

QUALITY

Quality, Suppliers and critical products

- Market needs
- Response to customer demands
- Certification
- Improvement of product QC

New QC Procedure for Eichrom Resins

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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 (e.g. 2005)
 - 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.
- D_w was measured under uptake and elution conditions.

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

What do these types of measurements tell you?

- Doublecheck if correct extractant was used
- Doublecheck if correct quantity of extractant was used

What do these types of measurements not tell you?

- Uptake of the analyte on the column/cartridge
- Breakthrough of the analyte
- Start of the analyte-elution from the column
- Analyte recovery in the final eluate
- Problems with insufficient separation of interferents

1) Support Resin

a) Flow rate test

- gravity flow rate specs for A and S grade established
- specs for S grade flow rates 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 and DAAP in development

b) LC/MS analysis

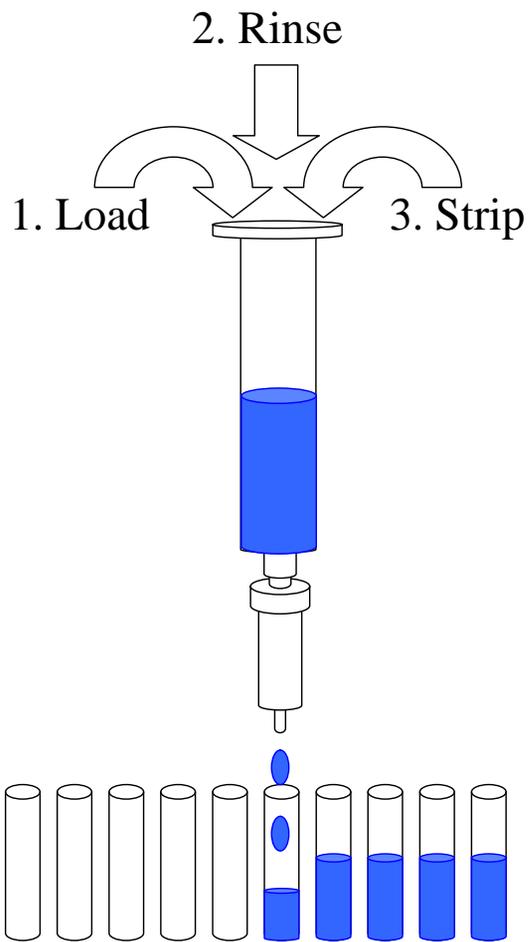
- determination and quantification of impurities
- 18-crown-6 and DGA complete

3) D_W test (uptake) on every batch of resin

Specs:

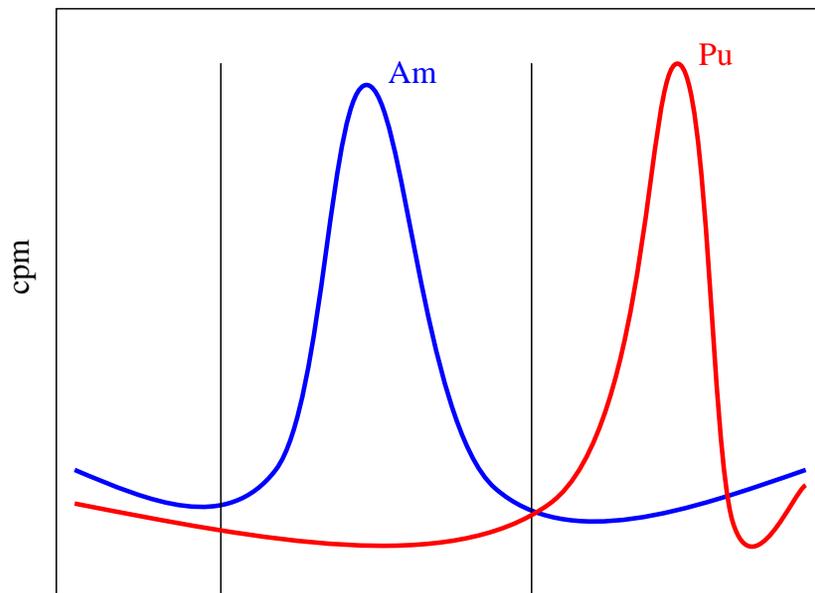
Resin	D_W spec.	Metal Ion
TRU	>60	Eu
TEVA	>200	Th
UTEVA	>100	Th
Sr	>100	Sr
Ln	>700	Eu
RE	>120	Eu
Actinide	>300	Bi

- 4) QC on Columns and cartridges
 - a) chromatogram
 - small fractions taken
 - b) vacuum box or gravity flow tests
 - samples are taken fraction by fraction (e.g. Am fraction, then Pu fraction)
 - c) QC specs on recoveries and cross-contaminations
- More demanding QC protocols and acceptance criteria result in greater assurance of inter and intra lot consistency

