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ORIGINAL PAPER

Optimization of the radiochemical procedure of ^{210}Po determination in small amounts of sediment samples

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Abstract

The radioactivity of alpha emitter radioisotope ^{210}Po can be used to determine the amount of ^{210}Pb radionuclide. This radioisotope is commonly used in the dating of marine as well as freshwater lake sediments. In this type of research, the collected samples are small, often 1.5–3 g (wet sediments), especially if we work with sediments taken from mountain lakes. This work presents the problems found with ^{210}Po determinations in a sample weighting 0.1 or 0.2 g. The analysis is based on the decomposition of samples, the concentration of the analyte, preparing source via spontaneous deposition on a silver discs, and alpha measurements of this source. The aim of this work was to optimize every stage of the procedure. The main assumption of the research was to minimize the weight of samples, the amounts of reagents taken for radiochemical determinations, and to eliminate hydrofluoric acid during the analytical process. The study focused on the optimization of the decomposition of sediments (microwave digestion with concentrated HCl and HNO_3 acids), the adsorption of polonium on the container wall in time, and the deposition conditions. The results show that there is a possibility to determine ^{210}Po in small amounts (0.1–0.2 g) of sediments without using hydrofluoric acid with the yield of radiochemical procedure above 90%. The procedure was checked by reference material, and good accuracy and precision were achieved.

Keywords Alpha spectrometry · Microwave digestion · Mountainous sediments · ^{210}Pb dating · Polonium

Introduction

Among many elements that are part of the earth's crust, radionuclides also exist. Due to their source, natural and artificial radionuclides are distinguished. A number of natural and artificial radionuclides are used as indicators for studying geochemical and biological processes in the natural environment (Isaksson and Raaf 2016).

Polonium ^{210}Po is a radioactive isotope that was discovered by Maria Skłodowska-Curie in July 1898. Natural polonium is a decay product in the uranium radium series. Polonium is an alpha emitter and decays directly to its stable daughter isotope, ^{206}Pb. Although ^{210}Po is the longest-lived radioisotope of polonium, in nature, its half-life is only 138.4 days. Other naturally occurring radioisotopes of polonium have half-lives between just 0.3 µs and 3 min. Some radioisotopes can be made through the alpha, proton, or deuteron bombardment of lead and bismuth in a cyclotron. Two long-lived polonium artificial radioisotopes can be distinguished. These are ^{208}Po and ^{209}Po. They are used as tracers in the determination of chemical yields in polonium analyses. ^{210}Po is always present in the environment, because it is maintained by its parent nuclide ^{210}Pb, which decays by beta emission through ^{210}Bi to ^{210}Po (Lehto and Hou 2011).

Due to the fact that it is a member of the ^{238}U series, it is widespread. So it can be determined in every part of the environment, including sediments. They are a very important component of the water ecosystem. On the one hand, they interact with water and, in consequence, they influence water quality. But on the other hand, sediments can be treated as a buffer that can reduce unfavourable changes and stabilize the system. Sediments consist of minerals and organic compounds, and they usually have good sorption properties and tend to accumulate toxins. Sediments reflect the relative contamination of aquatic environments (Karbassi and Amirnezhad 2004; Szarłowicz et al. 2011; Koiter

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et al. 2013). There are few reasons why polonium should be determined in sediments. The most common reason for its determination in sediments is ascribed to ^{210}Pb determination. ^{210}Pb could be determined via its daughter radioisotope (polonium) using alpha spectrometry. The deposition of ^{210}Pb into sediments can be used to measure sedimentation rates and sediment age (^{210}Pb method) (Sanchez-Cabeza and Ruiz-Fernandez 2012; Hamerlík et al. 2016; Szarlowicz et al. 2018). In addition, ^{210}Po, being an alpha emitter with high radioactivity, makes a major contribution to the natural radiation received by humans, because of its accumulation from food especially from fish or shellfish (Skwarzec 1988, 1997; Ugur et al. 2002; Lazorenko et al. 2002).

In the available scientific literature, there are research articles and reviews articles dedicated to the determination of polonium in environmental samples (Planinsek et al. 2013; Sethy et al. 2015; Fonollosa et al. 2015). There are several works that deal with the analytical problems of polonium determination (Smith and Hamilton 1984; El-Daoushy et al. 1991; Vesterbacka and Ikaheimonen 2005; Henricsson et al. 2011), but there are a few works that represent the determination in which quite small amounts (~ 1 to 2 g) of the sediment are used (Sanchez-Cabeza et al. 1998; Ebaid and Khater 2006; Vesterbacka et al. 2009).

