted vs Bed Volumes

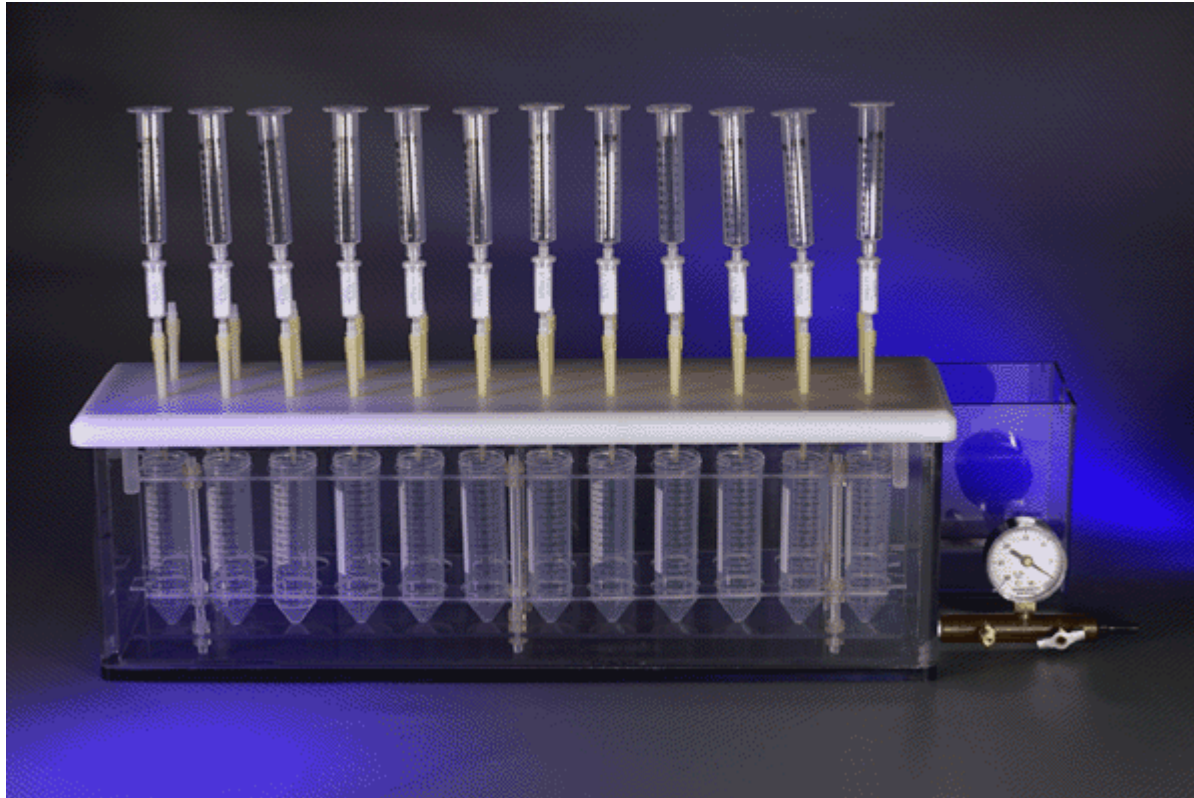


Bed Volumes



Important Parameters

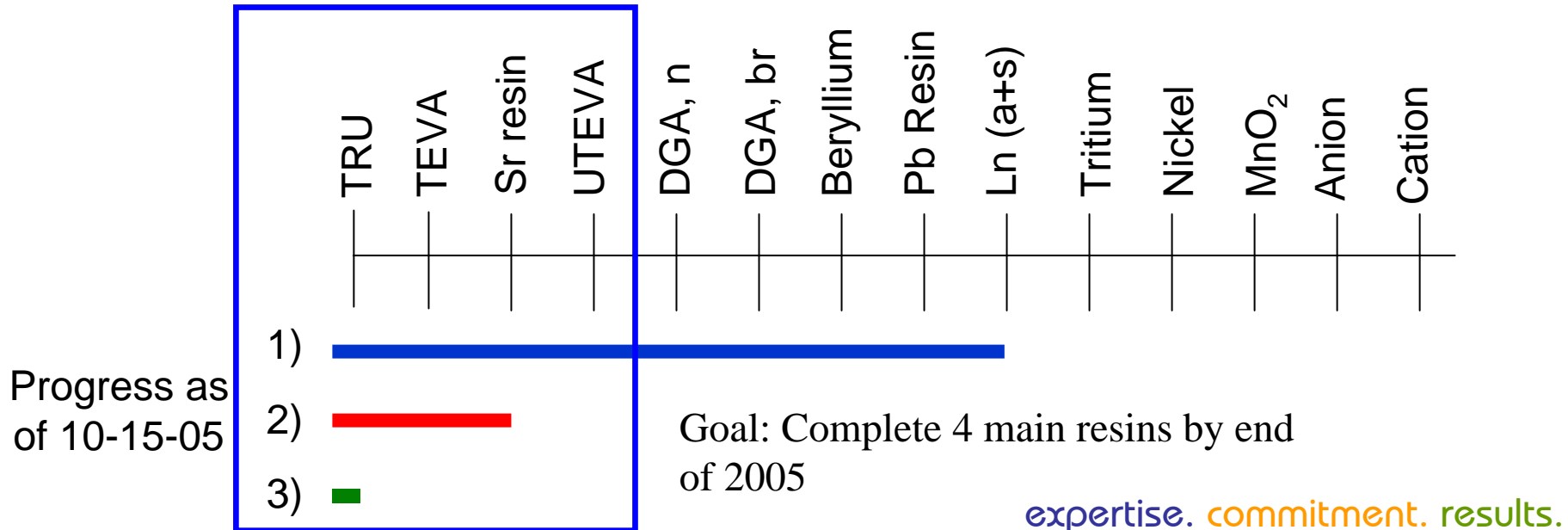
- 1) Recovery of analytes
- 2) Cross-contamination
- 3) Reproducibility



Essentially the same as the chromatogram test, however, fewer fractions are collected

Provides reproducibility data

- 1) Determine separation schemes to test each Eichrom resin.
- 2) Perform liquid-liquid extraction (extractants) and chromatogram and vacuum box/gravity tests on quality control retains to determine reasonable recovery/separation metrics for future lots.
- 3) Implement new QC procedure and provide improved certificates of analysis for each lot of Eichrom resin.

