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Determination of ^{210}Pb and ^{210}Po in water using the extractive scintillation cocktail PorexTM

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HIGHLIGHTS

- With this method Pb-210 and Po-210 can be determined with one measurement.
- Matrix interferences causing extraction problems are eliminated by washing with 1% HNO_3 .
- The chemical recovery of the lead can be used to calculate the Po-210 activity concentration.
- Uranium which can be extracted by PorexTM is removed washing the precipitate with 1% HNO_3 .

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ABSTRACT

Method validation was performed to achieve the accreditation for our determination method of ^{210}Pb and ^{210}Po in water. A $\text{Pb}(\text{NO}_3)_2$ carrier is added to the sample and lead is precipitated with $\text{Na}_2\text{SxH}_2\text{O}$. ^{210}Po is co-precipitated and the extractive scintillation cocktail PorexTM is used to determine ^{210}Po and ^{210}Pb . Uranium is also extracted by PorexTM. It can be removed by washing the precipitate with 1% HNO_3 . The ingrowth of ^{210}Pb from ^{222}Rn during transportation time must be calculated. It has to be subtracted from the original ^{210}Pb in the sample and taken into account for the calculation of the lower limit of detection.

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1. Introduction

The EU-Recommendation 2001/928/Euratom (Commission Recommendation 2001/928/Euratom, 2001) proposes reference values for ^{210}Pb and ^{210}Po in drinking water. The Codex Alimentarius Austriacus section B1 for drinking water (Österreichisches Lebensmittelbuch, 2007) states that, at activity concentrations of ^{210}Pb and ^{210}Po above 2 Bq/l and 1 Bq/L respectively, remedial actions should be recommended.

According to the Austrian Drinking water regulation ^{210}Pb and ^{210}Po do not have to be analysed in drinking water. However, the committed effective dose per unit intake via ingestion for adults (h) (Sv/Bq) of ^{210}Pb has the same value as ^{228}Ra (6.9×10^{-7} Sv/Bq) (Council Directive 96/29/Euratom, 1996). The committed effective dose per unit intake via ingestion of ^{210}Po is even higher with 1.2×10^{-6} Sv/Bq.

Therefore determinations of ^{210}Pb and ^{210}Po in water samples of different origins have been conducted in our lab for several

years. The results have already been published (Landstetter and Katzlberger, 2010). During the application of the method for various matrices it has been adapted because of problems occurring at the different sample types. Especially ground water samples caused problems during the extraction step.

To overcome these problems we looked at different methods which are used to determine ^{210}Pb and ^{210}Po . But even though the methods are called simultaneous determinations – because ^{210}Pb and ^{210}Po are determined from one sample with the same sample preparation – two measurement methods are used. ^{210}Pb is often analyzed by liquid scintillation counting (LSC) and ^{210}Po is measured by alpha spectrometry. (Vajda et al., 1997)

The determination of ^{210}Po with alpha spectrometry is an excellent method and hence, often used for different matrices (Sinojmeri et al., 2012; Zhichao and Zhongyu, 2009). However, using the alpha-spectrometry for the determination of ^{210}Po implies an additional measurement of ^{210}Pb with LSC. In monitoring studies up to 300 samples have to be analyzed whose cost plays a very important part.

Regarding the pros and the cons of the PorexTM extraction and measurement with LSC, we decided to use this method for all our water samples instead of switching to the alpha spectrometry.

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The pros of the Porex™ method are that a simultaneous determination of ^{210}Pb and ^{210}Po from one sample with just one measurement is possible. This is time saving and cost conservative.

The cons of the Porex™ method are that on contrary to the alpha-spectrometry just the determination of the chemical recovery for ^{210}Pb is possible before the extraction step. The efficiency of the extraction step cannot be controlled. Problems can occur during the extraction step because of matrix interferences. Therefore the solution for the extraction should be as matrix free as possible. The adoption and changes of the Porex™ method described previously were made to achieve this goal. The main improvement was to include an additional washing step with 1% HNO_3 after the precipitation of ^{210}Pb and ^{210}Po . This can eliminate most of the problems because uranium is removed from the precipitate with the washing solution. With an energy of 4.8 MeV, 4.4 MeV and 4.2 MeV from ^{234}U , ^{235}U and ^{238}U respectively, this nuclides especially ^{234}U overlap the ^{210}Po peak at 5.3 MeV in the LSC spectra. All adoptions of the method are described more accurately in the experimental section.

However, the lack of a determination of the chemical recovery in the extractive scintillation cocktail Porex™ after the extraction still exists.

Nevertheless, method validation of the Porex™ extraction method was performed using ground water, drinking water and raw water. The repeatability limit and the accuracy were determined and the linearity was tested using spiked ground water samples. Additionally the method was used during an interlaboratory comparison of natural radionuclides in spiked drinking water.

