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CANBERRA

Optimizing Performance of Alpha Spectrometers and Gas Flow Proportional Counters; Best Practices for Sample Preparation

Canberra Users Group Meeting
Boston, MA - July 15,2010

Larry Jassin, Mike Fern, Terry O'Brien
Eichrom Technologies

NUCLEAR POWER OUTFITTERS
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Outline

- Eichrom/NPO Company Overview
  (How we fit into the Nuclear Community)
- Ambient/Gross Sampling (Smears / Air Filters)
- Sample Preparation Requirements
- Extraction Chromatography (EXC) Fundamentals
  Separation Tools-Uptake Curves
  Methods – Results
- Filters for Alpha Spectroscopy

Eichrom / NPO

- Founded in 1990
  - Technology transfer from Argonne National Laboratory
  - 2008 Eichrom Technologies acquired Nuclear Power Outfitters
  - 2009 Eichrom moved entire company into one facility in Lisle, IL just ~30 miles of downtown Chicago

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…collaborating with the nuclear industry to protect people and the environment.

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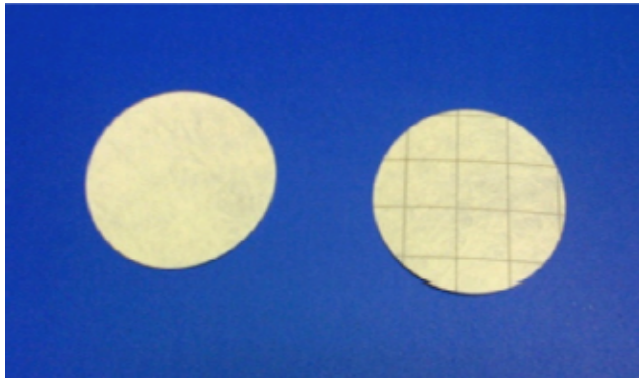
Collaborating Relationship

CANBERRA



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Health Physics Sampling Smears

Item Description	Number Per Box
Paper backed Smears for recording	500
Smears-Bulk packaged	2500

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Resolve® 47mm PTFE Filter for Air Monitoring

Issue	Characteristic
Quality spectra	•Low self background •Good resolution for alpha emitters • Collection of contamination on Surface (reduce self-adsorption)
Easy to Handle	Negligible curling and durable

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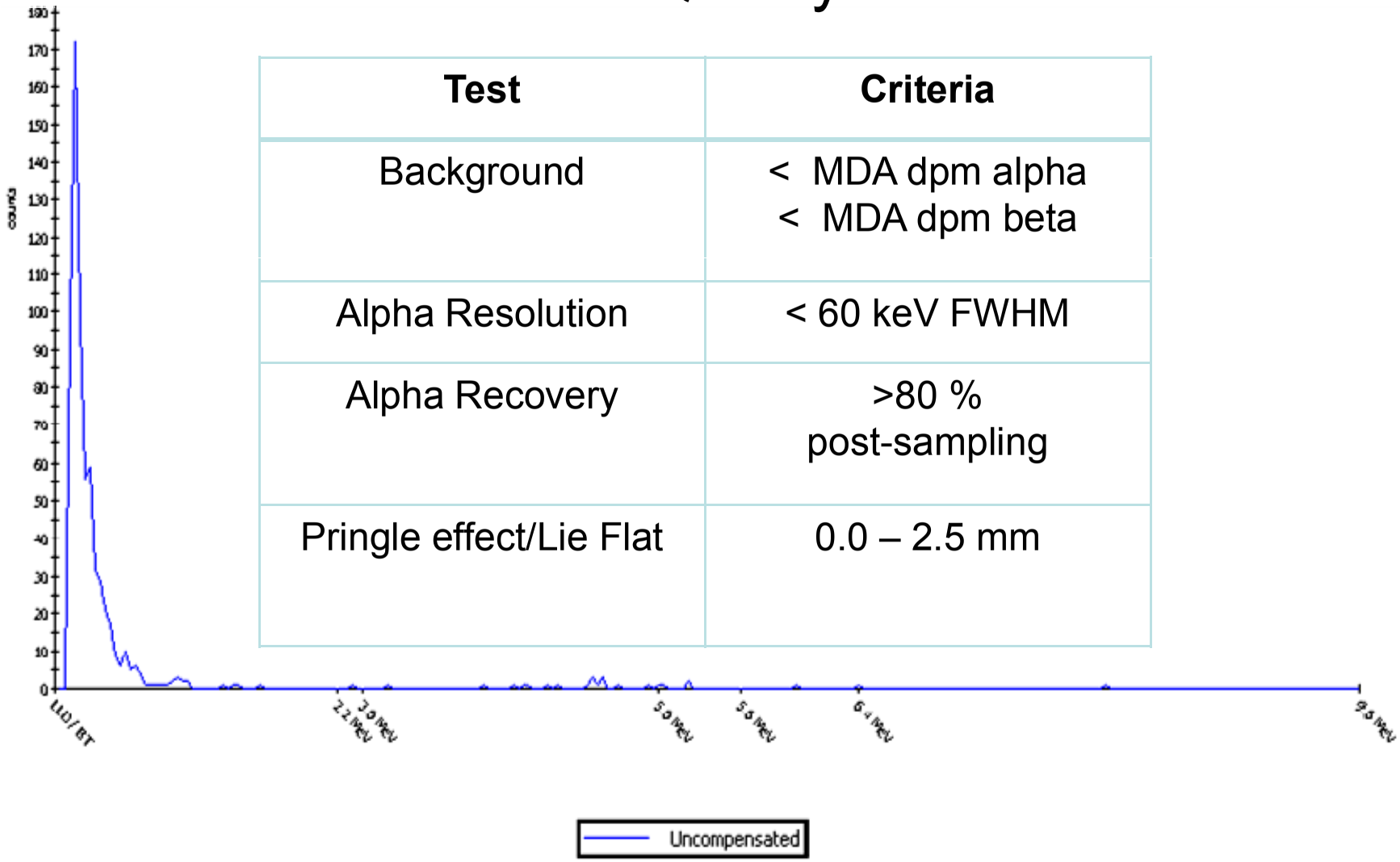
Filter Material – Quality Control Goals

Test	Criteria
Background	< MDA dpm alpha < MDA dpm beta
Alpha Resolution	< 60 keV FWHM
Alpha Recovery	>80 % post-sampling
Pringle effect/Lie Flat	0.0 – 2.5 mm



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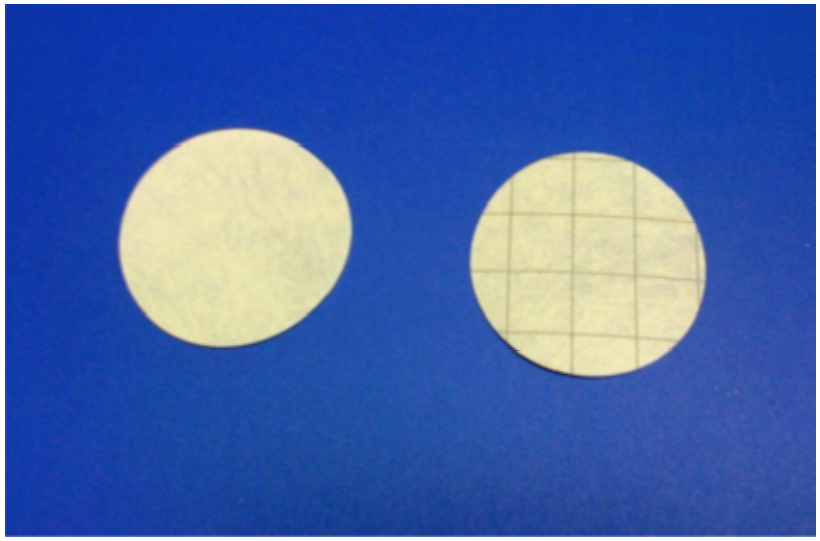

Resolve PTFE Filters
3 µm, PTFE Laminate
47mm Diameter

Polytetrafluoroethylene (PTFE) material
With a laminated stiff polymeric backing

Properties	
Diameter	47 mm
Reference Pore Size	3.0 µ nominal
Thickness	0.15 –0. 4 mm thick
DOP Efficiency	>99.99 % 0.3 um @5.33 cm/sec
Chemical Compatibility	Resistant to most chemicals and solvents
Air Permeability	6 ft^3/ft^2/min @ 0.5” H2O minimum

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Filter Backgrounds
Via Gas Flow proportional counting

	α - alpha Background MDA=2.24 (dpm)	**β - beta Background MDA=4.20 (dpm)**
Cellulose	0.78 ± 1.12	0.17 ± 1.30
Glass Fiber	2.00 ± 1.56	4.52 ± 3.44
Resolve PTFE Filter	0.24 ± 0.70	0.31 ± 3.54

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Spiked U+Am Alpha Comparison
Part 2: Post-Air Sampling
Spike Recovery (3 replicates)

Filter type	Alpha spike (dpm)	Measured before air sampling Alpha (dpm)	Initial % Recovery	Measured Alpha (dpm) after air sampling	% Recovery
Cellulose	474	106 ± 3.4	22 %	35.9 ± 20.2	8 %
Glass	474	237 ± 5.1	50 %	225 ± 18.8	47 %
Resolve PFTE	474	415 ± 6.9	88 %	397.8 ± 14.2	84 %

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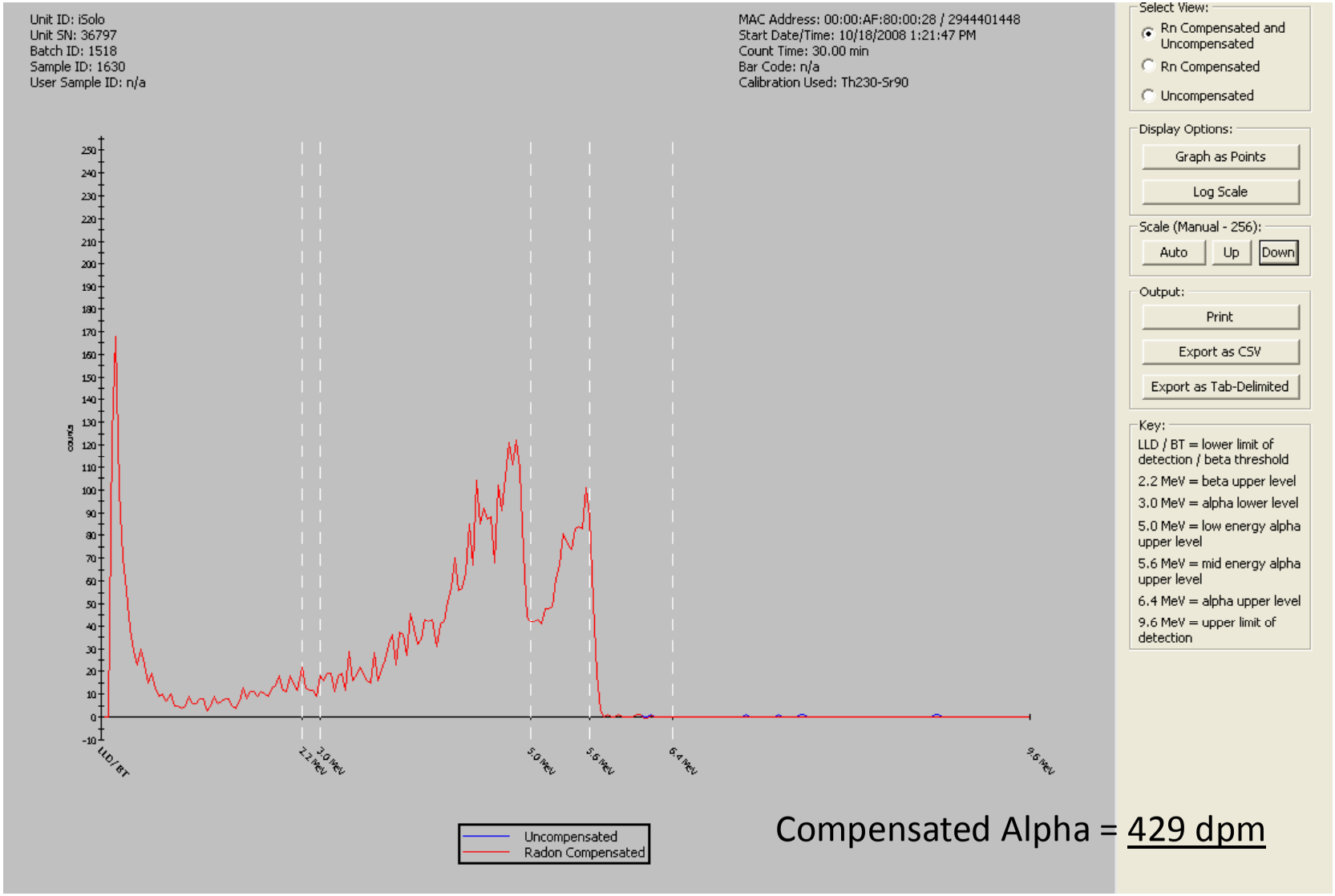
Spiked Resolve PTFE Filter Pre Air Sampling



Compensated Alpha = <u>429 dpm</u>

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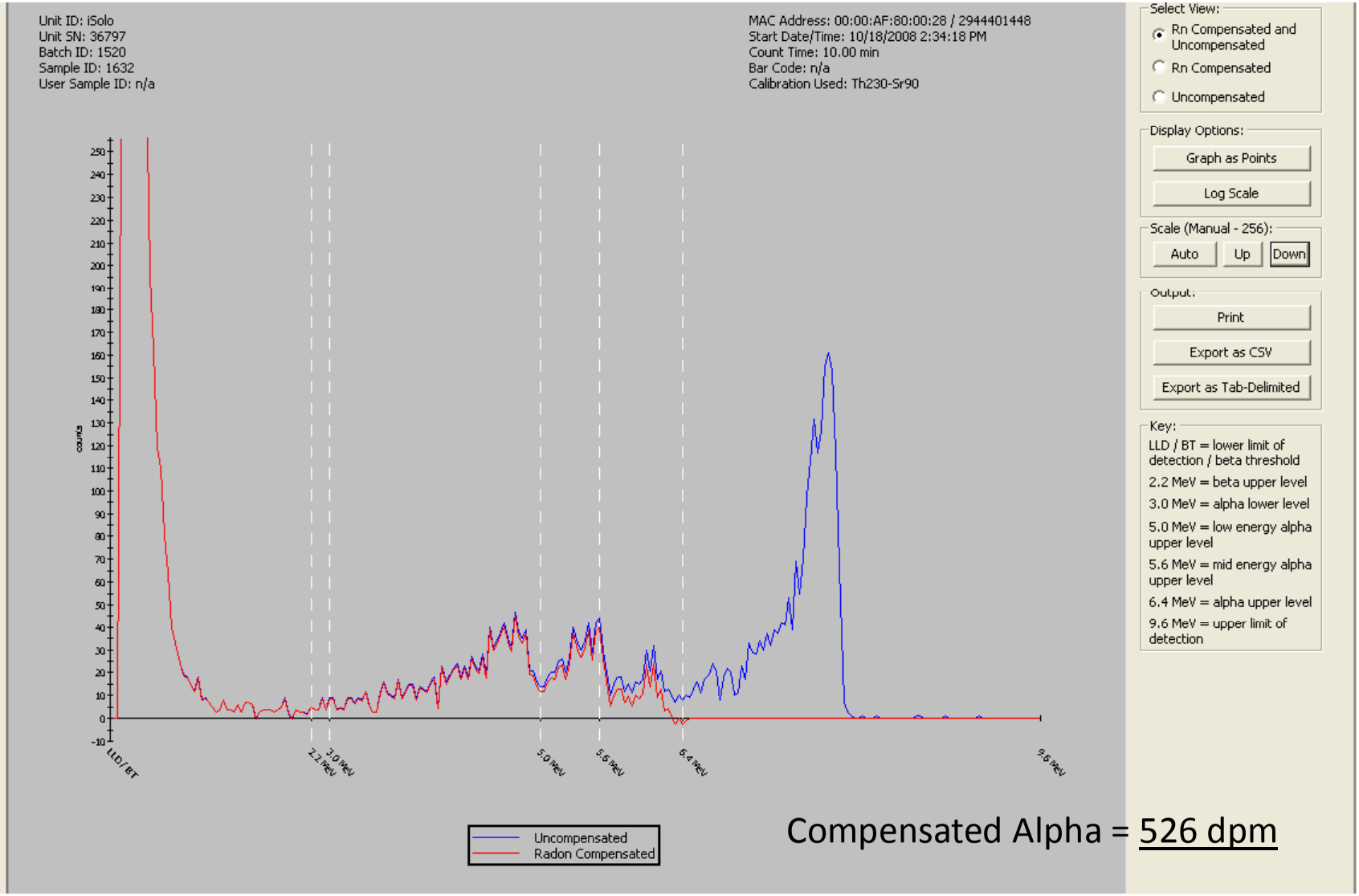
Spiked Resolve PTFE Filter
Post Air Sampling – 1st 10 min