There are many factors that can influence on polonium determination, and that is why there is the necessity to improve this method. The aim of this work was to present the environmental friendly procedure of polonium determination in small amounts (0.1–0.2 g) of sediments. In addition, some factors that influence polonium determination and present the quality and reliability of analytical methods are indicated.

Materials and methods

Generally, the radiochemical procedure of polonium determination includes the following stages: samples' decomposition, preparing alpha sources, and alpha measurements. It was decomposed in 0.1 or 0.2 g samples of sediments using a microwave oven. Then, the samples were centrifuged and evaporated with 2 mol dm^{-1} HCl, polonium was spontaneous deposited on a silver disc (Flynn 1968) in the presence of hydroxylamine hydrochloride or ascorbic acid within 3 h at temperatures of 85–88 °C. Such sources were measured in an alpha spectrometer. The alpha source preparation by autodeposition of ^{210}Po on a silver disc is a standard method for ^{210}Po determination. But there is no general method for sample preparation and radiochemical procedure.

Taking into account all stages, improvements or optimizations were developed. The details are described below.

To perform the analysis, the reference materials from IAEA (International Atomic Energy Agency) and NIST (National Institute of Standards and Technology) were used. Different kinds of materials with wide range of composition were used. Because of the similar composition in some soil materials, they were also used for optimization purposes. The reference samples analysed were the following: IAEA-447 moss-soil from an abandoned red marble mine in "Gerecse Mountain"; IAEA-327—the podsolic soil; 4354—freshwater lake sediment; IAEA-326 black soil was obtained from the Kursk region; and, IAEA 444 a soil from China. Additionally, the sediments from different kinds of lakes were used. All sediments samples were collected from mountain lakes using a Limnos corer (10 cm diameter). The sediment core was divided into a one-centimetre-thick layer. In the laboratory, the first step of analysis involves drying sediments in room temperature, crushing, grinding, and sieving through a 0.2 mm mesh. These samples were used for the microwave digestion process. An alpha spectrometer (Alpha Analyst, Canberra, USA) with PIPS (passivated implanted planar silicon) semiconductor detector was used for polonium measurements. The basic parameters of the detector are presented in Table 1. The energy and efficiency calibration of the detector was made using Standard Reference Source 99981, source with a ^{208}Po and AMR-33 source. All spectra were analysed using Genie-2000 software. Measurement time was 3 days for all sources.

Storage of the polonium ^{208}Po

In every analysis, the tracer ^{208}Po was used in order to control radiochemical procedure. The effect of the adsorption of the tracer on the walls of container was checked. The ^{208}Po solution $Po(NO_3)_4$ was stored in polyethylene and polystyrene containers in 1 mol dm^{-3} HNO_3. The radioactivity of the tracer solution was measured at the beginning of storage and after 1, 3, 6, 12, and 36 months for the polyethylene container and 1, 3, and 6 for polystyrene vial.

Optimization of the microwave digestion procedure

The microwave digestion process was carried out in a Plazmatronika (Uniclever) and Anton Paar (Multiwave Pro) system. For digestion, the 0.1–0.2 g of sediments and different

Table 1 Basic parameters of alpha spectrometer

Model	A450
Active area	450 mm^2
Active diameter	23.9 mm
Bias	39.9 V
Alpha resolution for the line 5,11 MeV	20 keV
Pressure	19 µm Hg
Absolute efficiency at 5 mm spacing with a source diameter of 12 mm	34%

reagents were used. The following mixture of concentrated reagents were used: 6 ml HNO_3, 6 ml HNO_3 + 200 µl $HClO_4$, 2 ml HCl + 6 ml HNO_3, 2 ml HCl + 4 ml HNO_3, 5 ml HNO_3 + 3 ml HCl, and 2 ml H_2O_2 + 4 ml HNO_3, 5 ml HNO_3 + 1 ml HF. All chemical reagents used in this study were of analytical grade. First, the sediments differing in organic matter content were digested, and the levels of samples digestion were visually assessed. The efficiency of the digestion process was checked by two procedures with different conditions (pressure and microwave power, time were optimized). Then, the best procedure (including reagents, pressure, microwave power, time) was chosen and the reference materials were digested. After microwave digestion, the adsorption of ^{210}Po (or ^{208}Po) on digestion vessel was checked by using a source from a blank solution after digestion.

