Determination of ^{210}Pb in Water

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AAHFS - Food Control and Research Vienna
(till 31.05.2002: “Federal Institute for Food Control and Research”)

Why measure Pb-210

- Chemotoxic
- Radiotoxic
- Long half life ($\tau_{½}$ = 22.3 years)
- accumulated in bone

Pb-210: Legislation

- **Austria**
  - 1.23 Bq/L Pb-210 in drinking water
    (Austrian Radiation Protection Ordinance, 1972)
- **EU**
  - current: Drinking Water Directive 98/83/EG (3.11.1998)
    ➜ Pb-210 exempted
  - recommendation: 2001/928/Euratom (20.12.2001)

✓ national reference values should be set
✓ remediation reference concentration: 0.2 Bq/L Pb-210

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Pb-210: Chemistry

U-238-Decay Series:

$$^{222}\text{Rn} \rightarrow \rightarrow {}^{210}\text{Pb} \xrightarrow[22.3\ \text{y}]{\beta^-,\gamma} {}^{210}\text{Bi} \xrightarrow[5.01\ \text{d}]{\beta^-} {}^{210}\text{Po} \xrightarrow[138.4\ \text{d}]{\alpha} {}^{206}\text{Pb (stable)}$$

- ^{210}Pb: $E_{\beta max}$= 16.6 keV (84%), 63.1 keV (16%)
  E_γ = 47 keV (4.1%)
- ^{210}Bi: $E_{\beta max}$= 1.2 MeV (100%)
- ^{210}Po: E_α= 5.3 MeV (100%)

Approach

Water sample

↓

Eichrom Pb resin column

↓

LSC Analysis

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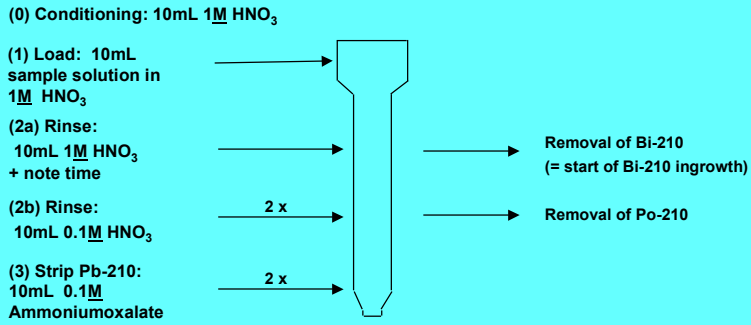
Sample Preparation

- 1.0 L / 2.0 L water samples
- acidified to pH 1.5
- 20 mg Fe-carrier added

Pre Column Separation

- stir and heat solution to 80°C for 1h
- precipitate Fe by adding ammonia solution 25%
- decant supernatant
- centrifuge and rinse $Fe(OH_3)$precipitate
- dissolve precipitate in 10mL HNO_3 1M
  (= load solution for Eichrom Pb resin column)

Pb Resin Column Separation



Counting Sample Preparation (1)

- collect eluate in teflon beaker
- evaporate to dryness
- add 5 mL HNO_3 65%
- evaporate to dryness (2 x)
- dissolve in 5mL HNO_3 0.1M (H-3 free)

Counting Sample Preparation (2)

- transfer to 20mL PE counting vial
- rinse beaker with 5mL HNO_3 0.1M (H-3 free)
- add rinsing solution to counting vial
- store sample for ≥ 5 days (Bi-210 ingrowth)
- add 10mL LSC cocktail (Zinsser QuickSafe 400™)

Recovery Rate

- during Bi-210 ingrowth (64-105 h after separation)
  92.0 ± 1.7 %
- in equilibrium (980-1020 h after separation)
  96.6 ± 2.9 %

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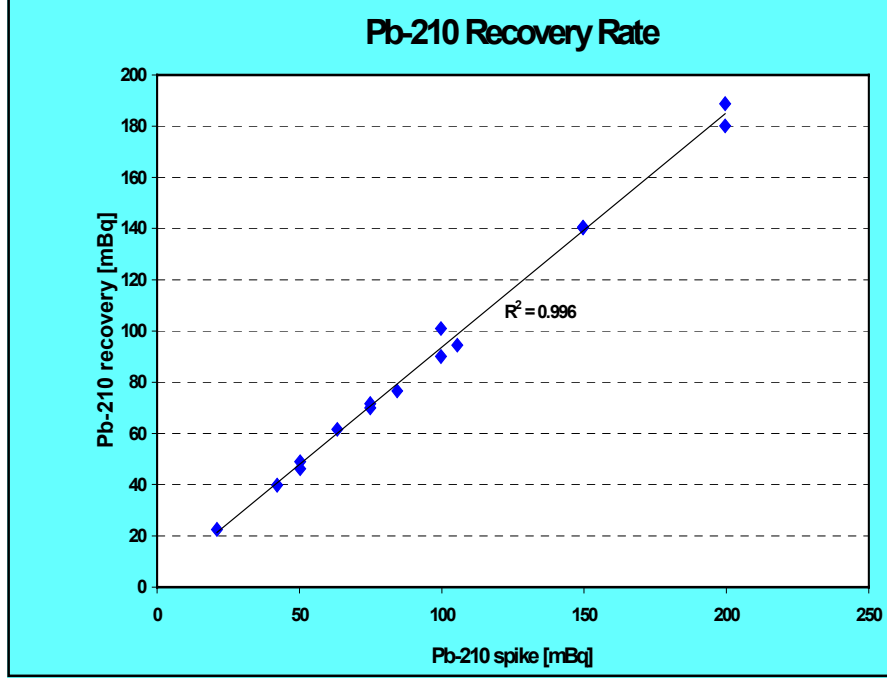