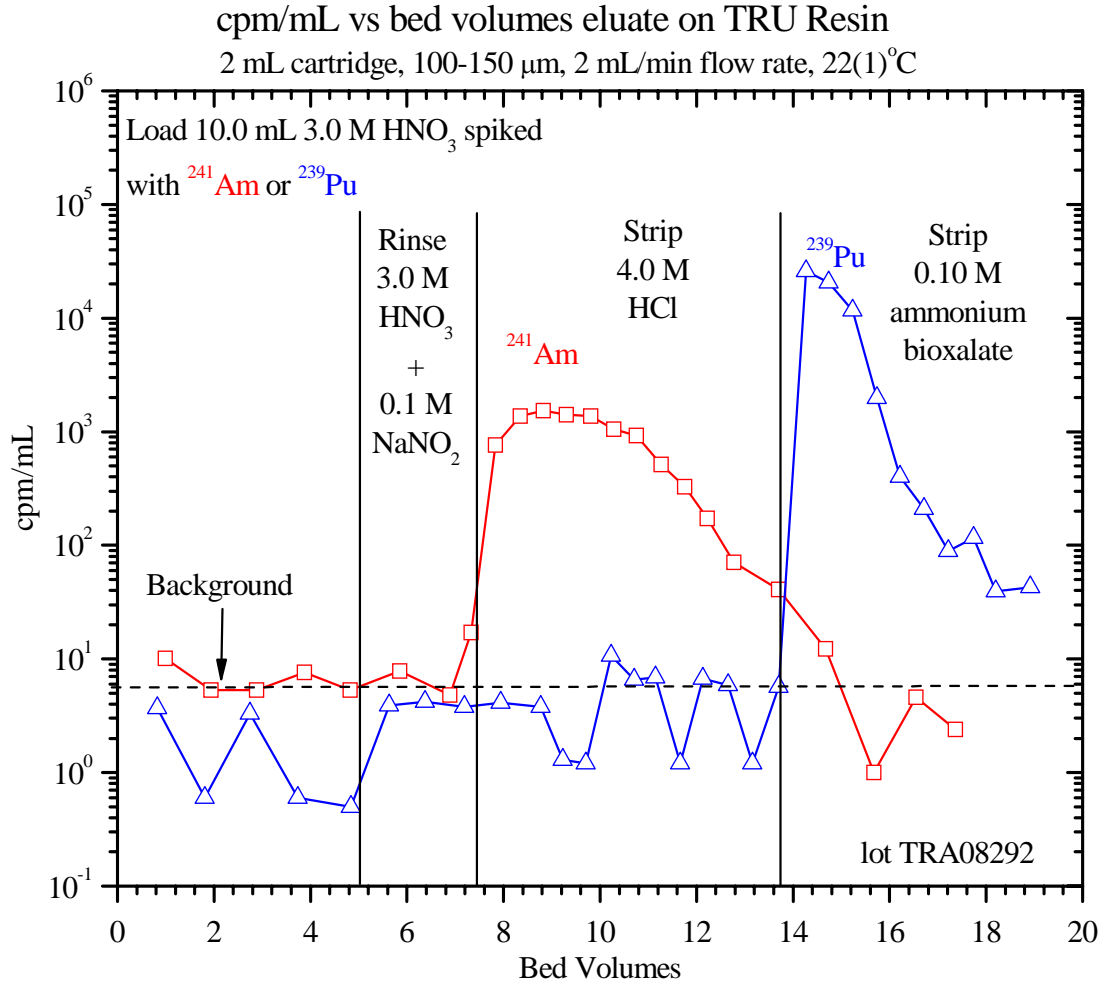


TRU: Load: Am and Pu from 3 M HNO₃

Rinse: 3 M HNO₃ + 0.1 M NaNO₂

Strip Am with 4 M HCl

Strip Pu with 0.1 M ammonium bioxalate



Summary of Column QC of 2.0mL TRU dry-packed cartridges^a

Lot	Analyte	% in	% in 4M HCl	% in 0.1M Bioxalate	column activity ^d	# replicates
		Load/Rinse	6 BV	5 BV		
TRA04164	Am ^b	0.2	99.5	0.2	4.4	1
	Pu ^c	0.2	0.4	95.0		
TRA04224	Am	0.5	99.3	0.2	3.8	1
	Pu	0.1	0.3	95.2		
TRA04264	Am	0.4	99.4	0.1	1.9	1
	Pu	0.1	0.2	98.0		
TRA07164	Am	0.1	99.6	0.2	3.7	1
	Pu	0.3	0.6	98.3		
TRA11024	Am	0.2(2)	97.3(9)	1.0(4)	0.3(1)	5
	Pu	0.1(1)	0.1(1)	97.9(3)	1.8(2)	
TRS01294	Am	0.2(1)	98.0(9)	1.3(8)	0.5(1)	5
	Pu	0.1(1)	0.1(1)	97.1(7)	2.7(7)	
TRS01274	Am	0.2(2)	98.4(7)	0.9(5)	0.5(1)	5
	Pu	0.1(1)	0.1(1)	97.0(9)	2.9(9)	
TRS05034	Am	0.3	99.4	0.3	1.9	1
	Pu	0.0	0.2	98.0		
TRS08244	Am	0.6	99.1	0.2	1.3	1
	Pu	0.0	0.1	98.6		
TRS11114	Am	0.3	99.4	0.2	4.2	1
	Pu	0.1	0.3	95.5		
TRF06294	Am	0.0	99.8	0.2	1.4	1
	Pu	0.0	0.1	98.4		

^aLoad 10mL 3.0M HNO₃ spiked with ²⁴¹Am or ²³⁹Pu, Rinse 5mL 3.0M HNO₃ + 0.1M NaNO₂, Strip

