

VALIDATION OF AN IMPROVED METHOD FOR THE SEPARATION AND MEASUREMENT OF Pb-210 AND Po-210

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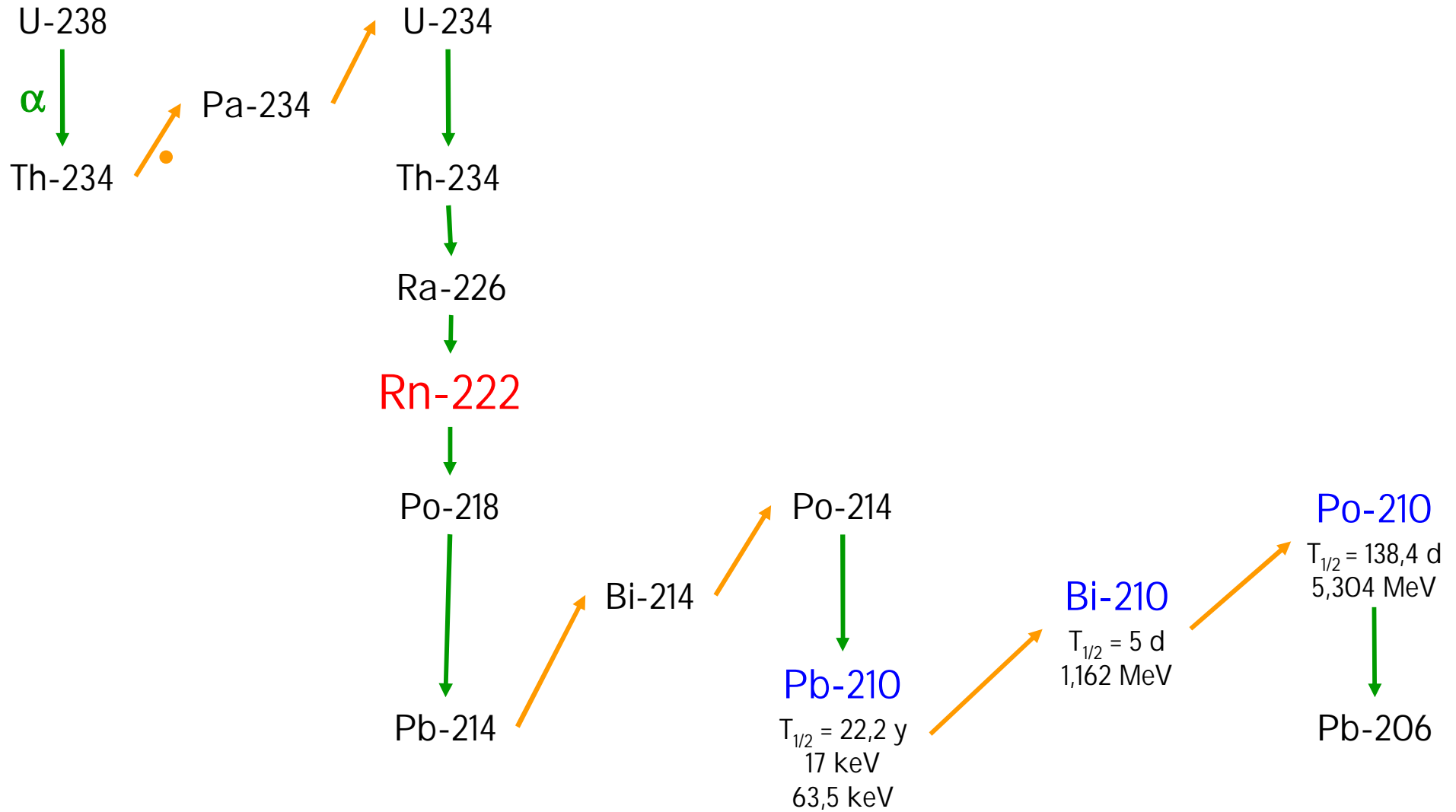
Outline

- § Why measurement of Pb-210 and Po-210 required?
- § Procedure
 - Former method
 - optimized method
- § Results of validation
- § Comparison Ag and Ni discs for ^{210}Po autodeposition
- § Conclusion

Why measuring Pb-210 and Po-210?