Compensated Alpha = <u>526 dpm</u>

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Resolve PTFE Filter

3.0 µ PTFE Laminate

47mm dia. (50 to a package)

- Quality Control Specification:
  - Background < MDA for α & β
  - Resolution verified <60 KeV FWHM
  - Minimal curling <2.5 mm deflection
- The filters have a easy to determine orientation:

  "Grid side down, Opposite Air flow"
- Performance demonstrated at Eichrom and externally
  - Lee Reagan of Canberra,
  - Peter Olsen of Washington Closure Hanford

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We Need Sample Preparation

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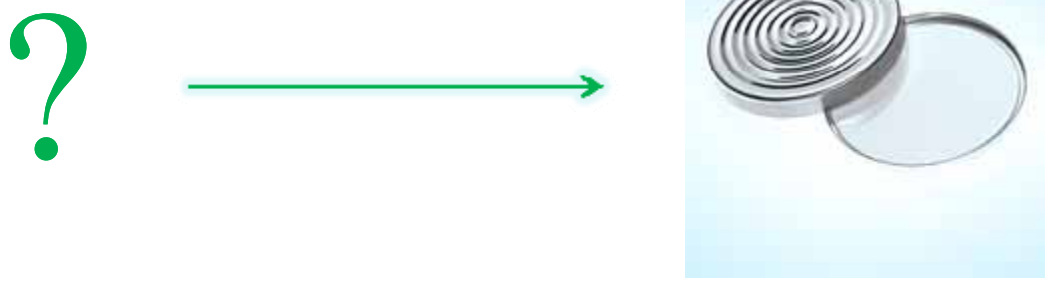
Steps in the Sample Preparation Pathway

- Sample Preparation Concepts/ Measurement Equipment Choice
- Extraction Chromatography (EXC) Fundamentals
- Separation Schemes and Results
- Importance of Filter Media
  - Rare Earth-Micro Co-Precipitation

Your goals for Sample Preparation

- Minimize sample prep time and waste generation
- Trade offs between
  - sample size, detection level, sample preparation rigor
- Data quality objectives
- Detection instrument selection
- Instrument's capabilities



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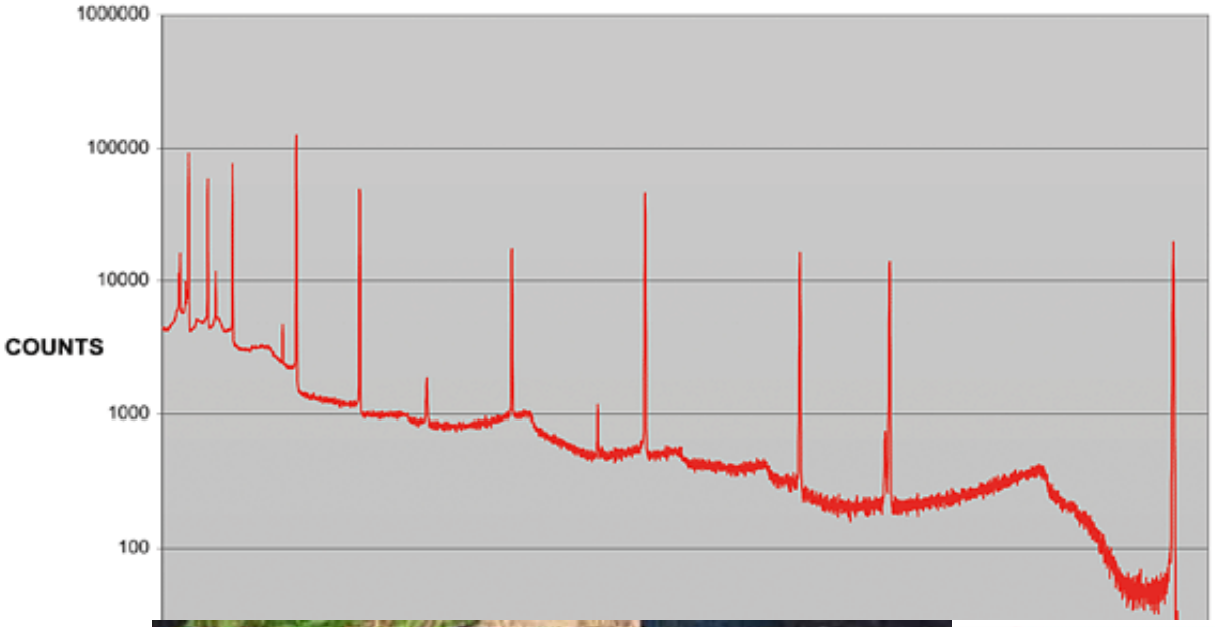
Measurement of Gamma Energy



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Radiologic Screening Counters

- Gas-Flow Proportional Counters
  - Alpha/Beta Weight Attenuation
  - Alpha/Beta Cross Talk Calibration
  - Alpha/Beta Isotope Calibration

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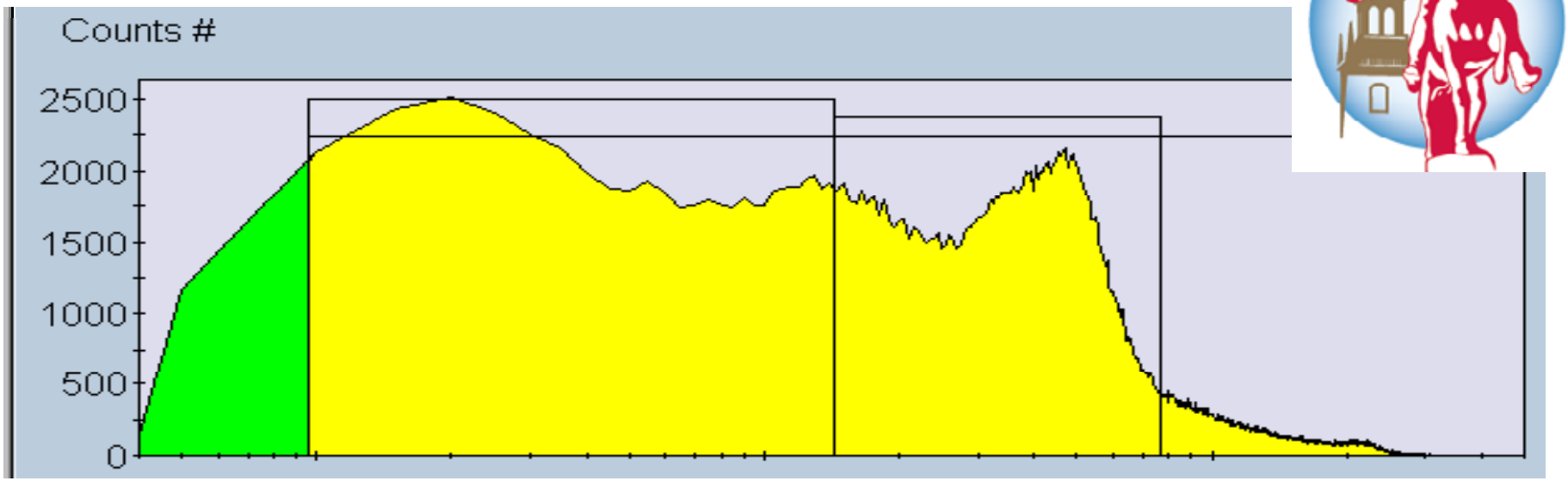
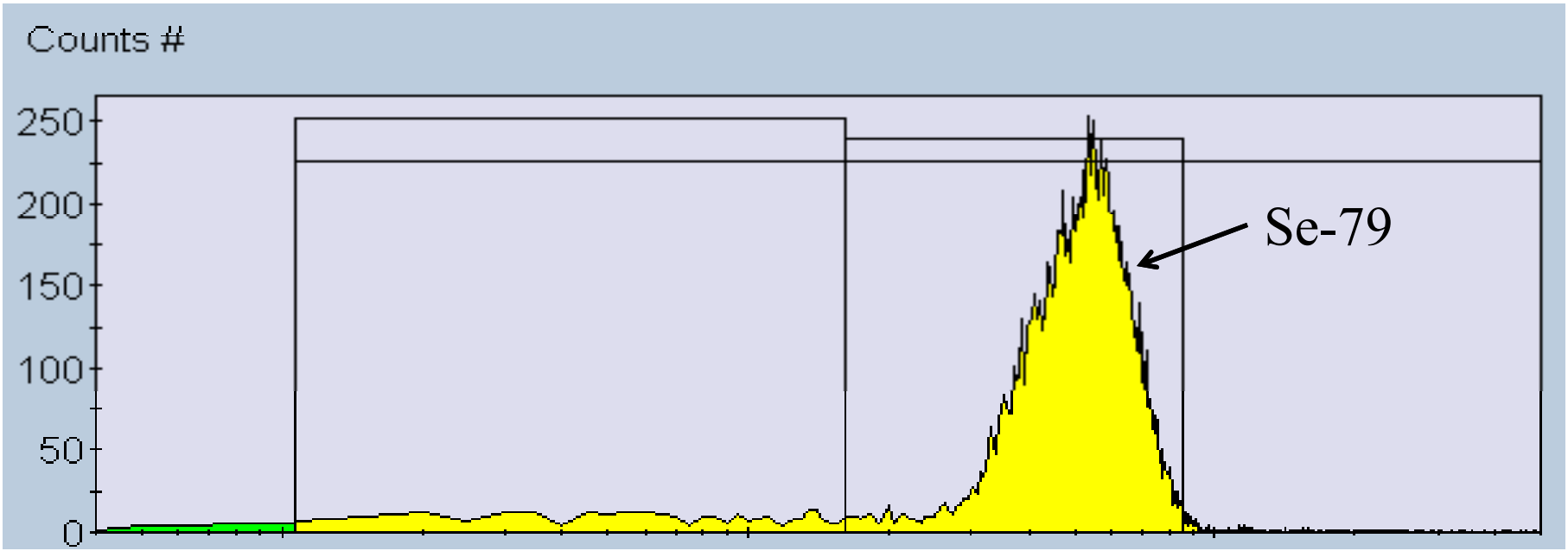
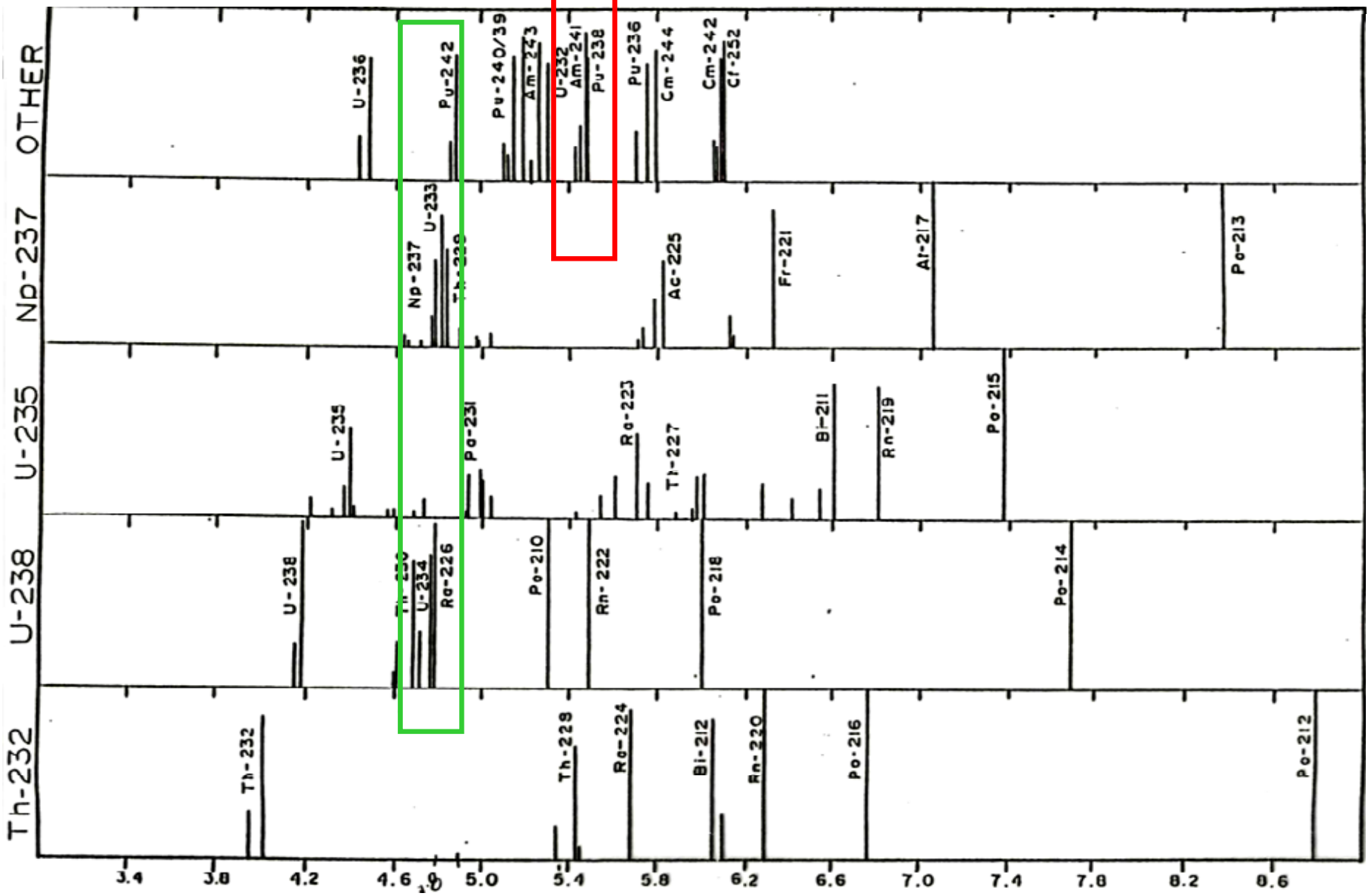
Measurement of Beta Energy





Measurement of Alpha Energy



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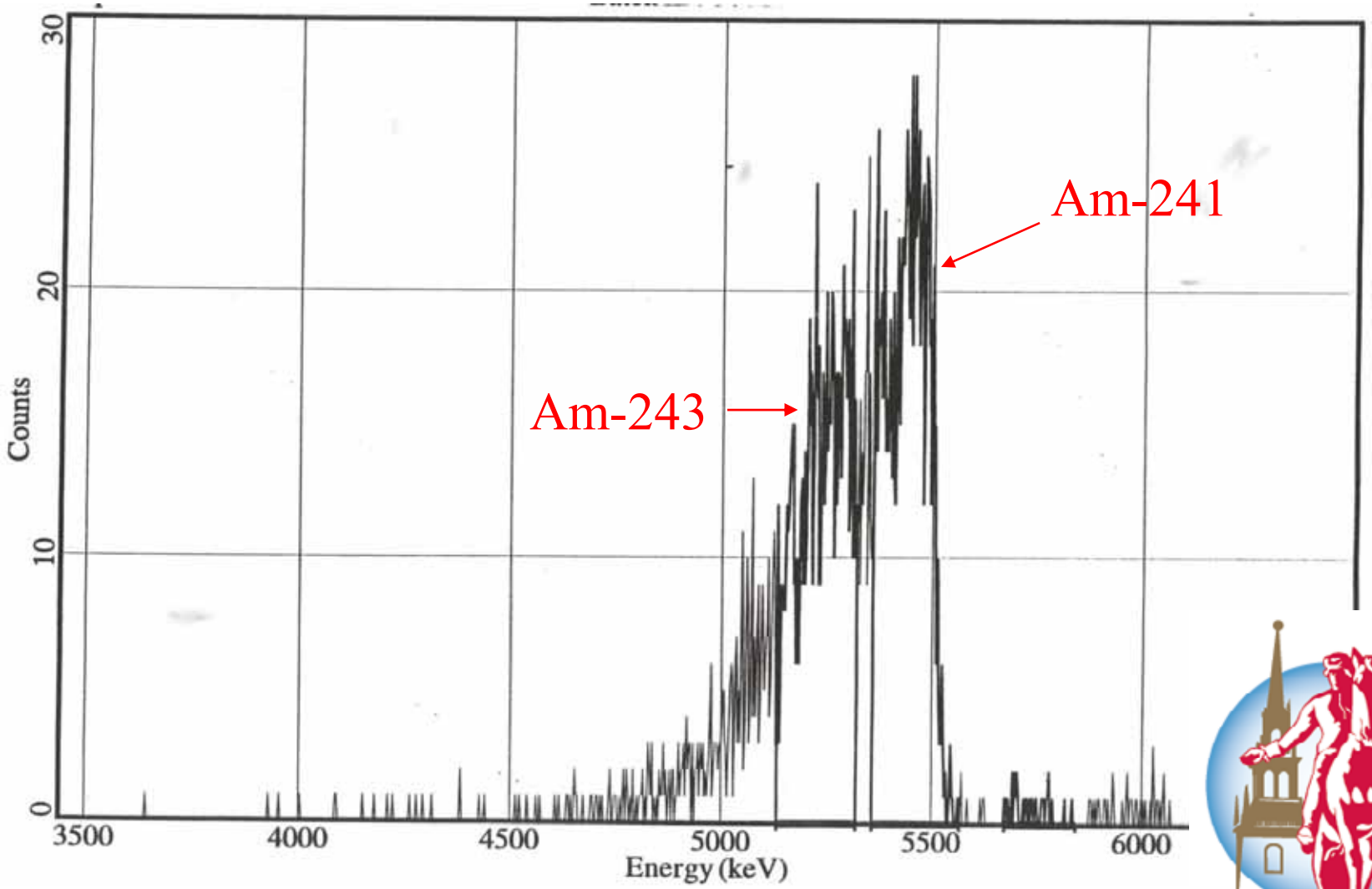
Americium Spectrum after TRU Resin Separation