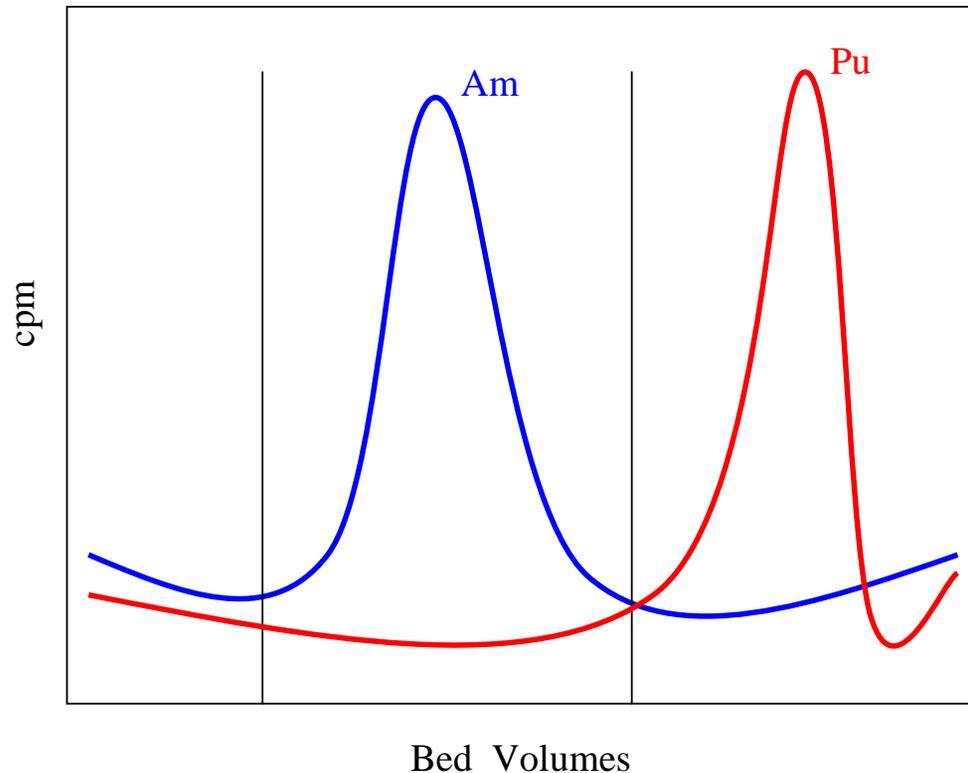


Aliquots taken for gamma, LSC or ICP-AES analysis

cpm or ppm plotted vs Bed Volumes



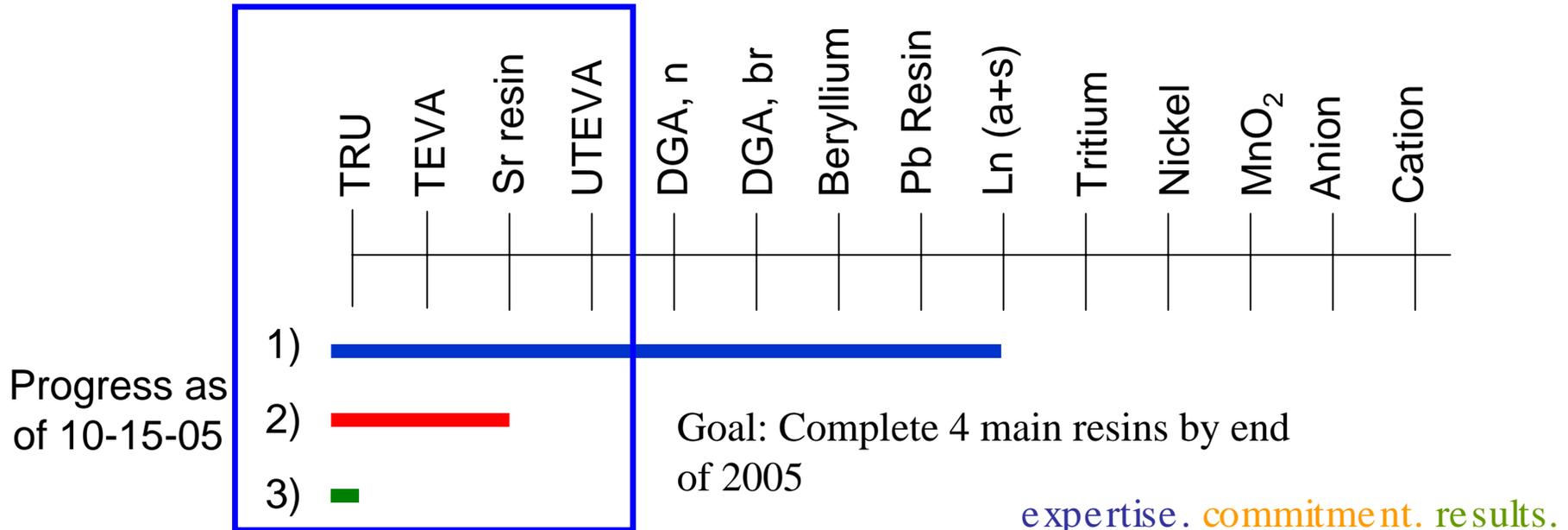
Bed Volumes



Obtained information:

