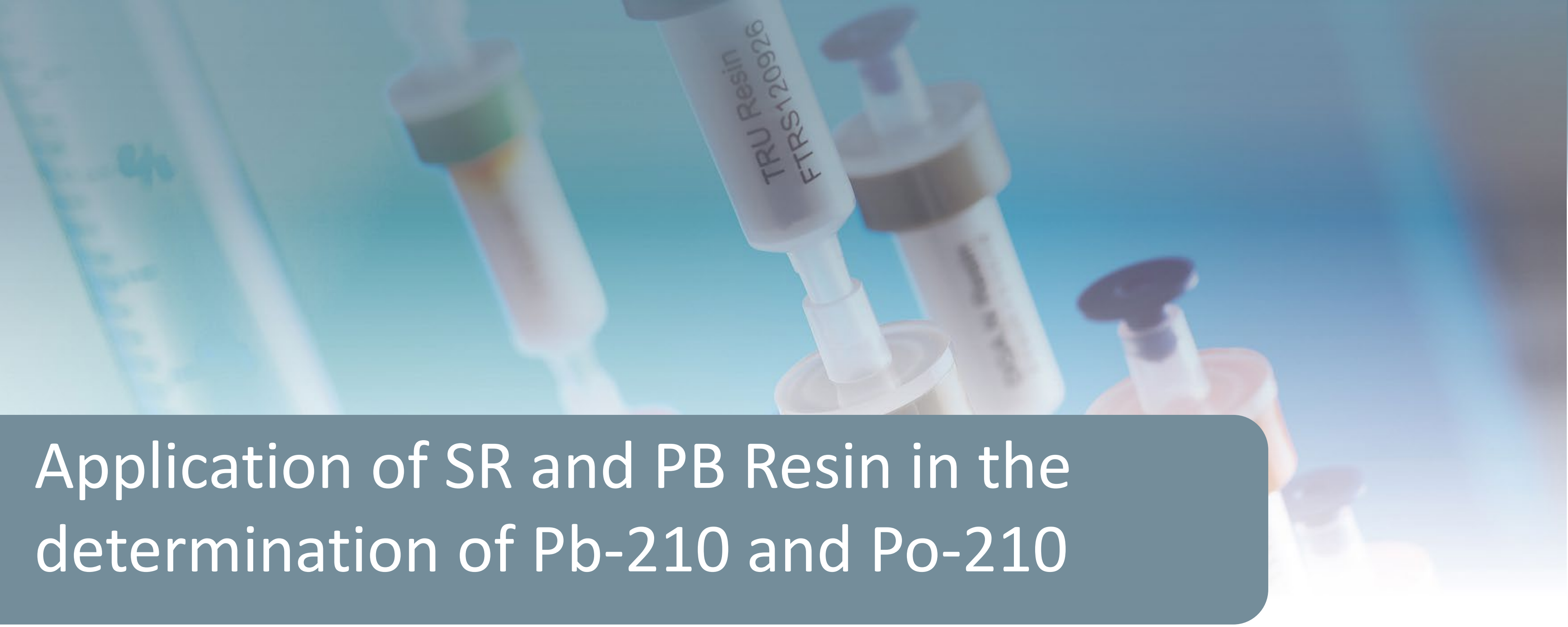


Application of SR and PB Resin in the determination of Pb-210 and Po-210

S.Happel – TrisKem

EVT2204728 “2023 Training Workshop of IAEA’s ALMERA Network on Advanced Topics in Radiochemistry Techniques: Lead-210 and Polonium-210”

11/10/2023

TRISKEM
Expertise in Separation Chemistry

TrisKem International

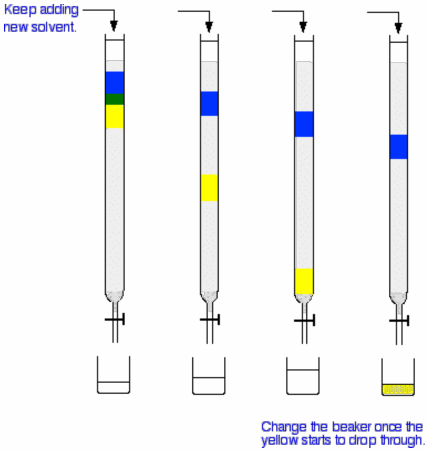
- Based in Rennes (France)
- Independent company since 02/07
  - Formerly part of Eichrom Europe
  - ISO 9001 since 2007
  - Two suppliers of SR, PB Resin (TrisKem and Eichrom) depending country
- Main product: extraction chromatographic resins
- Staff : 20
- R&D, QC and TechSupport group:
  - 4 RadChem PhD, 3 Technicians
- R&D: Development of new resins, techniques and applications
- Products used in several domains

Extraction chromatography

Combination of liquid-liquid extraction (LLX) and chromatography

- Liquid-liquid extraction: distribution of an element between 2 non-miscible phases
  - Most general case: organic and aqueous phase

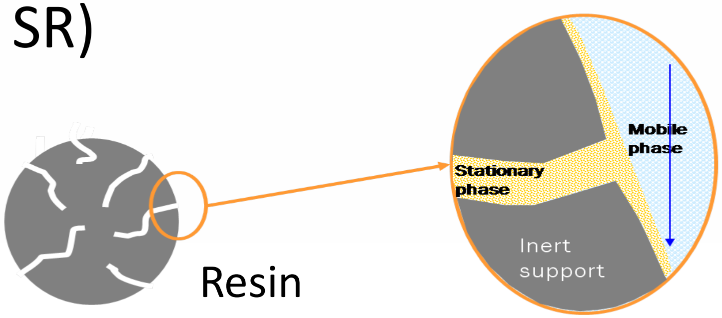
- Chromatography: distribution of an element between a solid and a liquid phase

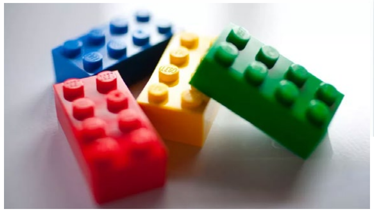


Extraction chromatography

Organic extractant impregnated onto inert support

- « Supported Solvent Extraction » / « Solvent Impregnated Resins »
  - Stationary phase impregnated onto inert support
  - Distribution between two non-miscible phases
  - High density of functional groups
  - Fast kinetics/small volumes => rapid separations
  - High variety of selectivities
  - Combining several cartridges can improve/facilitate separation
  - Bleeding might need to be addressed (e.g. Pb elution with 6M HCl from SR)





Inert Support

- Choice of inert support depending on application
  - Mainly polymers
  - Radiolysis stability, plastic scintillators,…
- Inert regarding chemical reactions with
  - Organic phase/extractant
  - Elements to be isolated
  - Mobile phase

- Stable regarding mechanical actions
- High specific surface
- Defined pore and particle size distribution
- Spherical particles

Stationary Phase

- Generally organic compounds
  - Few exceptions e.g. ABEC systems
- Non-miscible with water
- Non-volatile but soluble in volatile solvents
- Ideally fast kinetics
- High selectivity
  - Aim: selectivity for analyte, no selectivity for matrix/interferences
  - Pure extractants, synergetic mixtures, solid extractants dissolved in diluents, solids (e.g. DMG)
  - Influence of Diluents
- Chemically, physically and mechanically stable
- High capacity

Extraction chromatography

Types of Extractants

Acidic e.g. HDEHP (LN Resin)

$$M^{3+} + 3HY \rightleftarrows MY_3 + 3H^+$$

$$M^{3+} + 3(HY)_2 \rightleftarrows M(HY_2)_3 + 3H^+$$

Neutral e.g. CMPO/TBP (TRU Resin), DPPP (UTEVA Resin), SR & Pb Resins

$$M^{3+} + nE + 3X^- \rightleftarrows ME_n \bullet X_3$$

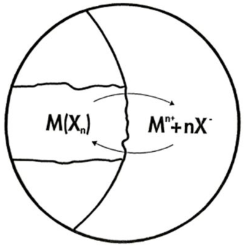
Basic e.g. Aliquat 336 (TEVA Resin)

$$R_3N + HX \rightleftarrows R_3NH^+X^-$$

$$R_3NH^+X^- + MX_3 \rightleftarrows R_3NH^+MX_4^-$$

$$X^- = NO_3^-, Cl^-$$

Metal Anion Complex Formation



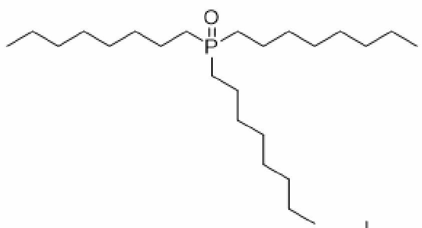
Metal + Anion $\rightleftharpoons$ Complex

Complex + Organic $\rightleftharpoons$ Extracted

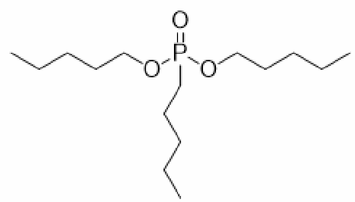
Horwitz et al.

Typically employed extractants

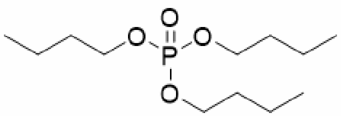
Trioctylphosphine oxide
(TOPO)

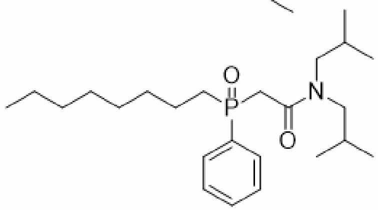


Dipentyl pentyl phosphonate
(DPPP)

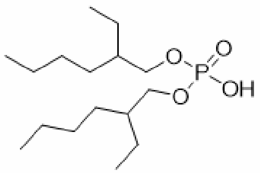


Tributylphosphate
(TBP)

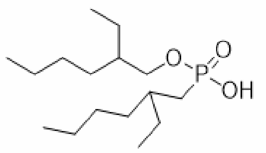




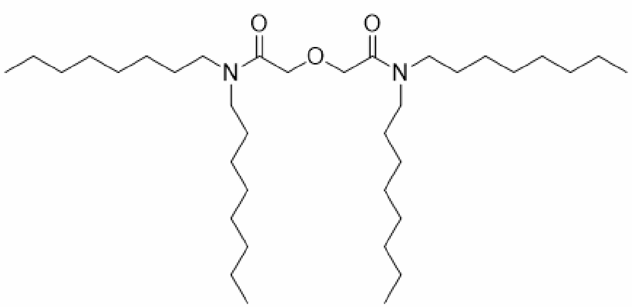
Octyl (phenyl)-N-N-diisobutyl-carbamoylmethylphosphine oxide
(CMPO)



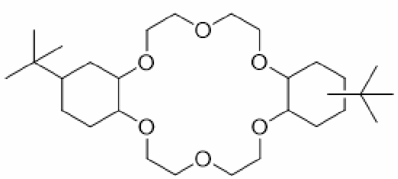
Bis(2-ethylhexyl) hydrogen phosphate
(HDEHP)



2-Ethylhexylphosphonic acid mono-2-ethylhexyl ester
(HEH[EHP])

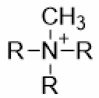


N,N,N′,N′-tetra-n-octyl-diglycolamide
(TODGA)



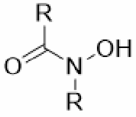
4,4′(5′)-di-t-butylcyclohexano-18-crown-6

Aliquat 336 (R = 8, 10)



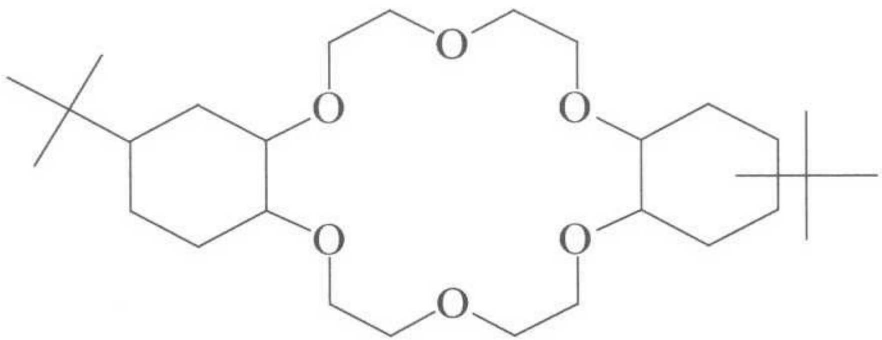
Tertiary amine

Phosphine sulfide

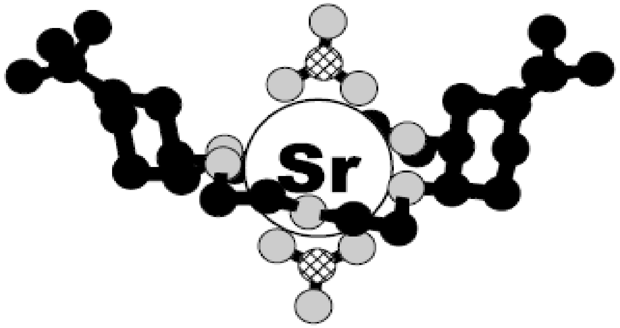


Hydroxamate

SR Resin



Diluent: 1-octanol



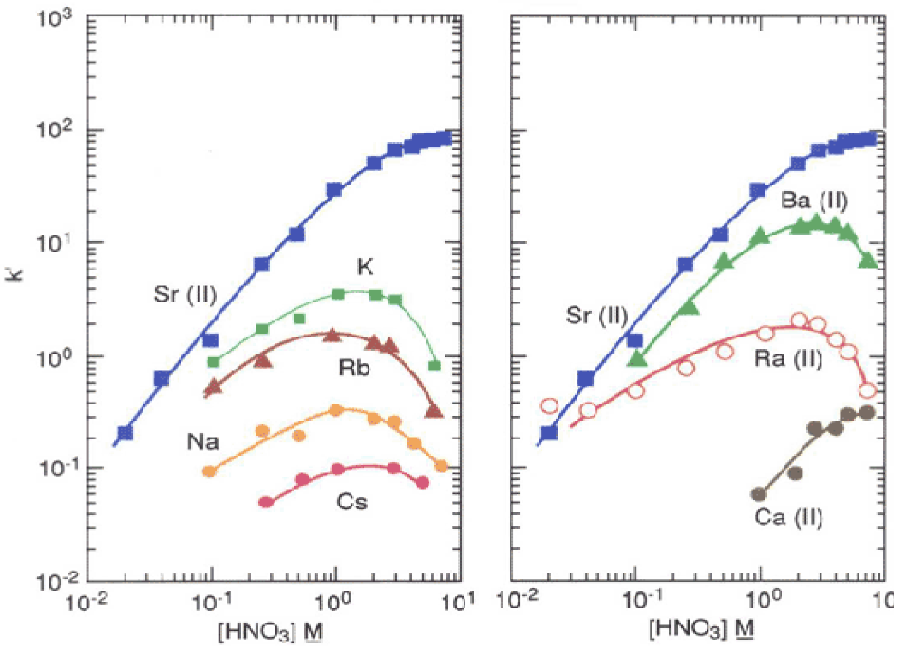
Dietz et al. 2004

- 1.0M 4,4'(5')-di-t-butylcyclohexano 18-crown-6 in 1-octanol.
- 40% (w/w) loaded onto inert chromatographic support.
- Sr max. capacity: 21 mg / 2mL resin bed,
- Advised working capacity: max. 8 mg / 2mL resin bed (Opt.: 5 mg)
- Capacity for Pb higher