presence of rare earths degrades spectrum- self absorption issues



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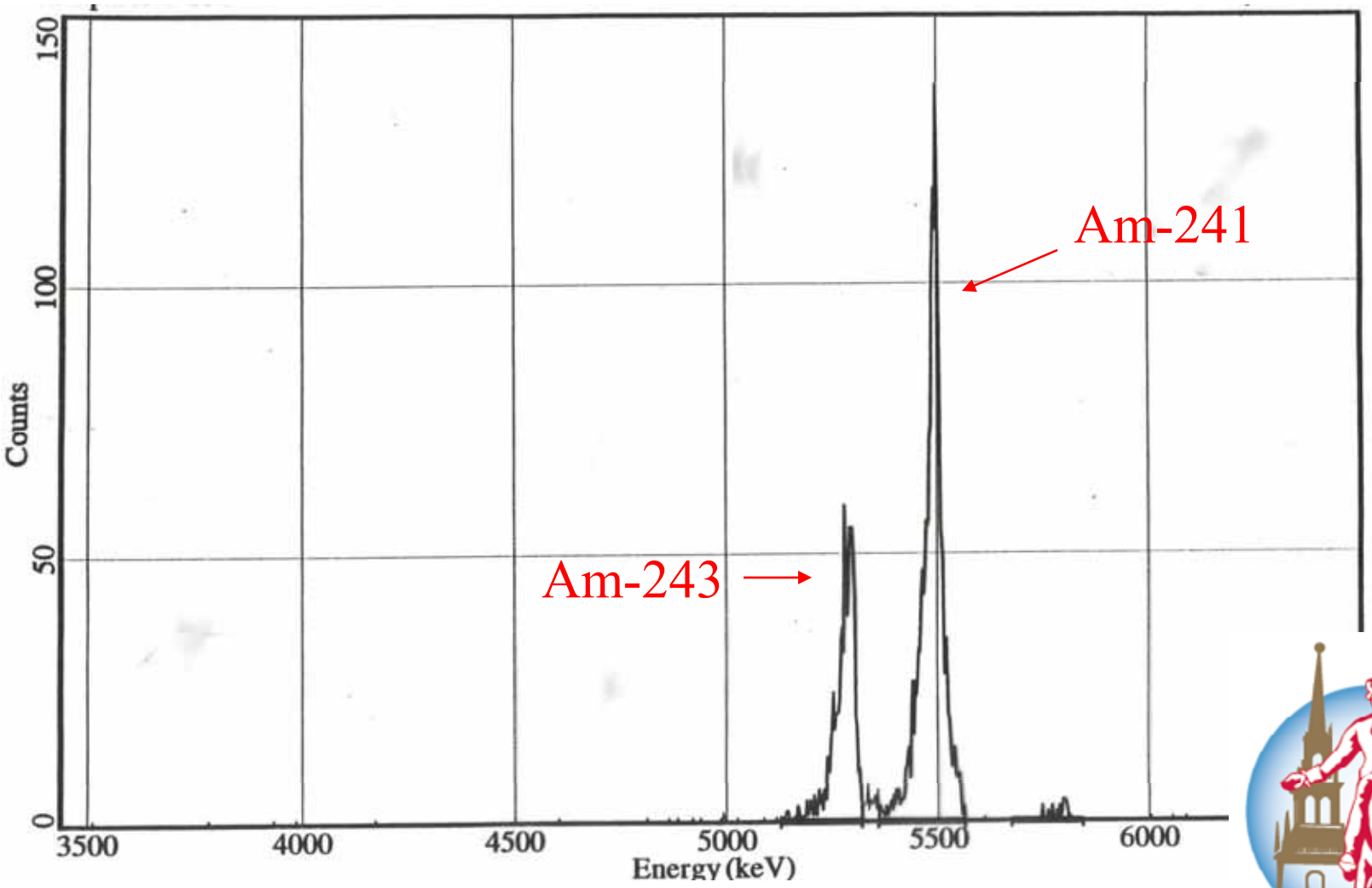
Am Spectrum after TEVA Resin Separation

Lanthanide elements removed - cleaner spectrum



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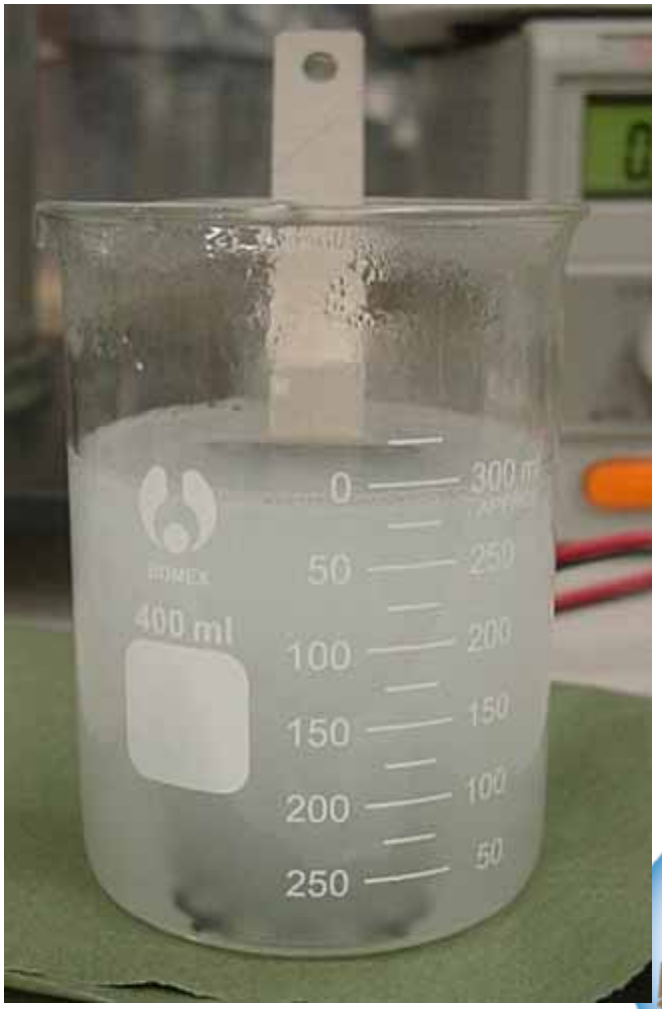
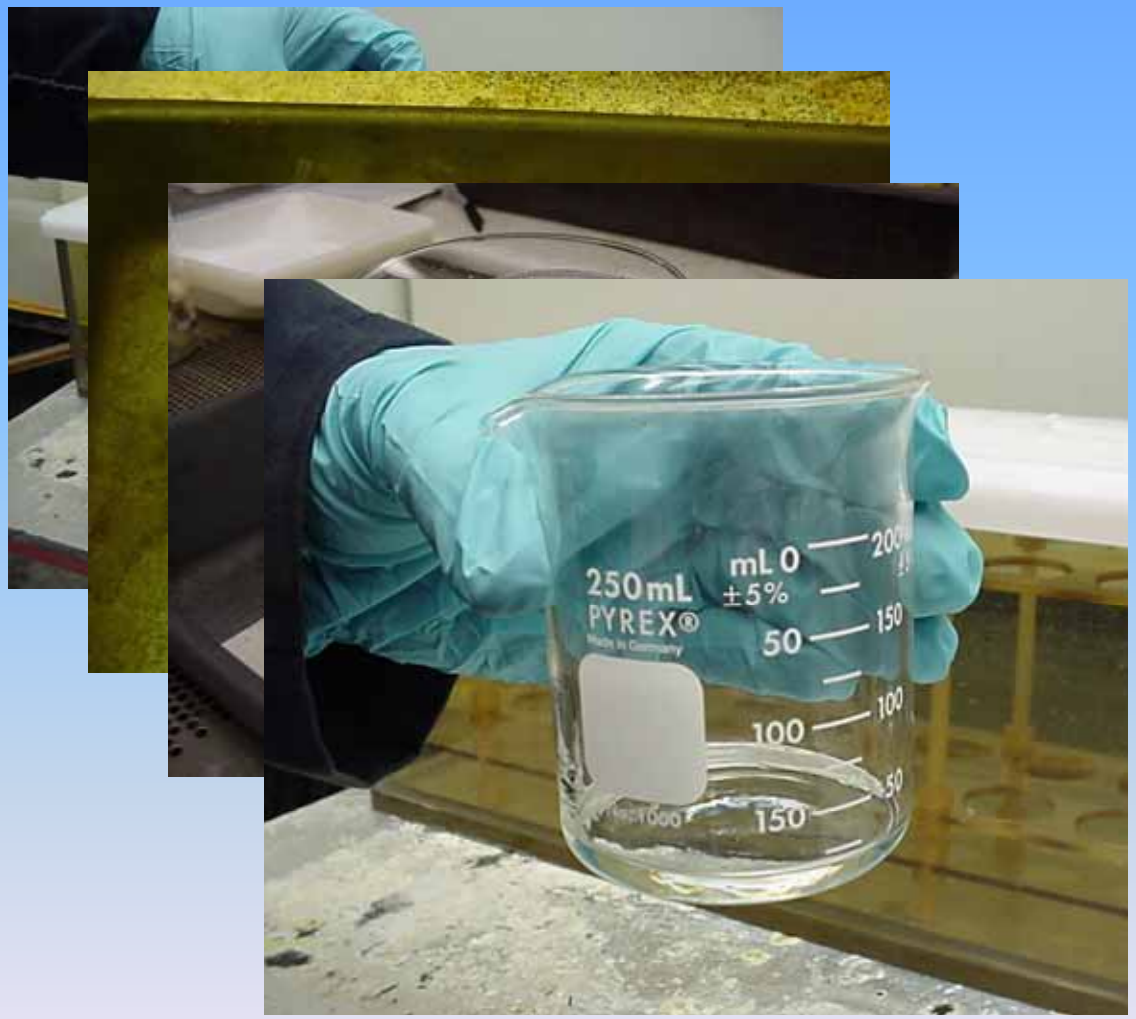
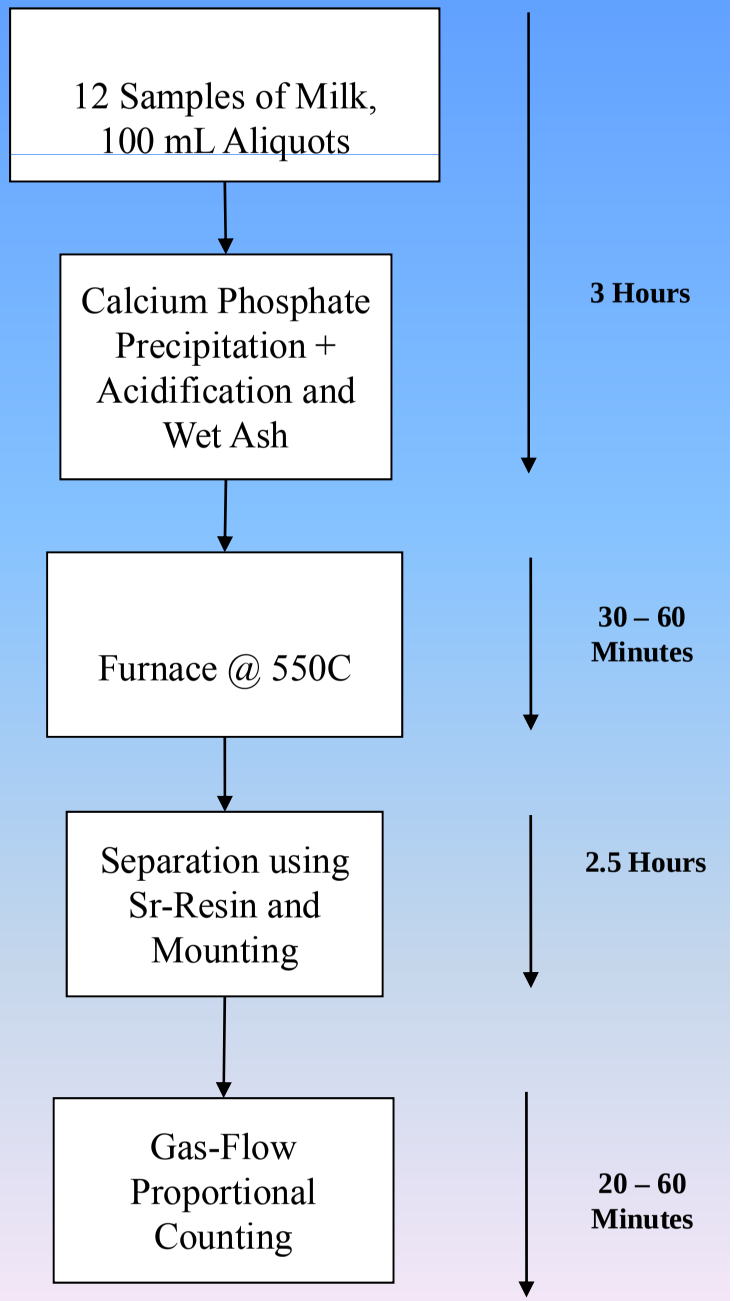
Sample Preparation Steps

- Sample Modification
  - Digestion
  - Leach
  - Fusion
- Volume Reduction
  - Precipitation
  - Evaporation
  - Ion Exchange



FlowChart of with time frames Radiostrontium Emergency Method At SRS





Maxwell III, SL and Culligan, B.K., "Rapid Method for Determination of Radiostrontium in Emergency Milk Samples", Journal of Radioanalytical and Nuclear Chemistry, Vol. 279, 3 (2009) 757-760

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Extraction Chromatography

Solvent Extraction

Column Chromatography

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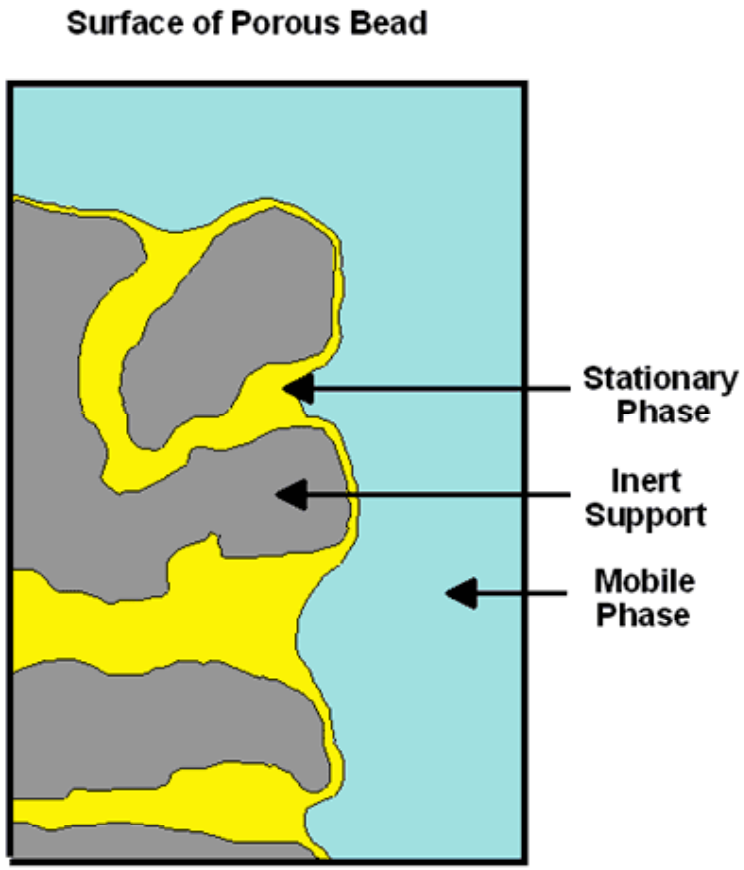
Extraction Chromatographic Resin