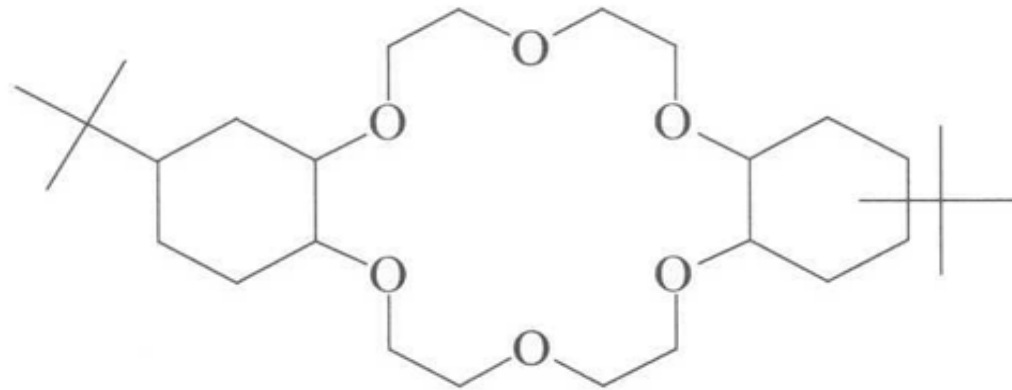
- § European drinking water directive 98/83/EC
 - WHO recommendations
 - Calculation of Total Indicative Dose (TID)
 - ^{210}Pb - ^{210}Po activities needed
- § Analysis of radioactivity in natural and drinking waters
 - Pb-210 and Po-210 part of the natural radionuclides
 - No normative method
 - Sensitive methods needed with low DL

Reminder



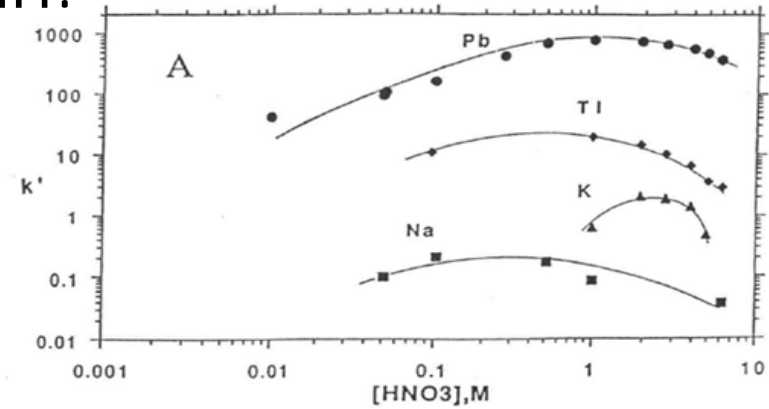
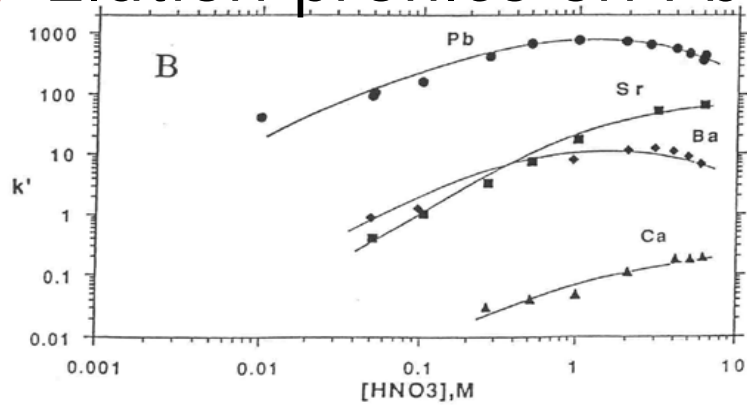
Existing data on Pb Resin (1/2)

- Extractant : crown ether (same as Sr Resin)