SR Resin – HNO_3 data

Figures 2 and 3

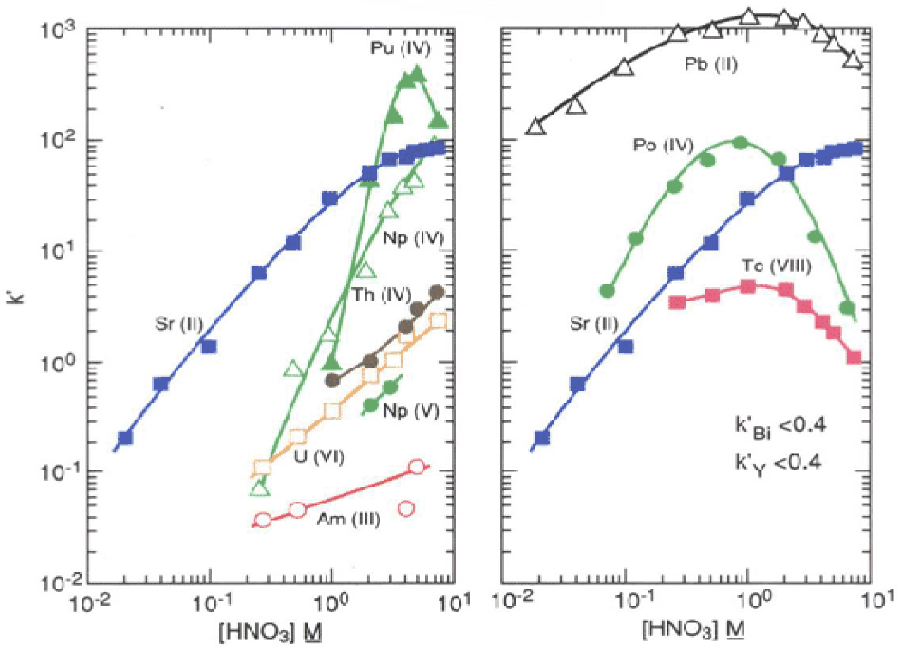
Acid dependency of k' for various ions at 23-25°C.
Sr Resin



Horwitz, et al., (HP292)

Figures 4 and 5

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



Horwitz (HP199)

- Called SR Resin but higher retention of Pb
- Pb well retained at low HNO_3, partial elution in water
- Attention: Po data in HNO_3

SR Resin – HCl data

TRISKEM
Expertise in Separation Chemistry

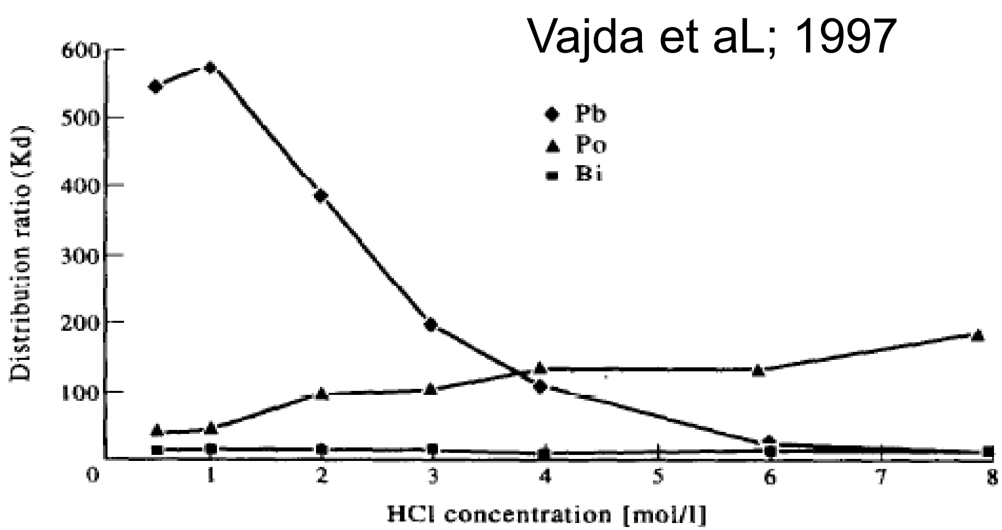


Fig. 1. Distribution ratios K_d of lead, bismuth and polonium in HCl solutions.

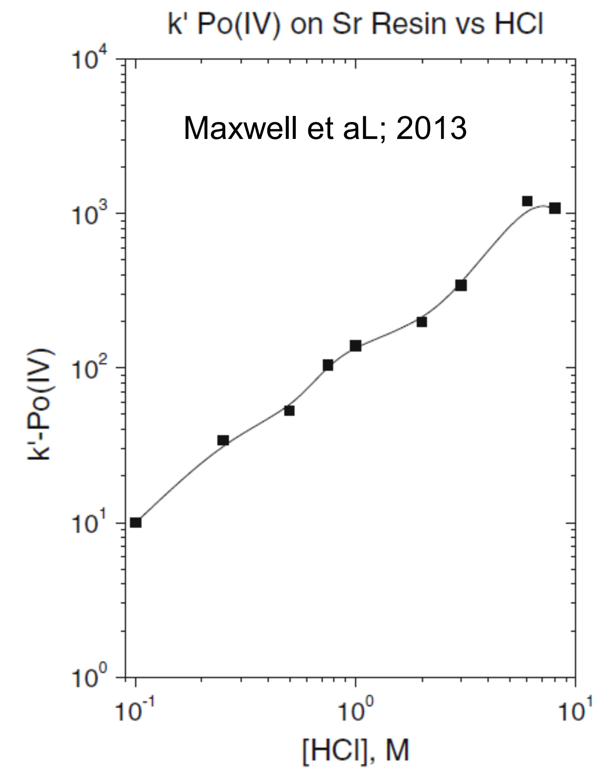
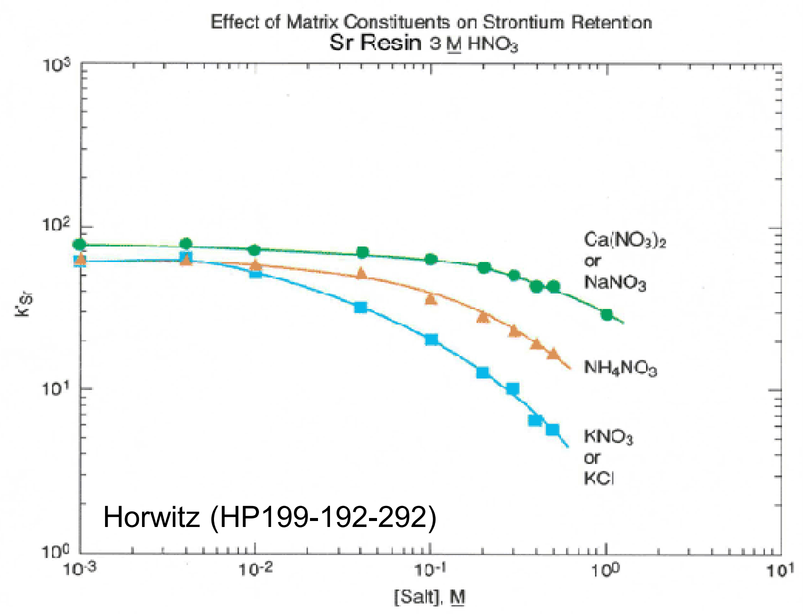
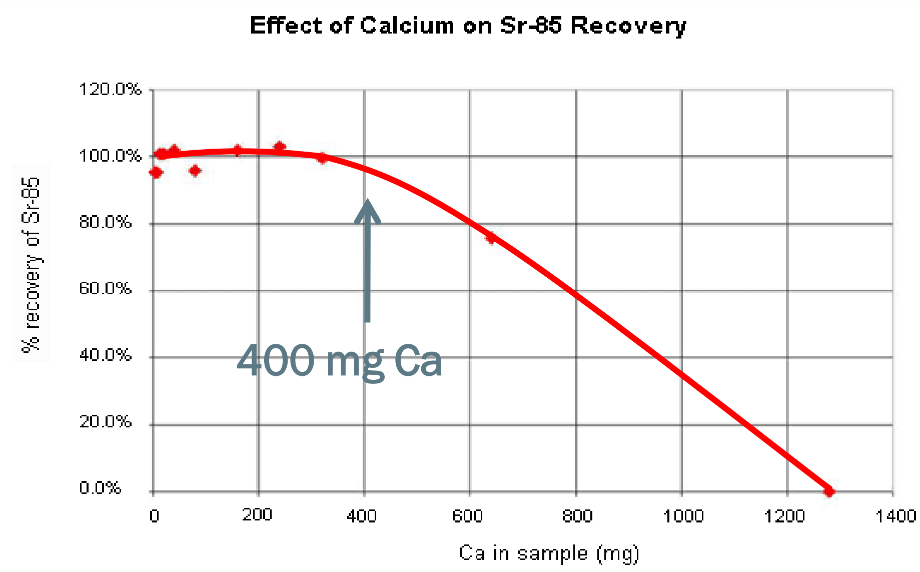


Fig. 3 Retention of Po^{4+} on Sr resin in HCl

- High Pb and Po uptake for 2M HCl
- Pb elution with high HCl (alternative: citrate)
- To be noted: A. Knight et al showed that octanol based resins (e.g. TK400) have very similar selectitivty for Po in HCl => diluent strongly involved in Po extraction

SR Resin

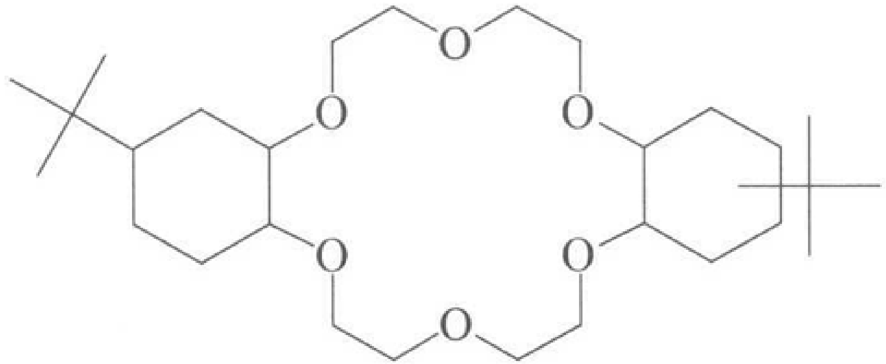




- **Major interferents**: Ca, stable Sr, K, Na => lower effect on Pb

- **Monovalent interferent elimination possible**: Coprecipitation with oxalate, phosphate or iron hydroxide

PB Resin



- **Extractant**: 0.75M crown-ether (same crown ether as in SR Resin)
- **Diluent**: Isodecanol

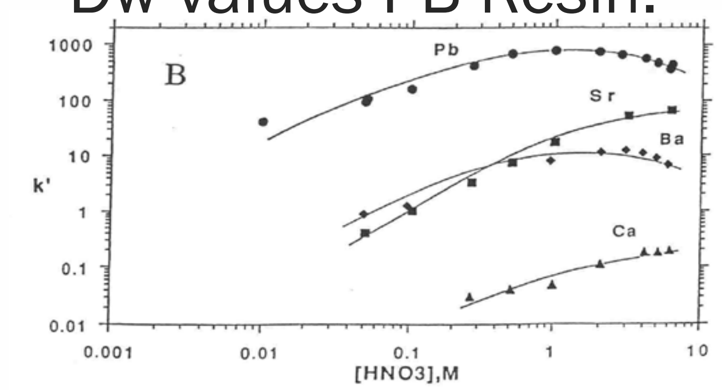
(longer carbonated chain for easier removal of Pb)

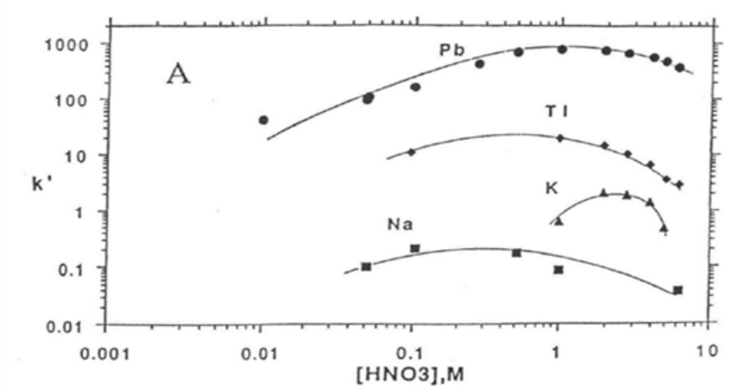
- **Pb capacity:** 29 $mg.g^{-1}$ PB resin (lower than SR Resin)
- **Main application**: Pb-210 in water
- Less bleeding than SR Resin (lower solubility of Isodecanol in aqueous solutions)

Comparison PB vs SR

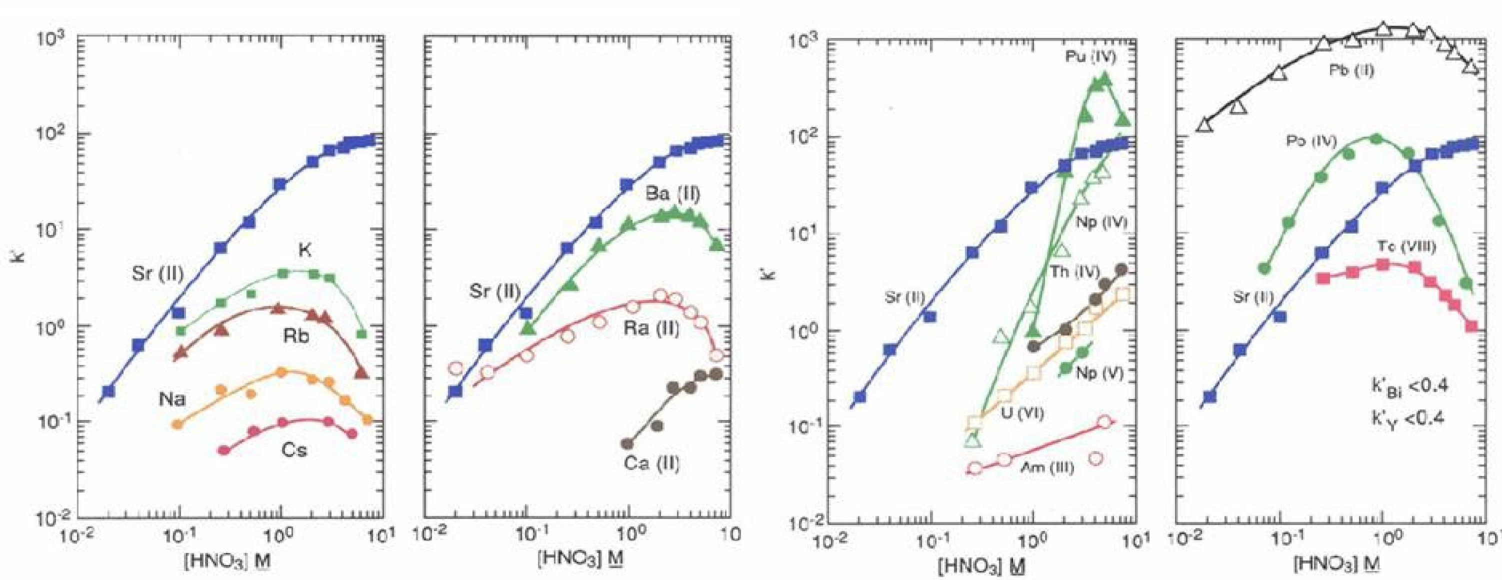
TRISKEM
Expertise in Separation Chemistry