In order to test the method with a real sample (not just with spiked samples) under realistic conditions, we were looking for a reference material containing ground water, ^{210}Pb , ^{210}Po and ^{222}Rn . Unfortunately we could not find any. Therefore we used raw water with high radon activity concentration to test the method with a real sample. Because of the natural decay chain ^{210}Pb is growing in from ^{222}Rn and ^{210}Po is growing in from ^{210}Pb after sampling. Subsamples of this real sample were used to test the chemical recovery and comparing the results of the Porex™ extraction method with the results obtained by alpha spectrometry measurements.

2. Experimental

The method of the ^{210}Pb and ^{210}Po determination is already published (Landstetter and Katzberger, 2005).

1 ml of $\text{Pb}(\text{NO}_3)_2$ solution (1.6 mg Pb^{2+}/ml) was added to 1 L of water sample and the sample was evaporated to near dryness. The residue was repeatedly dissolved in concentrated HCl and

evaporated. The residue is dissolved in 15 ml 1 M HCl. The precipitation was done at pH 1.5 with 1 M Na_2S . After centrifugation and filtration through a blue ribbon filter the filtrate was placed aside for radium determination and the precipitate was used for the ^{210}Pb and ^{210}Po determination. Therefore it was dissolved in concentrated HNO_3 and fumed off with concentrated HNO_3 and HCl. The sample was evaporated to near dryness, dissolved in 2 ml 1 M HCl and 12 ml 7.5 M H_3PO_4 and stored in the refrigerator until ^{210}Bi is in equilibrium with ^{210}Pb . After 2 weeks ^{210}Bi and ^{210}Po were selectively extracted with Porex™ and measured by LSC.

This method was used during the first survey of drinking water samples from Upper Austria in 2005. At the beginning 1 L of water was evaporated to near dryness and the residue was repeatedly dissolved in concentrated HCl and evaporated. This step had to be changed because in water samples with high sulfate concentrations the radium precipitates during this step and cannot be analyzed in the filtrate anymore. We usually measure radium in our water samples in addition to ^{210}Pb and ^{210}Po . Now the 1 L of water is just evaporated to 60 ml and the conversion into the chloride form by repeatedly dissolving in concentrated HCl and evaporating had to be canceled.

For the sulfide precipitation we use NaOH to adjust the pH to 1.5. After adding the 1 M Na_2S solution the pH reaches 10. This is necessary if thorium is present in the water. Within monitoring programs with lots of samples the acidified water can be stored for several weeks. During this time an ingrowth of ^{234}Th from ^{238}U occurs. The pH of 10 is necessary to precipitate thorium and remove it from the filtrate for the radium determination.

The above described method was used in our lab for several samples until we conducted a groundwater study in Austria. During these determinations a peak appeared below the ^{210}Po peak in the LSC alpha spectrum. Within this study we also performed ^{238}U analyses and could correlate this peak with high uranium concentrations in the sample. With an energy of 4.8 MeV, 4.4 MeV and 4.2 MeV from ^{234}U , ^{235}U and ^{238}U respectively, these nuclides especially ^{234}U overlap the ^{210}Po peak at 5.3 MeV in the LSC spectra. The uranium gets co-precipitated as hydroxide when the pH reaches 10 after the addition of Na_2S . Therefore uranium is in the extracted solution. This precipitation is not quantitative so we could not establish a quantitative correlation between the measured uranium with the LSC signal and the measured uranium concentration with the ICP-MS. Beside this it is not known how effective the extraction of uranium with Porex™ is. Fig. 1 shows the spectra with extracted uranium and polonium.

The extraction of uranium just occurred in the groundwater samples as mentioned before (Jokelainen et al., 2010). Additionally

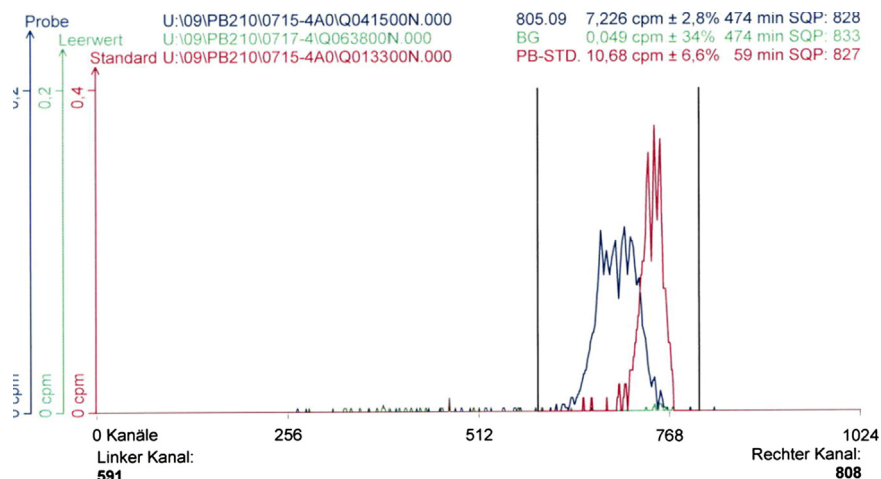


Fig. 1. Spectra of ^{210}Po of the standard solution (red) and the sample (blue) with a uranium concentration of 15 $\mu\text{g}/\text{L}$. It can be seen that uranium is extracted in addition to polonium.

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