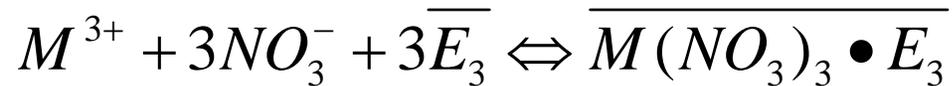
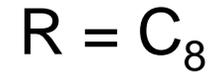
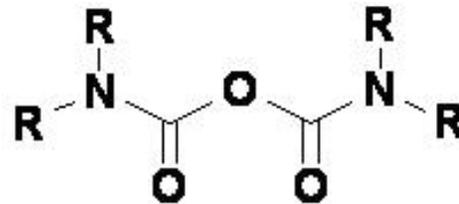


Application of  
extraction chromatography  
to the  
determination of radionuclides in  
soil and biological samples

## Outline

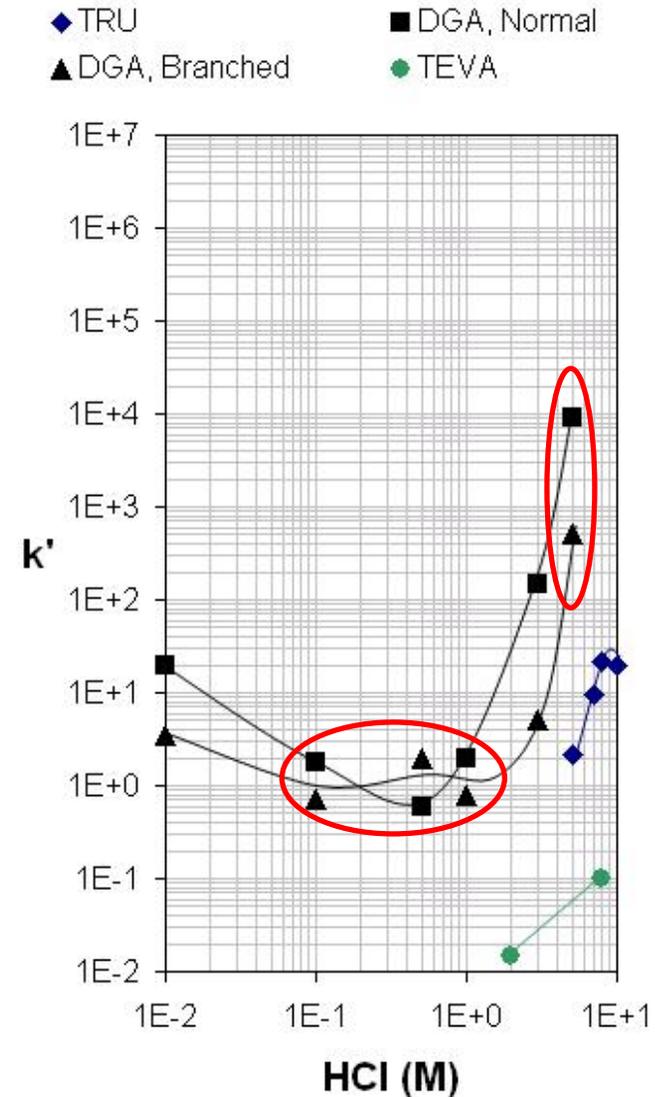
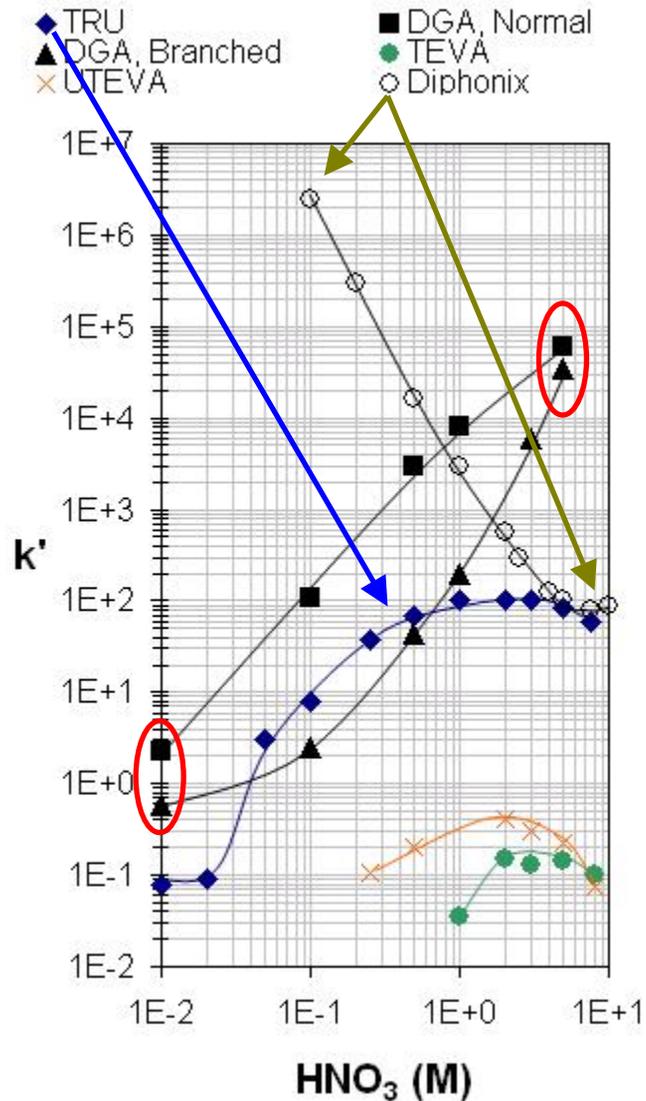
- DGA Resin
- Application to soil samples
  - Preconcentration
  - Separation
- Other matrix elimination steps
- Biological samples