- Dw values PB Resin:





- Dw values SR Resin:

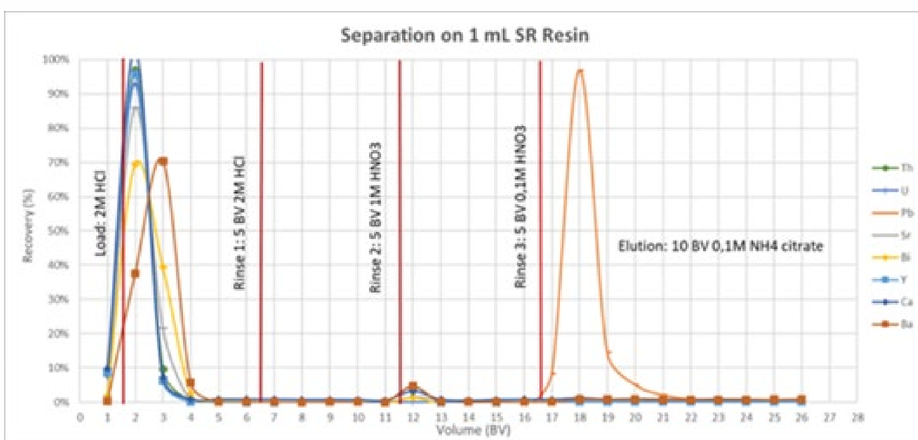


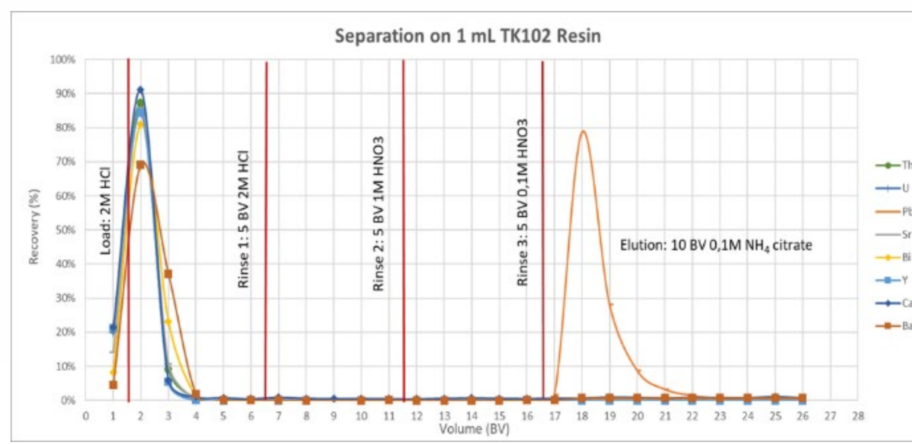
Differences SR and PB Resin

- PB Resin:
  - Lower concentration (and thus amount) of crown-ether
  - Use of isodecanol instead of octanol
    - Horwitz et al showed that solutions of crown-ether in alcohols with longer chains show lower Dw
    - Less bleeding
  - Deliberate choice:
    - Pb retention on PB Resin remains high
    - PB Resin allows Pb elution in water
      - Especially interesting in case of evaporation and GPC measurement
- SR Resin: typically higher Po yields
- If only Pb to be analysed PB Resin might be a good option (pricing, easier Pb elution)
- For Pb and Po (and high matrix samples) SR better choice

New: TK102 Resin

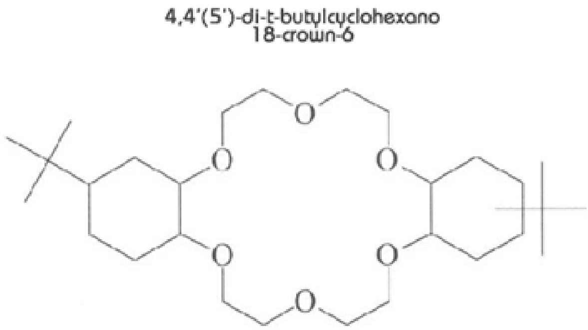
- Modified version of SR Resin
  - Same crown-ether but diluent, inert support and ratios different
    - Higher amount of crownether
    - Short chained, fluorinated alcohol
  - Generally higher Dw and capacity for Sr, Pb, Ba
  - Improved Ra/Ba separation
  - Use of more hydrophobic diluent instead of Octanol
    - Significantly less Bleeding
- Separation methods under optimisation

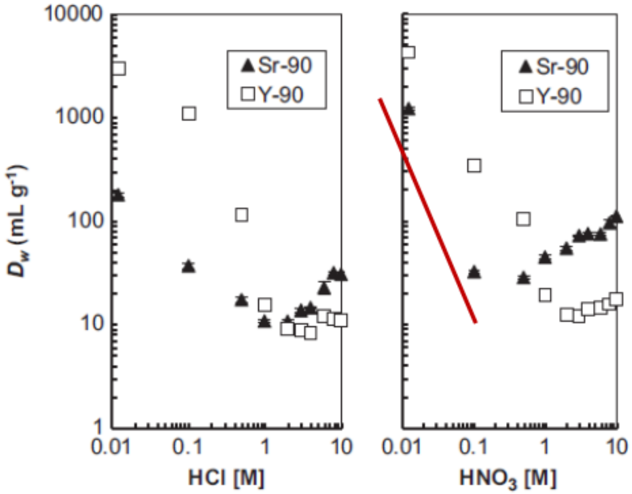




TK100/1 Resins

- Based on same crownether as SR Resin
  - Different diluents: TK100 => HDEHP, TK101 => short chained ionic liquid
  - Sr and Pb uptake also between pH 2 and 7
  - Concentration and purification on same column

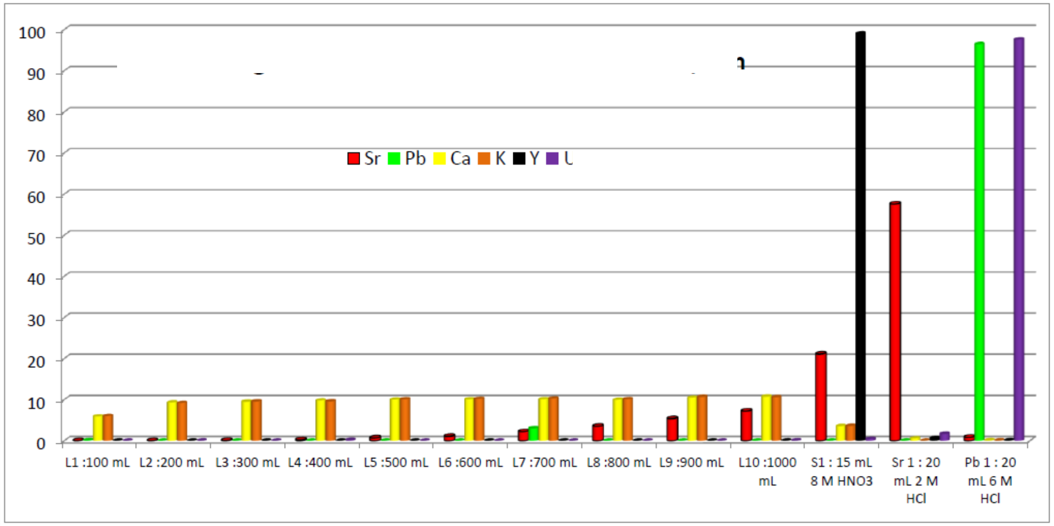


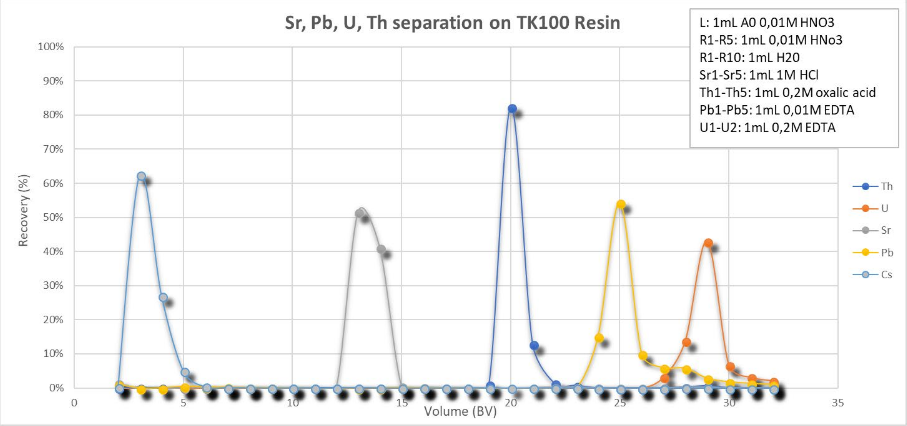


- Typical applications :
  - Pb-210 in water samples (up to > 5L per 2 mL column/cartridge)
  - Sr-89/90 in water samples (up to 0,5L per 2 mL column/cartridge)
  - Sr-90 by ICP-MS (very high Zr-90 decontamination) => NPL
  - Ra-226 by ICP-MS => NPL (Load and purification in one step)
    - Agilent application note

TK100 Resin

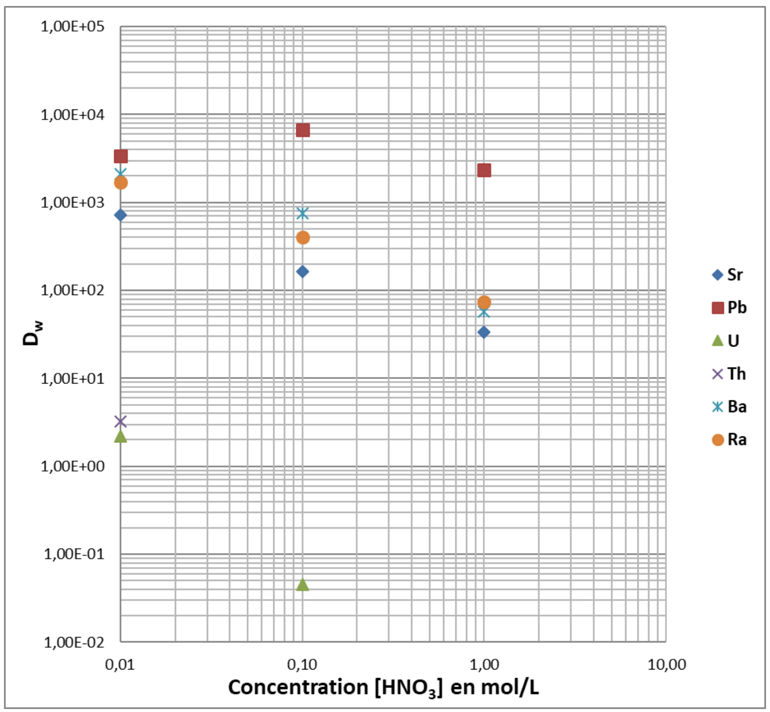
- Spiked water samples at pH 2
- 1L sample, 2 mL cartridge
- Load at 5 – 10 mL/min
- K and Ca not retained
  - Sr breakthrough starts at 600 mL
- Rinse:
  - 5 mL deion. water
  - 15 mL 8M HNO_3
- Sr Elution with 0.5 - 2M HCl
  - Th, Pb, U remain retained
  - May be eluted sequentially
- May be used for one-column sequential separation (Sr, U, Th, Pb, Ra,…)
- Using HDEHP as diluent allows for retention of Sr, Ba, Ra, Pb from acidified water samples (e.g. pH2)
- Stability issues with Sr retention



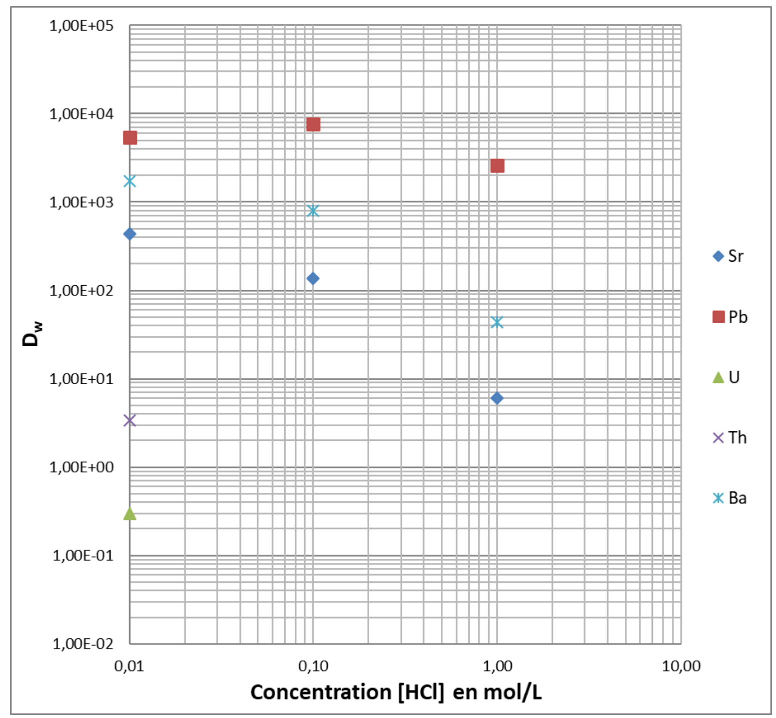


TK101 Resin

- Use of TK101 instead => HDEHP replaced by short chained ionic liquid
- High Ra & Pb retention from dilute acid, no Th, U retention