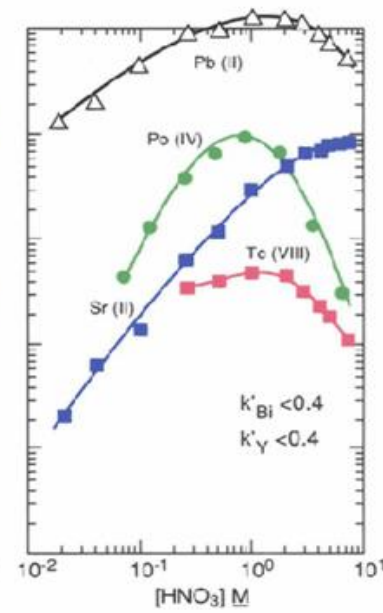
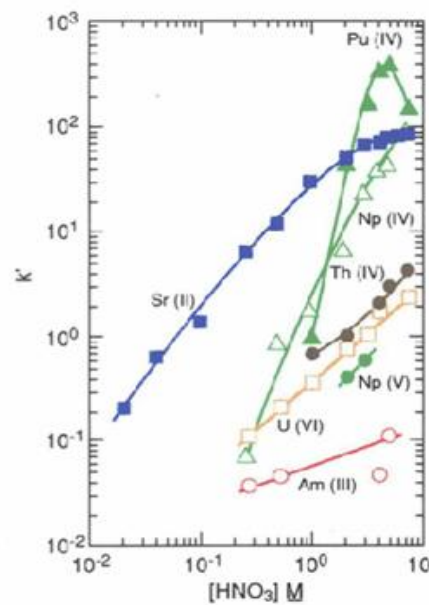
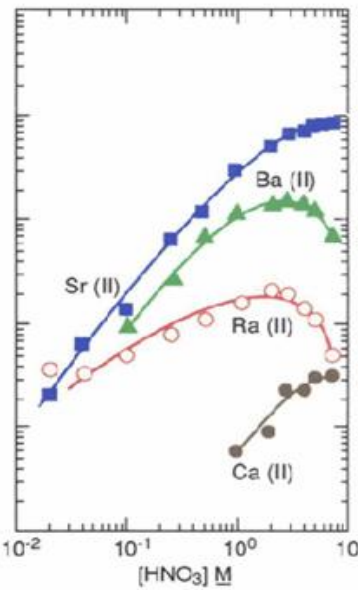
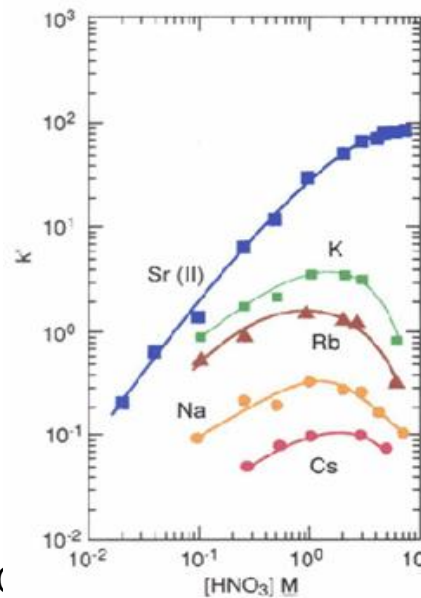


Existing data on Pb Resin (2/2)