## DGA Resins

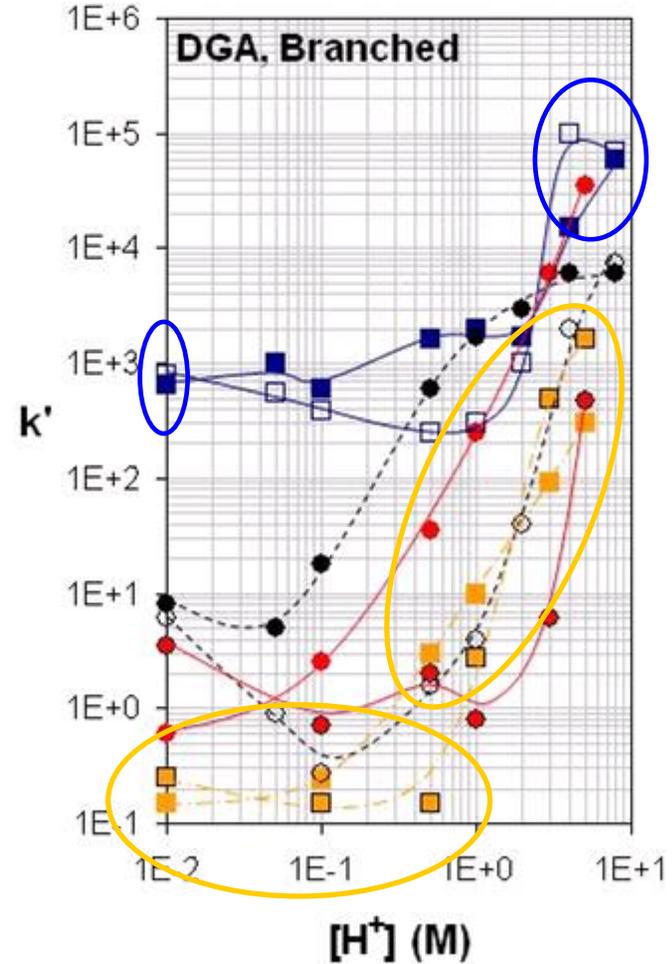
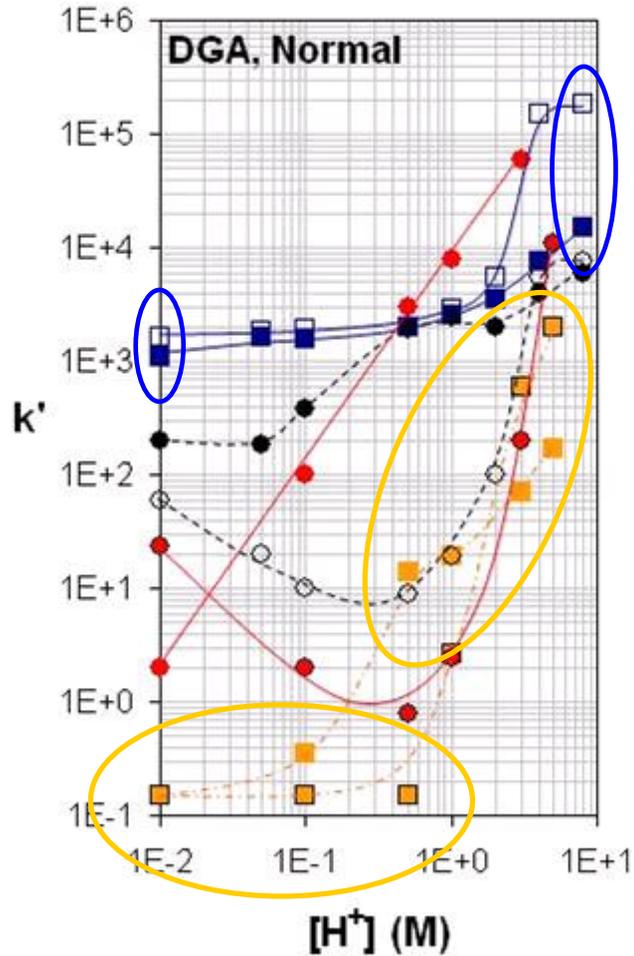


- DGA, Normal (*N,N,N',N'*-tetra-*n*-octyldiglycolamide)
  - Generally used
- DGA, Branched (*N,N,N',N'*-tetrakis-2-ethylhexyldiglycolamide)

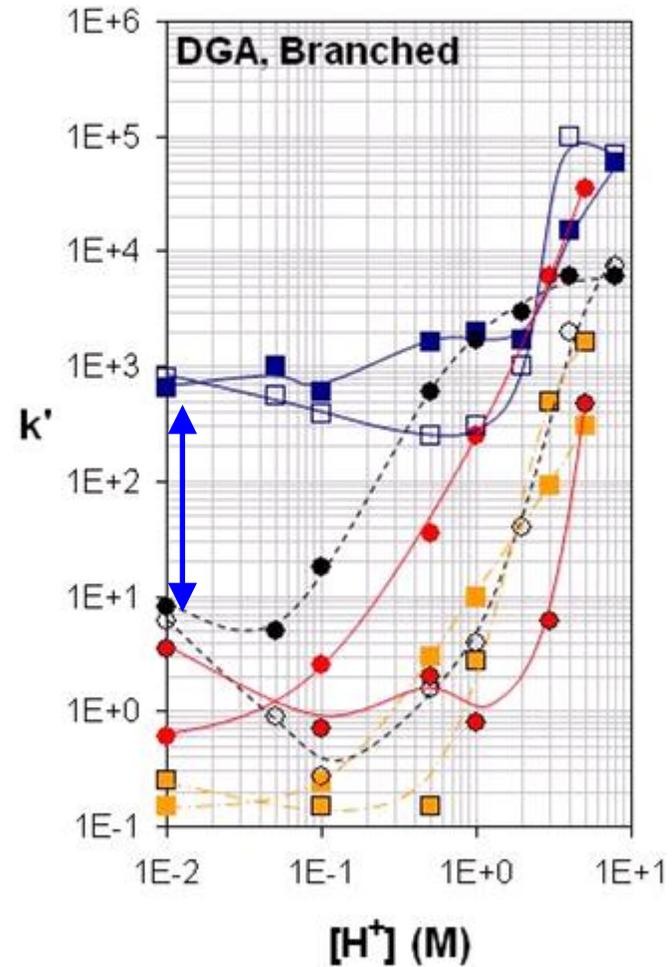
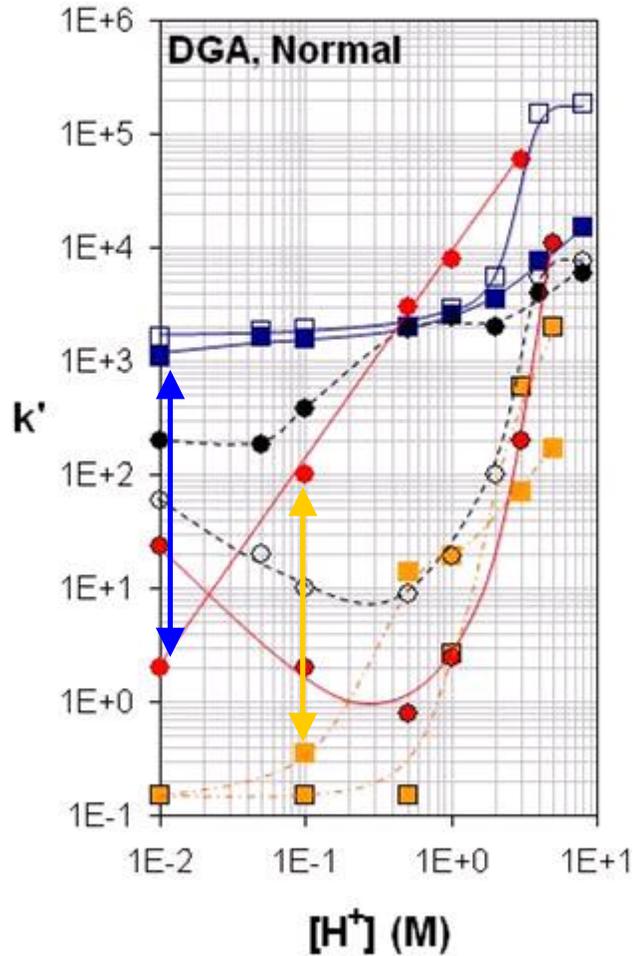
## Americium k' values