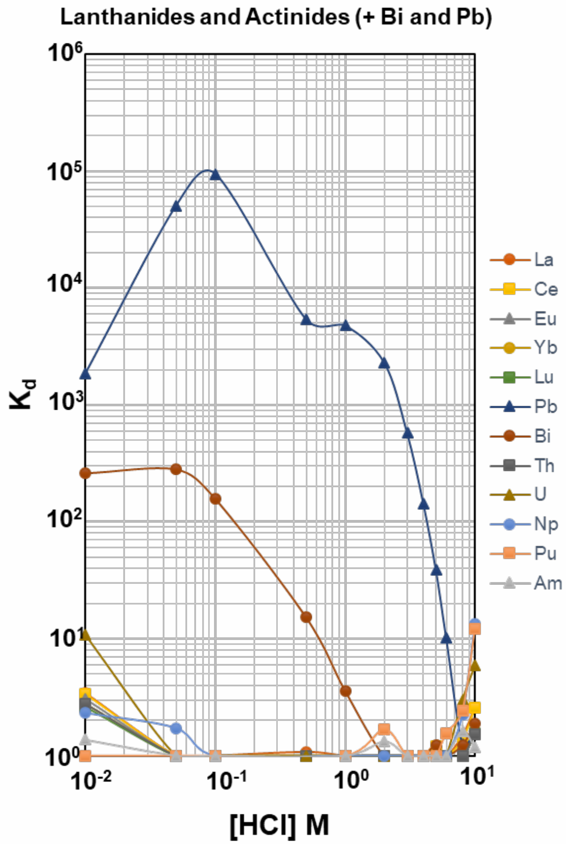


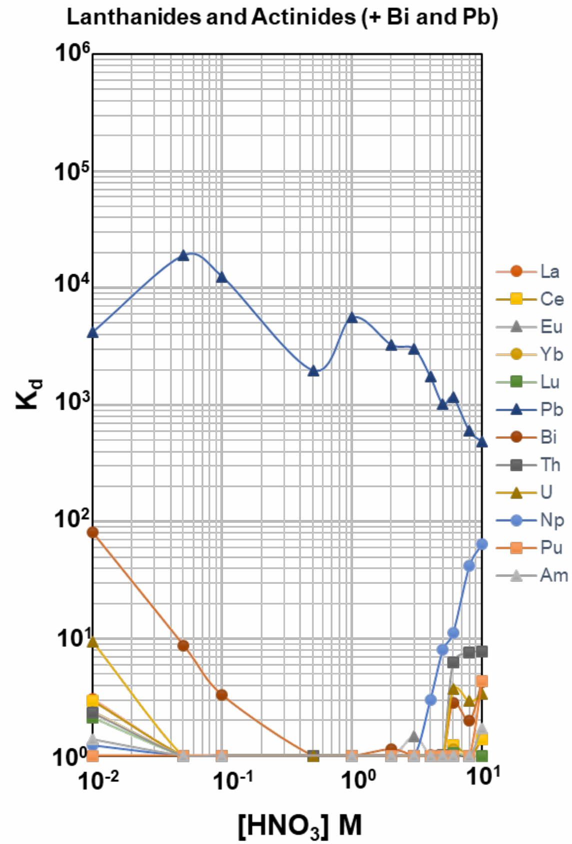
Dw TK101 in HNO_3,
selected elements



Dw TK101 in HCl,
selected elements

TK101 Lanthanides and Actinides (+ Bi and Pb)

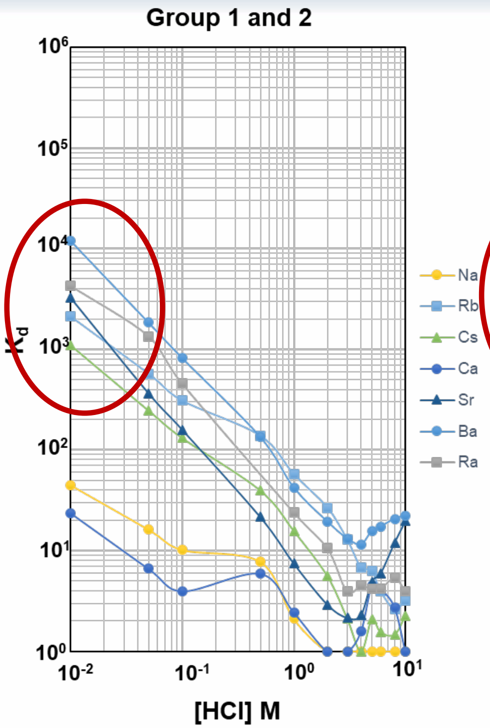


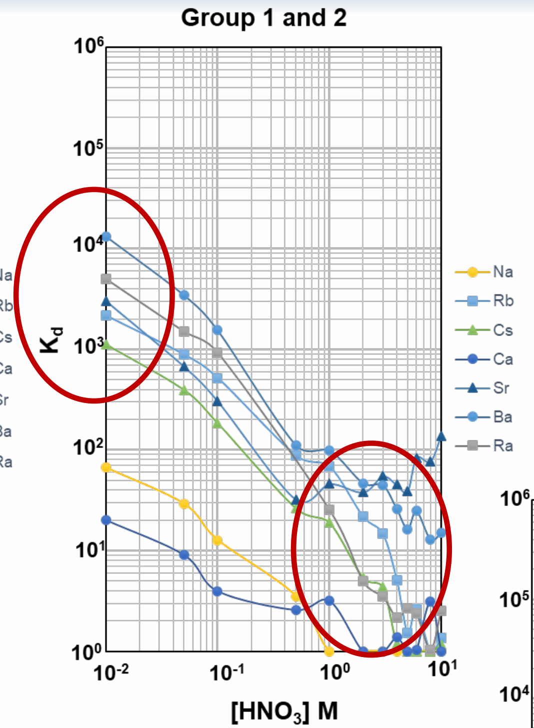


All data Russell et al NPL

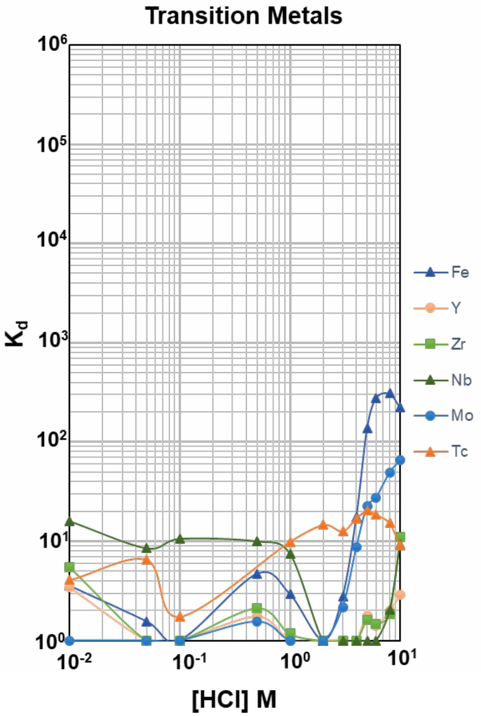
- Very strong Pb retention at all HNO_3 concentrations and HCl < 6M HCl
- Loading of >1L on 2 mL TK101 cartridge
- Easy purification of Pb from other éléments
- Elution in high HCl (or citrate)

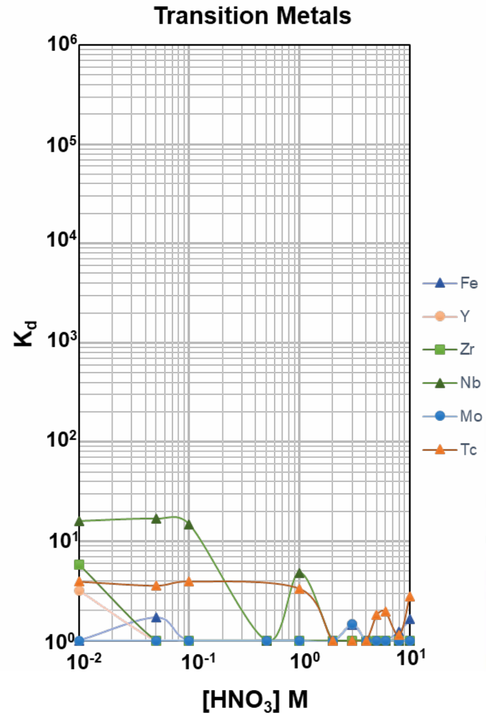
TK101 Resin





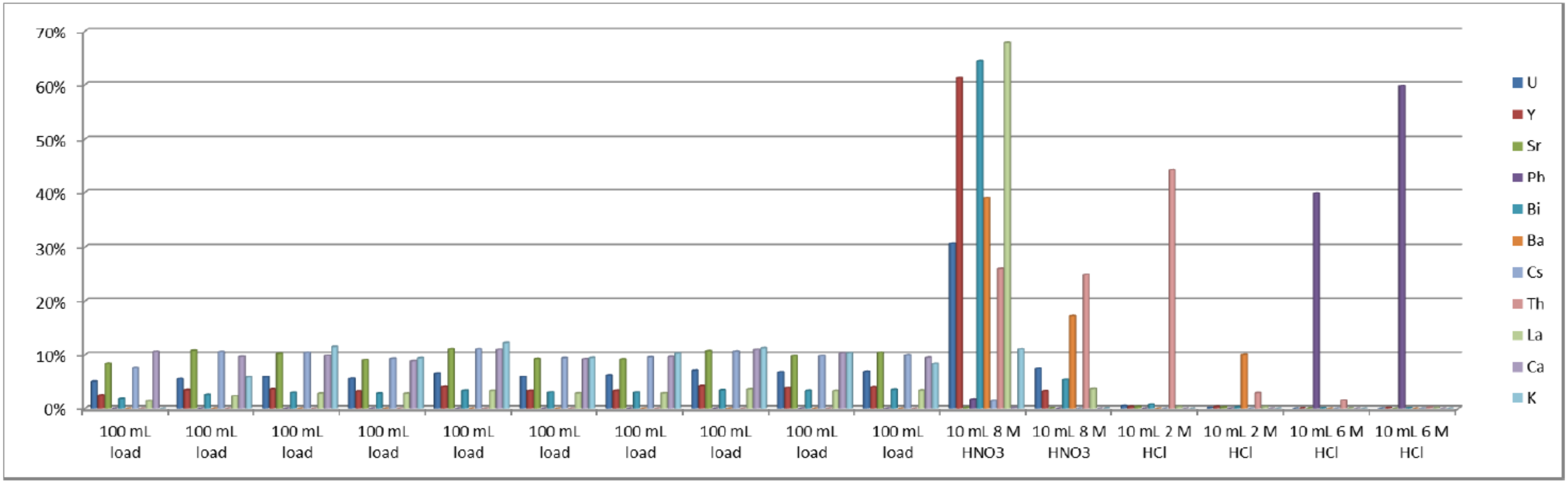
All data Russell et al NPL





Elution study TK101

- Initial testing
- 2 mL TK101 cartridge
- 5 – 10mL/min
- 1L sample (spiked tap water at pH2) – load in 10 100 mL aliquots
- Rinse with 8M HNO_3 and 2M HCl, Pb elution in 6M HCl (optional citrate)
- Automatisation?



Diluents

Conclusion:

- In case of crown-ether based resins diluents have strong impact on resin selectivity
- Can be used to modify/adjust resin selectivity
- SR vs PB
  - Different crown-ether concentrations, different alcohol chain lenghts
  - Facilitate Pb elution from PB Resin
  - PB Resin might be advantageous in case of Pb-210 only, especially in case of measurement by GPC (elution in water)
  - SR generally prefered in case of Pb-210 and Po-210 (higher Po yields) and in case of high matrix samples
- Using ‘cation exchanging’ diluents allows for direct loading of Pb (Po under examination) from acidified water samples

Example: Pb-210 and Po-210 determination in water samples

TRISKEM
Expertise in Separation Chemistry

Context:

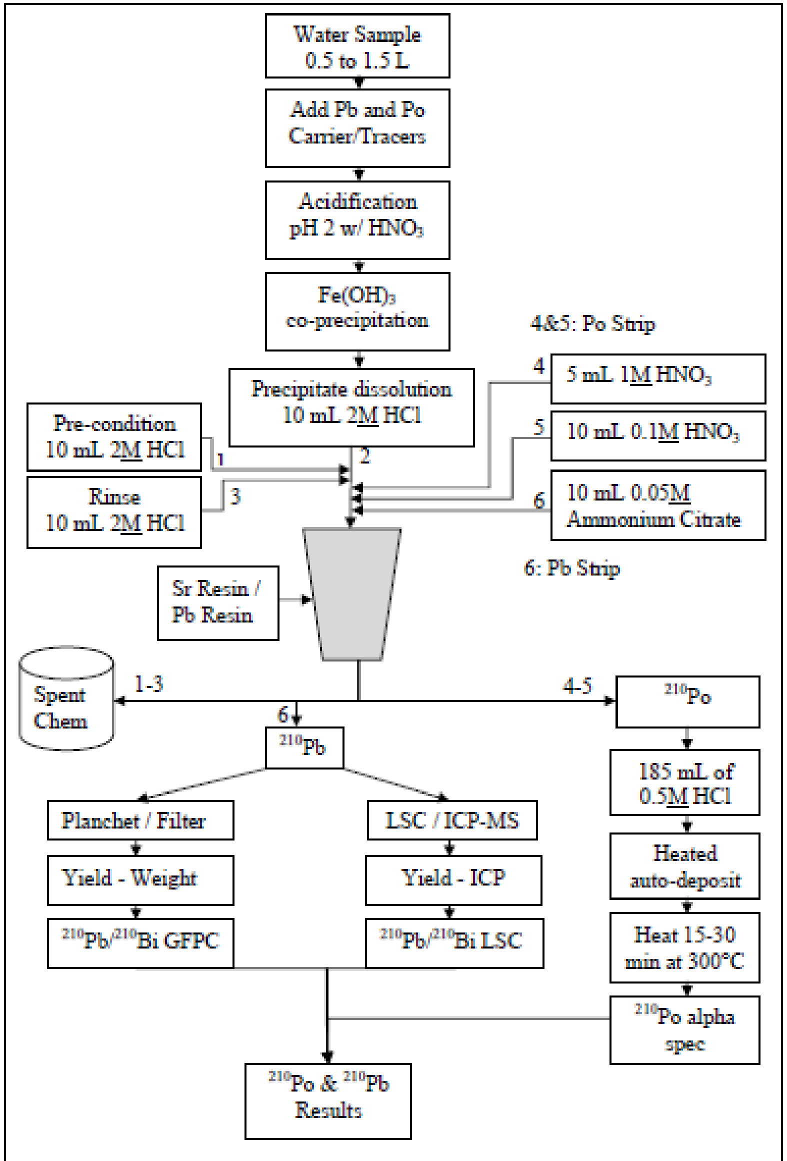
- European drinking water directive 98/83/EC
  - WHO recommendations
  - Calculation of Total Indicative Dose (TID)
    - ^{210}Pb-^{210}Po activities needed
- Analysis of radioactivity in natural and drinking waters
  - Pb-210 and Po-210 part of the natural radionuclides
  - Sensitive methods needed with low DL

Pb-210 / Po-210 in water samples

TRISKEM
Expertise in Separation Chemistry

- Sample volume 1.5L (pH 2)
  - Similar methods were tested for sea water samples
- Addition of 10 mg stable Pb and Po-208 or Po-209
- Pb-Po co-precipitation with $Fe(OH)_3$
  - Addition of 20 mg Fe(III)
  - Heating
  - Addition of 12 mL conc. ammonium hydroxide
  - Settling / filtration or centrifugation / rinsing
- Sequential separation of Po and Pb on Sr or Pb Resin
  - Only Pb: Pb resin advantageous (pricing, facile Pb elution)
  - Pb and Po: Sr resin advantageous (better Po yields)
- Po auto-deposition on Ag or Ni disc for α-spec