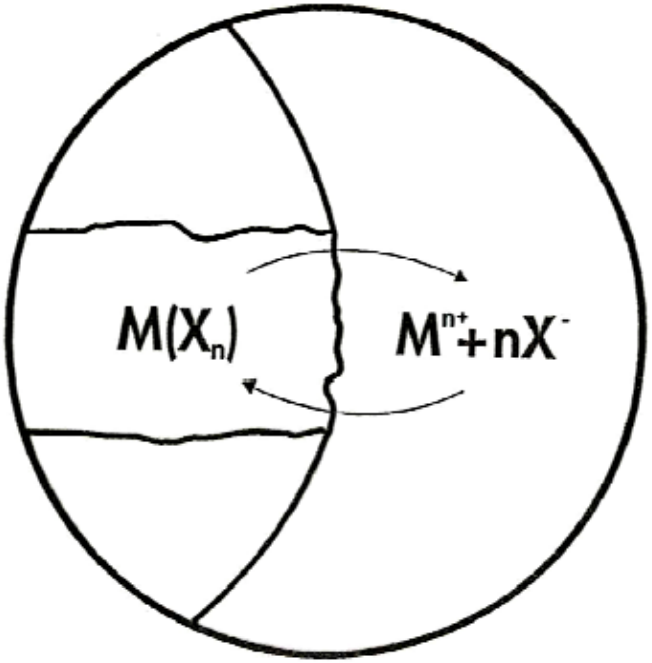
Inert support =

Macroporous Acrylic Resin

Example Stationary Phases

- **Crown Ether (Sr)**
- **CMPO (TRU)**
- **DAAP (UTEVA)**

Metal Anion Complex Formation



$$\text{Metal} + \text{Anion} \rightleftharpoons \text{Complex}$$

$$\text{Complex} + \text{Organic} \rightleftharpoons \text{Extracted}$$

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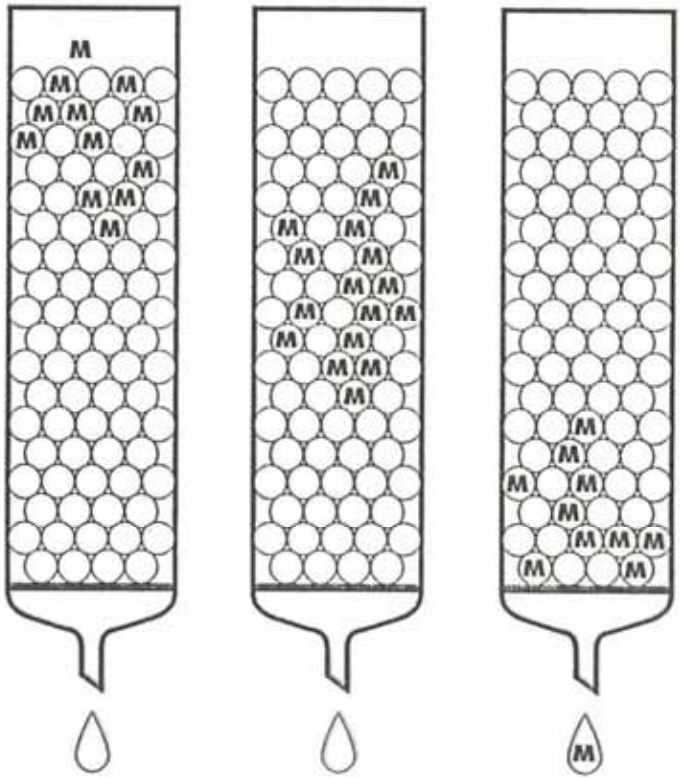
Elution of a Band (Chromatography)



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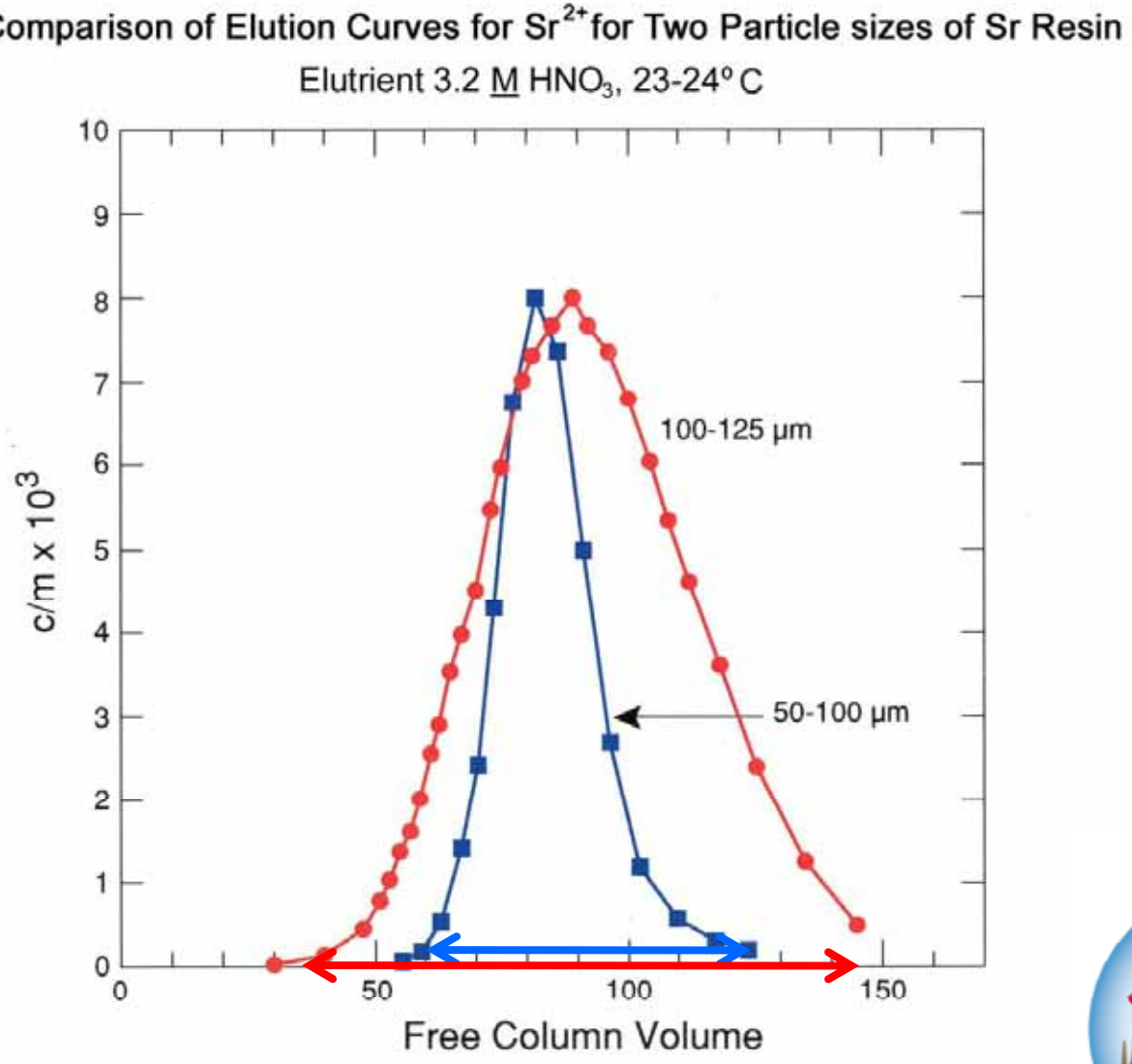
Smaller particles, sharper elution bands



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Choose the appropriate amount of resin

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Sr Resin
Part #: SR-B100-A
Lot: SRA03032
Amount: 100 grams

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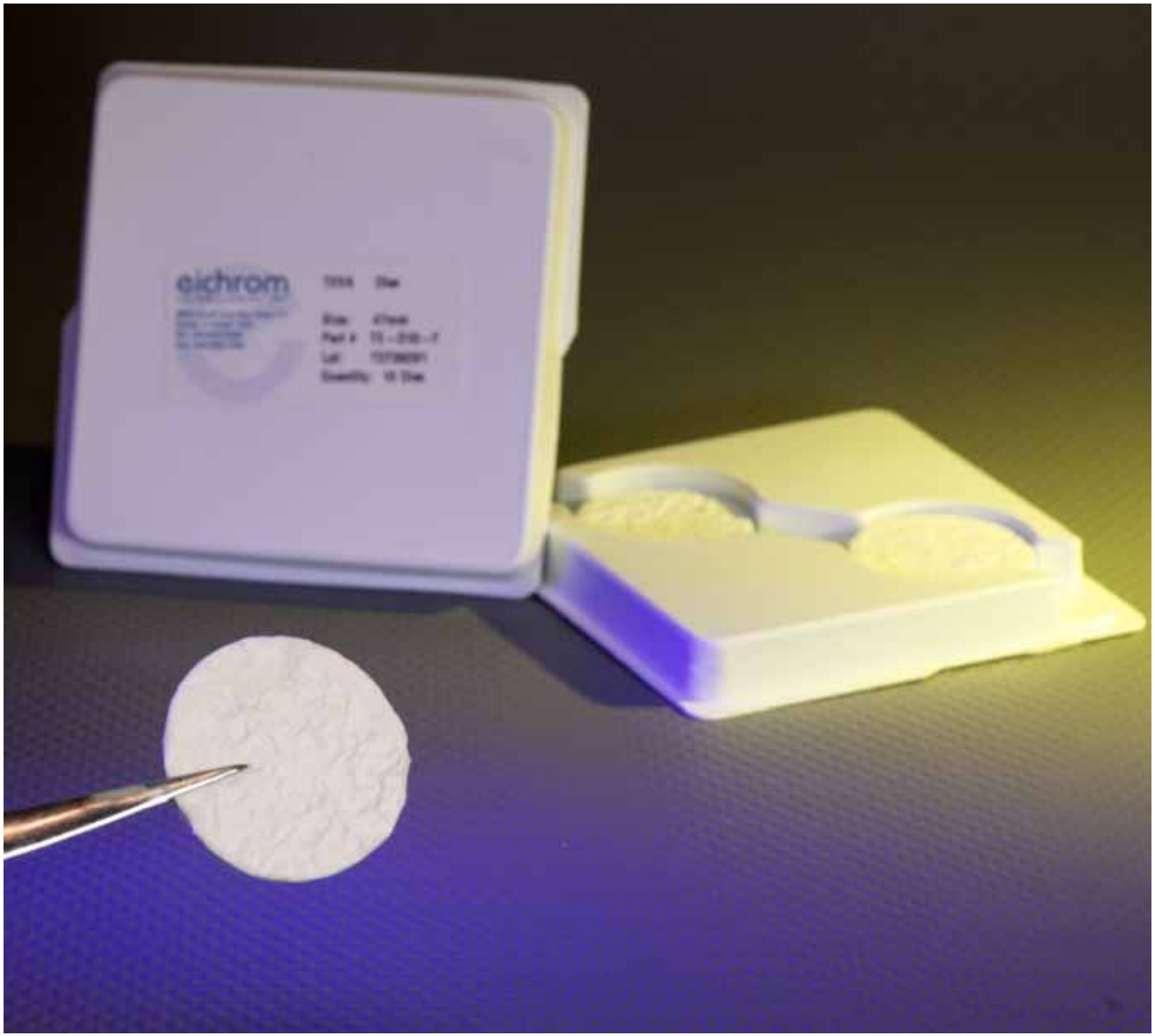

Gravity Flow vs. Vacuum Assisted

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LATEST NEWS: Nuclear Power Outfitters Featured Product: "T-Flex" Tungsten Shielding System

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Product Name	Color Code	Applications*		
Accessories		Plastic accessories for columns and cartridges	product info	part numbers
Resolve™ Filters		Alpha spectroscopy source preparation	technical info	part numbers
Resolve™ PTFE Filters		• NEW Radiological Air Monitoring	technical info	part numbers
Actinide Resin	● Yellow	**Group actinide separations/gross alpha measurements**	technical info	part numbers
Beryllium Resin		**Be**	technical info	part numbers
DGA Resin		Actinids, Lanthanides, Y, Ra	technical info	part numbers
Diphonix® Resin		Actinides and transition metals	technical info	part numbers
Ion Exchange Resins		Analytical grade cation and anion exchange resins	technical info	part numbers
Ln Resin	● Purple	**Lanthanides, Ra-228**	technical info	part numbers
MnO_2 Resin		**Ra**	technical info	part numbers
Nickel Resin	● Pink	**Ni**	technical info	part numbers
Pb Resin	● Black	**Pb**	technical info	part numbers
Pre-filter Material		**Organics removal**	technical info	part numbers
RE Resin		Th, U, Np, Pu, Am, Cm, **rare earth elements**	technical info	part numbers
Sr Resin	● Red	**Sr**, Pb	technical info	part numbers
TEVA® Resin	● Green	**Tc, Th, Np**, Pu, **Am/lanthanides**	technical info	part numbers
Tritium Column		**^3H**	technical info	part numbers
TRU Resin	● Blue	**Fe**, Th, Pa, U, Np, **Pu, Am, Cm**	technical info	part numbers
UTEVA® Resin	● Orange	Th, **U**, Np, Pu	technical info	part numbers

*Primary applications shown in **blue**.

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EXC Resin Extractant Choice

- Ionic Recognition Extractant based resin
  - Analyte retention related to charge and ionic radii
- Neutral and Anionic Extractant based resin
  - Analytes are directly hydrogen ion dependant, although anionic extractant based resins tend to show reduced analyte uptake as the acid competes
- Acidic Extractant based resin
  - Analyte retention is inverse hydrogen ion dep

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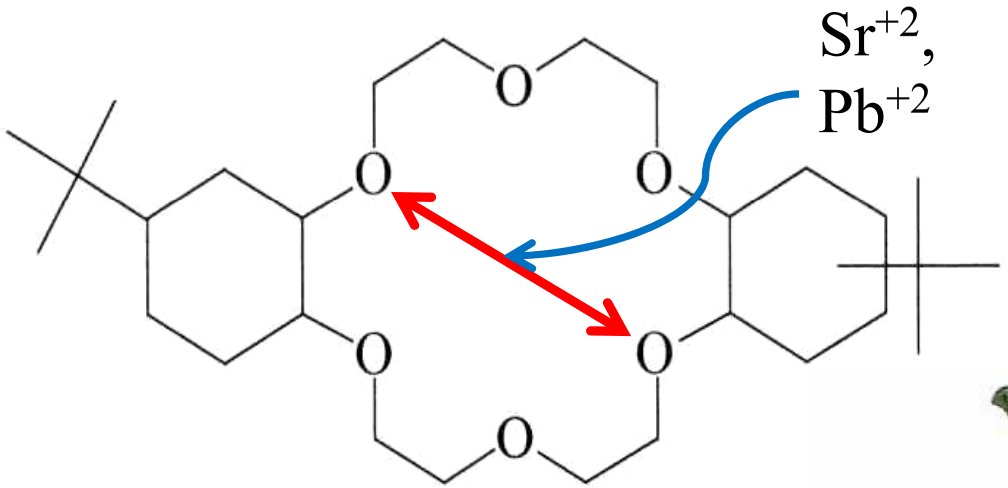
Sr Resin/ Ionic Recognition

di-t-butylcyclohexano 18-crown-6



Diluent: 1-octanol

Ionic
Recognition,
size and charge

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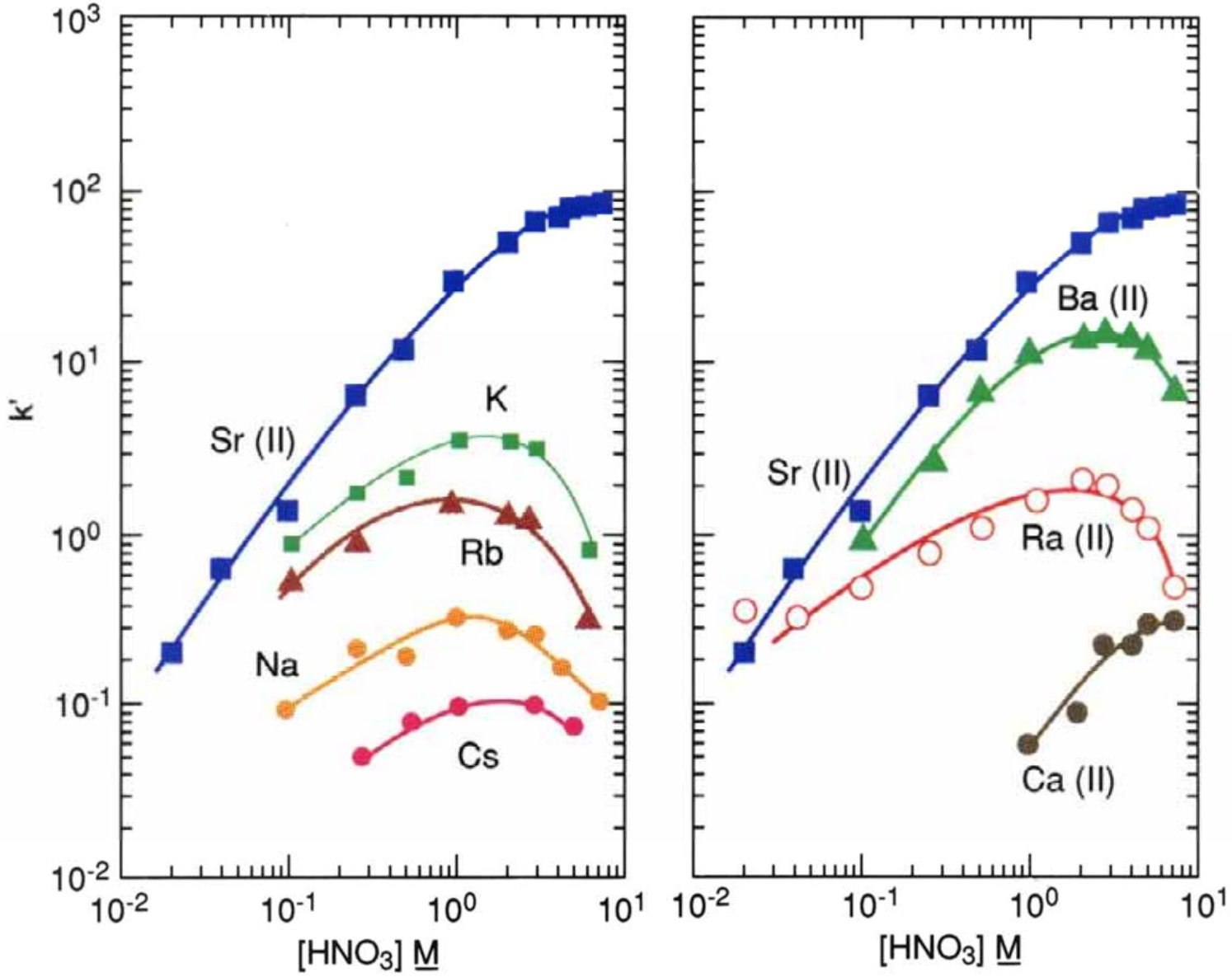
Acid dependency of k' for various ions at 23-25°C.
Sr Resin