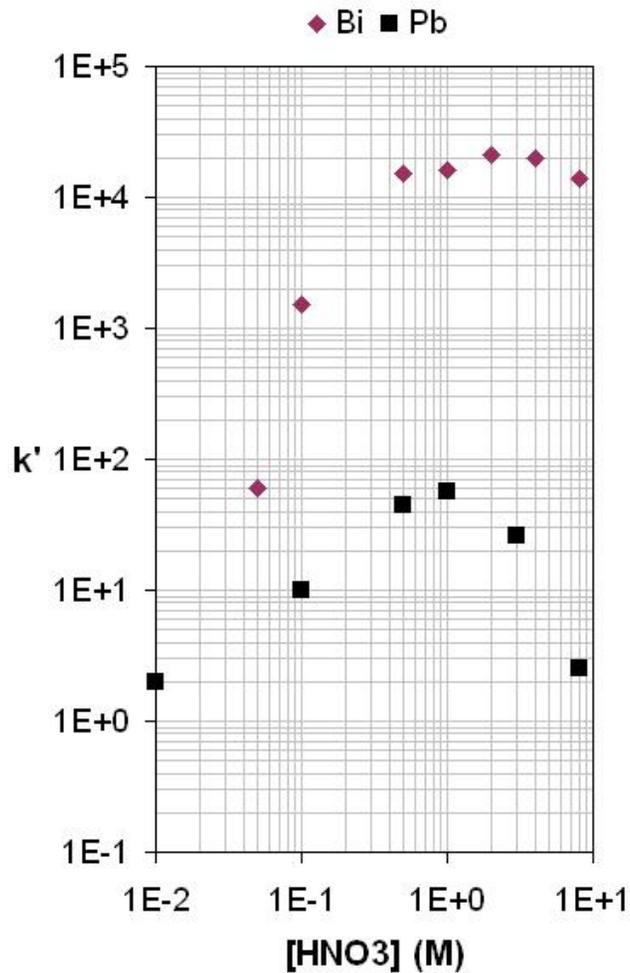
## Americium Separation



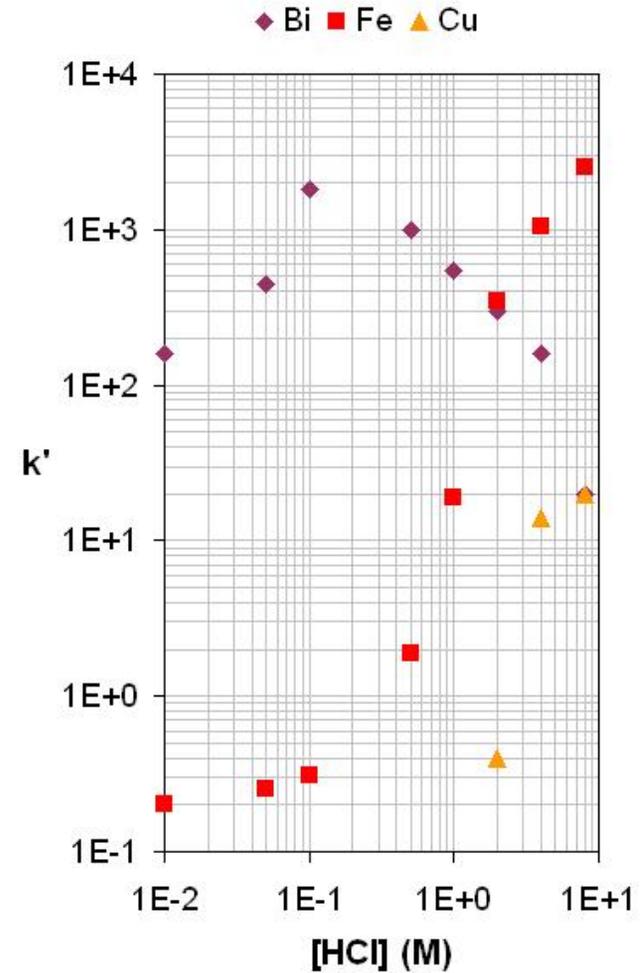
## Americium Separation



## Interferents



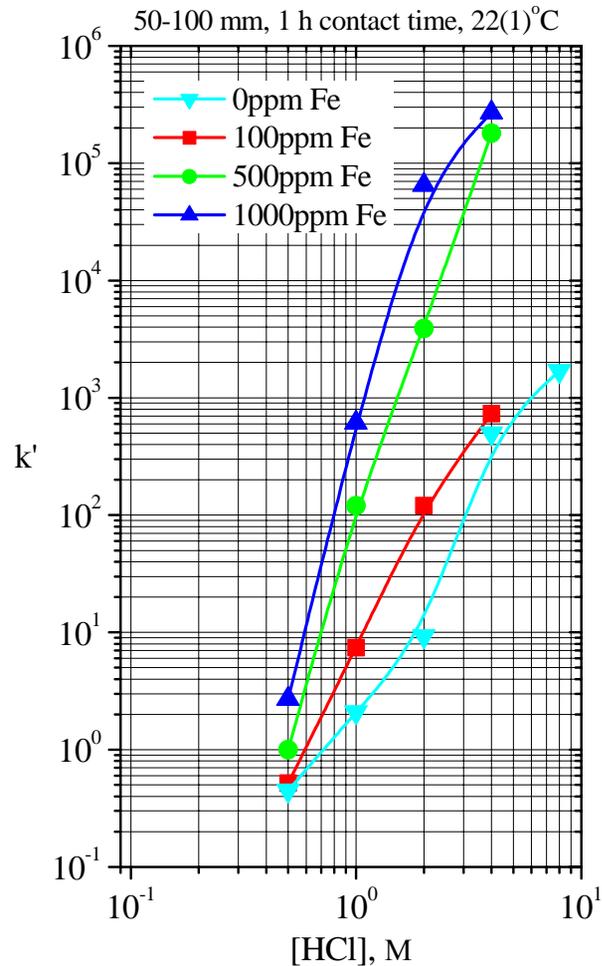
$k'$  for V(V), Ti(IV), **Al(III)**,  
**Fe(III)**, Co(II), Cu(II), Ni(II),  
 Zn(II) < 2 for all [HNO<sub>3</sub>]



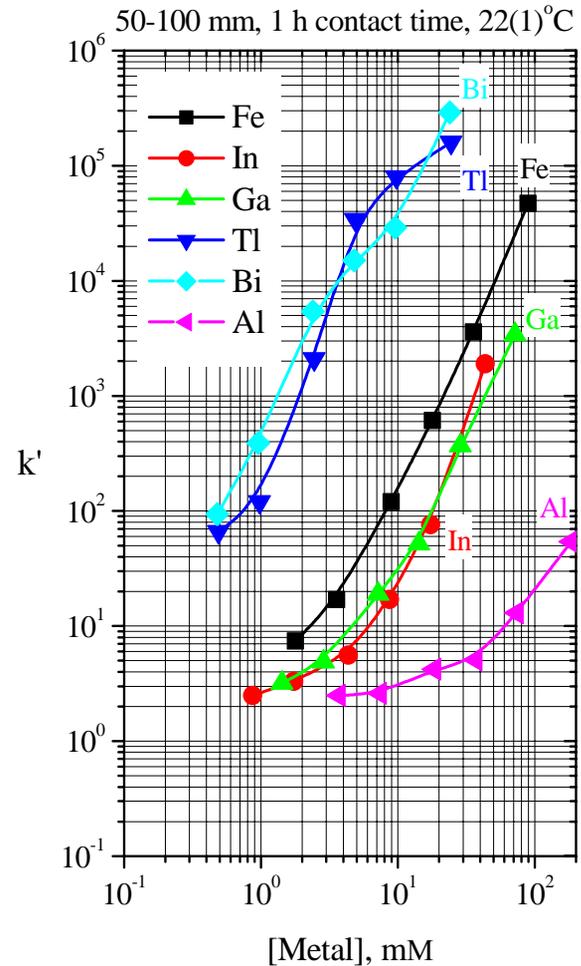
$k'$  for Ti(IV), **Al(III)**,  
 Co(II), Ni(II), Pb(II),  
 Zn(II) < 2 for all [HCl]