Pb/Po separation on Sr or Pb resin

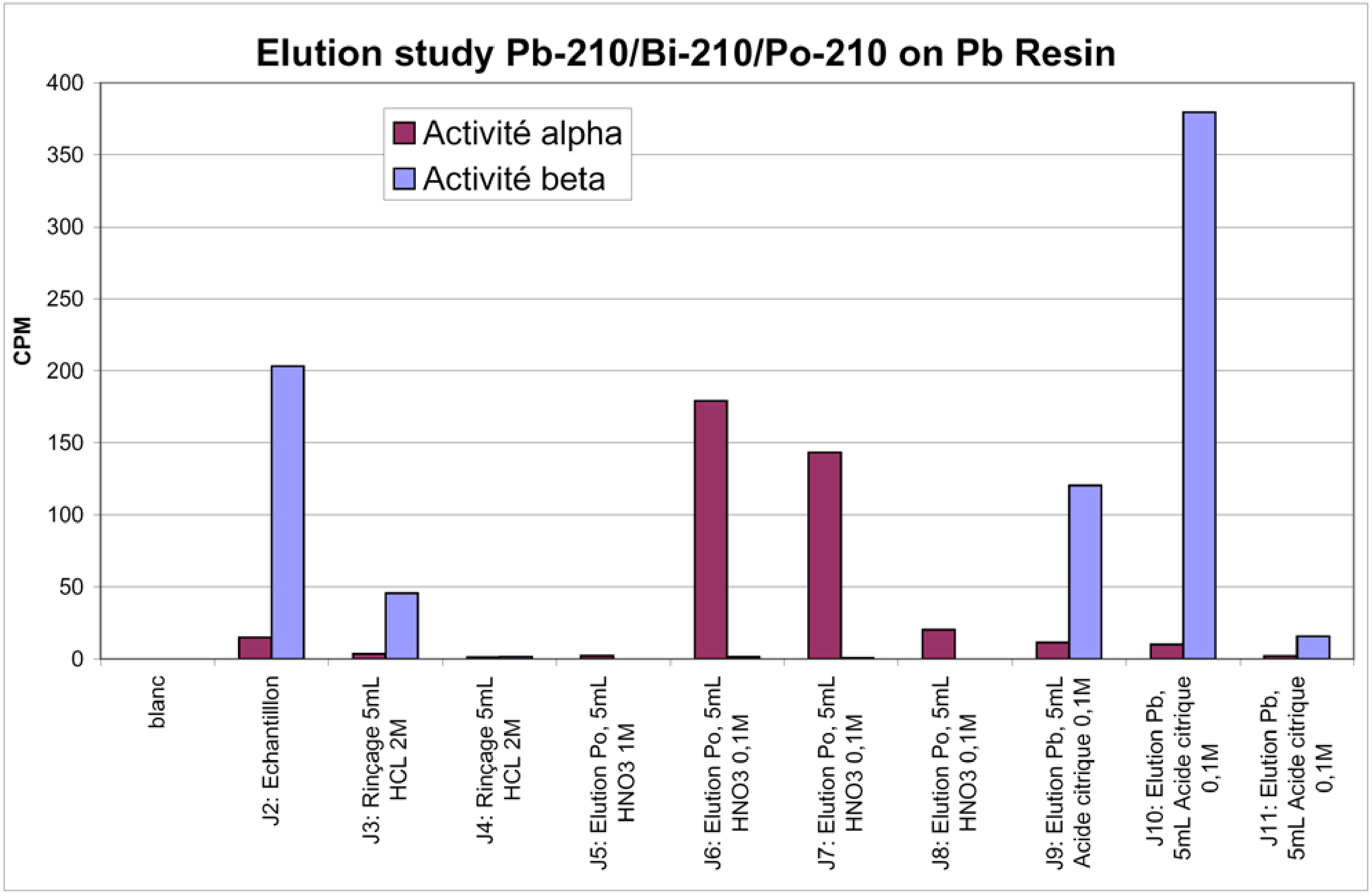


- After further optimisation introduced as application note/method
- Load from 2M HCl to retain Pb and Po
- Reminder, when loading from 1M HNO_3 only Pb retained
- Rinse with 1M and 0.1M HNO_3 to elute Po => autodeposition
- Pb elution in ammonium citrate
  - for LSC measurement
- In case of GPC measurement either
  - PB Resin => elution with water
  - SR Resin => elution in 6M HCl
    - Attn. Organics!

Pb-210 measurement

- Pb:
  - 1 mL for AAS(/ICP-MS) measurement → determination of chemical yield
  - 9 mL for LSC measurement (or GPC)
- Pb-210 Counting:
  - Direct counting of Pb-210 via LSC ($\varepsilon \approx 60$ %)
    - Time dependend calibration of LSC counter
    - Preferably 2 window method
  - LSC counting after Bi-210 ingrowth
    - Better ε, easier calibration, longer waiting time
  - Also possible: evaporation of Pb eluate and GPC
    - Problem: citratic acid, better elution with 20 mL dest. Water or 6M HCl

Method development – elution studies



Alpha/Beta discrimination LSC counting

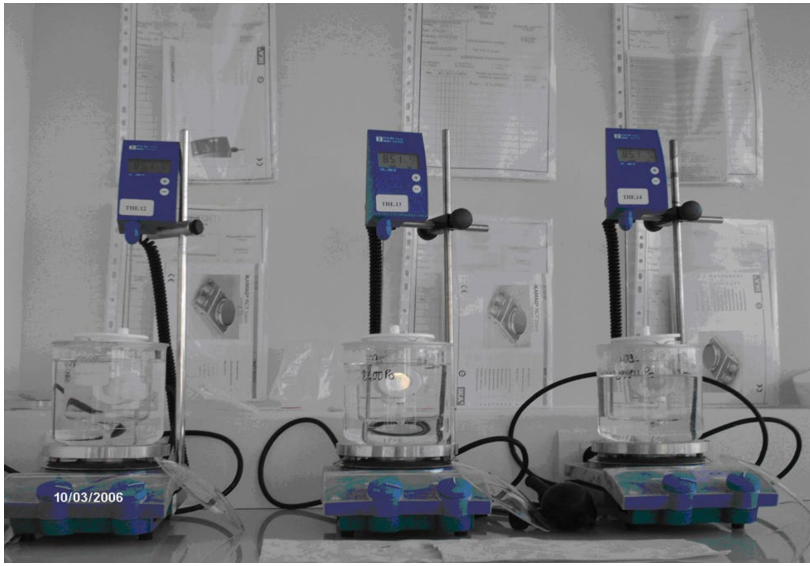
Selectivity

- Decontamination factors (DCF):
  - overall high for U, Th and Sr,
  - good DCF for Ra in Pb fraction,
  - rather low DCF for Ra in Po fraction - not problematic since Ra is not auto-deposited.

Interfering RN	Pb Fraction		Po Fraction	
	α DCF	β DCF	α DCF	β DCF
Ra-226	182	89	14	45
U-Nat	> 1000	> 1000	> 1000	> 1000
Th-Nat	> 1000	➢ 1000	> 1000	> 1000
Sr-90	NA	➢ 1000	NA	> 1000

Po-210 autodeposition

- Evaporate eluate to near dryness (T < 110°C)
- Convert residue into HCl form
  - Repeated evaporation with 5mL conc HCl
- Dissolve residue in 0.1M HCl
- Optional: addition of 100mg ascorbic acid
- Autodeposit Po onto Ag or Ni disc
  - T solution: 80 – 85°C
  - t between 90 min and 8 h
  - stirring



Comparison of ^{210}Po auto-deposition yields on Ni and Ag Discs

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	Recovery (%)		FWHM (keV)	
	mean	Std (%)	mean	Std (%)
Ni (N=10)	61	30	18.4	8.2
Ag (N=10)	99	8.6	19.1	15.8

- Ag discs
  - Quantitative auto-deposition
  - Good reproducibility
- Ni discs
  - Reproducibility not stable
  - Inbetween change of supplier
- Both discs show very good resolution

Performance data

- Precision
  - Repeatability s_r and Reproducibility s_R for Pb-210 and Pb-210 < 10% (k = 1)

	Pb-210	Po-210
Repeatability s_r (N=6)	2.2	8.9
Reproducibility s_R (N=12)	4.4	5.2

- Detection limits (DL) for V = 1.5 L
  - Pb-210 in the order of 20 $mBq.L^{-1}$ with counting time of 240 minutes
  - Po-210 in the order of 3 $mBq.L^{-1}$ with counting time of 1000 minutes

Accuracy

- Validation by succesful participation in intercomparison (BfS drinking water), comparison with accreditated method and analysis of spiked samples

	Pb-210					**Po-210**				
Sample ref.	Ref. A (Bq.L^{-1})	Uc A (Bq.L^{-1})	Exp. A (Bq.l^{-1})	Uc A (Bq.L^{-1})	t value	Ref. A (Bq.L^{-1})	Uc A (Bq.L^{-1})	Exp. A (Bq.l^{-1})	Uc A (Bq.L^{-1})	t value
Water 1+	9,61E-02	2,87E-02	7,00E-02	3,00E-02	0,6	1,30E-02	2;91E-03	1,33E-02	8,54E-03	0,1
Water 2+	< LD		< LD		N/A	2,03E-03	1,15E-03	1,60E-03	6,00E-04	0,3
Spiked Water 1	1,77E-01	1,93E-02	1,60E-01	3,30E603	0,9	1,60E-01	1,93E-02	1,39E-01	1,84E-02	1,0
Spiked Water 2	1,69E-01	5,15E-03	1,63E-01	8,09E-03	0,7	1,69E-01	5,15E-03	1,55E-01	1,94E-02	0,7
Spiked Water 3	1,66E-01	5,08E-03	1,58E-01	8,09E-03	0,9	1,66E-01	5,08E-03	1,50E-01	2,04E-02	0,7
Spiked Water 4	1,69E-01	5,18E-03	1,63E-01	8,09E-03	0,6	1,69E-01	5,18E-03	1,57E-01	1,80E-02	0,7
Mineralwater*	1,24E-01	1,69E-02	1,02E-01	3,20E-02	0,6	1,06E-01	1,30E-03	1,06E-01	8,76E-02	0,6

$$t = \frac{\left| C_{A,det} - C_{A,Ref} \right|}{\sqrt{U^2_{C_{A,det}} + U^2_{C_{A,ref}}}}$$

- $t \leq 1$ • No significant difference: accurate
- $t > 1$ • Results are not accurate

(t corresponds to E_n in ISO/IEC Guide 43-1)

Pb-210 / Po-210 in environmental and biological samples

Dell, A.N., Curtis, M.C.: The Sequential Determination of ^{210}Pb and ^{210}Po in Environmental Samples using an EIChroM Resin, 6th International Conference Cogema-La Hague, 1996

Vajda, N et al.: A Novel technique for the Simultaneous Determination of ^{210}Pb and ^{210}Po Using a crown Ether, J. Environ. Radioact. 37(3), 355ff, 1997

Horwitz, E.P. et al.: A novel Strontium-selective extraction chromatographic resin, Solv. Ext. Ion Exch., 10(2), 1992

M. Villa: Isolation of lead and polonium from seawater and determination of Pb-210 by LSC, Oral presentation, LSC08, Davos 2008

Pb and Po in milk, meat, crop and sediment samples (Dell et al.)

- Sample volume/mass: 100mL milk, 100g crop or meat, 10g sediment
- Addition of Po-208/9 and10 mg stable Pb
  - Preferably Po-209 (half-life, α-energy)
- Wet oxidation using HNO_3 and H_2O_2 at T< 110°C
  - low T to avoid Po losses
  - Use of HF, « aqua regia » for soil and sediments
- Separation of Po and Pb on Sr Resin (3g)
- Po auto-deposition on Ag or Ni disc
- α-spec

Elution behaviour of Bi, Pb, Po on 3g SR Resin

Vajda et aL; 1997

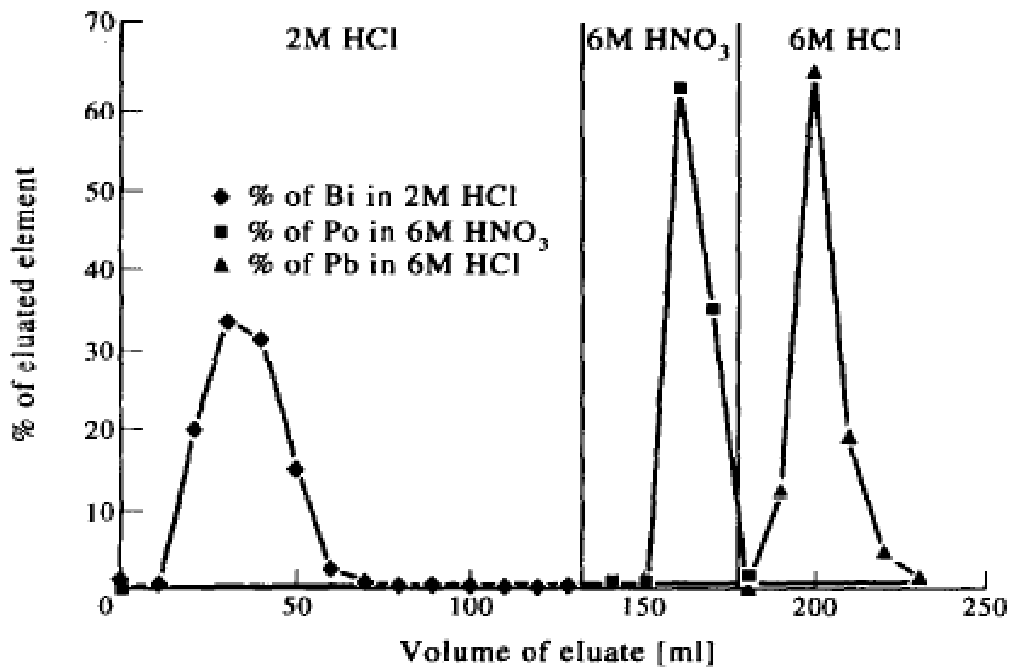
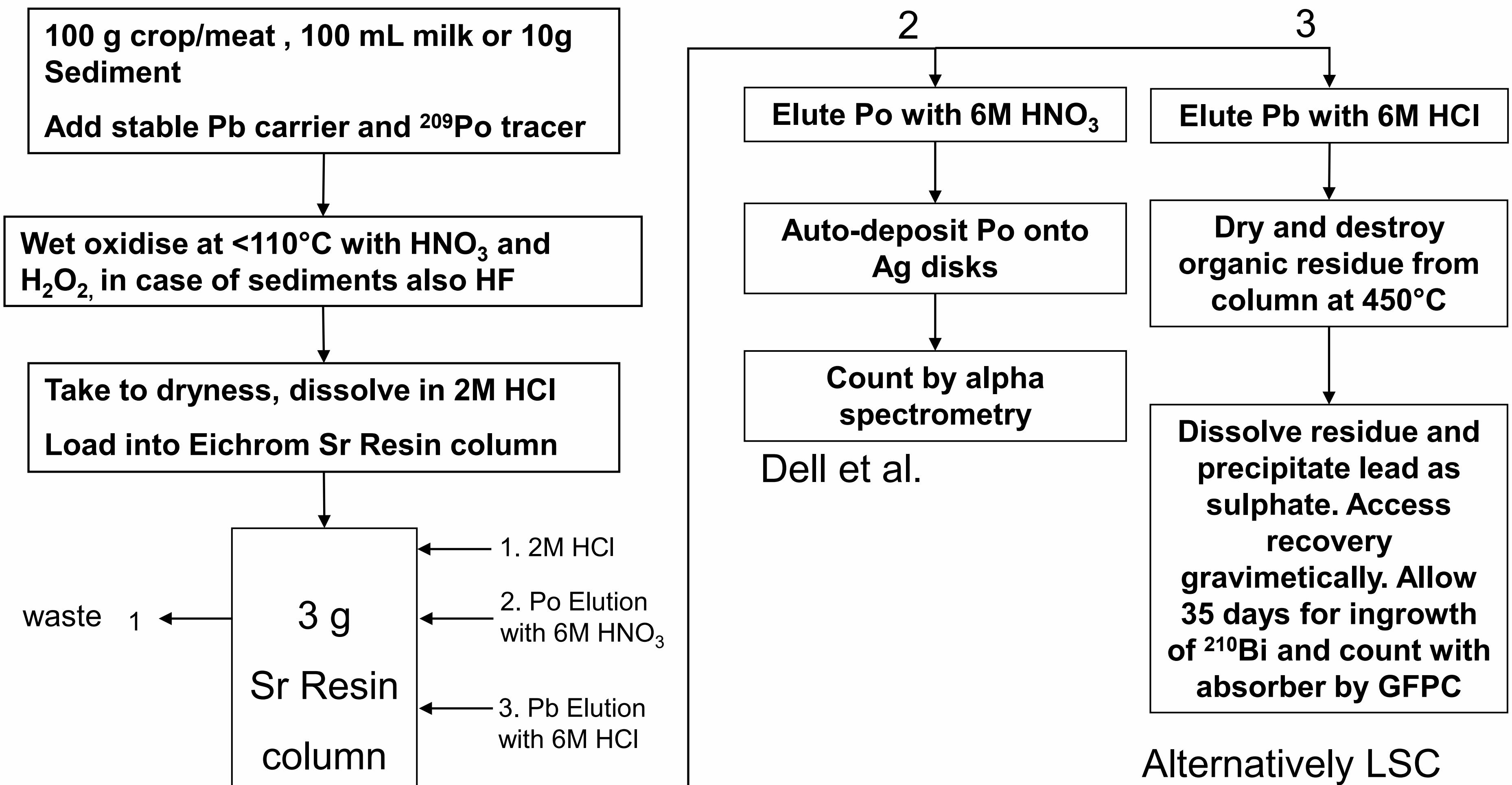


Fig. 2. Elution chromatogram of lead, bismuth and polonium on Sr. Spec.