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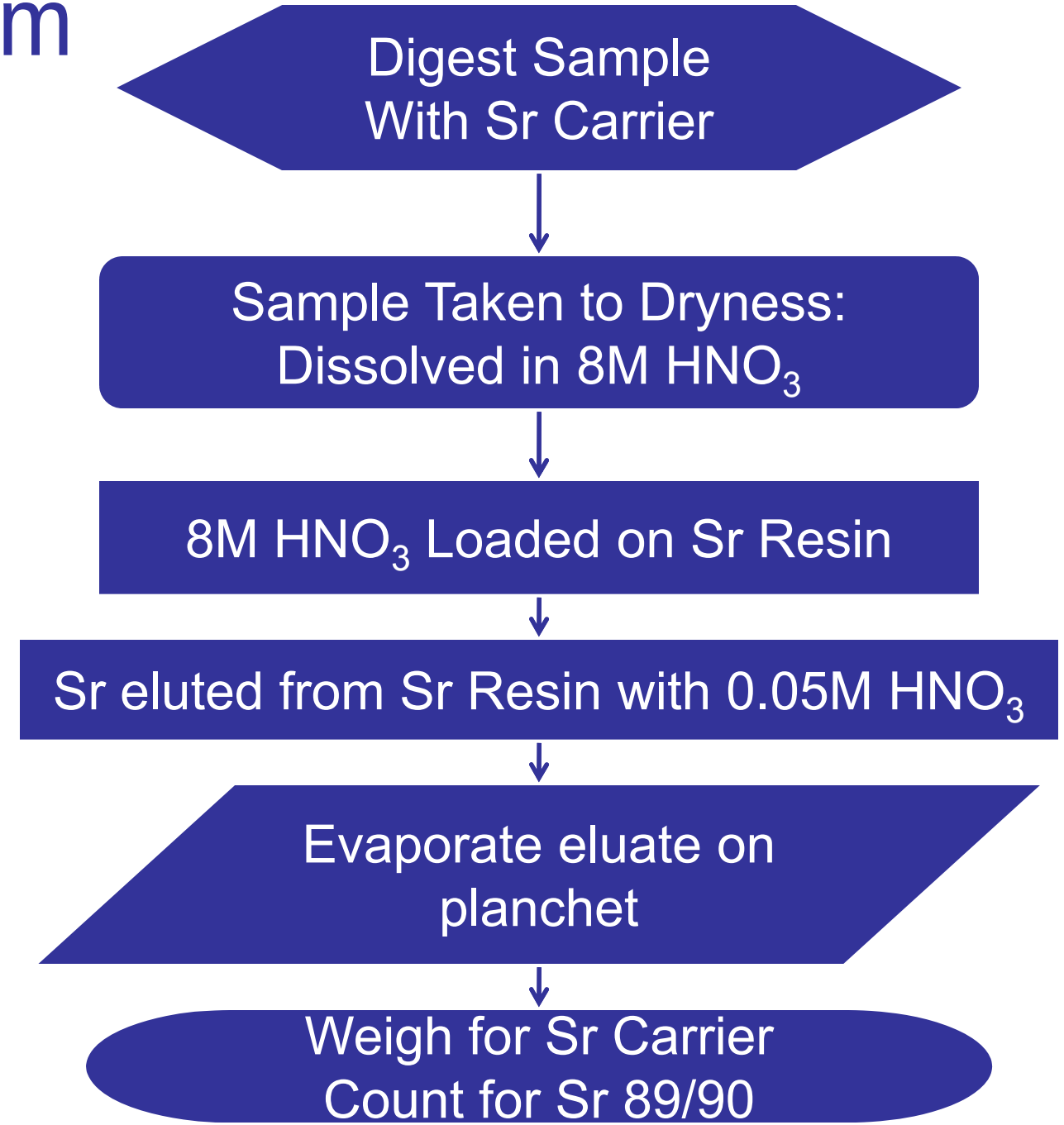
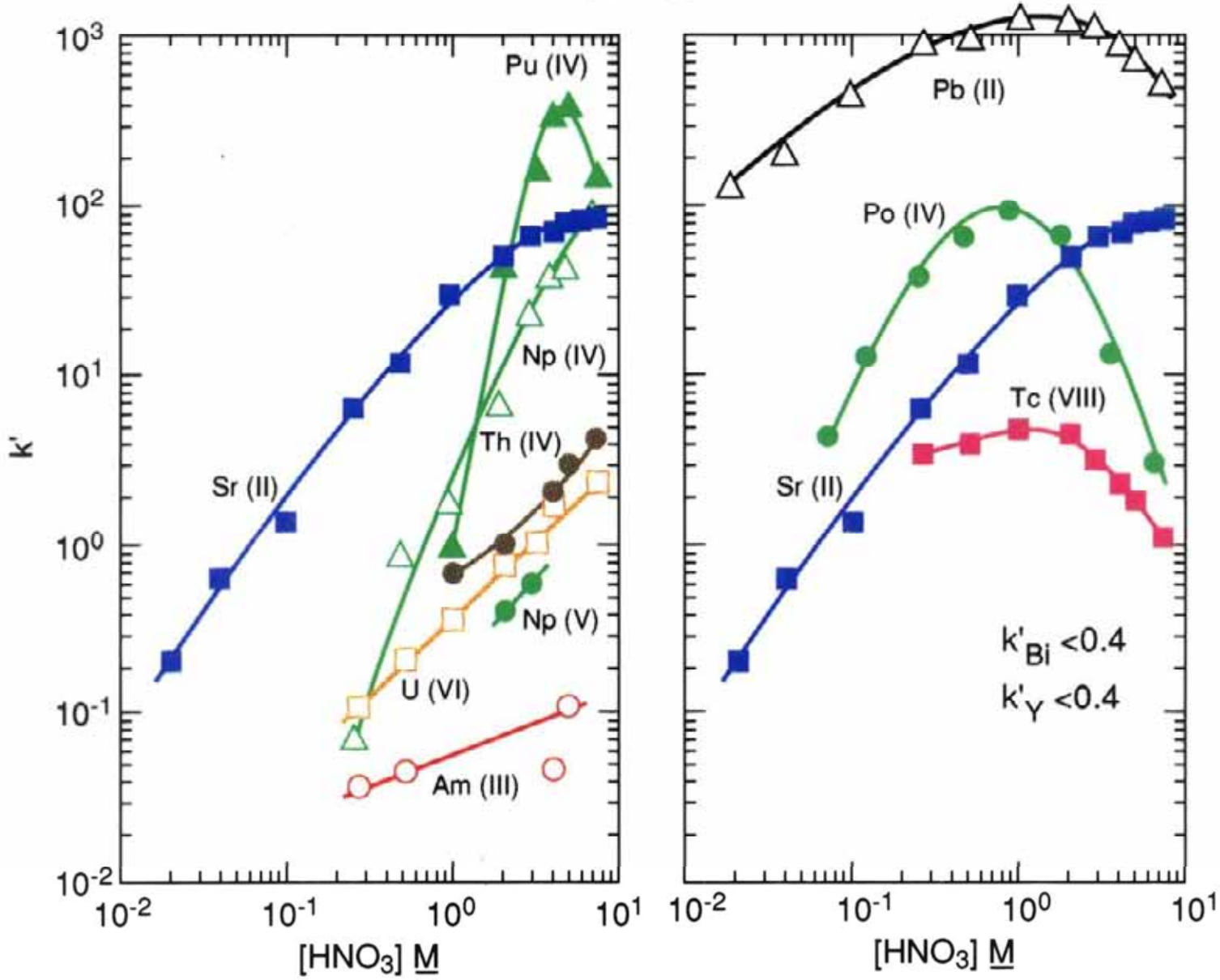

Digest Sample
With Sr Carrier
Sample Taken to Dryness:
Dissolved in 8M HNO_3
8M HNO_3 Loaded on Sr Resin
Sr eluted from Sr Resin with 0.05M HNO_3
Evaporate eluate on
planchet
Weigh for Sr Carrier
Count for Sr 89/90


Acid dependency of k' for various ions at 23-25°C.
Sr Resin
k'
[HNO3] M
Pu (IV)
Np (IV)
Th (IV)
Np (V)
U (VI)
Am (III)
Sr (II)
Pb (II)
Po (IV)
Tc (VIII)
Sr (II)
k'Bi <0.4
k'Y <0.4
[HNO3] M

Separation of Ni and Fe by anion exchange chromatography

Element	Content, %	
	Ni fraction	Fe fraction
Fe^{3+}	<0.001	>98.5
Ni^{2+}	>99.5	<0.001
Co^{2+}	<0.01	<0.1
Ba^{2+}	<7.5	<0.001
Eu^{3+}	>99.8	<0.001
Cs^{+}	>99.5	<0.001
Sr^{2+}	>99.5	<0.001

- Ni can be completely separated from Fe, Co, Cu, Zn, U, Pu, etc.
- Fe can be separated from Ni, Cr, Mn, Th, etc.

- Ni cannot be efficiently separated from Cr, Eu, Sm, Mn, V, Sc, Ti, Zr, Ba, Th, Am. Of them, the radioisotopes of Eu, Sm, Ba, Zr, Mn, Cr and matrix elements of Cr, Mn V in metal and alloy seriously interfer the determination of Ni-63.

- Fe cannot be completely separated from Zn, Co, Cu, Pu, Np, especially when a large amount of Fe (>10 mg) is loaded on the column.

Thus: a further purification for both Ni and Fe is needed.

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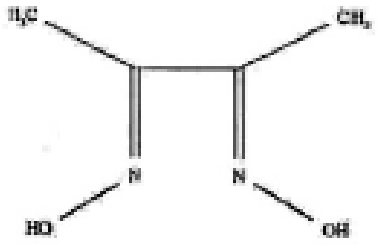
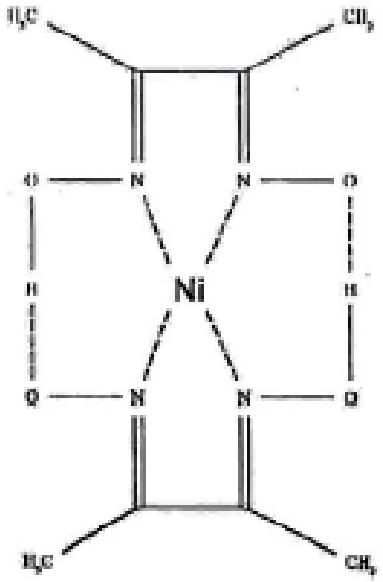
Nickel Resin

Figure 1



DMG



Ni-DMG Complex

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Physical Characteristics of Eichrom's Nickel Resin

- 11% (w/w) DMG loading
- Resin Density ~ 0.25 g/mL
- Working capacity ~ 3mg Ni

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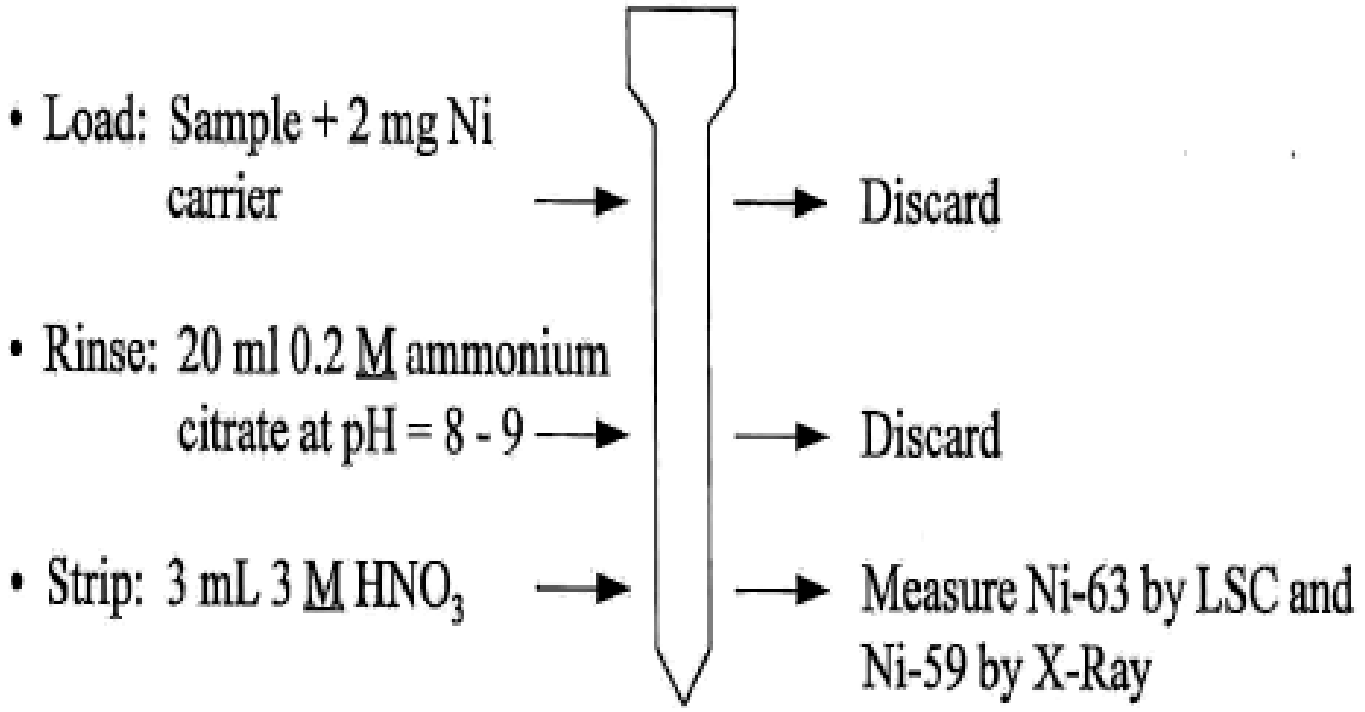
Typical Nickel Procedure

Figure 2
Typical Procedure



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Decontamination Factors -RWCU Resin Sample

Cr-51	>37,000
Mn-54	270,000
Co-58	110,000
Co-60	113,000
Nb-95	13,700
Cs-134	>9,000
Cs-137	58,000

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Cobalt Issues

- If DF for Co is not adequate
  - Treat sample through 1X-8 Anion Exchange Resin loading from 10 M HCl. The Co forms a chlorocomplex and is retained on the column while the Ni passes through in the load and rinse
  - OR add 5 mg of Co to sample this isotopically dilutes the radioactive Co