Yield of polonium deposition

Chemical yield of polonium deposition as a function of time deposition was checked. The reference material IAEA-RGU-1 was used in this test. A dozen depositions were made at different times (0.5, 1, 1.5, 2, 3, 6, and 7 h). The majority of depositions were made with mixing the solution; however, how no mixing of the solution affected the deposition process was also verified. The appropriate reducing reagent and its amount were also selected.

Results and discussion

Regarding storage time, the following results were obtained. The radioactivity of polonium measured after storage time in polyethylene container was in the same range with the initial radioactivity (479 Bq kg^{-1}±2.1%). According to Vesterbacka, glass bottles were better than plastic bottles. The author indicates a 3–4% decrease in polonium radioactivity after 5 months (Vesterbacka and Ikaheimonen 2005). In this research, there was no adsorption on polyethylene container. Moreover, in the 1 mol dm^{-3} HNO_3 solution, no decrease in time in polonium radioactivity (including radioactive decay) was observed. But, in polystyrene containers, clear adsorption of ^{208}Po during 6 months of storage was observed. The measured radioactivity was 20% lower than the initial radioactivity. Therefore, no long-term storage of this solution was done in this vessel. Such researches dedicated to polonium storage in 1 mol dm^{-3} HNO_3 in polyethylene vial were carried out by an independent laboratory and they confirmed the presented results.

For alpha analysis, samples have to be decomposed. Microwave sample digestion provides an efficient and clean sample preparation for multi-element analytical techniques as well as alpha spectrometry. Among presented research, authors have recently used microwave digestion for polonium analysis in sediment samples more often. As a medium for wet-digestion methods, mixtures of acids [different acids have been tested such as HNO_3, HNO_3:HCl:HF:H_2O (5:2:3:10), HNO_3:HF (1:1), mixture of HF, HNO_3, and H_3BO_3, in different proportions with varying pressure/temperature conditions and digestion times] were used (Wen et al. 1997; Sanchez-Cabeza et al. 1998; García and Kahn 2001; Ebaid and Khater 2006; Lubis 2006; Henricsson et al. 2011; Sert et al. 2012).

Other methods are focused on leaching polonium from the samples using a mixture of HNO_3:HCl:H_2O and H_2O_2, HF and HNO3 and with H_2O_2 and HNO_3 (Vrecek et al. 2004; Begy et al. 2015). Most of the presented analyses were done with a large amount of sediments, around 1–2 g and more. If the authors use the leaching method, the mass was up to 20 g. Such amounts are very hard to obtain from mountain lakes, especially if we collect the sediment core and sliced them into thin layers (~ 1 cm) such as for geochronology research. That is why this research was focused on the digestion of 0.1 g or 0.2 g of the samples in different mixtures with the elimination hydrofluoric acid (hazardous reagent). For the mixtures, the following facts can be observed: 5 ml HNO_3 + 1 ml HF—completely decomposed sample, 6 ml HNO_3, 6 ml HNO_3 + 200 µl $HClO_4$, 2 ml HCl + 6 ml HNO_3, 2 ml HCl + 4 ml HNO_3, 5 ml HNO_3 + 3 ml HCl and 2 ml H_2O_2 + 4 ml HNO_3—visually more or less decomposed samples, with the presence of silica.

There was no polonium adsorption on the wall of the digestion vessel. The silicates were centrifuged and the solution was evaporated with 30 ml of 2 mol dm^{-3} HCl under an infrared heater in Teflon evaporators.

The deposition of polonium was carried out on a silver disc (chemical purity Ag3N) with a diameter of 14 mm. The silver disc was placed in a holder and immerse in solution for different times. After 90 min, the yield of deposition is 90% as shown in Fig. 1. In these experiments, the solution was rotated during the deposition time. The rotational velocity was 1000 rpm. At a stationary condition, the efficiency of the deposition of polonium at 85–88 °C was around 90% after 180 min.