- 3g columns require rather large elution volumes not very suitable for citrate elution
- Typically Pb eluted with 6M HCl => requires post-tratment

Pb and Po in milk, meat, crop and sediment samples (Dell et al.)



Recoveries Pb: 50 - 100 %, Po: 55 – 75 %

Pb-210 in soil samples (Vajda et al.)

- Sample preparation
- Add tracers/carrier at earliest convenience
  - 30 mg Pb in case of gravimetric yield determination
  - 1 – 10mg for spectrometry
- In case Po-210 to be determined mineralisation at < 110°C
  - If no Po higher temperatures may be employed
- 2 Options:
  - Complete dissolution
  - Leaching

Pb-210 in soil samples (Vajda et al.)

- Complete dissolution (volumes per 5g sample):
  - 3 evaporations with 10 mL conc. HNO_3 and 40 mL conc. HF
  - 3 evaporations with 30 mL conc HNO_3
  - Addition of 2g H_3BO_3 and 3 evaporations with 30 mL conc HCl
  - Dissolution in 30 – 50 mL 2M HCl
- Leaching (volumes per 5g sample):
  - 3 evaporations with 30 mL conc. HNO_3 and 5 mL 30% H_2O_2
  - 3 evaporations with 30 mL conc HCl
  - Dissolution in 30 – 50 mL 2M HCl
- Dissolution: warming for 30 min, followed by filtration

Pb-210 in soil samples (Vajda et al.)

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- Separation chemistry:
- 3g Sr resin column preconditioned with 2M HCl
- Load from 2M HCl
- Rinse beaker with 2 x 5 mL 5M HCl
- Rinse column with 90 mL 2M HCl (Bi removal)
- Rinse column with 60 mL 6M HNO_3 (Po removal)
- Elute Pb with 60 mL 6M HCl

Pb-210 in soil samples (Vajda et al.)

- Preparation of the sample for LSC measurement
  - Evaporation to dryness (yellow/brown residue will remain)
  - 3 evaporations with 2 mL conc HNO_3
- Option a.) Gravimetric yield determination
  - Dissolve residue in 20 mL 1M HNO_3
  - Add 400 mg ocalic acid
  - Adjust pH to 6 – 7
  - Filter, dry and weight
  - Redissolve in dilute acid (1 mL 6M HNO_3 or 10 mL 0.1M HNO_3)
  - Add cocktail

Pb-210 in soil samples (Vajda et al.)

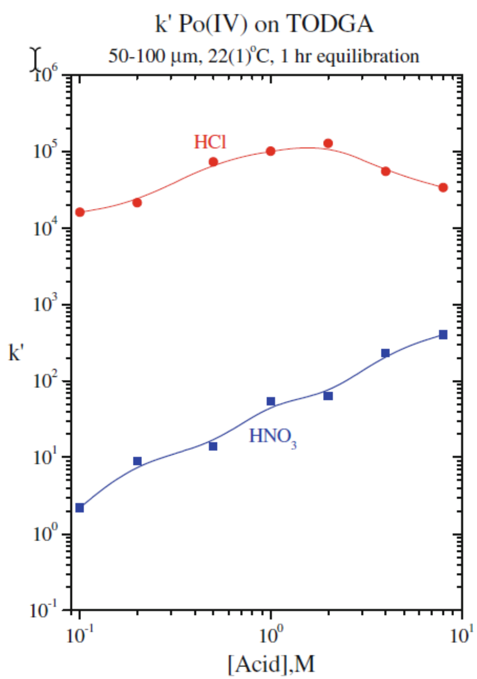
- Option b.) Spectrometric yield determination
  - Dissolve residue in 10 mL 0.1M HNO_3
  - Withdraw aliquot for yield determination
    - In case of yield determination by ICP-MS and addition of small amounts of Pb carrier it is preferable to control Pb content of original sample to prevent over-estimation of the chemical yield
  - Add cocktail
- Measurement by LSC

Pb-210 in soil samples (Benedik et al.)

- Preparation of the sample for GPC measurement
  - Addition of 2 – 3 mL conc H_2SO_4,
  - Heating for 30 min and filtration
  - Drying of filter and weighing
  - Gravimetric yield determination
  - Filter counting after Bi ingrowth

Po-210 via DGA Resin and MP

- Sherrod Maxwell et al.
- Rapid methods for water samples
- Co-precipitation with CaPhosphate
- Po separation via DGA Resin
- Sample preparation for alpha specrometry by microprecipitation with Bi-Phosphate



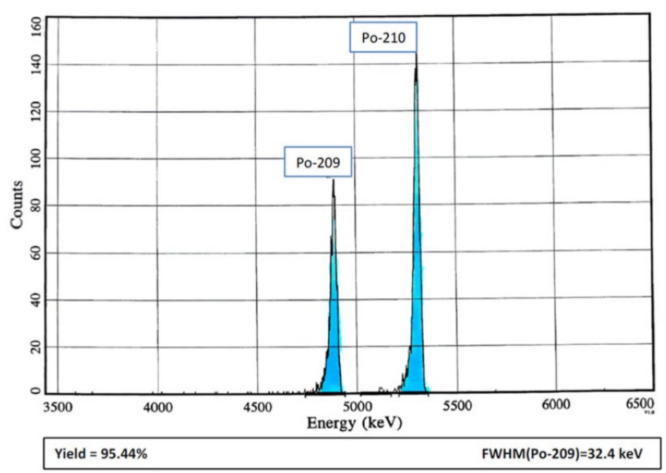
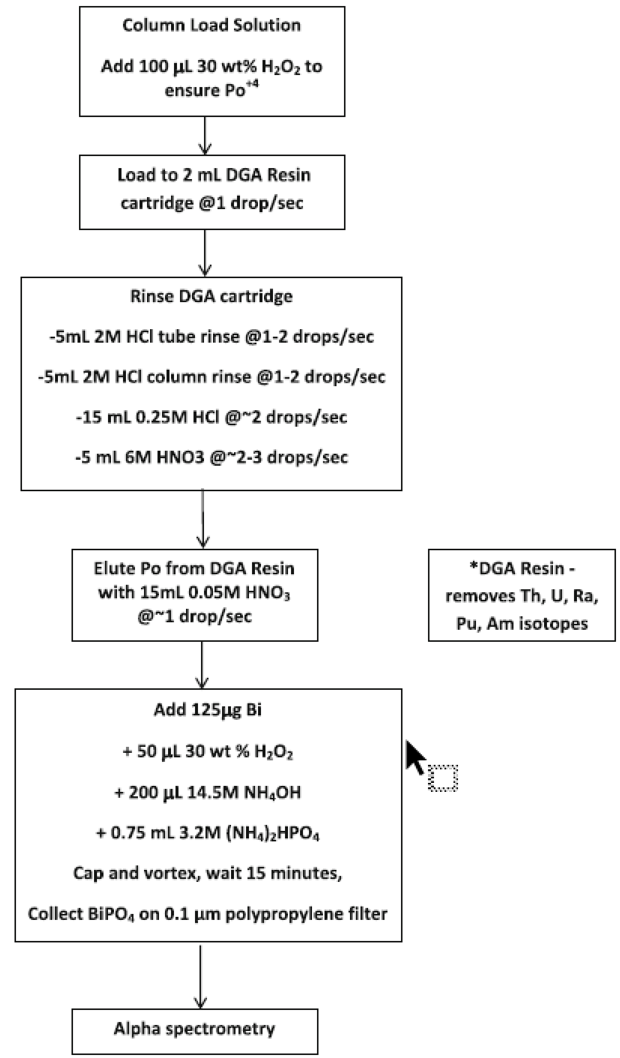


Fig. 8 Alpha spectra for Po isotopes using bismuth phosphate (100 µg Bi)



Thank you for your attention!

shappel@triskem.fr

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Elution experiment

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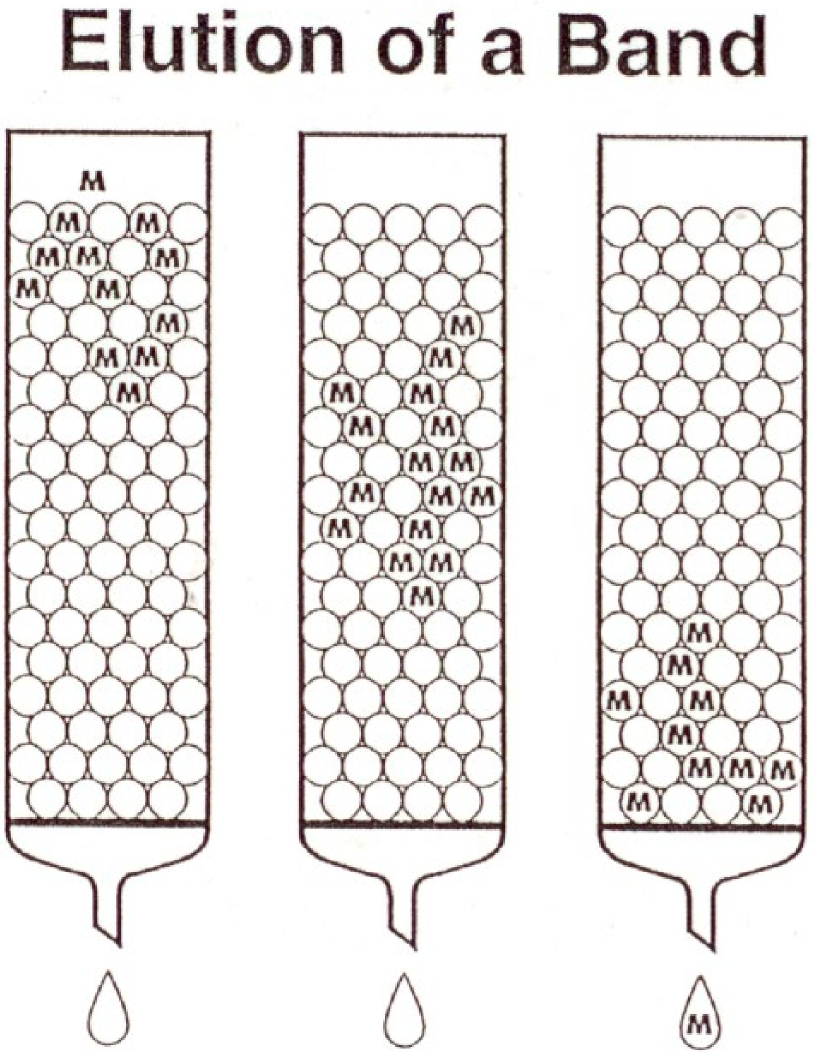
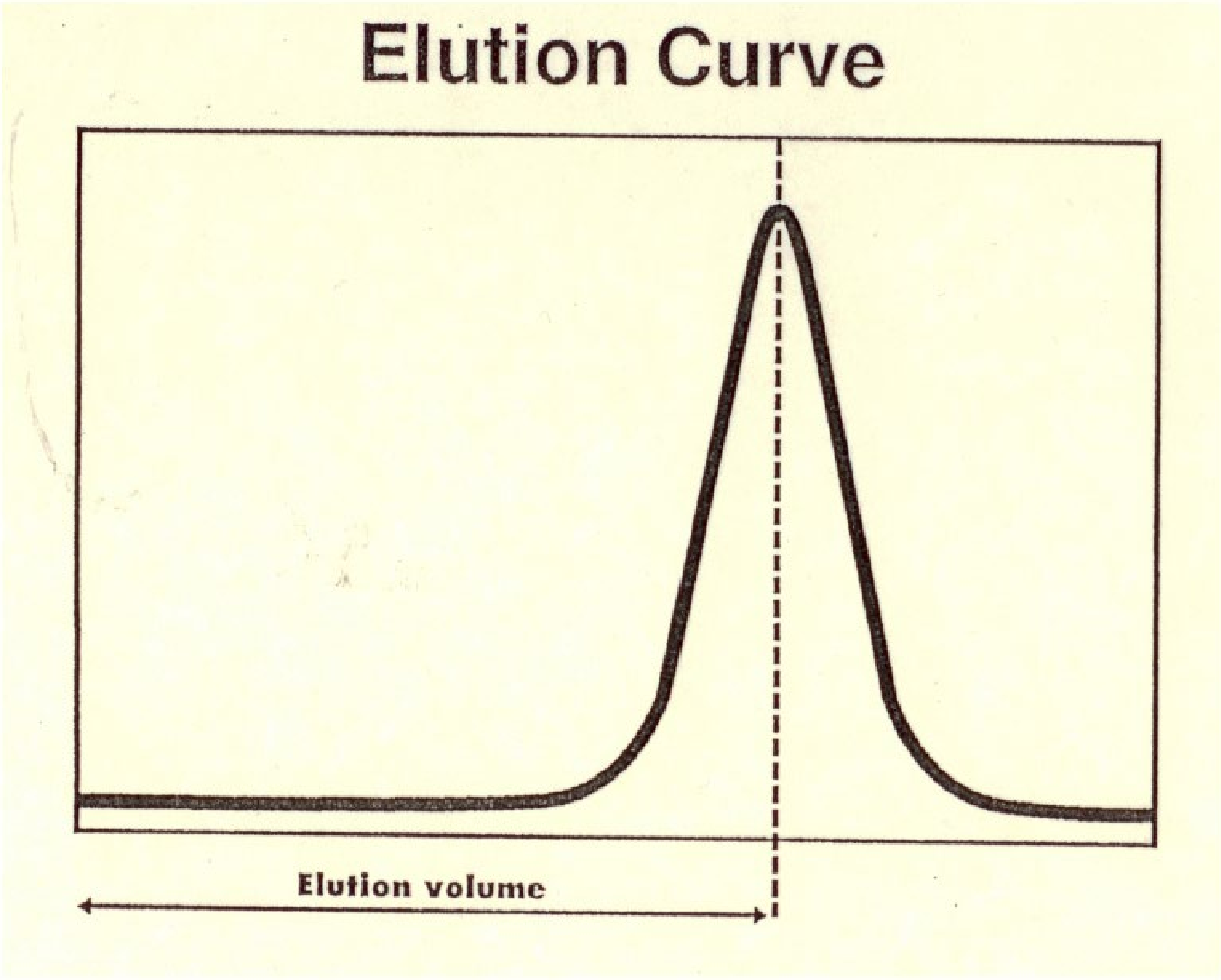


Detector

Stolen from
Bill Burnett!