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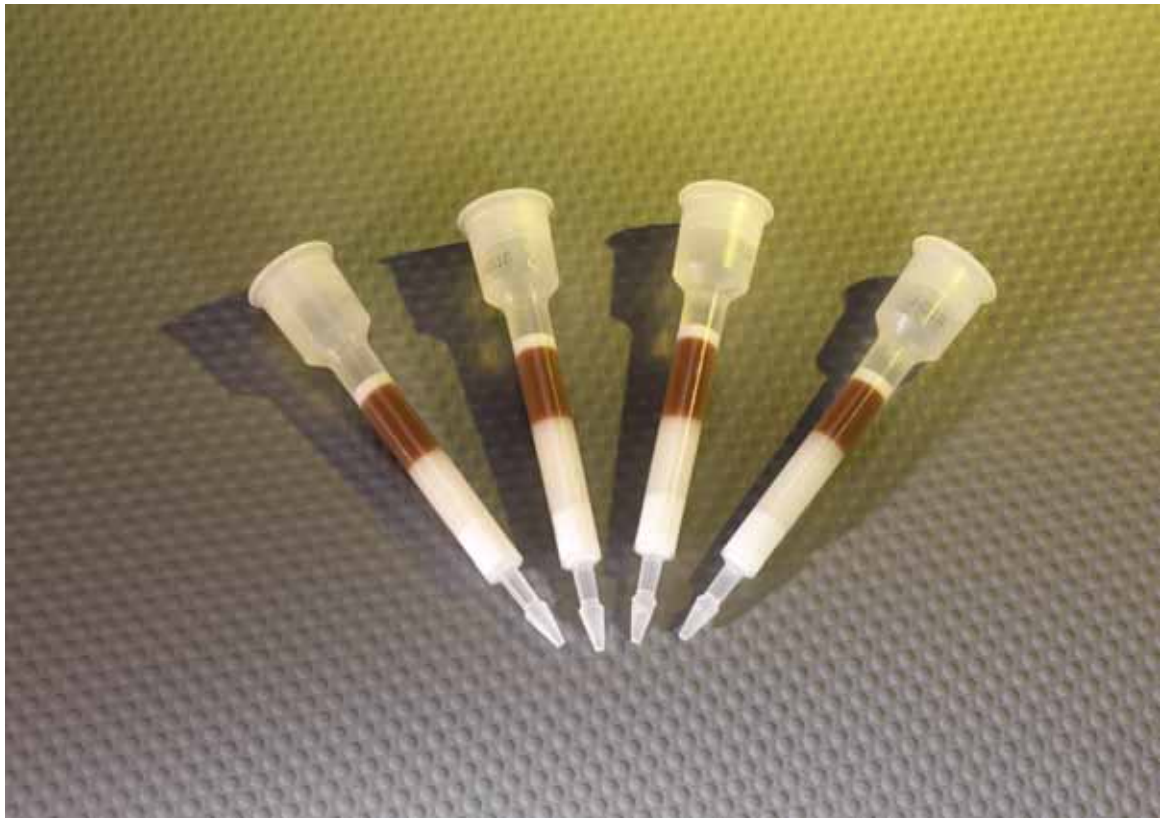
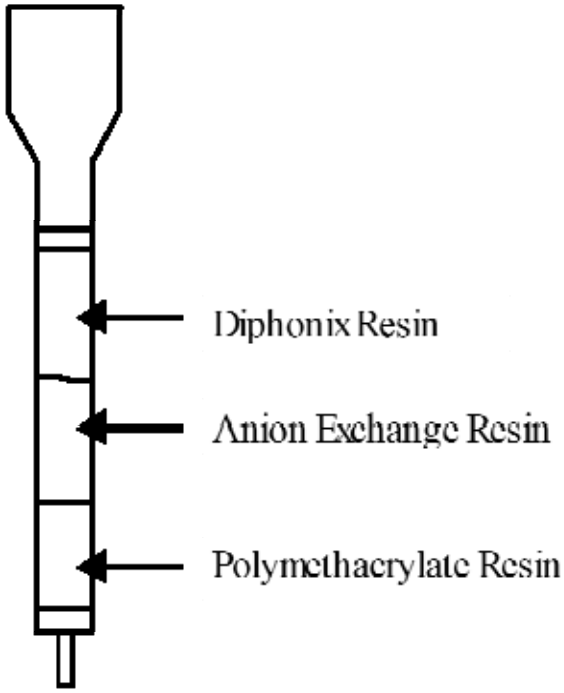
Tritium Columns





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Tritium Column Recoveries and Decontamination

Spike Recovery Results using Tritium Column			
Sample Type	Volume	3H Spike	% Recovery
Distilled water	15 mL	97.2 Bq	96.4% (n=4)
Distilled water	25 mL	20.2 Bq	87.6% (n=4)
Distilled water	25 mL	19.5 Bq	94.9% (n=7)
Ground water	25 mL	17.1 Bq	91.2% (n=5)
Sea water	25 mL	4.0 Bq	90.0% (n=6)
Urine	25 mL	85.1 Bq	91.1% (n=4)

Carolina Power & Light Samples		
Isotope	PWR Sample	BWR Sample
Cr-51	2,900	1,900
Mn-54	518	5,590
Co-58	4,740	4,960
Fe-59	109	---
Co-60	392	5,990
Sn-113	230	---
Nb-95	4,220	116
Zr-95	2,210	---
I-131	14,200	---
Cs-134	1,1200	---
Cs-137	1,320	---
La-140	---	1,550
Ce-144	---	203
D. Cahill, Carolina Power & Light, New Hill NC		

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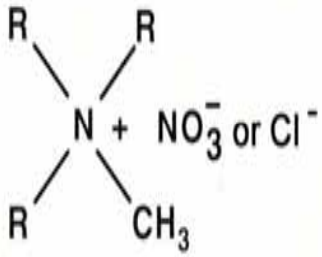
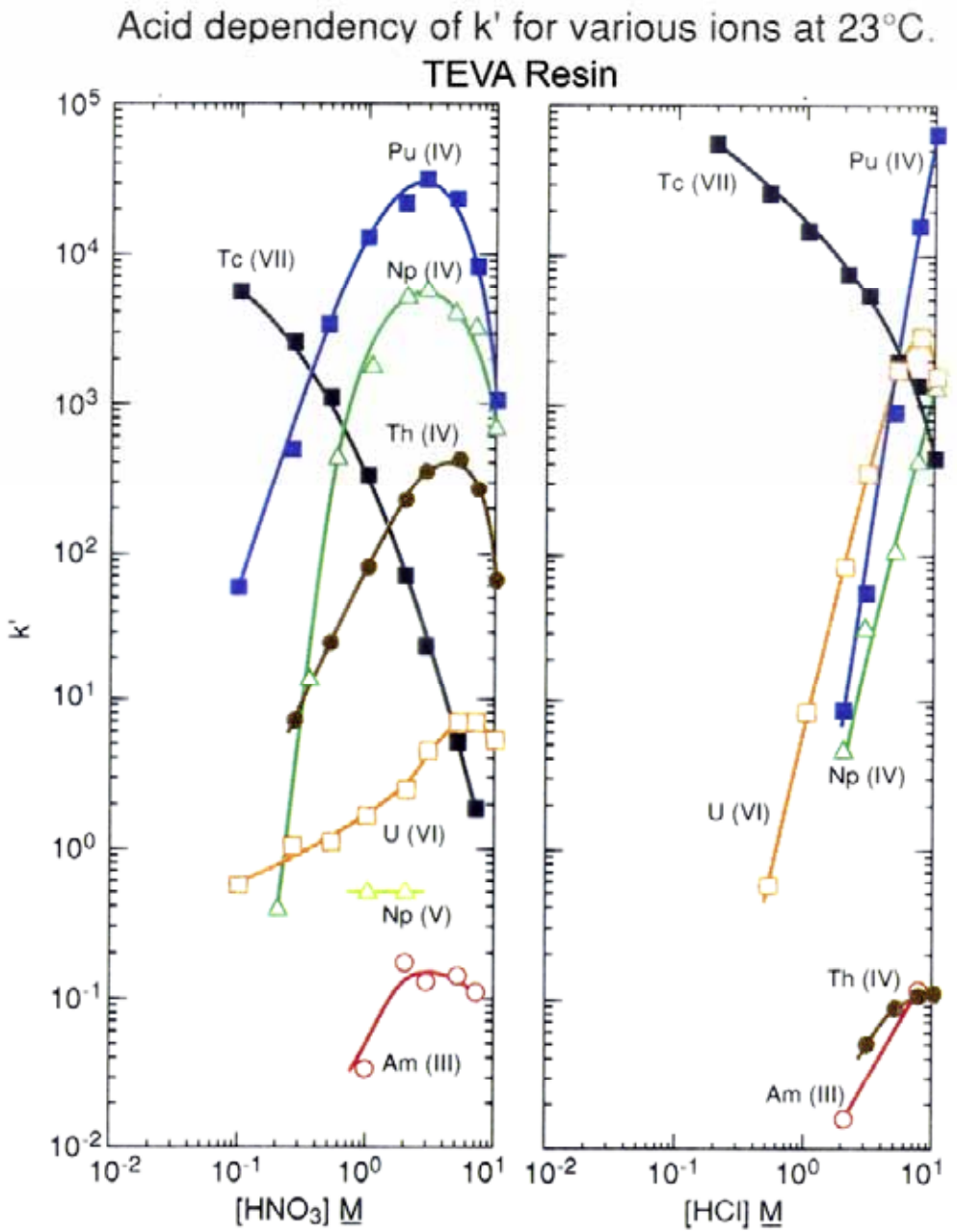
Anionic Extractant
TEVA Resin

Trialkylmethylammonium Nitrate or Chloride



R = C_8H_{17} and $C_{10}H_{21}$



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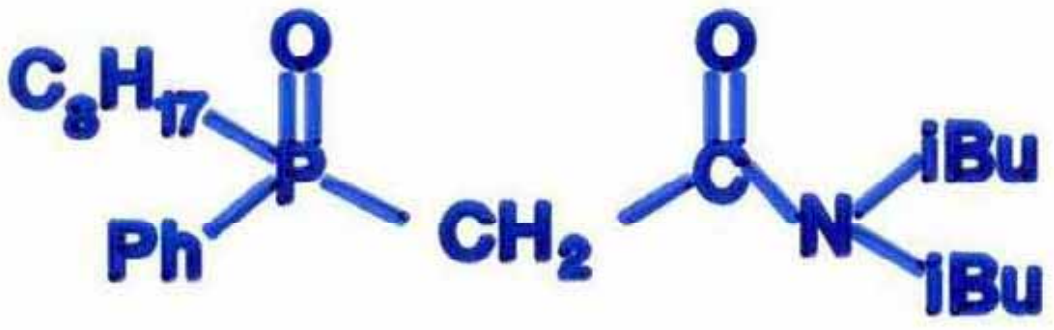
TRU Resin, Neutral Extractant

Octyl(phenyl)-N,N-diisobutylcarbamoylmethylphosphine oxide

(CMPO)



Diluent: tri-*n*-butyl phosphate

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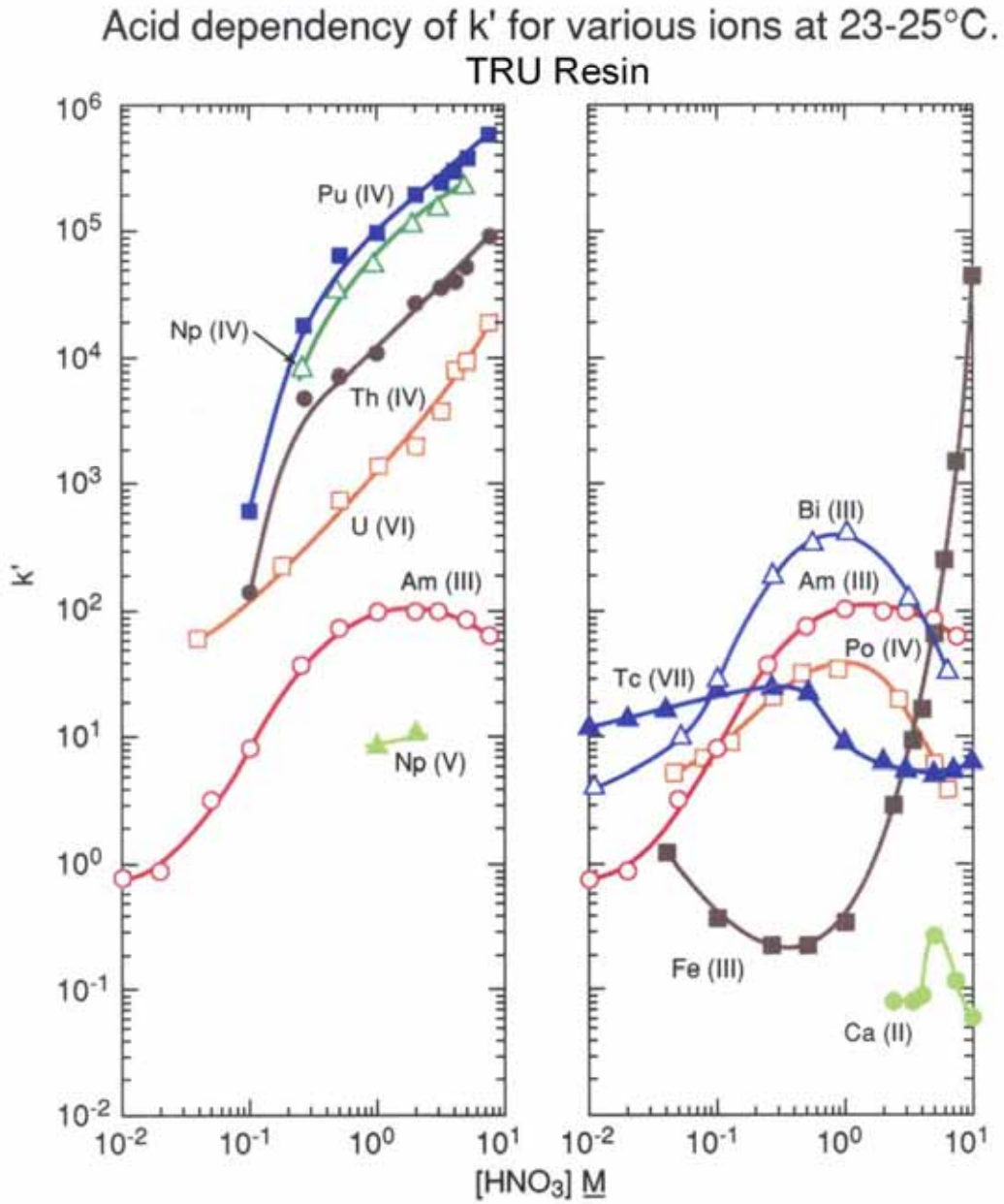
Acid dependency of k' for various ions at 23-25°C.
TRU Resin
k'
Pu (IV)
Np (IV)
Th (IV)
U (VI)
Am (III)
Np (V)
Bi (III)
Am (III)
Po (IV)
Tc (VII)
Fe (III)
Ca (II)
[HNO3] M

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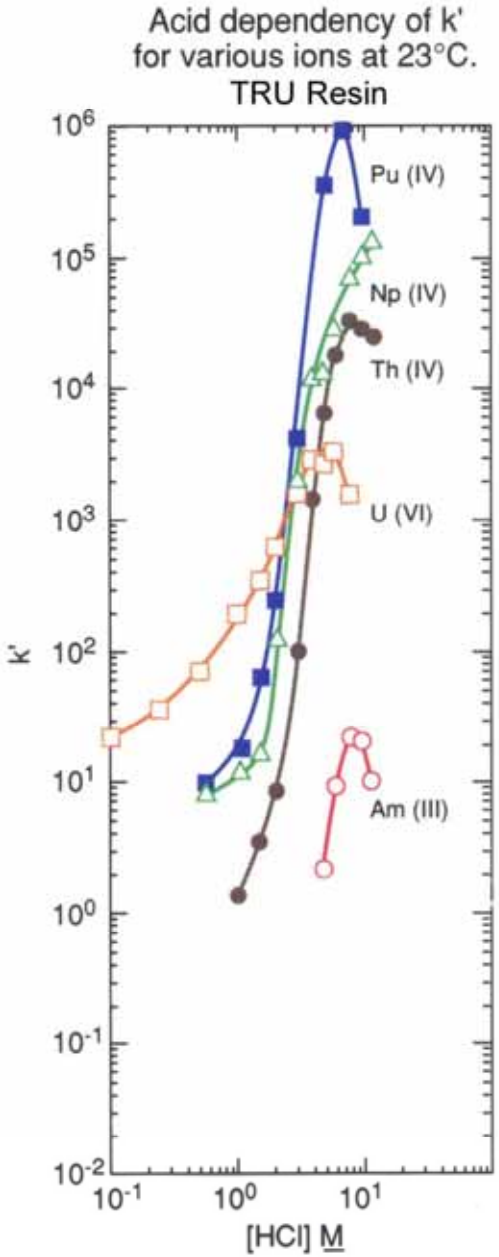
Acid dependency of k'
for various ions at 23°C.
TRU Resin
Pu (IV)
Np (IV)
Th (IV)
U (VI)
Am (III)
k'
[HCl] M