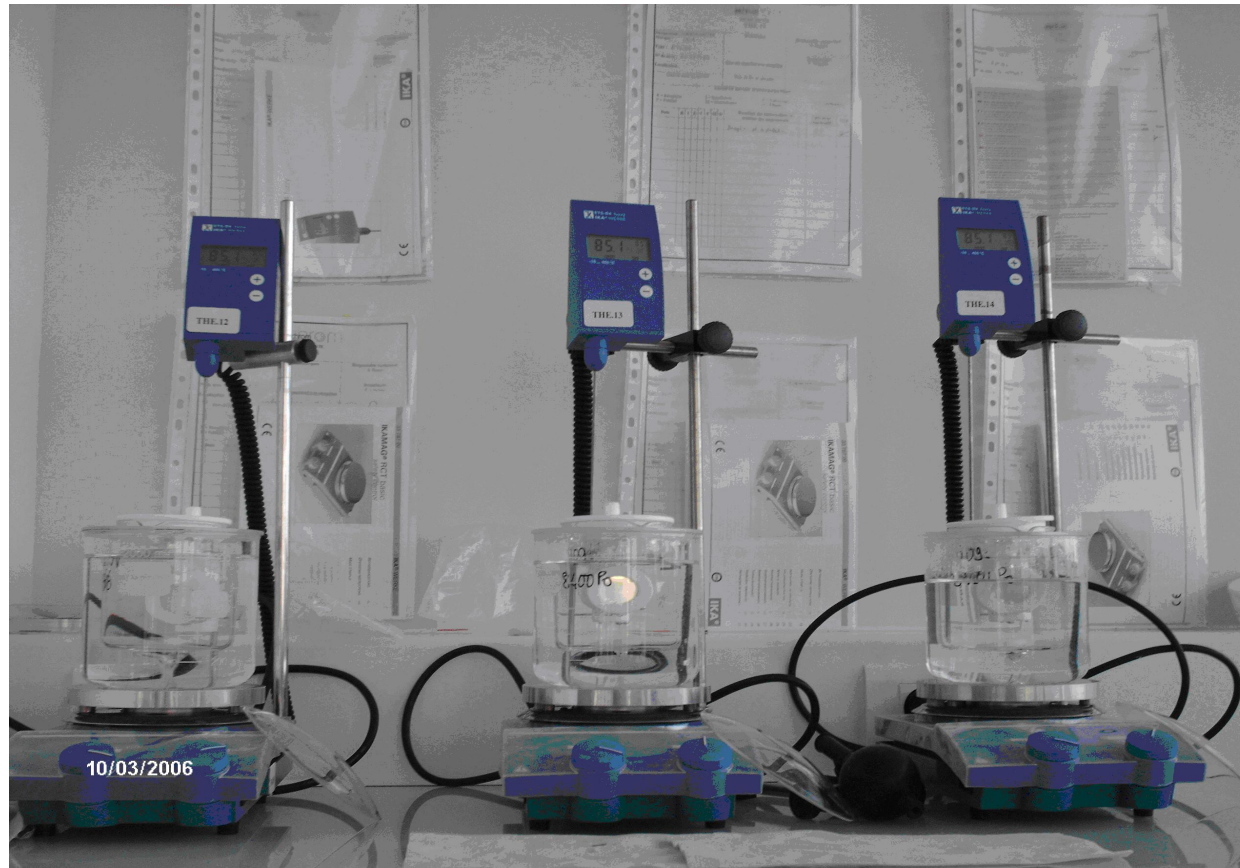
§ Elution profiles on Pb Resin:



§ Elution profiles on Sr Resin:



Experiments



^{210}Pb - ^{210}Po separation procedure: method 1

- § Pb-Po pre-concentration on cationic resin (C8-B500-M-H)
- § Sequential separation on Sr Resin of Po and Pb