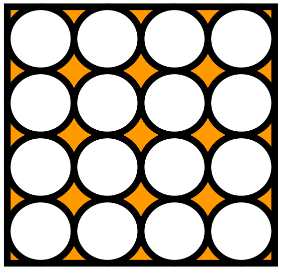
Elution experiment



Stolen from
Bill Burnett!

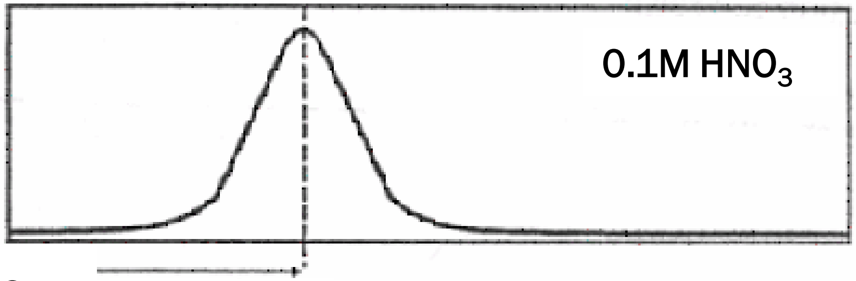
Free Column Volume (FCV)

- Volume of mobile phase in the resin bed:



- For extraction chromatography typically 0.65 mL per mL column bed
- Capacity factor k’ expressed in FCV for comparison of methods using different column geometry - normalisation

Elution curves => k'