²⁴¹Am with 12mL 4M HCl, Strip ²³⁹Pu with 0.1M ammonium bioxalate

^bDetermined by gamma counting

^cDetermined by liquid scintillation counting

^d% of Pu activity remaining on column following 0.1M bioxalate strip

>95% yields for Pu and Am

Good separation of Pu and Am

<1% Pu in Am

<1.5% Am in Pu

Very reproducible

different lots

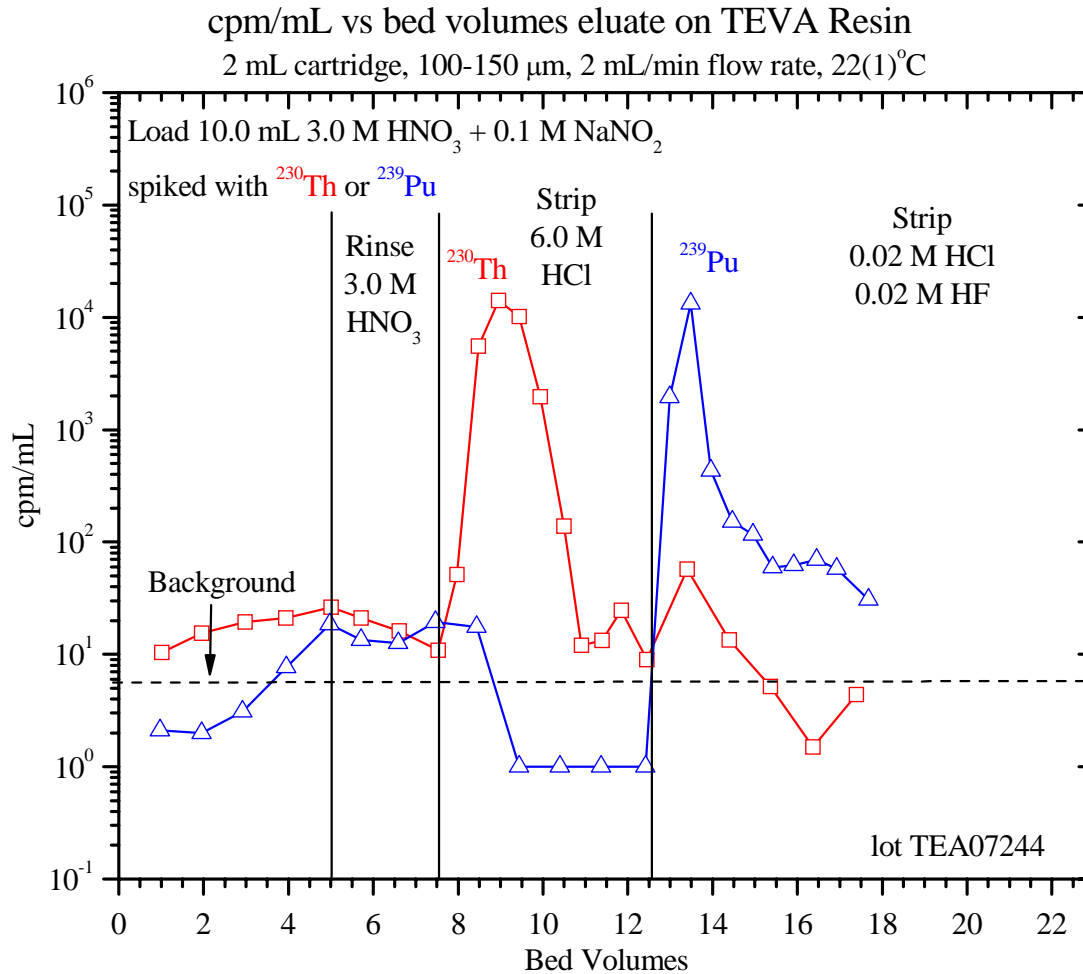
same lot

TEVA: Load: Th and Pu from 3 M HNO₃ + 0.1 M NaNO₂

Rinse: 3 M HNO₃

Strip Th with 6 M HCl

Strip Pu with 0.02 M HCl + 0.02 M HF



Summary of Column QC of 2.0 mL TEVA dry-packed cartridges^a

Lot	Analyte	% in		% in 0.02 M HCl + 0.02 M HF	column activity ^c	# of replicates
		Load/Rinse	% in 6 M HCl			
TEA10144	Th ^b	0.6	98.5	0.4	5.7	1
	Pu ^b	1.0	0.3	91.2		
TEA10114	Th ^b	1.5	95.7	0.4	3.1	1
	Pu ^b	1.0	0.7	94.1		
TEA07244	Th ^b	1.0(1)	97.2(9)	1.8(9)	0.1(1)	3
	Pu ^b	1.1(1)	0.3(1)	95.8(7)	2.2(7)	
TEA07154	Th ^b	0.7	97.2	0.2	8.6	1
	Pu ^b	0.8	0.3	90.5		
TEA04014	Th ^b	0.5(2)	97.2(9)	2.0(9)	0.1(1)	3
	Pu ^b	1.1(1)	0.2(1)	98.2(1)	0.2(1)	
TEA03254	Th ^b	0.5	98.6	0.4	2.6	1
	Pu ^b	0.9	0.4	95.2		
TES10044	Th ^b	0.2(1)	97(2)	2.0(9)	0.1(1)	2
	Pu ^b	0.9(1)	0.2(1)	98.3(1)	0.1(1)	
TES11124	Th ^b	0.5	98.4	0.6	3.9	1
	Pu ^b	0.8	0.3	95.2		
TES11204	Th ^b	0.6	98.0	1.1	4.9	1
	Pu ^b	1.0	0.5	93.3		
TES11124	Th ^b	0.5	98.4	0.6	3.9	1
	Pu ^b	0.8	0.3	95.2		
TEF10254	Th ^b	0.2(1)	94(3)	5(3)	0.1(1)	2
	Pu ^b	0.3(1)	0.1(1)	98.6(1)	0.5(1)	
TEF01274	Th ^b	0.2(1)	98.6(2)	1.0(1)	0.1(1)	2
	Pu ^b	0.3(1)	0.1(1)	98.8(1)	0.3(1)	