- § Po auto-deposited on Ag disc for α -spec
- § Pb measurement
 - 1mL for AAS measurement • determination of chemical yield
 - 9mL for LSC measurement

Synop1_Pb

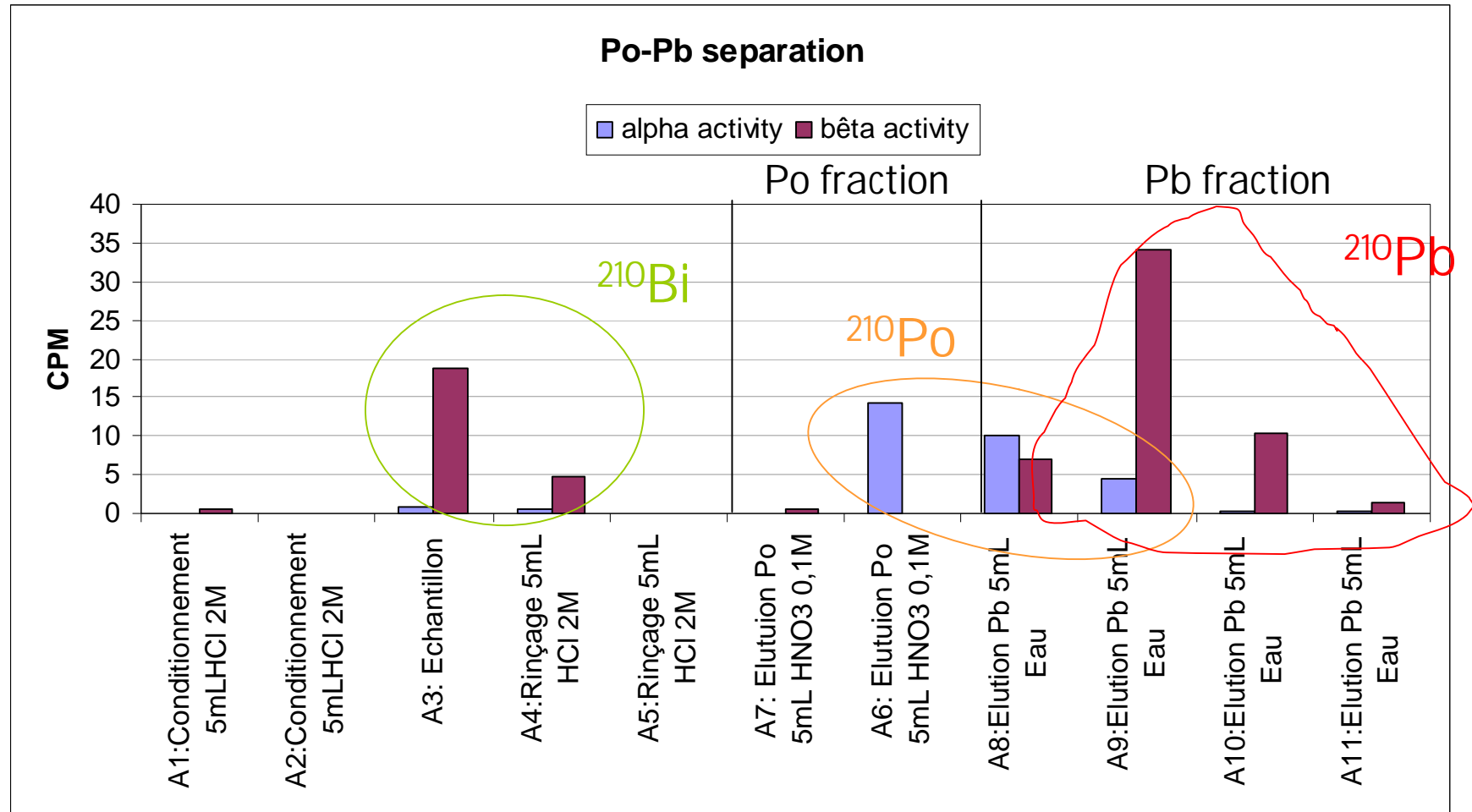
^{210}Pb - ^{210}Po separation procedure: 2nd method