LSC Analysis

- Instrument: WALLAC 1220 Quantulus™
- counting time: 300 min
- α/β discrimination: PSA Level = 95
- counting efficiency: 100%
- Spectrum analysis window: CH# 30 - 770
- LLD (CL=95%) : **2 mBq/L Pb-210**

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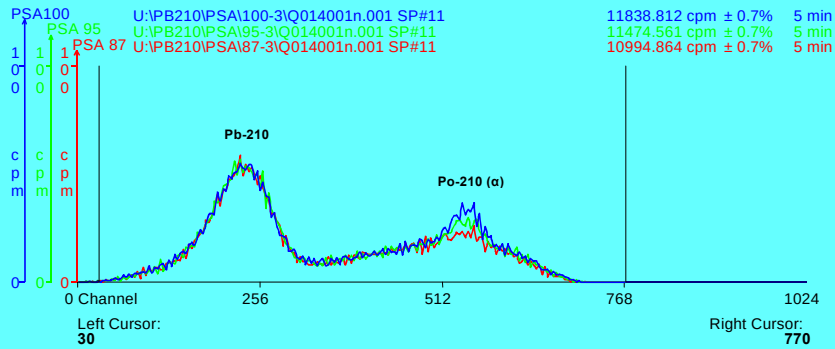
PSA-Level Test (QS400)

sample volume: **0.01 L**
recovery rate: **1**
standard act.: **100** Bq ± **5** % (1σ)

reference date: **31.08.2001 10:00**
measurement date: **29.05.2002 13:55**
decay correction: **---**

Efficiency: **100 %**

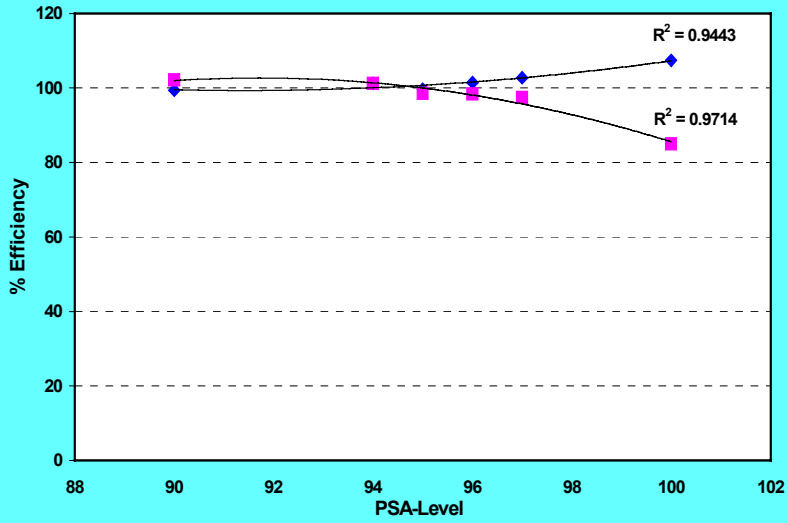


PSA-Level 87-95-100

Pulse-Shape-Analysis Level

Wallac 1220 Quantulus, QuickSafe 400®10mL+10mL HNO_3 0.1M

◆ % Efficiency Beta Pb-210+Bi-210 ■ % Efficiency Alpha Po-210



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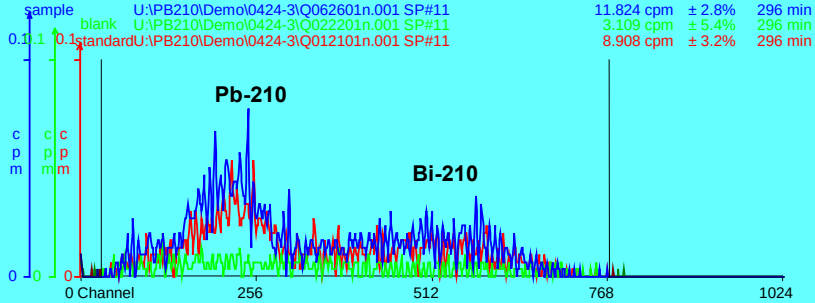
Pb-210 in Water

sample volume: **0.01 L**
recovery rate: **1**
standard act.: **75** mBq ±**5** % (1 σ)

reference date: **11.02.2002 12:45**
measurement date: **26.04.2002 03:34**
decay correction: **---**

Efficiency: **100**%

Sample: 73.9 ± 3.1 mBq/L (1.65σ)



Left Cursor: **30**

Right Cursor: **770**

groundwater (artesian well)

LSC Analysis: Time and Cost

- **Total time:** **2 weeks**
  - **sample preparation:** **5 days**
  - **samples/preparation:** **4**
  - **Bi-210 ingrowth:** **7 days**
  - **counting time:** **2 days (4 samples)**
- **Total Cost :** **EUR 182.- /sample**

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Summary

- ✓ <2 mBq/L ^{210}Pb detection limits achievable
- ✓ ^{210}Pb recovery rates >90%
- ✓ reasonable time and cost effort
- ✓ (future) EU legistic requirements met