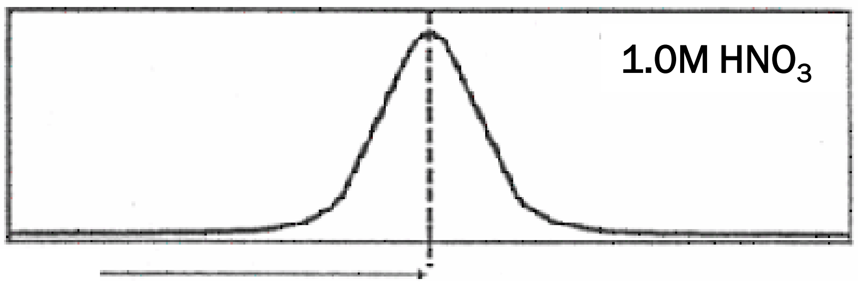
Elution volume $V_1 \propto k_1$

$k_1 = V_1/FCV$

FCV = free column volume

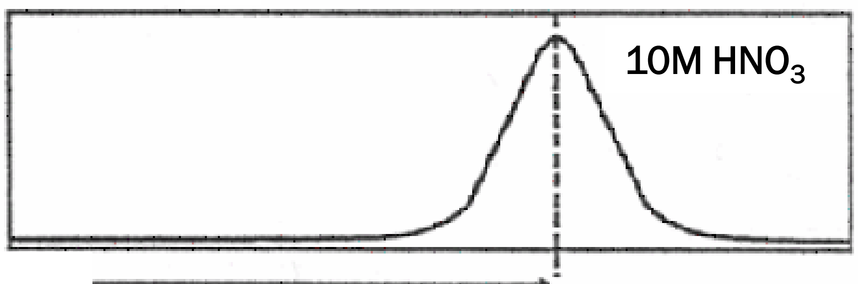
k' = 5 => V_1 ~ 6,5 mL

- **Need min. 15 mL for elution**



Elution volume $V_2 \propto k_2$

$k_2 = V_2/FCV$



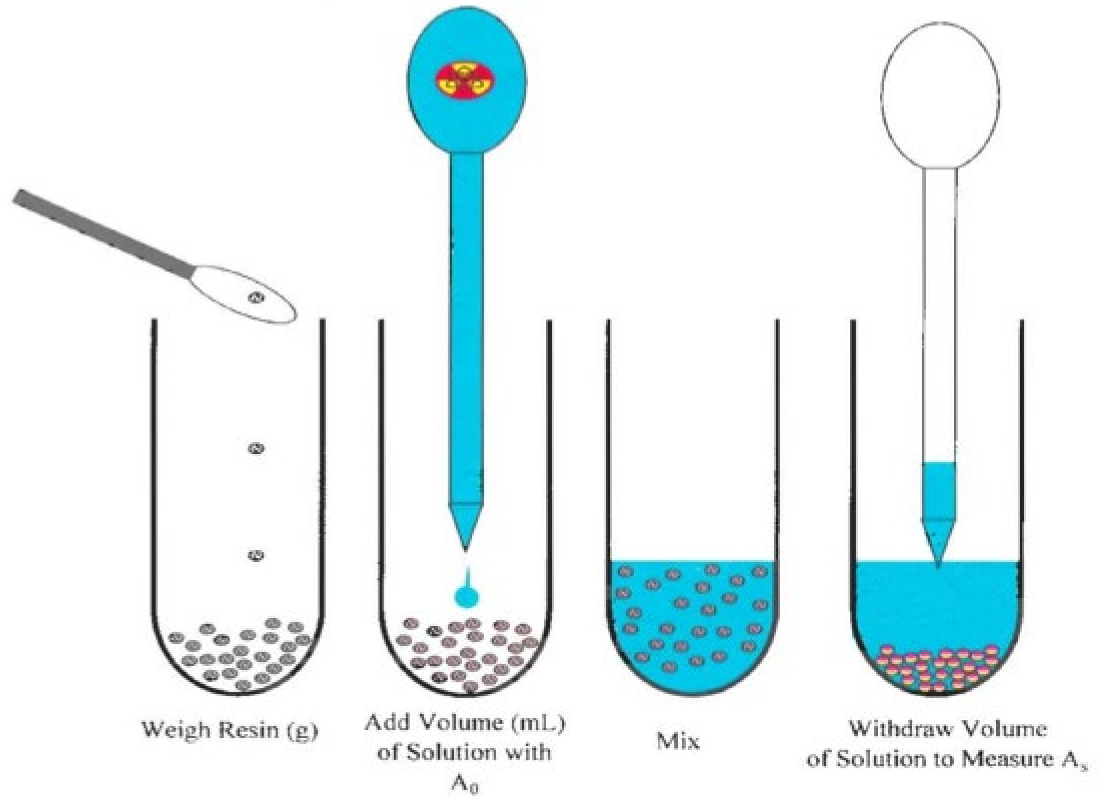
Elution volume $V_3 \propto k_3$

$k_3 = V_3/FCV$

For k' = 1000

- **V_3 ~ 1,3L!**

Dry Weigh Distribution Ratio

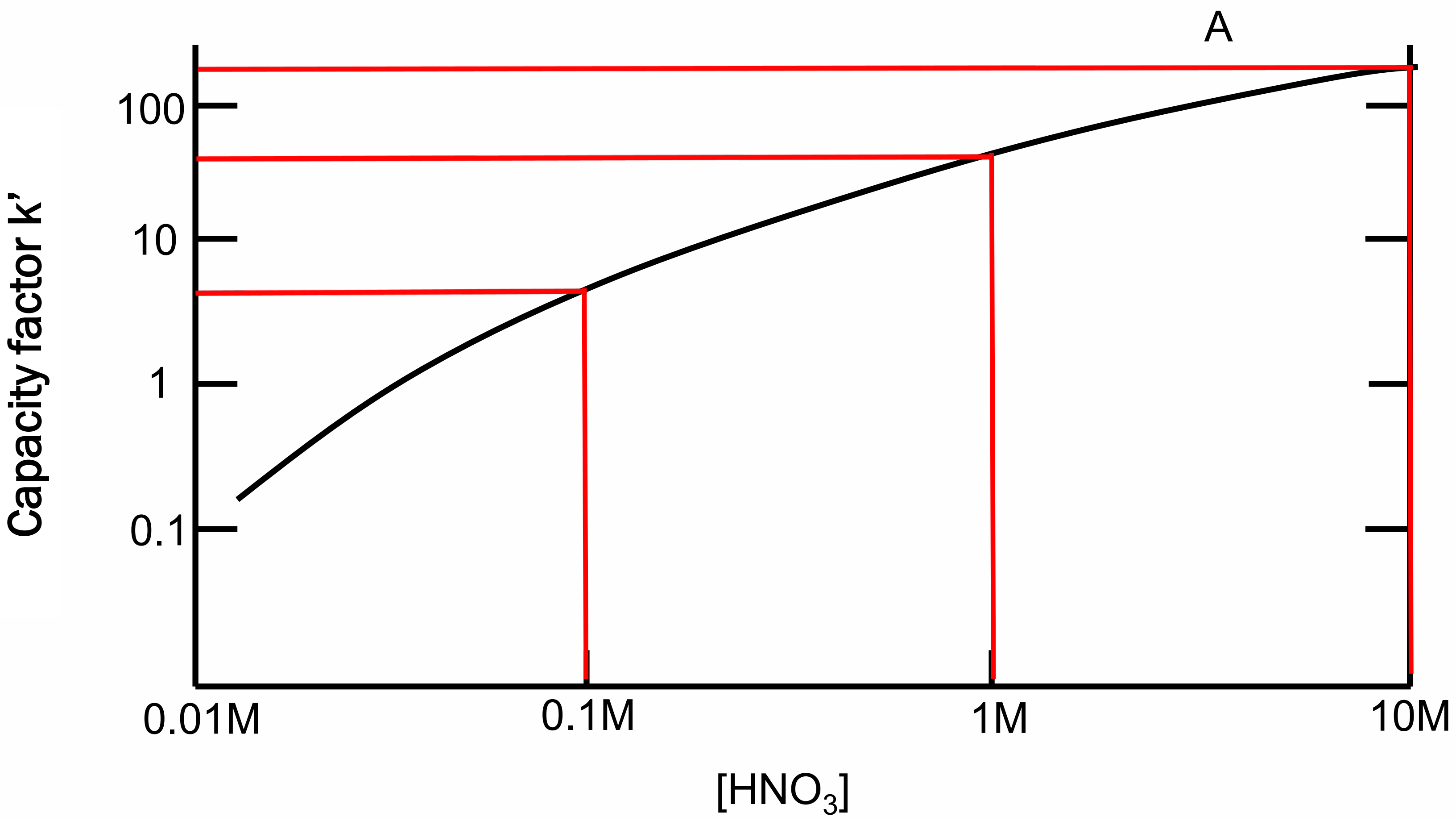


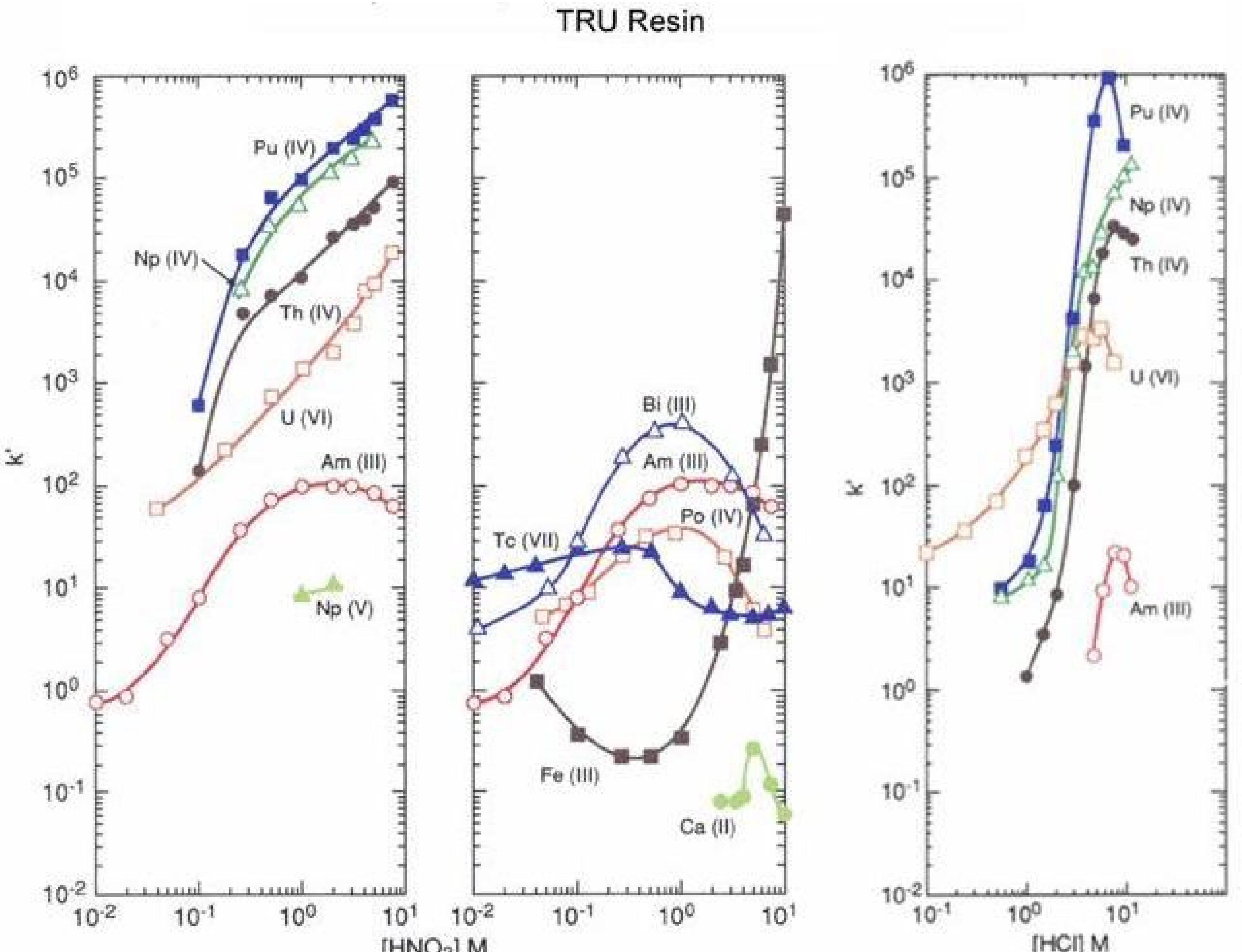
Horwitz

$$D_W = \frac{A_0 - As}{w(g)} \Big/ \frac{A_s}{v(mL)}$$

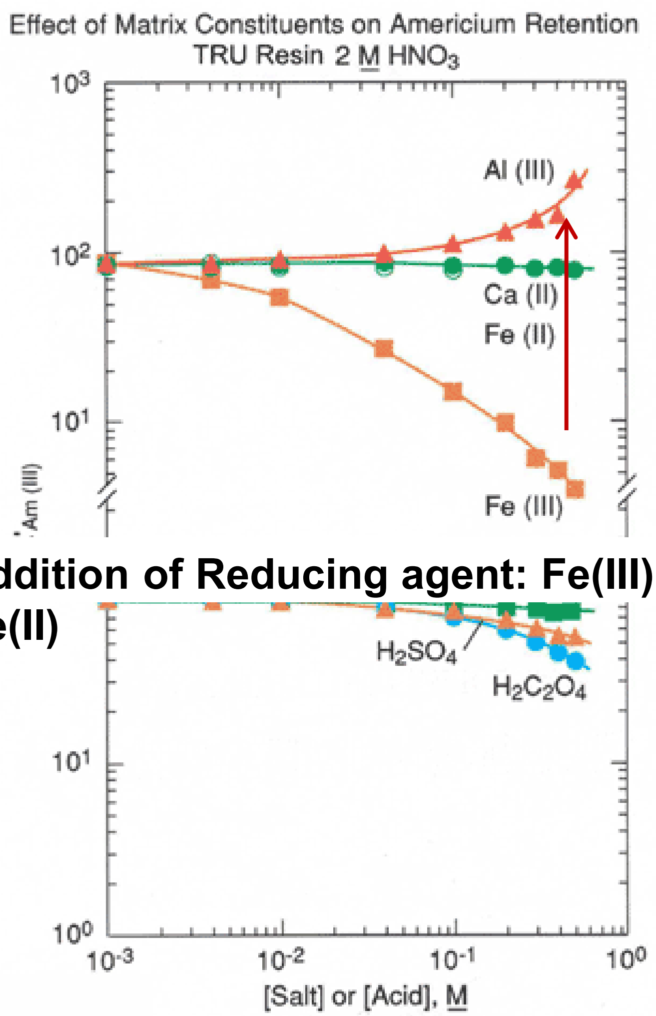
$k' \sim D_W * 0.5$

k’ = f ([acid])

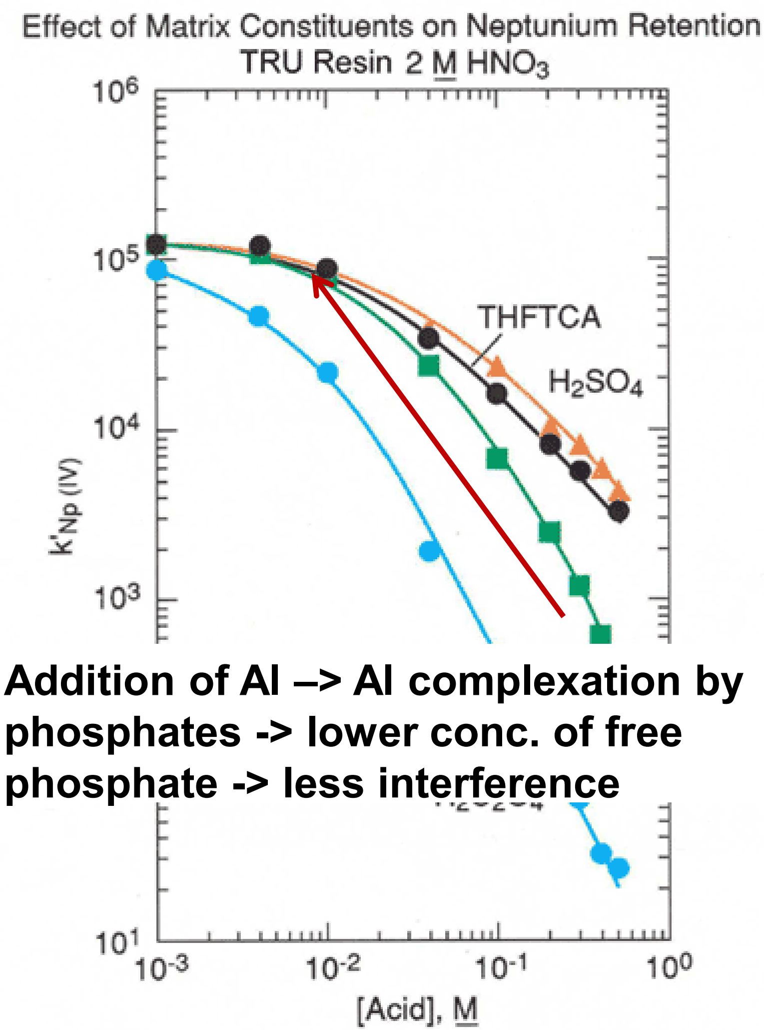




From Horwitz (1)



Addition of Reducing agent: Fe(III) -> Fe(II)

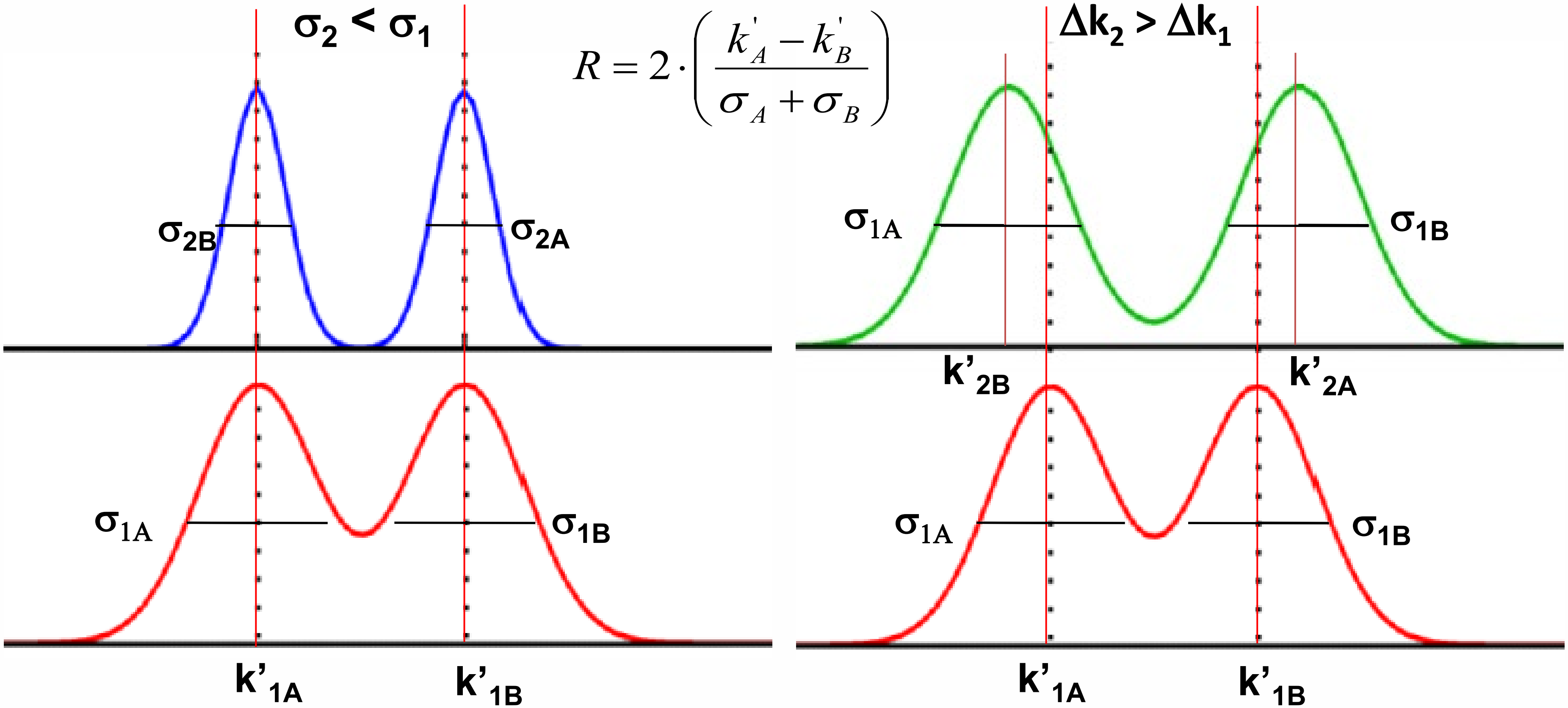


Addition of Al –> Al complexation by phosphates -> lower conc. of free phosphate -> less interference

(HP193)

From Horwitz (1)

Resolution R

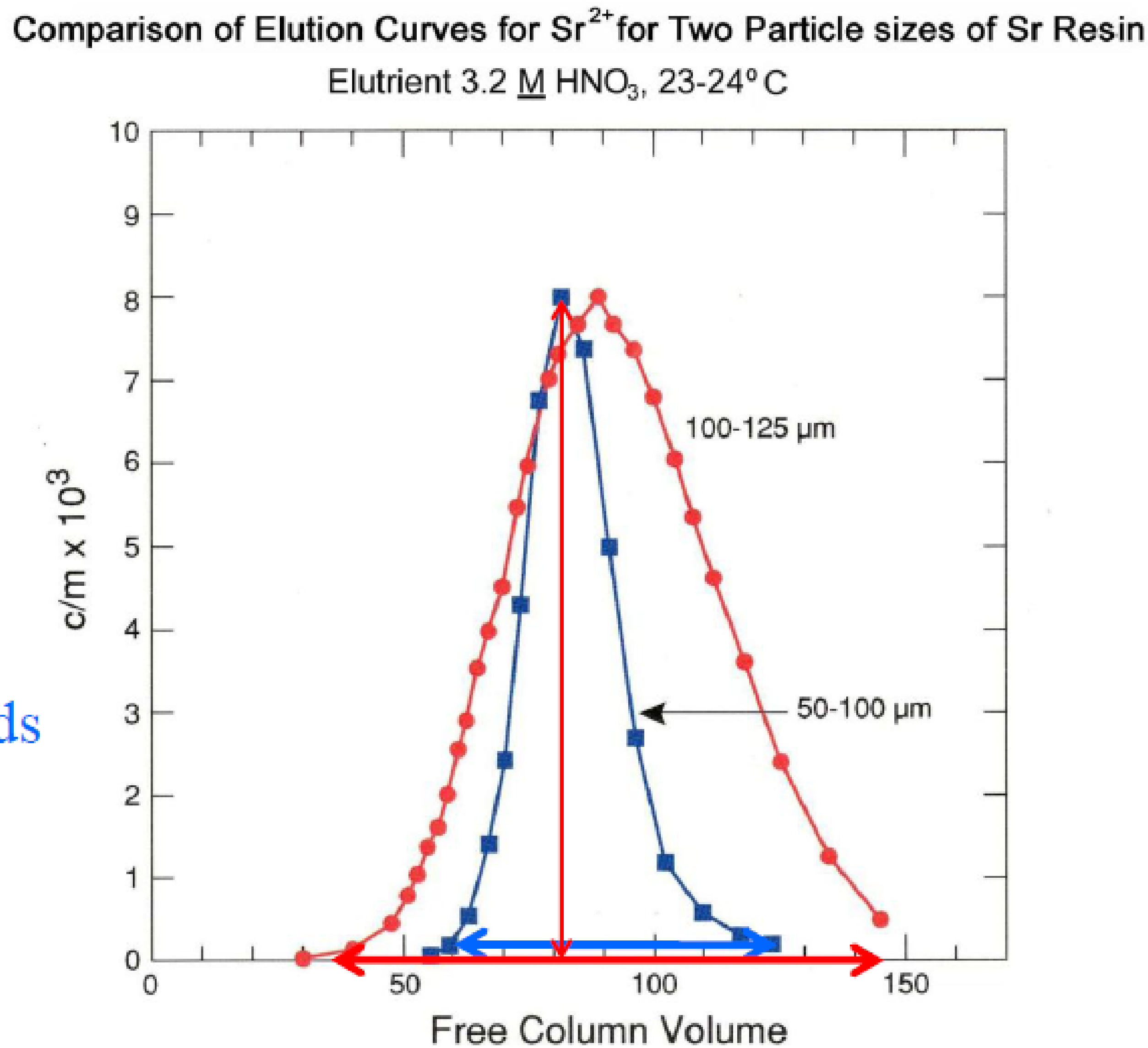


$$R = 2 \cdot \left(\frac{k'_A - k'_B}{\sigma_A + \sigma_B} \right)$$

Improvement of resolution R

- Decrease peak width σ: e.g. particle diameter, flow rate
- Increase difference of k' values : modification of stationary or mobile phase, valence, auxiliary compexants

- Smaller particles, sharper elution bands
- Peak maximum corresponds to k'



Taken from Horwitz, 2011

Results (Vrecek et al.)

Table 1
Survey of results for ^{210}Pb and ^{210}Po in IAEA-300 using procedure (a)–(d)

Procedures used in the processing of a sample of IAEA-300 reference material	^{210}Pb ($Bq\,kg^{-1}$)[a]	^{210}Po ($Bq\,kg^{-1}$)[a]
(Baltic Sea sediment)	(Pb recovery in %)	(Po recovery in %)
Total sample dissolution (a)	352±42 (91)	401±40 (12)
Dissolution after equilibration (b)	351±42 (93)	313±31 (19)
Leaching with HF and HNO_3 (c)	348±41 (88)	337±34 (42)
Leaching with H_2O_2 and HNO_3 (d)	343±26 (84)	347±31 (54)
Recommended value	360	340.5
Confidence interval	(339–395)	(273.6–361.0)

[a] Error margins derived from three replicate measurements.

Table 2
Comparison of ^{210}Pb and ^{210}Po results by radiochemical procedure (d) and gamma spectrometry

Sample	^{210}Pb ($Bq\,kg^{-1}$)[a] (radiochemical: procedure d)	^{210}Pb ($Bq\,kg^{-1}$)[b] (non-destructive: direct γ-detection)	^{210}Po ($Bq\,kg^{-1}$)[a] (radiochemical: procedure d)
River sediment 1	63±8	60±13	65±7
River sediment 2	69±8	66±11	68±7
River sediment 3	141±7	144±6	145±6
Soil near TPT 1	126±13	121±12	124±12
Soil near TPT 2	110±13	100±10	111±9
Soil near TPT 3	75±9	80±8	107±9
Needles near TPT	68±11	—	69±17
Grass near TPT	36±4	—	32±6
IAEA 368	26±4	21±2	20±2
(sediment)	*23 (15%)*[c]	*23 (15%)*[c]	
IAEA 307	56±7	63±3	60±10
(sea plant)	*59 (40–91)*[c]	*59 (40–91)*[c]	
IAEA 327	63±7	57±2	57±9
(soil)	*59 (8%)*[c]	*59 (8%)*[c]	

Note: ^{210}Pb and ^{210}Po are considered as being in equilibrium.
[a] Error margins derived from three replicate measurements.
[b] Error margin derived from the statistical variation of a single sample analysed.
[c] Certified values in italics.