- § Pb-Po co-precipitation with $\text{Fe}(\text{OH})_3$
- § Sequential separation on Pb Resin of Po and Pb
- § Po auto-deposited on Ag disc for α -spec
- § Pb measurement
 - 1mL for AAS measurement • determination of chemical yield
 - 9mL for LSC measurement

Optimisation of method 2 (A)

§ Conditions

- Resin conditioning with 10 mL 2M HCl
- Load of sample
- Rinsing with 10 mL 2M HCl
- Elution of Po with 10 mL 0.1M HNO₃
- Elution Pb with 20 mL H₂O



Optimisation of method 2 (A)

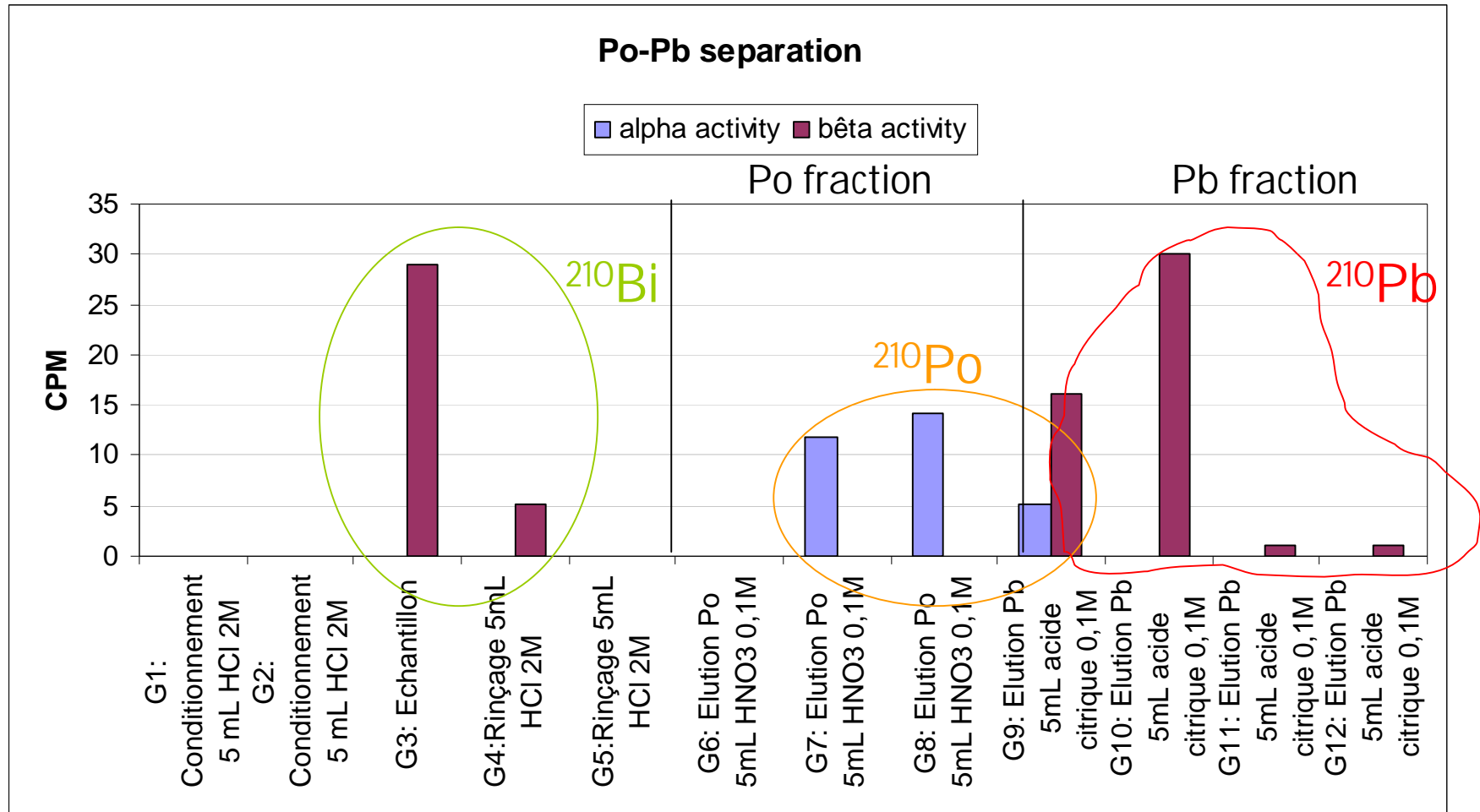
§ Results

- ^{210}Bi in load and rinsing fractions
- Pb elution with H_2O :
 - 100% Pb
 - 39% Po

Optimisation method 2 (B)

§ Conditions

- Resin conditioning with 10 mL 2M HCl
- Load of sample
- Rinsing with 10 mL 2M HCl
- Elution of Po with 15 mL 0.1M HNO₃
- Elution Pb with 20 mL 0.1M citric acid



Optimisation method 2 (B)