>93% yields for Th and >90% for Pu

Good separation of Pu and Th

<1% Pu in Th

<2% Th in Pu

Very reproducible

different lots

same lot

^aLoad 10 mL 3.0 M HNO₃ + 0.1 M NaNO₂ spiked with ²³⁰Th or ²³⁹Pu, Rinse 5 mL 3.0 M HNO₃,

Strip ²³⁰Th with 10 mL 6 M HCl, Strip ²³⁹Pu with 10 mL 0.02 M HCl + 0.02 M HF

^bDetermined by liquid scintillation counting

^c% Pu or Th remaining on column following 0.02 M HCl + 0.02 M HF Strip

UTEVA: Load: Th and U from 4 M HNO₃

Rinse: 3 M HNO₃

Strip Th with 5 M HCl + 0.05 M oxalic acid

Strip U with 1 M HCl

Sr: Load: Sr from 8 M HNO₃

Rinse: 8 M HNO₃

Rinse: 3 M HNO₃/0.05 M oxalic acid

Rinse: 8 M HNO₃

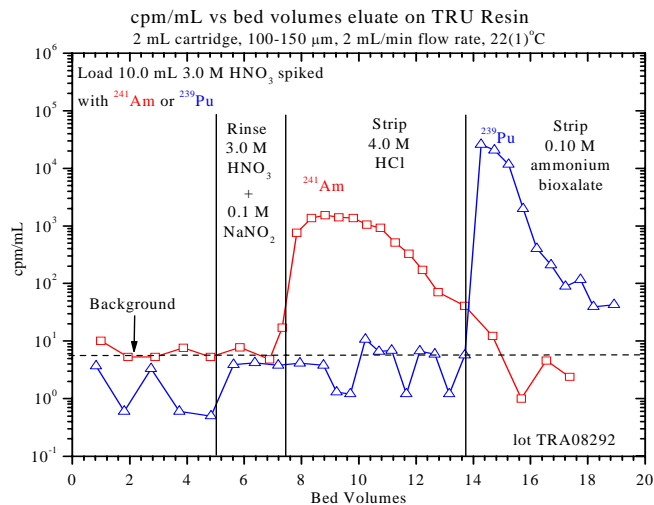
Strip Sr with 0.05 M HNO₃

Product: Eichrom's TRU Resin
Lot #: TRA08292

Eichrom's TRU Resin Lot # TRA08292 has been manufactured according to Eichrom's standard operating procedures. This lot has been tested in accordance with Eichrom's quality assurance policy and procedures. This lot of product meets all Eichrom quality specifications.

ANALYSIS RESULTS

Test Value	Specification	Result
Appearance	White beads	Pass
Distribution Ratio, Eu in 3.1 N nitric acid	≥ 60	88
% ^{241}Am stripped in 4M HCl		
Least acceptable mean (5 rep)	95.0	97.5
Max. standard deviation	2.5	1.2
% ^{241}Am stripped in 0.1M ammonium bioxalate		
Maximum acceptable mean (5 rep)	1.0	0.2
Max. standard deviation	0.5	0.2
% ^{239}Pu stripped in 0.1M ammonium bioxalate		
Least acceptable mean (5 rep)	95.0	98.0
Max. standard deviation	2.5	1.1
% ^{239}Pu stripped in 4M HCl		
Maximum acceptable mean (5 rep)	1.0	0.4
Max. standard deviation	0.5	0.3
Extractant distribution ratio, ^{241}Am strip in 0.05M HNO_3	<0.50	0.31
Raw support resin gravity flow rate, mL/min	0.60-0.80	0.72



Eichrom recommends the use of gravity flow with 100-150 μm beads, "A" grade.

Conclusions

- Eichrom is implementing a new QC program.
- The new QC program will include liquid-liquid extraction tests (extractants), D_w tests (every resin lot) and column elution tests (resin lots with new raw materials).
- So far, TRU, TEVA and Sr have been tested to generate data from which metrics to evaluate new resin lots will be determined.
- UTEVA Resin tests are next, followed by DGA and the remaining Eichrom products.