Determination of $^{210}$Pb and $^{210}$Po in Water Samples

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Abstract

A fast procedure for determination of $^{210}$Po and $^{210}$Pb in natural water samples, based on spontaneous deposition and extractive chromatography is presented. The activities were determined by low-level alpha spectrometry for the polonium and liquid scintillation spectrometry for the lead. The minimal detectable activities estimated were for $^{201}$Po 0.2 mBq/L and 2.5 mBq/L for $^{210}$Pb. The procedure is therefore sufficiently sensitive for investigation of polonium and lead activities concentrations in aquifers at environmental levels.

1. INTRODUCTION

Lead-210 and Polonium-210 are naturally occurring members of the Uranium-238 decay series. They could be found in various environmental samples, such as groundwater, fish shellfish and various foodstuffs [1, 2]. Due to the importance of the water for the human life, its quality has to be controlled. For the determination of $^{210}$Po and $^{210}$Pb activities in drinking water different radiochemical techniques, ion exchange, co-precipitation, electrochemical separation and solvent extraction [3 – 6]. These methods are somewhat complicated, elaborate, time consuming and producing toxic organic wastes. In order to develop simple, fast and sensitive method for sequential determination of $^{210}$Po and $^{210}$Pb in water samples, we examined the combination of spontaneous deposition; co-precipitation and extraction chromatography.

2. EXPERIMENTAL

The flow chart of the radiochemical separation of $^{210}$Po and $^{210}$Pb in water samples is presented in Figure 1. All the reagents used, unless otherwise stated, were p. a. grade and the water was deionized.
2.1. Description of the Analytical Procedure

2.1.1. Spontaneous deposition of $^{210}$Po

Spontaneous deposition on metals as silver, copper or nickel is a simple and efficient method to concentrate polonium from acidic media. The deposition from HCl is more smoothly and quantitative compared to HNO$_3$ or H$_2$SO$_4$ [7].

Two liters of water were acidified in adding 8 mL of concentrated HCl per liter. The sample was transferred into a beaker and heated to 55 °C. After addition of the yield tracer $^{208}$Po (0.02 Bq), the sample was stirred for one hour in order to obtain a good isotopic exchange. For the spontaneous deposition of polonium polished copper disks were used. The deposition was carried on at 55°C under continuous stirring for 6 hours. The disks were then removed, washed with water, left to dry at room temperature before being submitted to alpha spectrometry.

2.1.2. Separation of $^{210}$Pb

The water sample after polonium electrochemical deposition was brought to near boiling and Fe(II), and Pb(II) carriers were added. Under the stirring process the iron hydroxide was precipitated by addition of concentrated ammonia. The supernate then was discarded and precipitate washed with water. Finally the precipitation was dissolved in 1 M HNO$_3$.

Further the lead was separated by extraction chromatography with Sr Spec - Eichrom Inc. resin. The columns were conditioned with 1 M HNO$_3$. The sample was loaded on the column followed by washes with 1 M HNO$_3$ and 0.1 M HNO$_3$. The second wash removed any Sr(II) and Po(IV) that could interfere with liquid scintillation counting (LSC). Finally the lead was eluted with 40 mL water. Two milliliters of concentrated HNO$_3$ were added to the lead eluate and the solution was gently evaporated. The residue was dissolved in 0.05 M HNO$_3$. An aliquot was taken for determination of stable lead by means of electro-thermal atomic
absorption spectrometry (ET-AAS). The rest of the solution was transferred to 20 mL scintillation vial.

3. RESULTS AND DISCUSSION

Alpha spectrometry of $^{210}$Po was performed on Canberra 7401 alpha spectrometers with 900 mm$^2$ active surface PIPS detectors. A typical spectrum of $^{210}$Po and yield tracer $^{208}$Po is presented in the Figure 2. The energy resolution is better that 25 keV. This fact points out that the quality of the prepared by spontaneous deposition sources for alpha-spectrometry was high.

![Figure 2]({https://example.com/image.png})

**Figure 2.** a) Spectrum of $^{210}$Po (5.30 MeV) and the yield tracer $^{208}$Po (5.14 MeV).

b) LSC spectra of $^{210}$Pb standard solution, $^{210}$Pb in sample and the background.

The Lead-210 activity was determined by liquid scintillation spectrometry (Gardian Wallac Oy). The solution containing $^{210}$Pb was directly mixed with the scintillation cocktail OptiPhase HiSafe 3. The beta spectra of the standard solution of $^{210}$Pb, sample and background are presented in the Figure 3. In order to estimate the chemical yield of the separation procedure of $^{210}$Pb, stable lead was used. Its content in the final analytical fraction was determined by means of ET-AAS. For the environmental samples, corrections for the efficiency and quenching are necessary to be performed. A number of lead-210 sources were prepared and quenched with different stable lead concentrations. In the Figure 3 a) the variation of the
efficiency is presented and in Figure 3 b) the quenching influence of the stable lead carrier is presented.

![Graph a) Efficiency of the Pb-210 registration by Liquid Scintillation Counting.](image1)

![Graph b) Quench dependency of the Pb-210 registration. Efficiency vs. stable lead carrier content.](image2)

**Figure 3.** a) Efficiency of the Pb-210 registration by Liquid Scintillation Counting.  
b) Quench dependency of the Pb-210 registration. Efficiency vs. stable lead carrier content.

### 3.1. Minimal detectable activity

The minimal detectable activity (MDA) of $^{210}$Po was defined by following equation [8]:

$$L_d = \frac{2.71 + 4.66\sqrt{B\cdot t}}{\eta \cdot Y \cdot V \cdot t}, \quad Bq/m^3$$

(1)

- $B$ – background counts, cps
- $\eta$ – detector efficiency
- $Y$ – chemical yield
- $V$ – sample volume, $m^3$
- $t$ – counting time, sec

The results obtained for the chemical yield and MDA for sample size of 2 liters are presented in Table 1.
Table 1. Radiochemical yield and MDA for $^{210}$Po and $^{210}$Pb.

<table>
<thead>
<tr>
<th>Radionuclide</th>
<th>Yield, %</th>
<th>Limit of detection, Bq/m$^3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{210}$Po</td>
<td>88.3 ± 6.0</td>
<td>0.2</td>
</tr>
<tr>
<td>$^{210}$Pb</td>
<td>85.5 ± 8.0</td>
<td>2.5</td>
</tr>
</tbody>
</table>

4. CONCLUSIONS

The method developed was tested by standard addition of different $^{210}$Po and $^{210}$Pb activities to real water samples. The results showed good correspondence between values added and that measured. The obtained minimal detectable activities of 0.2 Bq/m$^3$ for $^{210}$Po and 2.5 Bq/m$^3$ for $^{210}$Pb are sufficiently low. The method proved to be simple for implementation, fast with reproducible yields and applicable for low-level radioactivity control of natural waters.

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REFERENCES