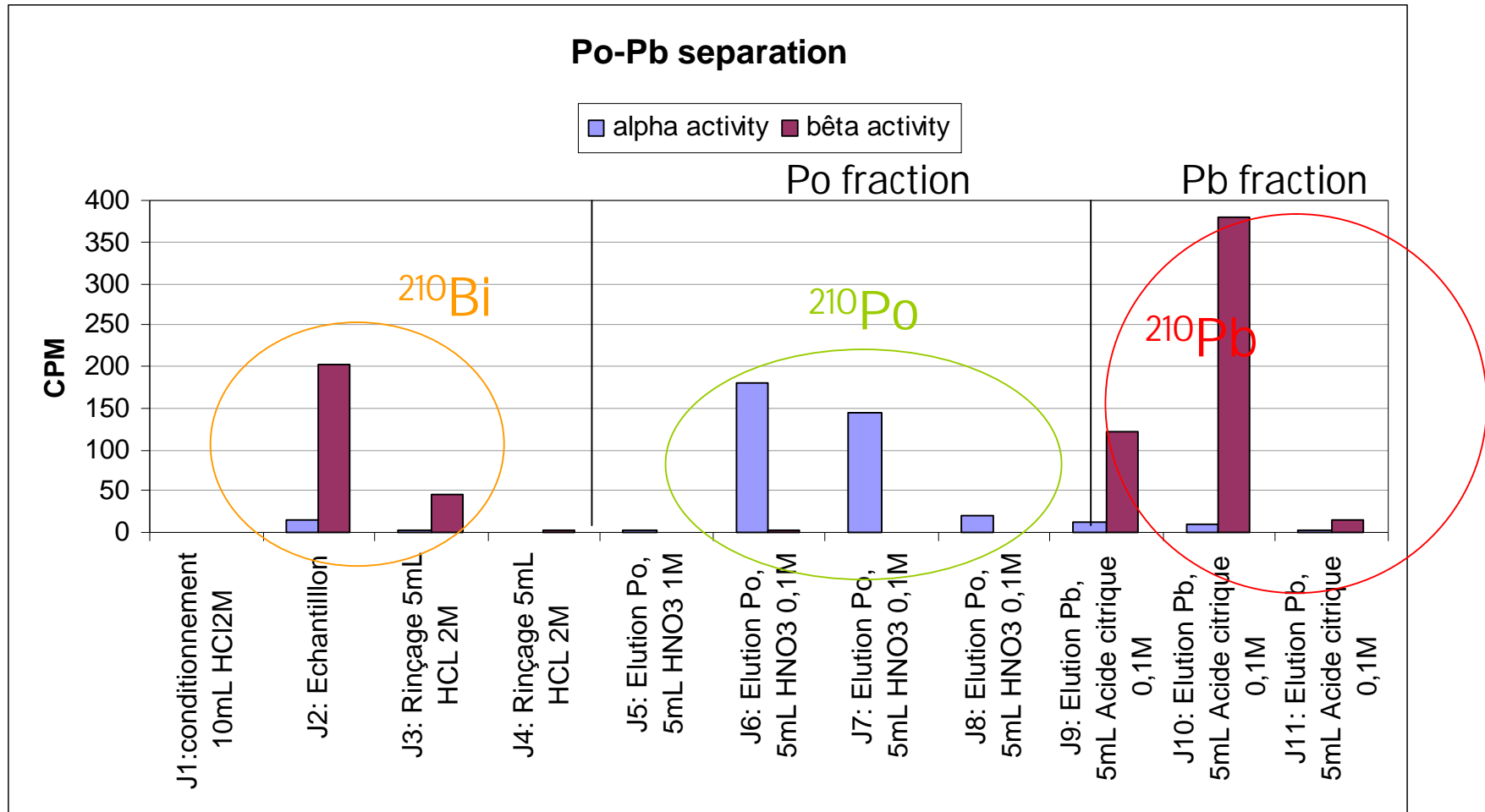
§ Results

- Bi in load and rinsing fractions
- Po elution : less than 0.1% Pb in Po fraction
- Pb elution :
 - 17% Po in 1st Pb fraction
 - 67% Pb recovery with no Po in fraction

Optimisation of method 2 (C)

§ Conditions

- Resin conditioning with 10 mL 2M HCl
- Load of sample
- Rinsing with 10 mL 2M HCl
- Elution of Po with 5 mL 1M then 10 mL 0.1M HNO_3
- Elution Pb with 15 mL 0.1M citric acid



Optimisation of method 2 (C)

§ Results

- Bi in load and rinsing fractions
- Po elution : less than 0.2% Pb in Po fraction, 83% Po recovery
- Pb elution : quantitative Pb recovery with 6% Po in Pb fraction

Preliminary conclusions

- § Differences between methods 1 and 2:
 - Different pre-concentration method
 - Different resin
 - 2M HCl media instead of 1M HNO₃
 - Higher recovery yields for Po (80% instead of 50%) with method 2
- § Pb elution with H₂O possible
 - ∅ but larger volume needed compared to citric acid
- § Pb eluted with 20mL 6M HCl with method 1
- § Pb eluted with 10mL citric acid (97% of total Pb) with method 2

Synop2_Pb

Results of validation for method 2: 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

Results of validation – method 2: Accuracy

§ t values • 1 for all analysed samples

Sample ref.	Pb-210					Po-210				
	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	3.30E-03	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
CRM (PTB)	8.00E-02	4.00E-03	8.70E-02	2.20E-02	0.3	8.00E-02	4.00E-03	8.50E-02	2.10E-02	0.2

+sample previously measured using accredited method

*mean of results obtained by other laboratories

Definition of Accuracy with t value

$$t = \frac{|C_{A,\text{det}} - C_{A,\text{Ref}}|}{\sqrt{U_{C_{A,\text{det}}}^2 + U_{C_{A,\text{ref}}}^2}}$$

§ $t \leq 1$ • No significant difference: accurate

§ $t > 1$ • Results are not accurate

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

Results of validation

§ Precision

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

- ∅ s_r and/or $s_R \leq 15\%$ • results OK
- ∅ s_r and/or $s_R > 15\%$ • results not accepted

§ 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

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

	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
- § Both discs show very good resolution

Conclusion

- § 2 methods available for sequential separation of ^{210}Pb - ^{210}Po
- § Auto-deposition of Po on either Ag or Ni discs