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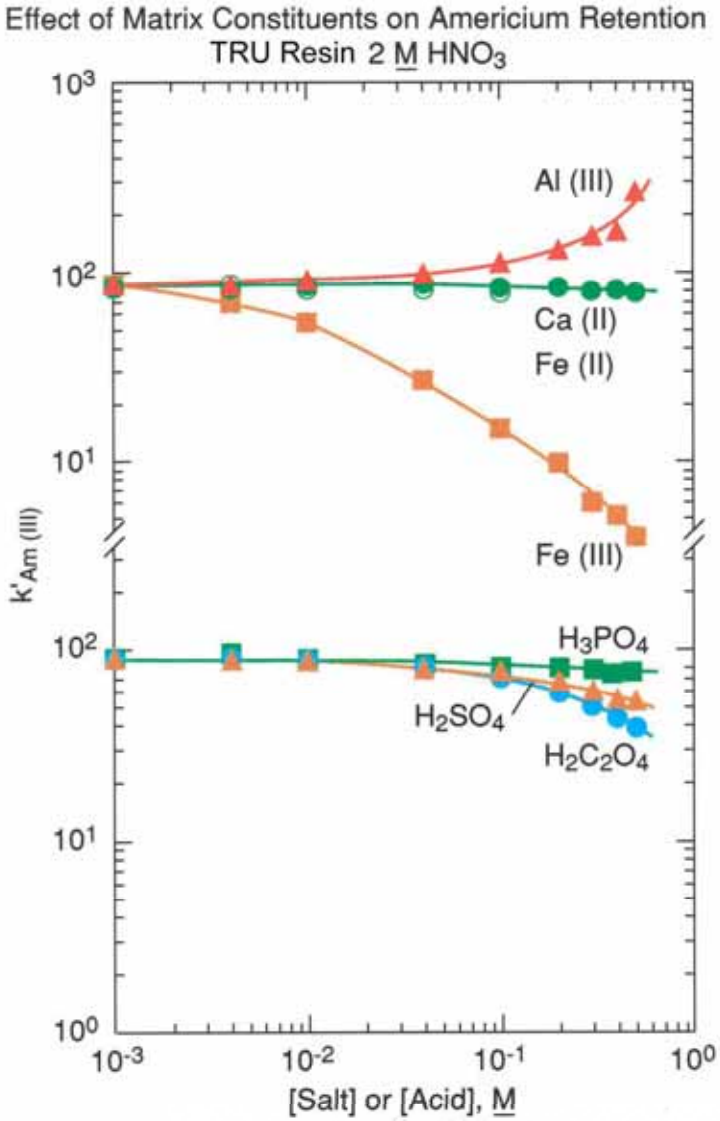
Effect of Matrix Constituents on Americium Retention
TRU Resin 2 M HNO3
k'Am (III)
Al (III)
Ca (II)
Fe (II)
Fe (III)
H3PO4
H2SO4
H2C2O4
[Salt] or [Acid], M

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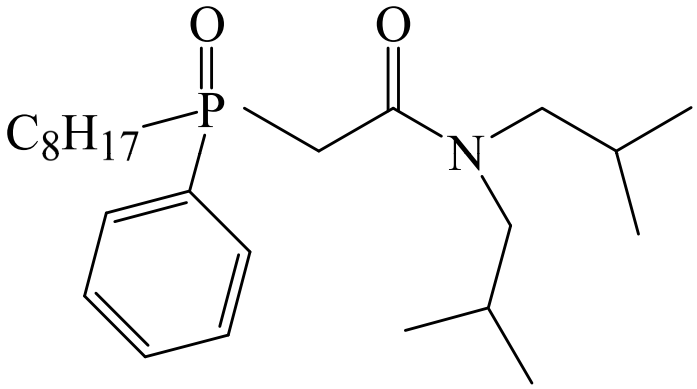
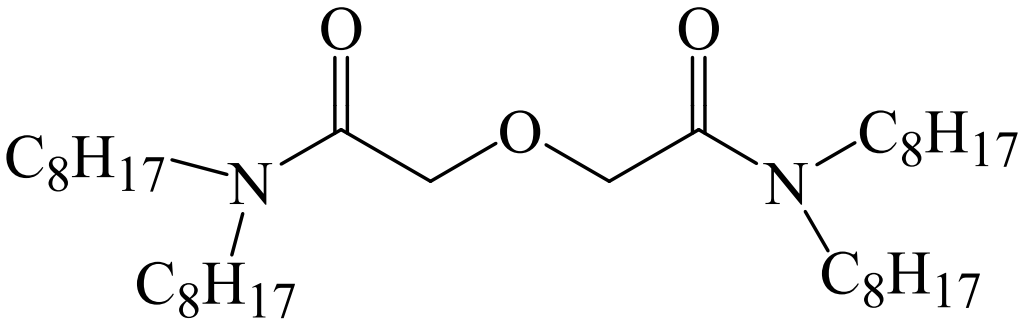
TRU Resin: Neutral Extractant

DGA Resin: Neutral extractant/ ionic recognition



TRU (CMPO)

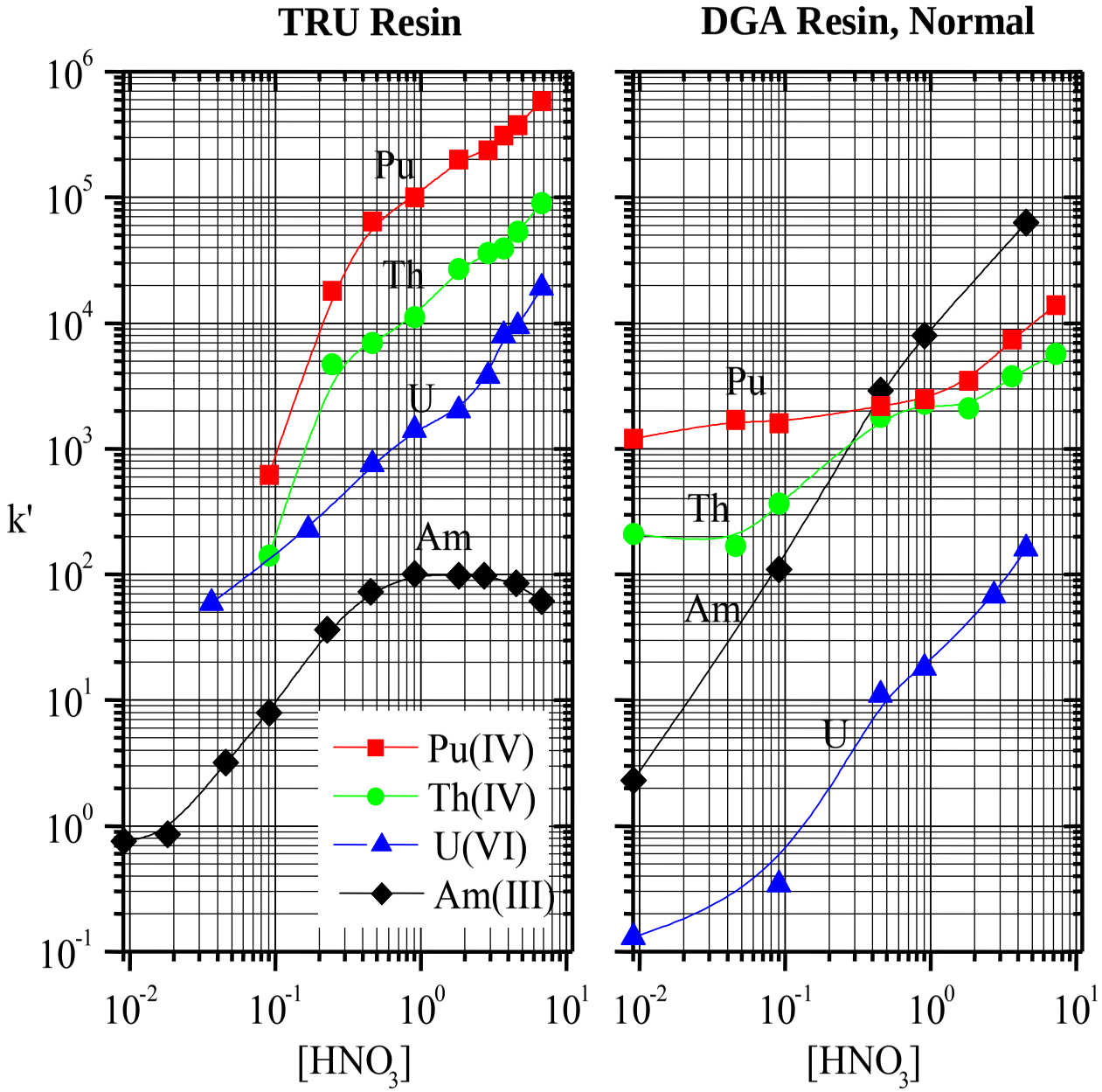


DGA

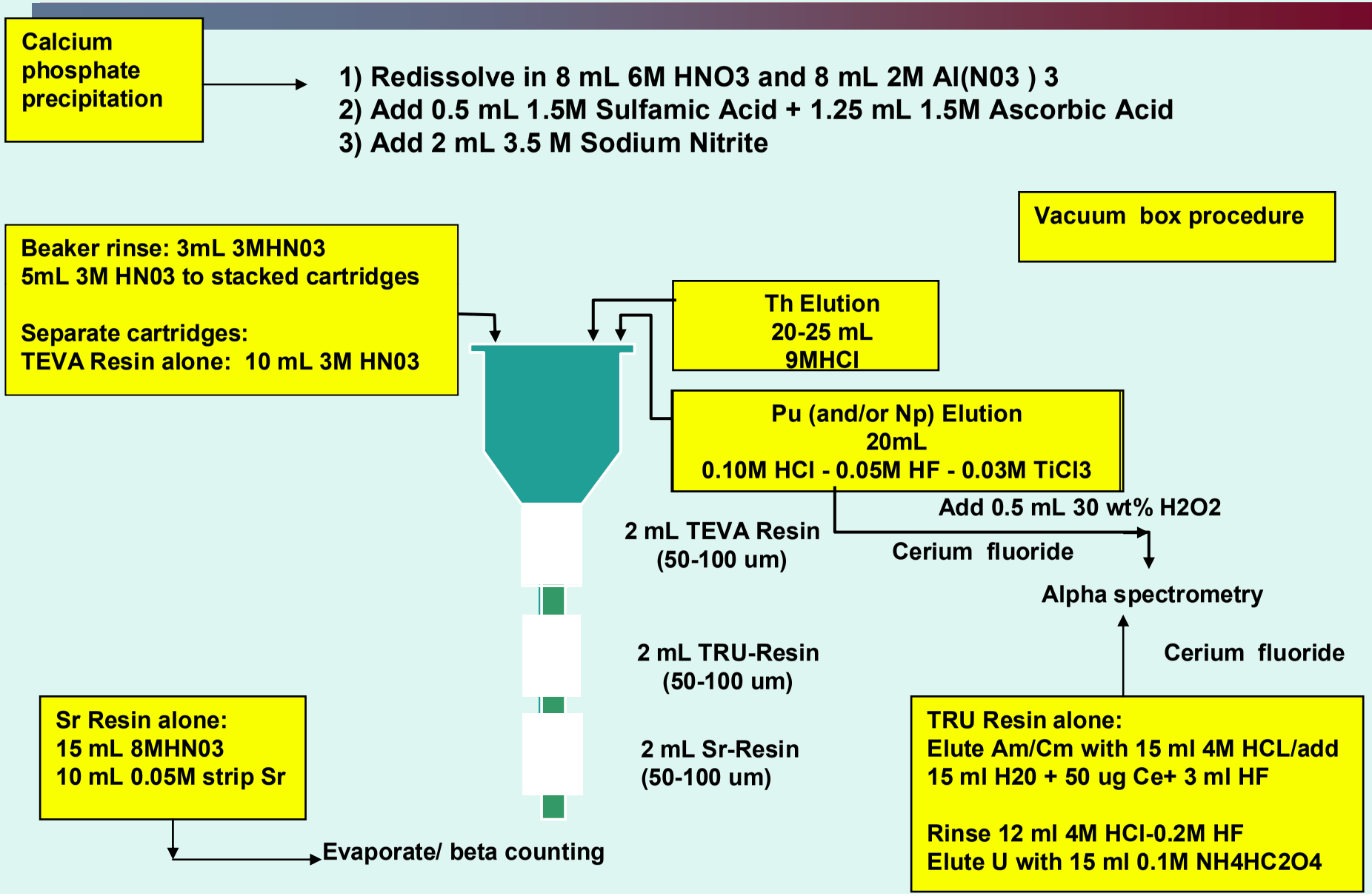
$$Am^{3+} + 3X^{-} + \overline{3E} \leftrightarrow \overline{AmX_3E_3}$$

$$X = Cl^{-} \text{ or } NO_3^{-}$$

Actinides on TRU vs. DGA out of HNO_3



Actinides and Sr-90 in Urine-NRIP 2008



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Pu in 100 ml Urine (alpha)

	Pu-236	Pu-239	Pu-239	% Diff	Pu-238	Pu-238	% Diff
	%	pCi added	pCi		pCi added	pCi	
1	111	22.5	19.6	-12.89	0.98	0.97	-1.02
2	104	22.5	22.4	-0.44	0.98	0.93	-5.10
3	108	22.5	22.2	-1.33	0.98	0.964	-1.63
4	107	22.5	22	-2.22	0.98	1.02	4.08
Avg	107.5	22.50	21.55	-4.22	0.98	0.97	-0.92

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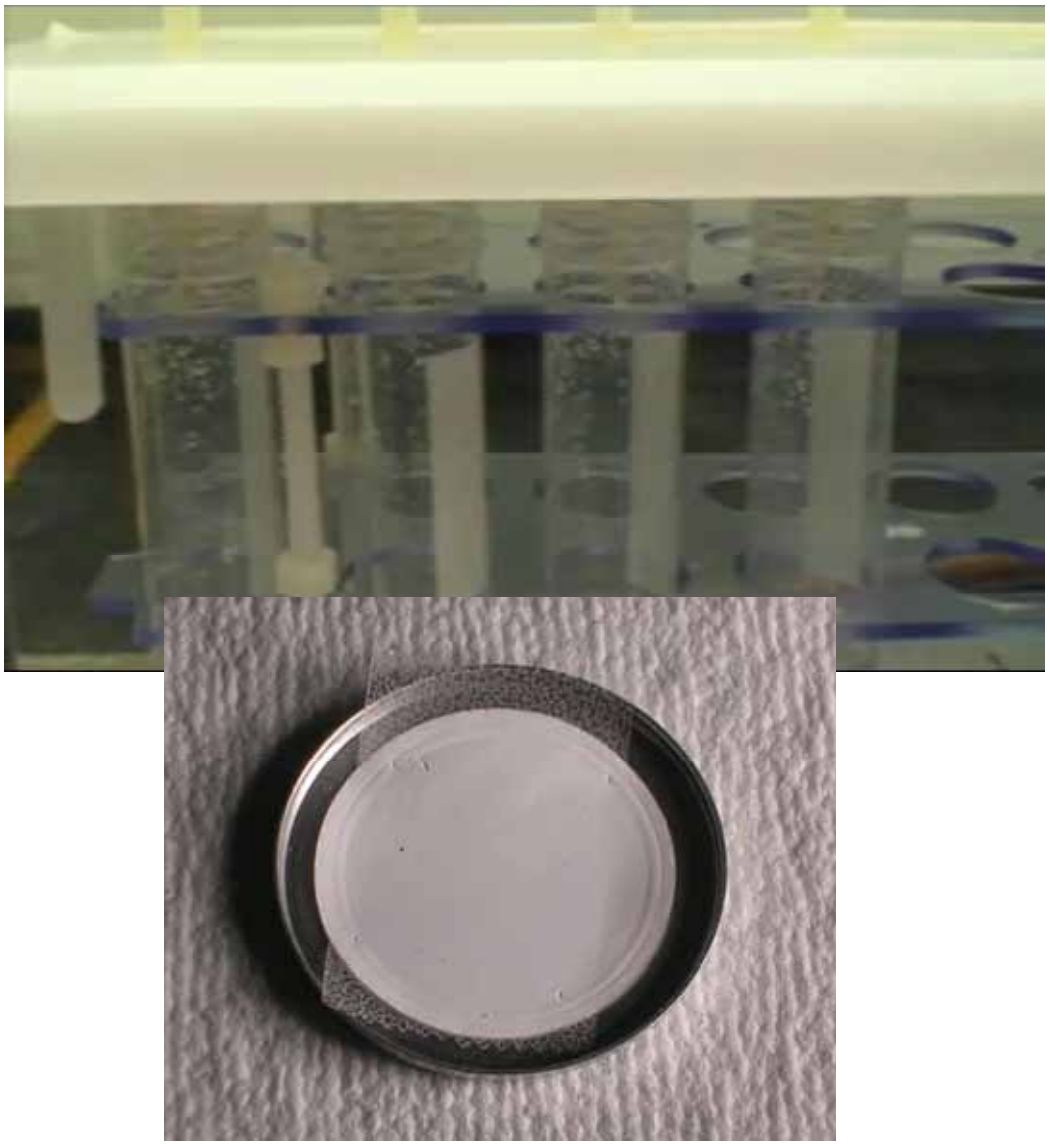
Am/ Cm in 100 ml Urine (alpha)

	Am-243	Am-241	Am-241	% Diff	Cm-244	Cm-244	% Diff
	%	pCi added	pCi		pCi added	pCi	
1	96.7	1.00	1.05	5	0.93	0.90	-2.9
2	100.1	1.00	0.89	-10.7	0.93	0.79	-15.1
3	98.7	1.00	0.90	-10	0.93	0.91	-2.2
4	95.6	2.00	1.81	-9.5	1.86	1.70	-8.6
5	98.3	2.00	1.81	-9.5	1.86	1.61	-13.4
Avg.	97.9			-6.9			-8.4

Rare Earth Co-Precipitation for Actinide Sample Alpha Source



Starting Sample

↓

Add Rare Earth Carrier (La, Ce, or Nd)