# EICHROM ENVIRONMENT

Acid Dependency of  $k'$  for Am from HCl + Fe on DGA

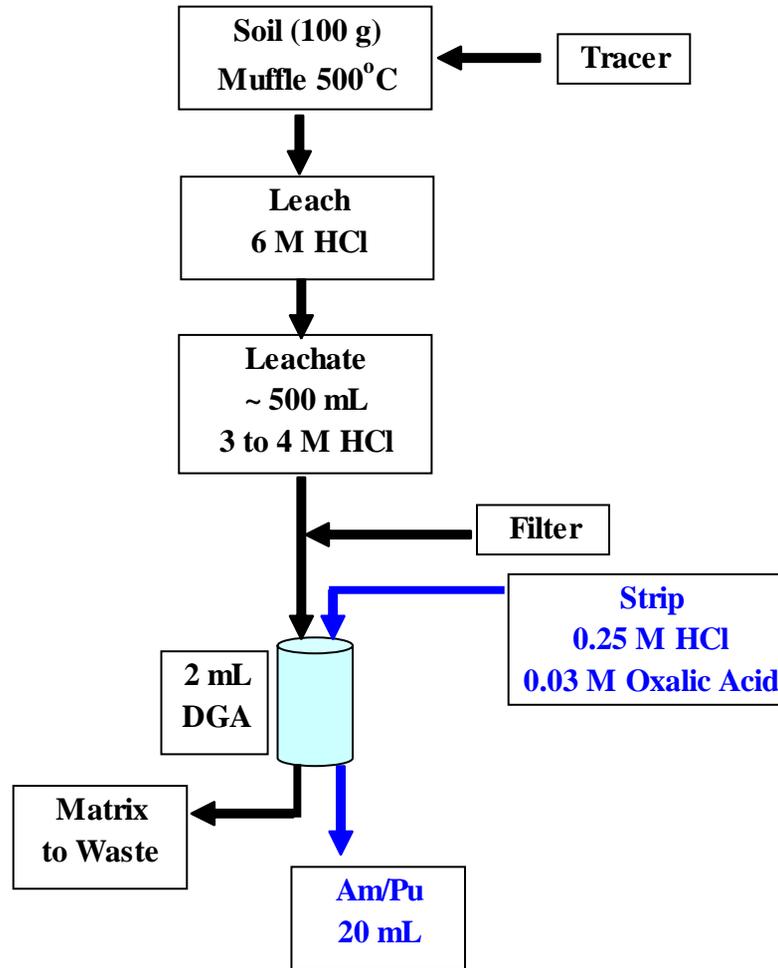


$k'$  for Am(III) on DGA from 1 M HCl + M(III)