- 1) Recovery of analytes
- 2) Cross-contamination
- 3) Elution profile
 - Start/end of elution
 - Peak position
 - Peak shape

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

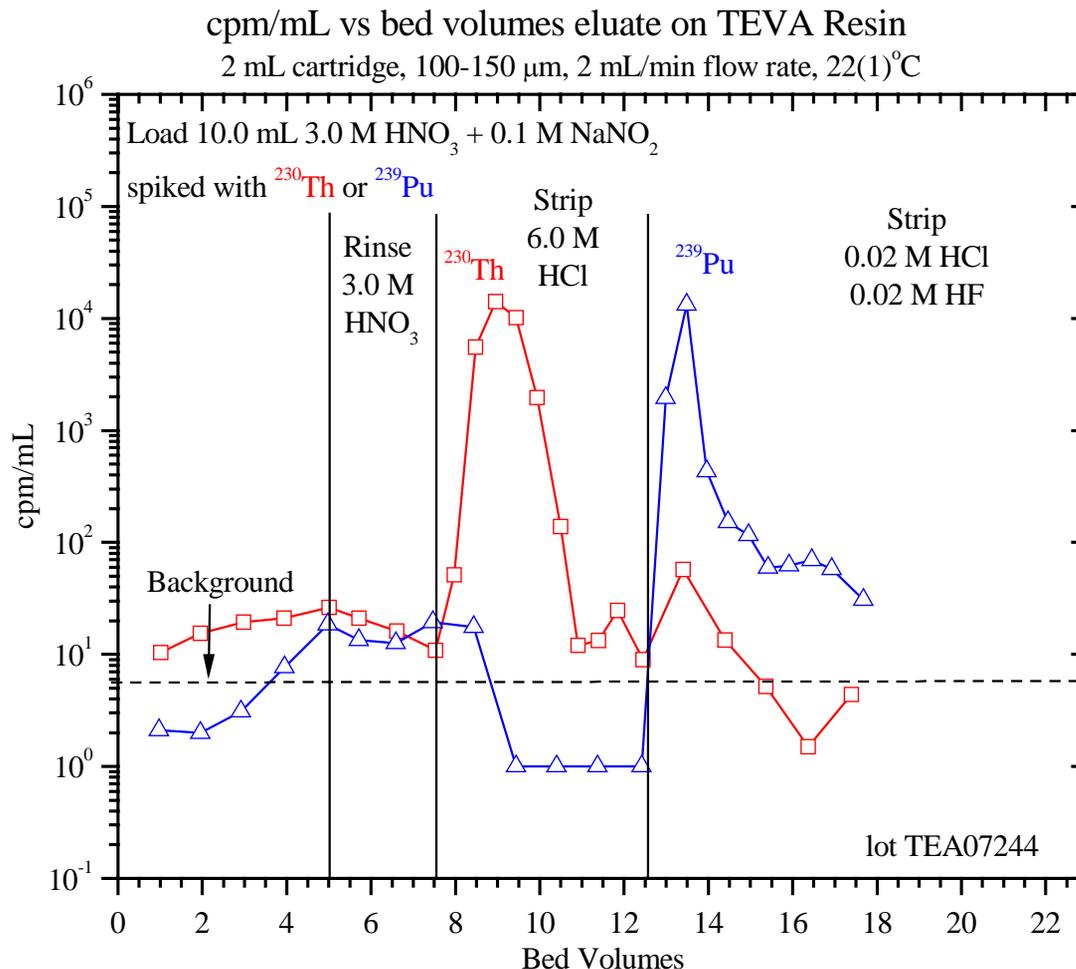


TEVA: Load: Th and Pu from 3 M HNO₃ + 0.1 M NaNO₂

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 Load/Rinse	% in 6 M HCl	% in 0.02 M HCl + 0.02 M HF	column activity ^c	# of replicates
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

Good reproducibility

- inter lot
- intra 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

- Eichrom is implementing a new QC program.
- The new QC program includes:
 - Flow rate specs on support
 - Liquid-liquid extraction tests on extractants,
 - Dw tests on every resin lot,
 - Column/cartridge elution tests incl. Chromatogram
- TRU, TEVA and Sr have already been tested in order to generate data, acceptance criteria are determined.
- UTEVA Resin tests are next, followed by DGA and the remaining Eichrom products.
- New CofA already available for TRU resin