↓

Add Conc. HF acid

↓

Wait ~30 minutes

↓

Filter through 0.1µ Resolve Filter

↓

Mount and Count

eichrom Quality Control for Alpha Spec

Figure 1

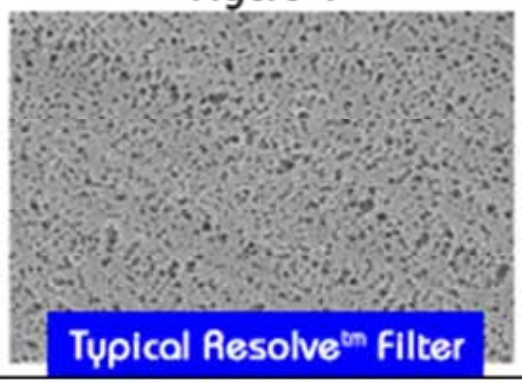


Figure 2

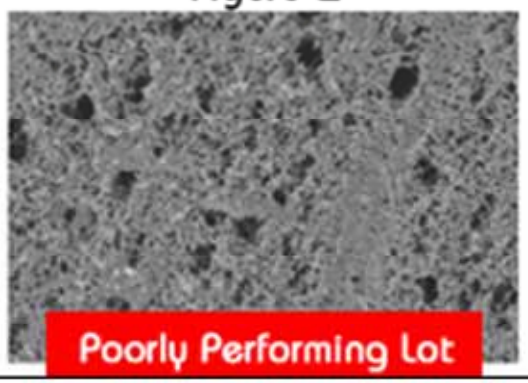


Figure 3

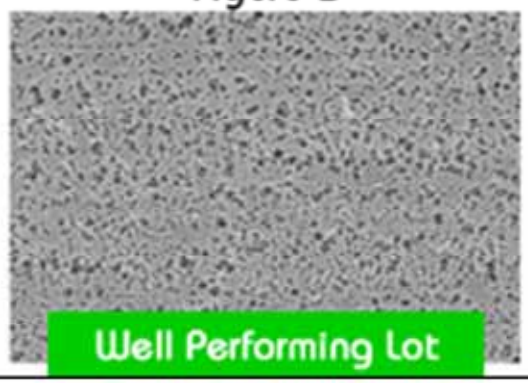


Figure 4

$^{241/243}$Am Resolution on Resolve Filter

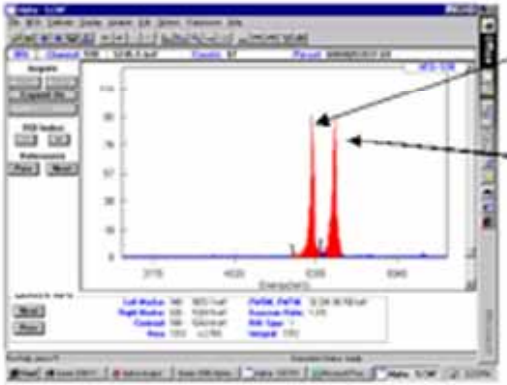
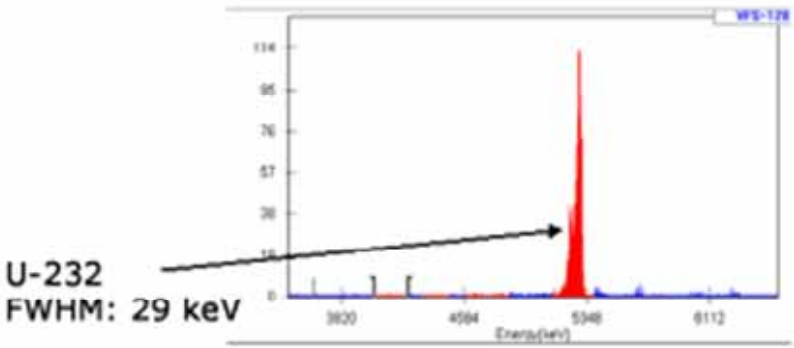


Figure 5

^{232}U Resolution on Resolve Filter



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Co-Precipitation testing of Resolve Filters

Sample	Substrate?	Orientation	FWHM (keV)	243Am Recovery	241Am Recovery
1	No	Glossy down	28	98.4%	98.4%
2	No	Glossy down	25	95.7%	95.5%
3	No	Glossy up	22	101.9%	97.6%
4	No	Glossy up	26	100.2%	99.3%
5	Yes	Glossy down	25	100.5%	96.1%
6	Yes	Glossy down	23	98.6%	97.0%
7	Yes	Glossy up	25	95.1%	94.8%
8	Yes	Glossy up	26	97.6%	95.0%

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Your separation resin tool box!

Hows

Versatility

Results

eichrom
expertise. collaboration. results.

…collaborating with the nuclear industry to protect people and the environment.

NUCLEAR POWER OUTFITTERS
a subsidiary of eichrom

NPO

NPO Products

Installation time/cost :: dose reduction :: lifecycle cost

- Shielding Materials of Construction
  - Lead
  - Tungsten
  - Steel
  - Water
  - Composites/ Laminates
  - High Temperature

Relative Thickness (approx.) of Gamma Shielding Materials (centimeter)

Water	Steel	Tungsten (solid)	NPO's "T-Flex"	Lead (solid)	Lead Blanket Layers (15#)	"Rule-of-Thumb" % Gamma Transmission Impact from shielding/typical ave. shut down Gamma Energy of 0.9 MeV
8 cm	1.3 cm	0.5 cm	1.0 cm	0.6 cm	1 (0.73kg/100cm²)	55% to 60%
10 cm	1.6 cm	0.6 cm	1.3 cm	0.9 cm	1 - 1/2	45% to 50%
14 cm	2 cm	0.8 cm	1.6 cm	1.1 cm	2	35% to 40%
18 cm	2.7 cm	1.1 cm	2.2 cm	1.5 cm	2 - 1/2	25% to 30%
30 cm	4.5 cm	1.8 cm	3.5 cm	2.5 cm	4	10% to 15%
35 cm	5.1 cm	2.1 cm	4.2 cm	3 cm	5	< 10%

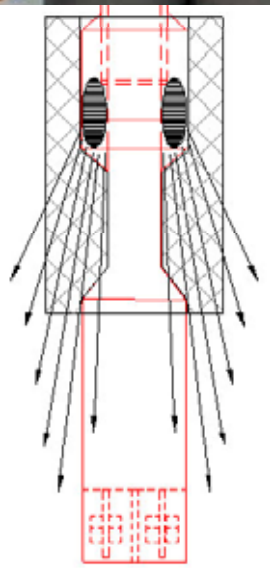
Measured Vs. Calculated Co-60 Attenuation

	1” cpm measured	Attenuation Measured %	Attenuation % Calculated at 1332 KeV	Attenuation % Calculated at 1173 KeV
No Shielding	67,500	N/A	N/A	N/A
¼” Pb Sheet	43,500	35.6%	33%	36%
T-Flex 7/16”	46,000	31.9%	34%	36%
15 # Pb Blanket	43,500	35.6%	33%	36%

Shielding Considerations

- Shielding Placement in line with maximum source term (molded to fit specific components)
- Installation and removal time critical
- Weight of individual shields sized to not exceed worker lifting capacity (integral hooks, cinch straps)
- Use of rack systems designed for shielding
- Use of Lattice Swivel Hook Systems for multi-tiered lead blankets
- Water tight storage containers for placement inside containment
- Track systems developed for easy transport of shielding when and where needed

Effectiveness::Positioning



Installation Time :: Dose

Integral
End-caps

ALARA Initiatives Saving Exposure

- Moldable lower cost siliconized tungsten iron blend
- 35 minute installation time on letdown piping (14 feet tall)
- No need to remove insulation
- Dose reduced from 900 mR to 135 mR

One 2 Meter Long Blanket = Two 1 Meter Blankets

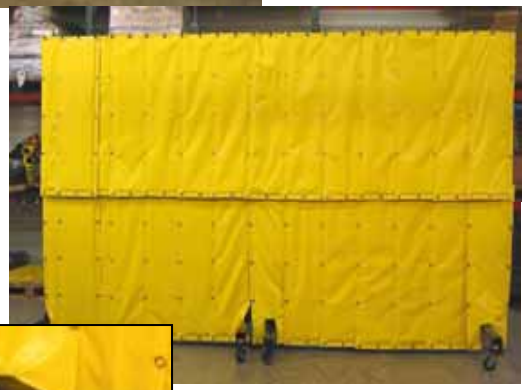
Unless you are carrying them!

Integral Hook system for end-to-end connection of lead blankets.

Flexible Designs :: Optimum Shielding Placement

LINKABLE MOBILE RACKS, INCLUDING TELESCOPING

- SINGLE AND DOUBLE TIERED, LENGTHS 3', 4' AND 6'
- ALL CONSTRUCTED FOR THREE LAYERS OF BLANKETS PER SIDE
- DIFFERING LEG HEIGHTS PERMITS "L" SHAPED SHIELD WALLS
- ALL LEGS INCLUDE PROVISIONS FOR OUTRIGGERS (OPTIONAL)
- TELESCOPING SINGLE AND MULTI-TIERED MOBILE RACKS ARE AVAILABLE



No Floor :: No Problem

Lattice Swivel Hook Systems
Create multi-tiered lead blanket support.
Will be self supporting on vertical piping (bare pipe 8” and larger

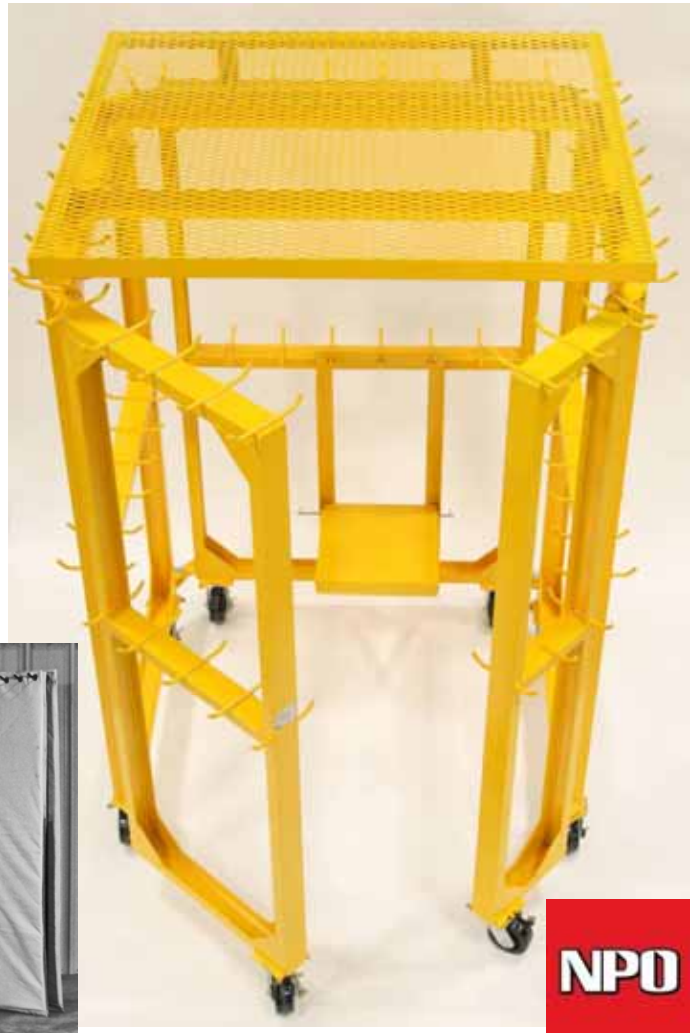
CONTAINERS OF ALL TYPES
SPECIFICALLY DESIGNED FOR THE APPLICATION

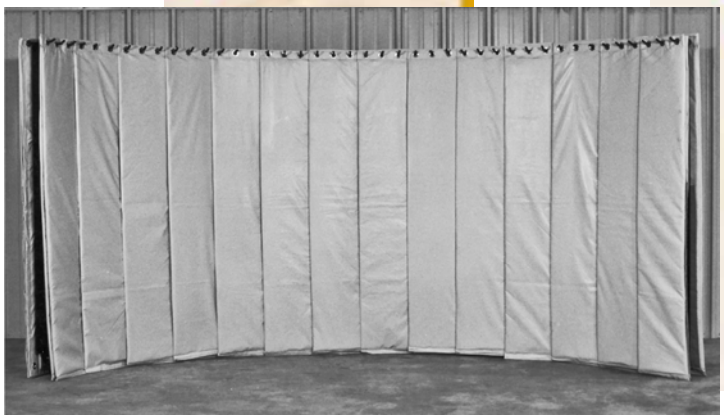
FABRICATED FROM EPOXY COATED STEEL OR ALL STAINLESS STEEL.AS SHOWN ABOVE. TYPICAL CAPACITIES OF 7,000+ POUNDS AND STACKABLE TO THREE HIGH. DESIGN PERMITS SECURING OF CONTAINER TO EXISTING STRUCTURE FOR IN CON-TAINMENT APPLICATIONS. OPTIONAL LIGHT WEIGHT ALUMINUM COVER (ABOVE).

REMOVABLE SIDE PANELS
- **PANELS SLIDE IN AND DOWN OVER FASTENERS THROUGH KEY HOLE SLOTS.**
- **ALL ENDS AND COVER ARE REMOVABLE**
- **FASTENERS ARE CAPTURED.**

Utilizing our lead blankets better!

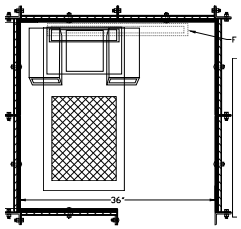
NPO's Mobile Frisker/Low Dose Waiting/Rest Area Booth System





Specialized Shielding Systems

Frisker Booths, PCM Booths and Hand and Foot Monitor Booths



NPO's low profile and flexible track system

Application of Track Systems for Reduced installation time and dose. Permanent Storage of conventional shielding materials in containment.

NPO's low profile and flexible track system, Regen. HX Application

END VIEW

ELEVATION VIEW

FULLY ENCASE SHIELD PANELS (1" LSE) SIMILAR TO NPO'S RX HEAD SHIELD SYSTEM

STAINLESS STEEL CLAMPING HARDWARE AND PIVOTING TROLLEY UNIT

PLAN VIEW

REGEN. SHIELD SYSTEM CONCEPT OVERVIEW:

- SHIELDING STORED IN DOT TYPE STRONG BOX VERTICAL CABINET LOCATED IN LOWER DOSE RATE AREA.
- SHIELDING TRAVELS ON SPECIALIZED TRACK THAT ALLOWS FOR DIRECTIONAL BENDS AS ILLUSTRATED.
- SHIELDING, AS ILLUSTRATED, PROVIDES 1" OF LSE WITH OVERLAPPING PIVOTING PANELS.
- SHIELDING PANELS SHOWN ARE SIMILAR TO NPO'S REACTOR HEAD SHIELD PANELS.
- SAME TRACK/STORAGE APPROACH IS AVAILABLE USING RIGID LEAD ENCASED STEEL PANELS.
- PANELS PIVOT AND OVERLAP PROVIDING ACCESS TO ANY AREA BEHIND SHIELDS.
- TRACK SUPPORTED BY 4 OR 5 VERTICAL SUPPORTS AND LATERALLY TO EXISTING STRUCTURE.

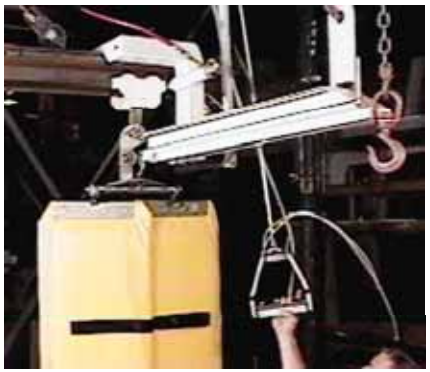
SHIELD PANELS SIMILAR TO NPO'S REACTOR HEAD SHIELD SYSTEM AS PREVIOUSLY INSTALLED AT INDIAN POINT NUCLEAR PLANT

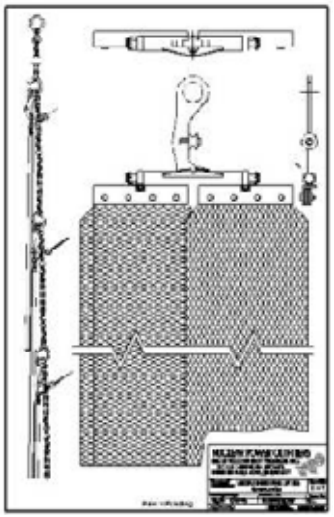
EFFECTIVE SHIELDING OF 1" OF LEAD PRIVIDING ATTENUATION IMPACT OF APPROX. (5X) OR TRANSMISSION OF 20%.

NPO

Specialized Shielding Systems

NPO's Reactor Head Shield System for PWRs





Summary

- Recognized Worldwide, Eichrom and NPO deliver solutions tailored to your specific applications
- Solutions built from a combined 50 years of nuclear experience in collaboration with you
- We invite you to participate in the next era of successful ALARA solutions