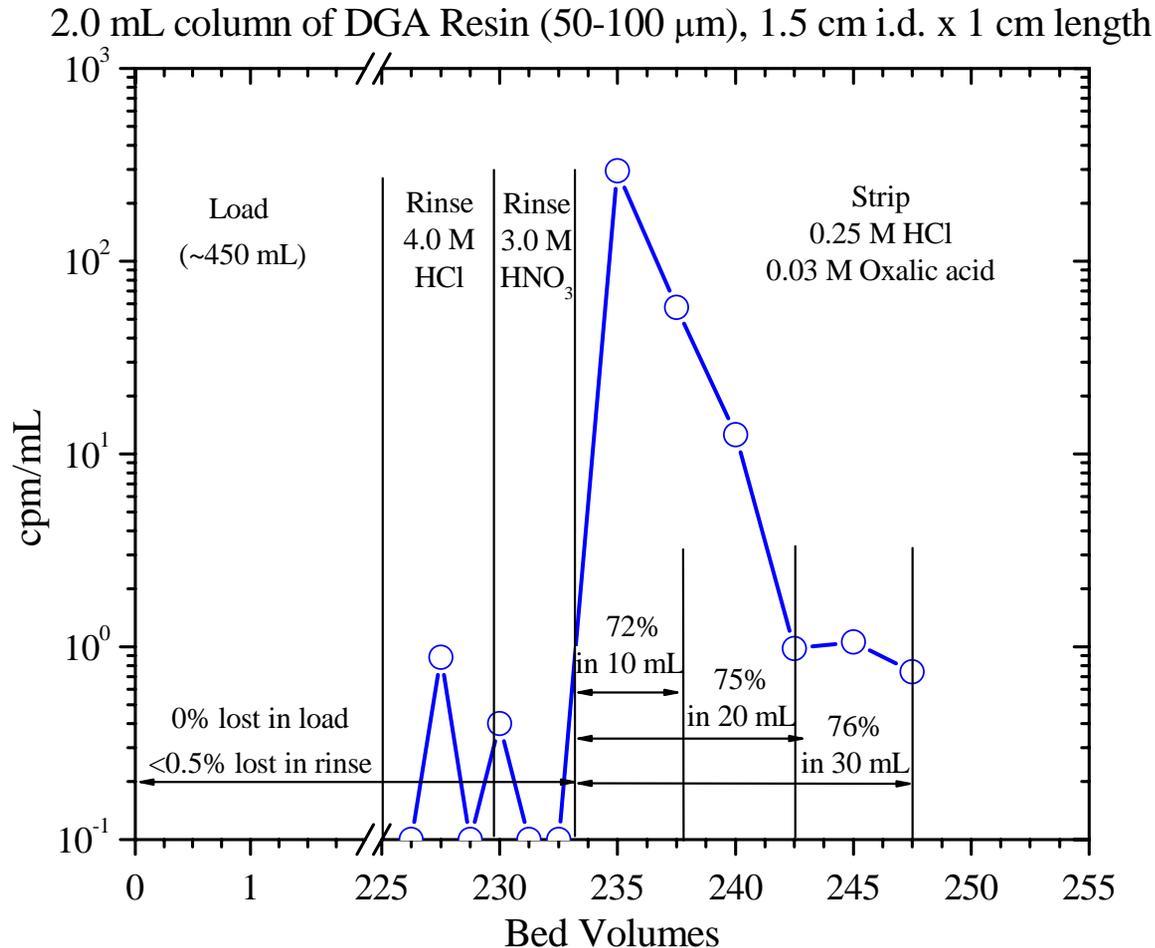
# EICHROM ENVIRONMENT

## Flowchart for the Preconcentration of Am and Pu from 100 g of Soil



# EICHROM ENVIRONMENT

## Elution of Am and Pu from 100 g Soil Leach on DGA Resin



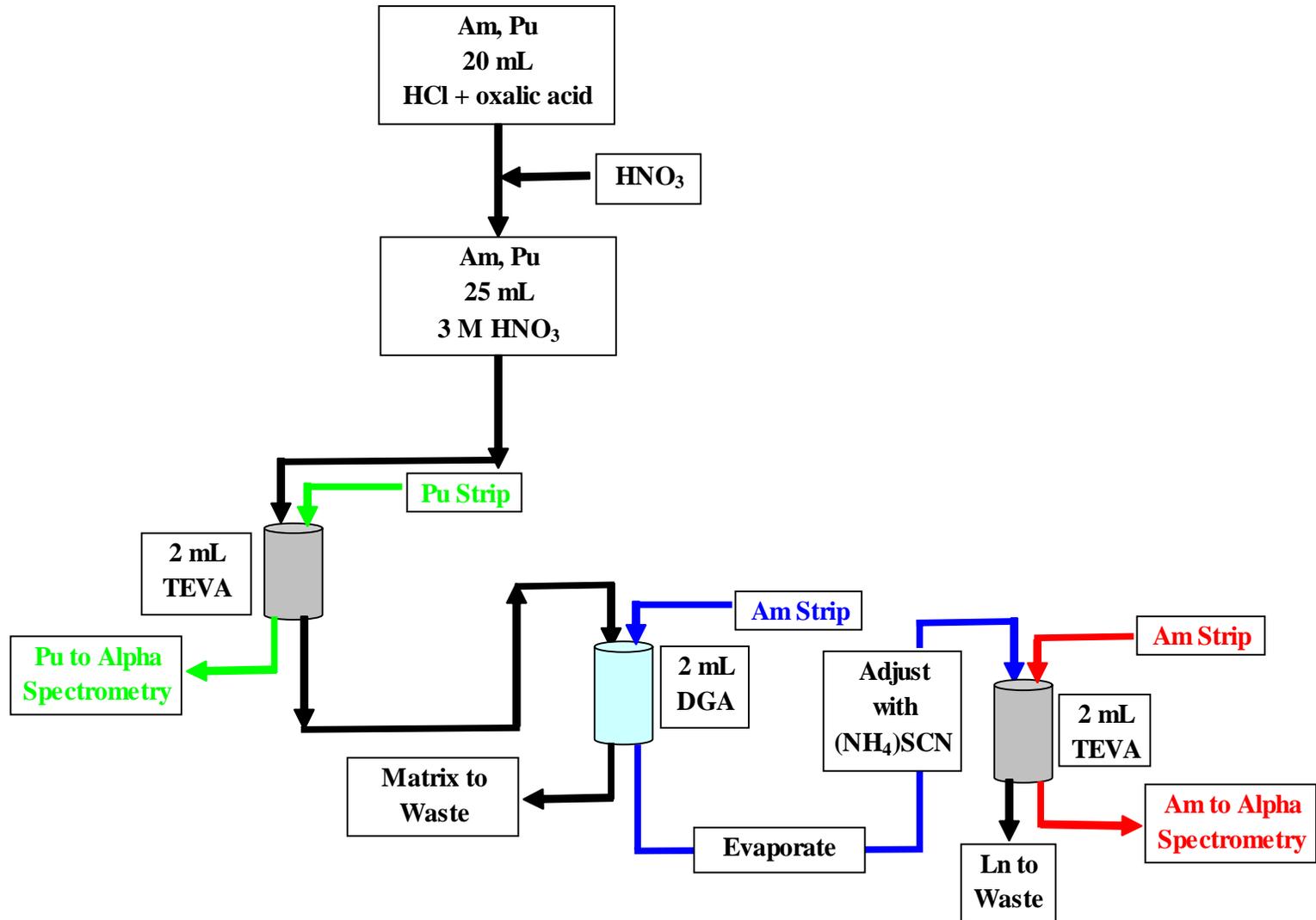
# EICHROM ENVIRONMENT

## Fraction of Major and Minor Constituents Removed from Am/Pu Fraction

<b>Major Constituents</b>	<b>Fraction Removed</b>
<b>Al</b>	<b><math>3 \times 10^4</math></b>
<b>Ca</b>	<b><math>3 \times 10^1</math></b>
<b>Fe</b>	<b><math>4 \times 10^4</math></b>
<b>Mg</b>	<b><math>6 \times 10^4</math></b>
<b>P</b>	<b><math>&gt;10^2</math></b>
<b>Lanthanides</b>	
<b>La-Nd</b>	<b>~2</b>
<b>Sm-Eu</b>	<b>negligible</b>
<b>Minor Constituents</b>	
<b>Ba, Cu, Mn, Na</b>	<b><math>&gt;10^2</math></b>
<b>Ni, Pb, Si</b>	<b><math>&gt;10^2</math></b>

# EICHROM ENVIRONMENT

## Flowchart for the Separation of Pu and Am from Preconcentrate



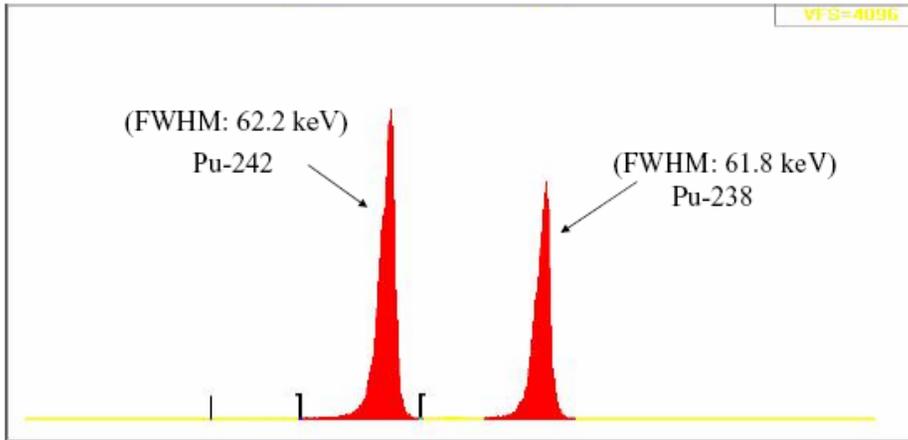
# EICHROM ENVIRONMENT

## Results of Rapid Soil Method (100 g Soil)

Isotope	MAPEP-04		MAPEP-05	
	Found (Bq/kg)	Reported (Bq/kg)	Found (Bq/kg)	Reported (Bq/kg)
$^{238}\text{Pu}$	31.2	35.4	59.0	60.8
$^{241}\text{Am}$	68.4	69.0	107	94.0
Results based on : 50 to 60% recovery of $^{243}\text{Am}$ 40 to 65% recovery of $^{242}\text{Pu}$				

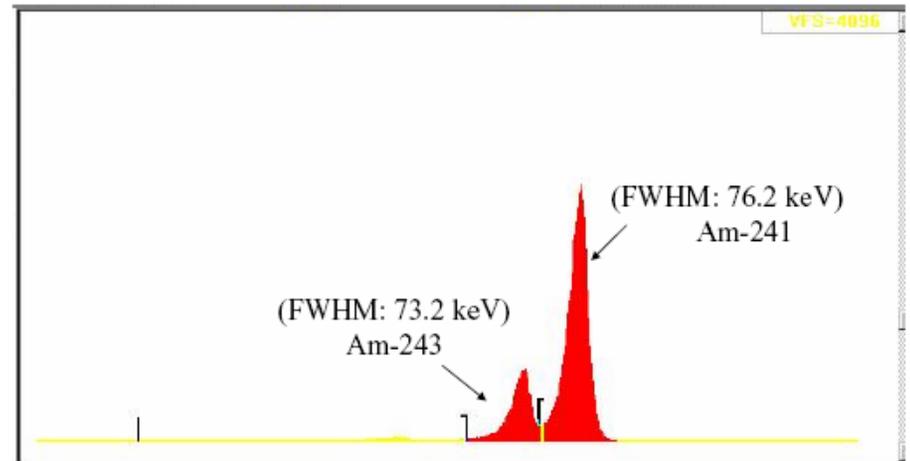
# EICHROM ENVIRONMENT

Pu fraction: 100 g MAPEP-05-MaS14 Soil Sample



MARKER INFO			
Next	Left Marker: 334 : 4506.9 keV	FWHM, FWTM: 62.218, 135.141 keV	
Prev	Right Marker: 476 : 4977.6 keV	Gaussian Ratio: 1.131	
	Centroid: 434 : 4838.2 keV	ROI Type: 1	
	Area: 68144 ± 0.38%	Integral: 68144	

Am fraction: 100 g MAPEP-05-MaS14 Soil Sample



MARKER INFO			
Next	Left Marker: 540 : 5034.4 keV	FWHM, FWTM: 73.213, 164.473 keV	
Prev	Right Marker: 627 : 5305.6 keV	Gaussian Ratio: 1.232	
	Centroid: 602 : 5228.9 keV	ROI Type: 1	
	Area: 20266 ± 0.70%	Integral: 20266	

# EICHROM ENVIRONMENT

## Other matrix elimination steps for soil samples

- Sherrod Maxwell (SRS laboratories)
  - $\text{CeF}_3$  co-precipitation
  - TEVA/TRU/DGA
  - complete dissolution (Pu/Am/Np)
    - ( $\text{Na}_2\text{O}_2$  / NaOH fusion) up to 10 g soil
    - Rapid method (results in 24h possible)
    - High recoveries (> 80%)
  - leached soil samples up to 100 g (Pu/Am)
    - leaching  $\text{HNO}_3/\text{HCl}$
    - Recoveries 60 – 80%