Some authors prefer the deposition time around 6 or 4 h (Vrecek et al. 2004; Sert et al. 2012), but this research indicates that any deposition times longer than 3 h negatively affected the quality of the measurement source. For instance, after 6 or 7 h, the alpha sources were thick, and they had a white coating and were characterized by the worse peak in resolution (Fig. 2).

It is well known that some interference, e.g., ions of Mn, Fe, Se, Cr, or Te, exists in environmental samples and disturbs polonium deposition. Due to the fact that iron is by mass the most common element on Earth, it is the most interfering element during the analysis of sediment samples.

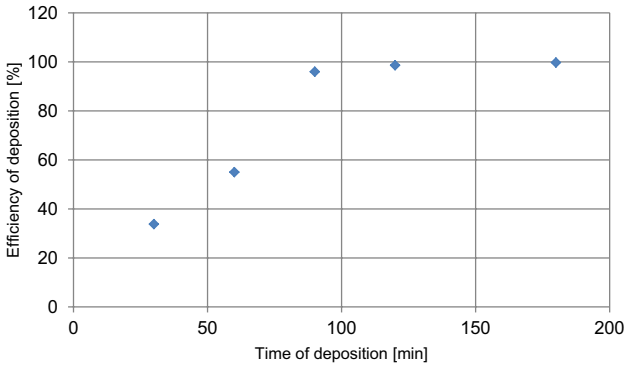

Fig. 1 Changes of efficiency of deposition in studied period of time

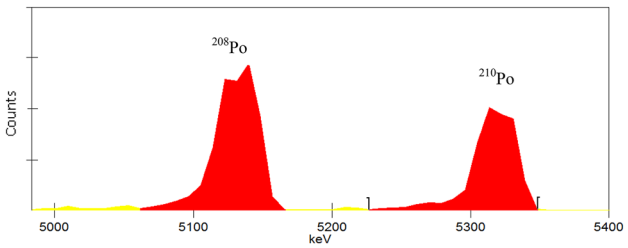

Fig. 2 Spectrum with worse peak resolution (45 keV)

If the concentration of iron is above 10,000 ppm, ascorbic acid is added as a reducing agent (1 or 2 drop of saturated solution). However, if the concentration of iron is below 10,000 ppm, hydroxylamine hydrochloride is added to reduce Fe^{3+}–Fe^{2+}, and sodium tricitrate is added as a masking agent to sufficiently protect against oxidation. In these conditions, very good sources with high peak resolution (20–23 keV) were obtained (Fig. 3).

Taking all laboratory work done in the field of the entire radiochemical analysis, it is stated that minor silica residues do not affect the value of polonium in the sediments. Several replicates were made for samples with different organic matter contents. For both digestion mixtures with HF and without them, the obtained radioactivity values were in the reference range. The obtained results were characterized by very good accuracy and repeatability. Therefore, the use of a harmful reagent was discontinued. The best procedure for sediments with major organic matter content was 2 ml HCl + 6 ml HNO_3, and, for more inorganic samples (presence of silica, organic matter < 20%), the mixture of 3 ml HCl + 5 ml HNO_3 was sufficient. The overall scheme of the radiochemical procedure of polonium determination in sediments is shown in Fig. 4.

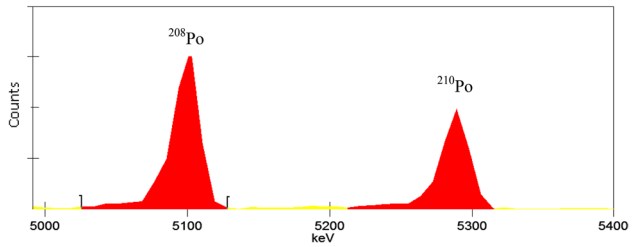

Fig. 3 Spectrum with high peak resolution (20 keV)

To check these procedures, tests using the reference material proceeded. The reference standard material was used in which polonium is in equilibrium with its mother radionuclide ^{210}Pb. All obtained data were in the range presented in the reference sheet. The accuracy (relative error %) and precision (% RSD relative standard deviation) are presented in Table 2. Moreover, MDA (minimum detectable activity) was determined using the Curie definition (1968) MDA that depends on the mass of the samples. For the samples around 0.1 g, this was MDA ~ 1.7 Bq kg^{-1}; $m = 0.2$ g MDA ~ 0.89 Bq kg^{-1}.