## Other matrix elimination steps for soil samples

- Varga/Vajda
  - Pu and Am in soil samples (0,5 – 3 g) via ICP-SFMS
  - CaF<sub>2</sub> co-precipitation
  - Am and Pu recoveries 81 – 94%
  - DL (1g samples): Pu and Am ca. 0,1 mBq
- Kim/Burnett/Dell/Maxwell
  - Diphonix resin
  - up to 50 g (leached samples)
  - Elution or destruction of Diphonix resin
  - Pu, Am (Th, U possible)

# EICHROM ENVIRONMENT

## Sherrod Maxwell (SRS) Actinides and Sr-89/90 in Animal Tissue

- 200 g fish
- Pu, U, Am, Cm, Np, Sr
- Wet ash /furnace
- Dissolve residue in 12 ml 6M HNO<sub>3</sub> + 12 ml 2M Al(NO<sub>3</sub>)<sub>3</sub> + 3M HNO<sub>3</sub> as needed (~40-45 ml load solution)
- Load onto TEVA+TRU+DGA
  - DGA: good Am/Cm recovery
  - Adjust valence
- Collect load/rinse for Sr determination
- Evaporate and redissolve later in 15 mL 8M HNO<sub>3</sub> plus 5 – 10 mL 3M HNO<sub>3</sub>
- Load onto Sr Resin (3 mL)

# EICHROM ENVIRONMENT

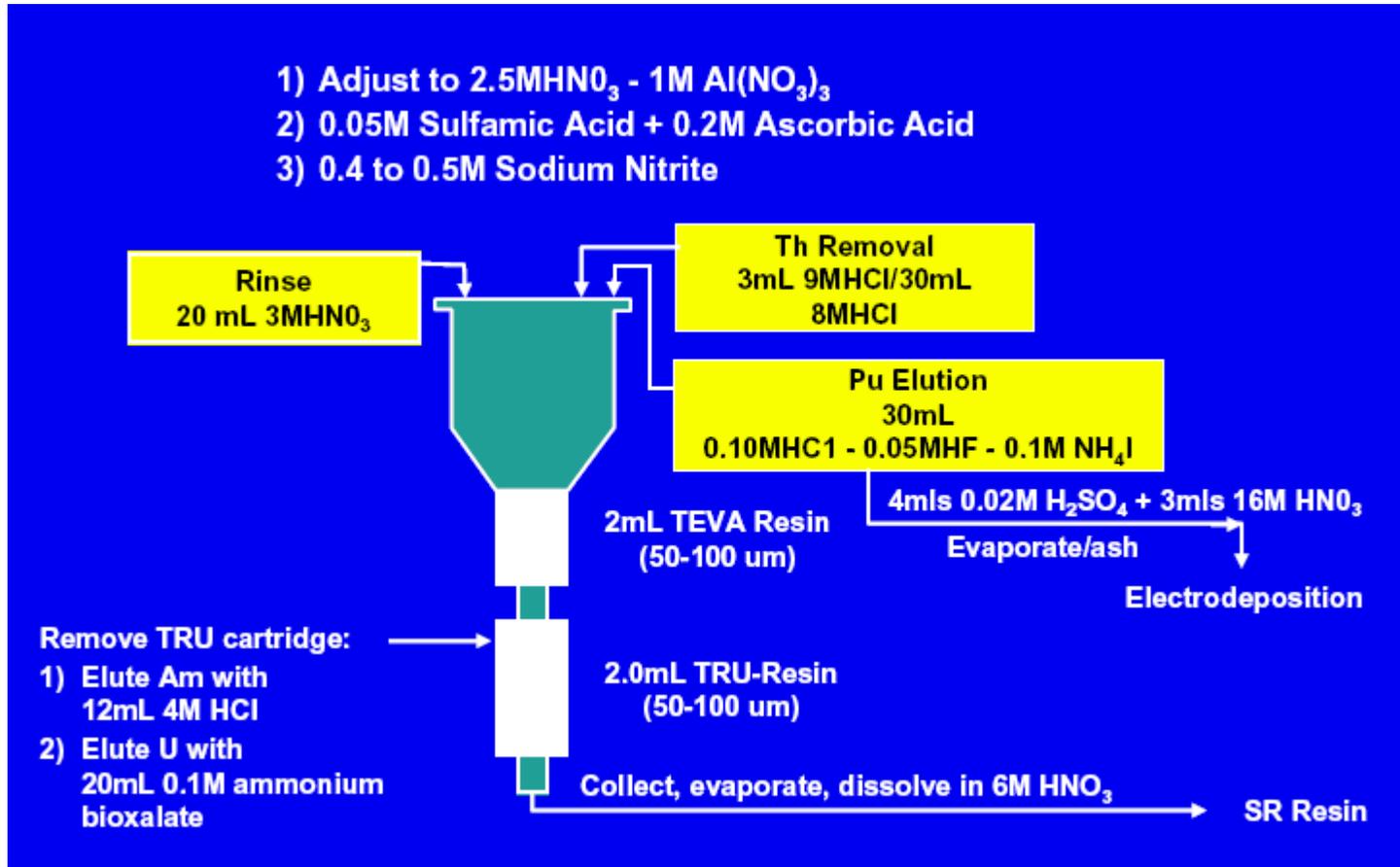
<b>Tracer/carrier</b>	<b>Avg. Recovery</b>	<b>MS Recovery</b>
<b>Pu-236</b>	<b>99.8%</b>	<b>100% (Pu-238)</b> <b>90.0% (Np-237)</b>
<b>Am-243</b>	<b>109%</b>	<b>94.1% (Am-241)</b> <b>94.3% (Cm-244)</b>
<b>U-232</b>	<b>97.1%</b>	<b>91.1% (U-235)</b>
<b>Sr carrier</b>	<b>84.9%</b>	<b>97.7% (Sr-90)</b>

## Urine and fecal samples

- SRS laboratories
- Urine (Pu, Np, U, Am, Sr)
  - 500 mL
  - Sample preconcentration via Ca-Phosphat co-precipitation
  - TEVA, TRU and Sr Resin
  - Recoveries: > 80% for Pu, Np and Am
- Fecal samples
  - 10 g (leached samples)
  - Matrix removal via Diphonix resin
  - Microwave digestion of Diphonix resin
  - Pu and Am recoveries: 70 – 100%

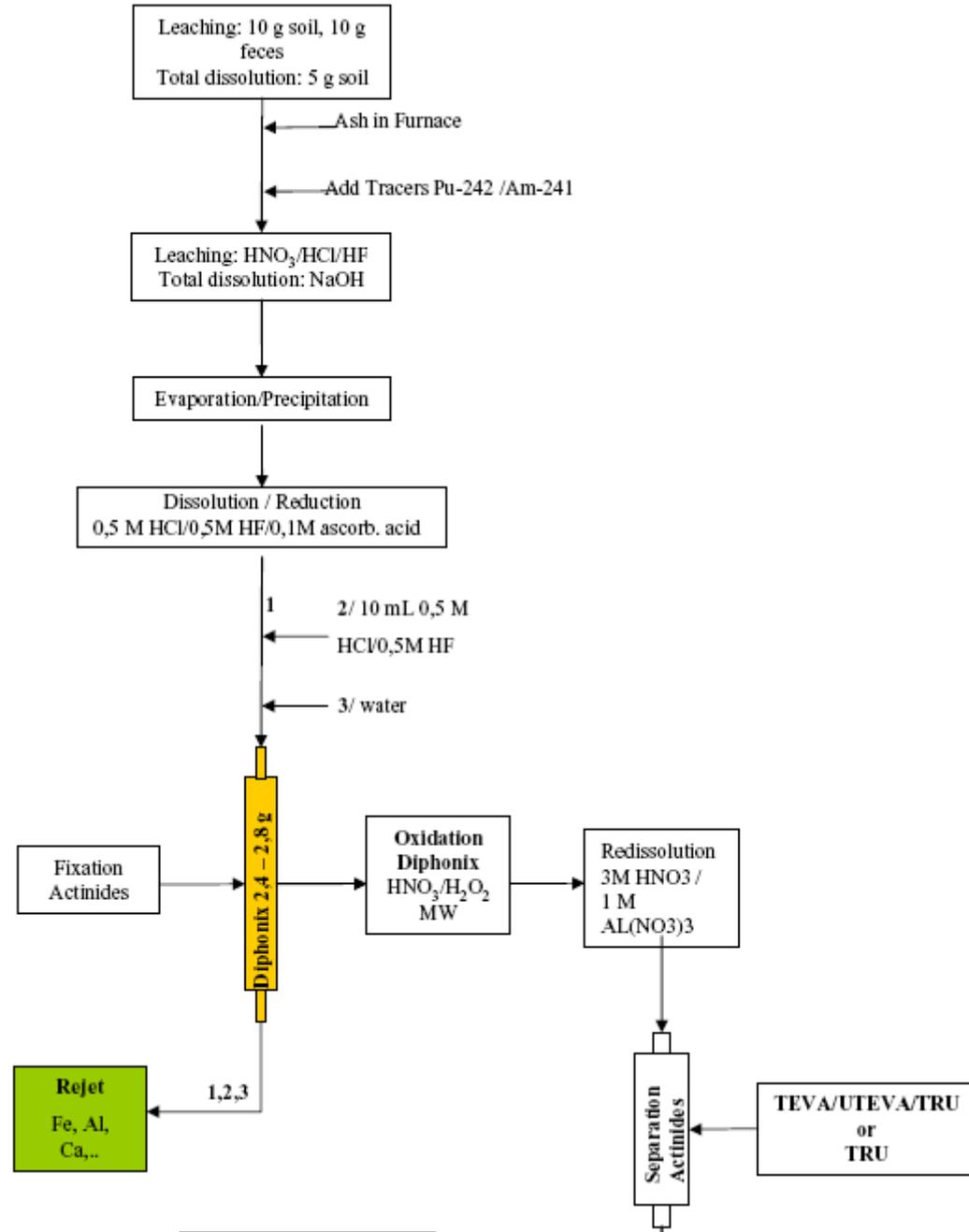
# EICHROM ENVIRONMENT

## Pu, Np/Am, U, Sr on TEVA/TRU/SR RESINS (URINE)



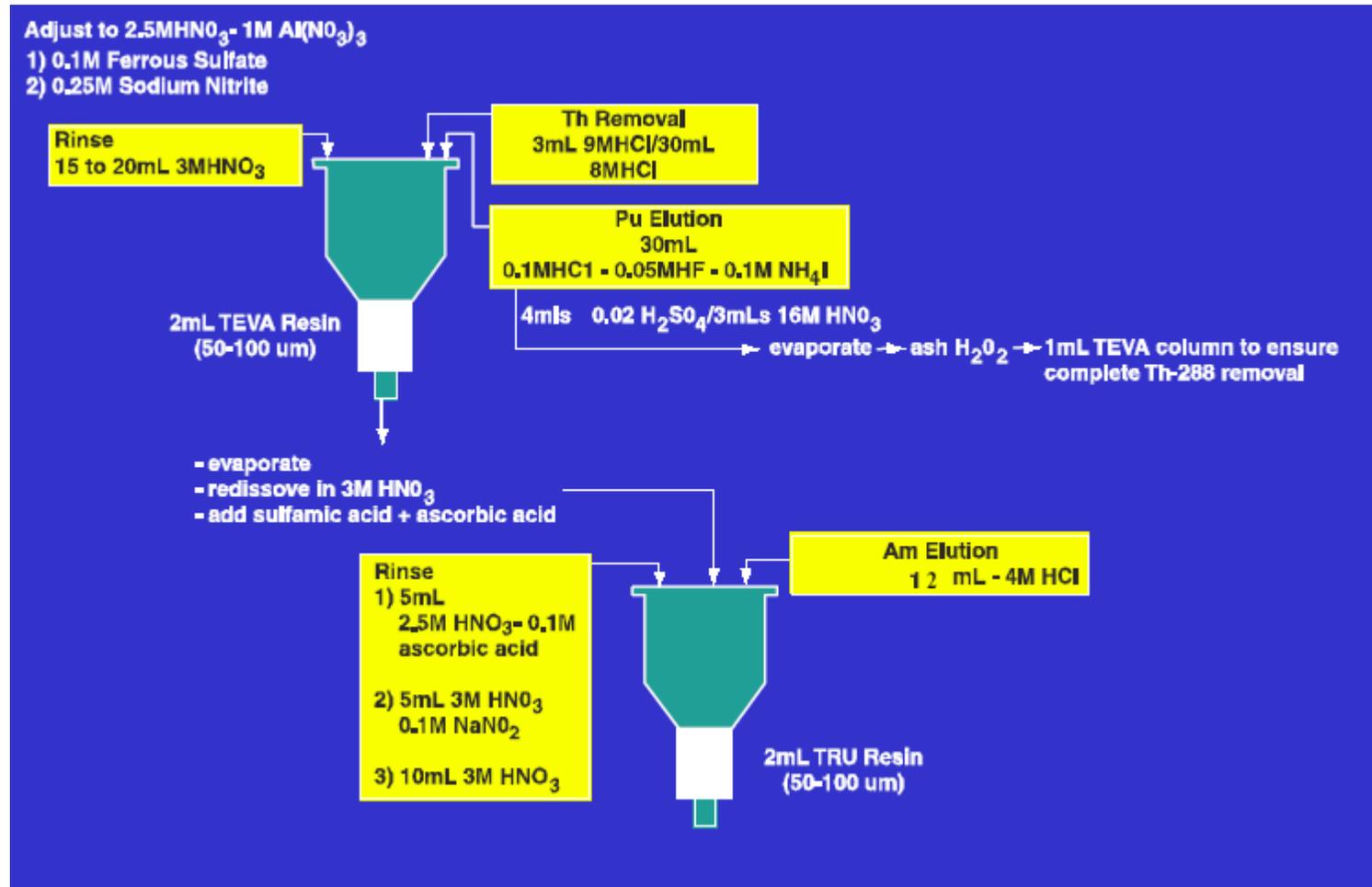
# EICHROM ENVIRONMENT

## Sample preparation scheme fecal samples DIPHONIX



# EICHROM ENVIRONMENT

## Pu, Am on TEVA/TRU RESINS (FECAL METHOD)

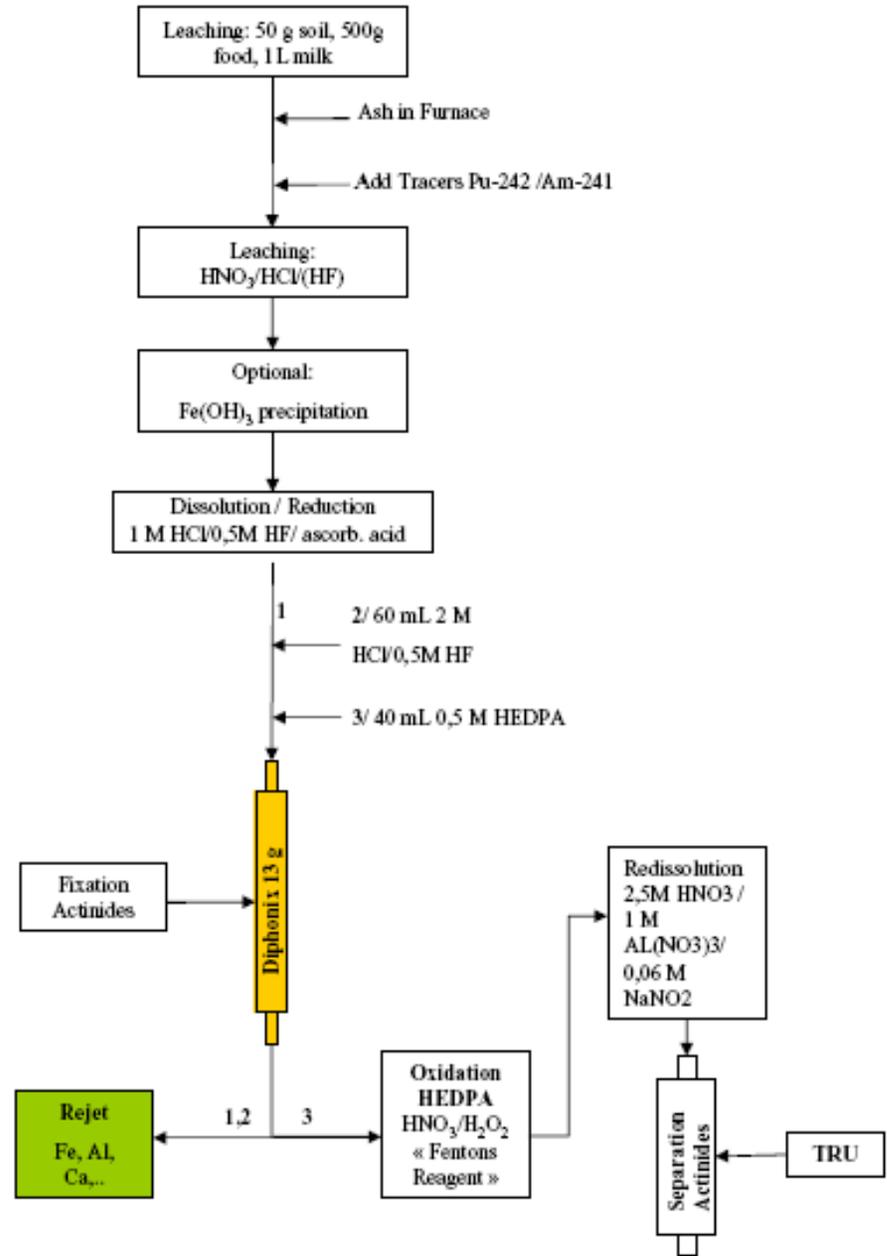


## Food and milk samples

- Kim/Burnett/**Dell**
- Pu and Am
- 500 g liver, apples, root vegetables
- 1000 mL milk
- Diphonix elution with HEDPA
- HEDPA oxidation via « Fentons Reagent »
- Yields for all matrices in the order of 75 – 100 %

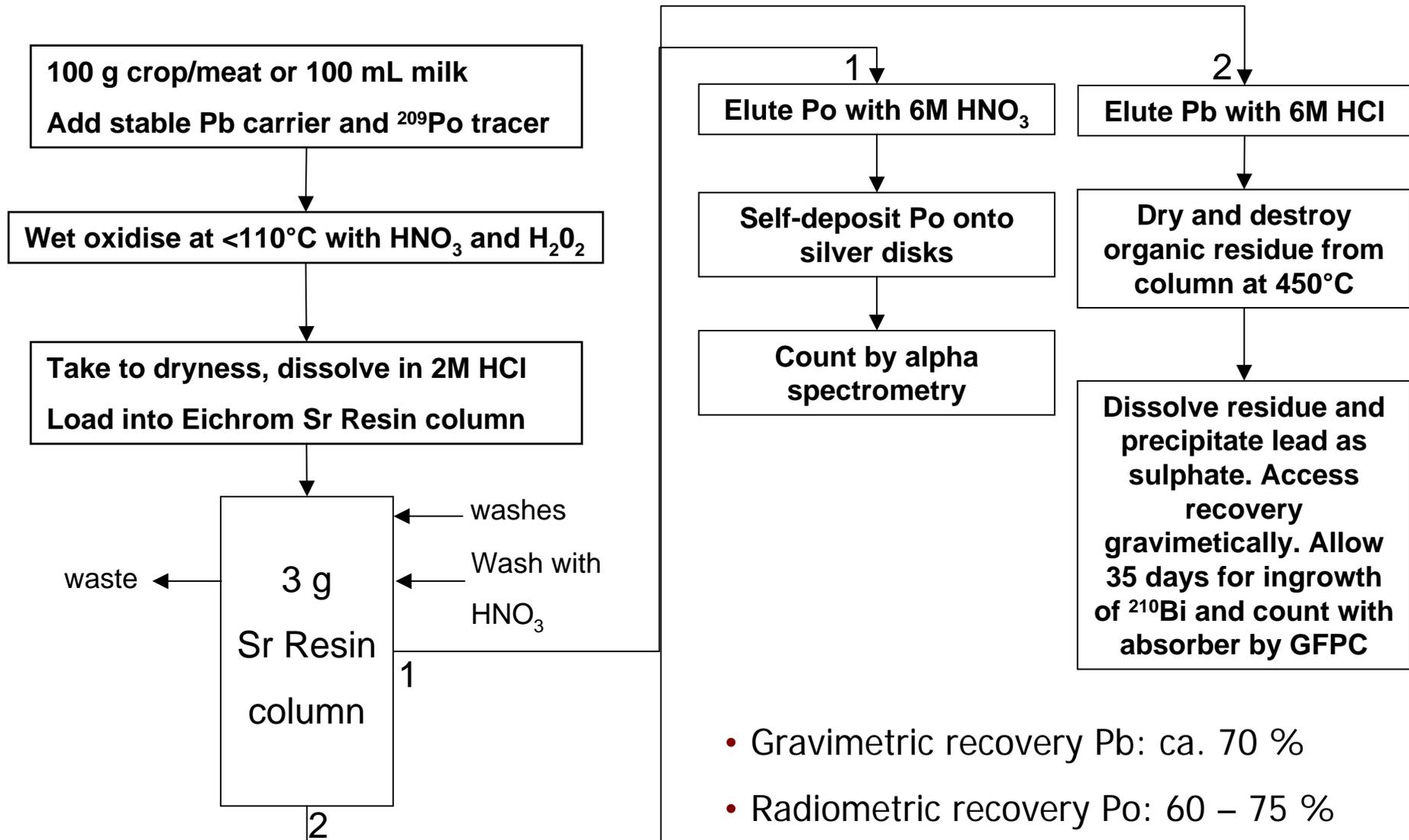
# EICHROM ENVIRONMENT

## Sample preparation scheme food samples DIPHONIX



# EICHROM ENVIRONMENT

## Pb and Po in milk, crop and animal samples (Dell)



# EICHROM ENVIRONMENT

---

## New contact:

### EICHROM ENVIRONMENT

---

Parc de Lormandière - Bât. C - Rue Maryse Bastié - Campus de Ker Lann - 35170 Bruz - FRANCE

Tél. : + 33 (0)2 99 05 00 09 - Fax + 33 (0)2 99 05 07 27

E-mail : [contact@e-environment.fr](mailto:contact@e-environment.fr) - [www.eichrom.com](http://www.eichrom.com)

SAS au capital de 40.000 euros - SIRET : 493 848 972 00011 - APE 511C - TVA Intra communautaire : FR 65 493 848 972