Using Eq. 1, the radioactivity of polonium (A_{Po}) was calculated.

$$A_{Po} = \frac{(CPS_s - CPS_{bl}) \cdot 1000}{\varepsilon \cdot Y \cdot m} \,[\mathrm{Bq\,kg^{-1}}] \quad (1)$$

where CPS_s counts per second for sample, CPS_{bl} counts per second for blank, ε efficiency of the detector, Y yield of radiochemical procedure, and m mass of the sediments [g].

Uncertainties were evaluated by use of the law of propagating uncertainty. The uncertainty of the radioactivity measurements was around 5–10%.

As it was shown above, determining ^{210}Po levels requires the application of the radiochemical procedure. Based on results, the advantages of the suggested solution as compared to the existing methods can be discussed. Conventional open wet sample digestion (large volume of acids, long period) was replaced by microwave digestion. All experimental activities bring less acid consumption (Fig. 4), no exposure of analysts to acid fumes, the prevention of the loss of volatile elements, no risk of contamination from external sources, hydrofluoric acid elimination and greater efficiency. The proposed improvements allow including this radiochemical procedure to so-called green chemistry method. The use of a tracer with a shorter half-life also contributes to reducing the production of radioactive materials.

Microwave digestion 0.1 g or 0.2 g sediment with ^{208}Po	
organic matter>20% 2ml HCl+6ml HNO_3	Organic matter<20% 3ml HCl+5ml HNO_3
Centrifugation **Evaporation with 30 ml 2mol·dm^{-3} HCl** **^{210}Po deposition**	
Fe<10000ppm 2ml 0.5 mol·dm^{-3} HCl 3ml 40% hydroxylamine hydrochloride 2ml sodium tri-citrate 2x2 ml 0.5 mol·dm^{-3} HCl	Fe>10000ppm 10 ml 0.5 mol·dm^{-3} HCl 1 or 2 drops of saturated solution of ascorbic acid
Alpha measurement ~259200 sec	

Fig. 4 Steps in radiochemical procedure of ^{210}Po determination

Table 2 Some metrological parameters for ^{210}Po determinations in reference materials and sediment samples

Certified material	Certified radioactivity (Bq kg^{-1})	Experimental radioactivity (Bq kg^{-1})	Accuracy (%)	Precision (%)
IAEA-447	424 ± 20	418 ± 21	1.4	1.0
NBS4354	120	118.2 ± 8.0	1.5	1.7
IAEA-326	53.3 ± 4.5	50.7 ± 3.6	5.1	2.5
IAEA-327	58.8 ± 4.9	61.4 ± 4.0	4.2	2.3
IAEA-444	48.0 ± 1.6	47.1 ± 2.8	1.9	2.5
Sediment with known radioactivity	784 ± 42	771 ± 36	1.7	2.0

Conclusion

The following can be concluded:

1. 0.1 g or 0.2 g sediments with the mixture of concentrated HCl and HNO_3 and microwave digestion is sufficient for polonium determination and, in such conditions, there were no adsorption of polonium on container wall.
2. The presence of silica does not influence the polonium determination, so the HF during digestion can be eliminated.
3. The best deposition time for alpha polonium deposition is 3 h, and, in this process, thin, uniform alpha sources with very good peak resolution (20–23 keV) are produced.
4. The proposed procedure gives results with the yield of radiochemical analysis between 90 and 100%.

To summarize, sediments are a valuable source of information about the state of the aquatic ecosystem. Determinations of radionuclides in sediments are important from the point of view of the level of exposure as well as their use to estimate the age of individual layers. The analysis of radioisotope levels seems to be a much easier matter, especially since the amount of sediments taken for analysis can be incomparably greater than in geochronological studies. Quite often, the dating of sediments is made from hard to reach reservoirs, where the sampling process is already a challenge. Additional restrictions in obtaining more samples are related to the size of the sampler. It should be emphasized that the reservoirs are often located in protected areas, which also imposes a restriction on the amount of taken sediment. The developed procedure is environmentally friendly. On the one hand, HF acid was eliminated and the amount of reagents used was reduced, which also allowed reducing the cost of the analysis. It can be successfully used in geochronological research (^{210}Pb method) to determine ^{210}Pb using polonium. Of the available methods for the determination of ^{210}Pb, this is the most sensitive measurement method.

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