

## DIFFICULTIES WITH $^{228}\text{Ra}$ DETERMINATION FROM THE LOW-ENERGY BETA SPECTRUM COMPONENT IN WATER SAMPLES VIA LSC

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**ABSTRACT.** This project results from studies of radium isotopes in natural water samples carried out in the Nuclear Physics and Its Application Department at the University of Silesia using the liquid scintillation (LS) technique. This paper investigates the  $^{228}\text{Ra}$  activity calculation. Theoretically, the activity of the  $^{228}\text{Ra}$  isotope can be determined both from the low-energy beta spectrum component coming directly from  $^{228}\text{Ra}$  and the high-energy beta component from the  $^{228}\text{Ac}$  counts. Until now, the activity of  $^{228}\text{Ra}$  in water samples was calculated using the part of the spectrum related to  $^{228}\text{Ac}$ , which is in radioactive equilibrium with  $^{228}\text{Ra}$ . Samples were measured for ~600 hr to minimize the contribution of activity due to  $^{224}\text{Ra}$ . Since the half-life of  $^{228}\text{Ra}$  is 5.7 yr, the activity of this isotope is constant only for a short period of time. Taking this into account, we decided to determine the activity of this isotope from its low-energy beta spectrum component. Several samples were prepared using standard radium isotopes obtained from the International Atomic Energy Agency (IAEA). The chemical procedure for determining radium isotopes by liquid scintillation counting (LSC) was based on the Polish Norm PN-89 Z-70072 (1989). The  $^{228}\text{Ra}$  activity can be determined from the first few measurements of the low-energy beta part of the spectrum, when accounting for the efficiency of the chemical procedure and detection, as well as the sample volume. We discuss analytical difficulties related to this method.

### INTRODUCTION

The long-lived radium isotopes  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  are responsible for the presence of radioactivity in natural waters.  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  have a half-life equal to 1620 and 5.75 yr, respectively. These isotopes, during their disintegration, create a series of  $\alpha$ - and  $\beta$ -radioactive daughters. At the Department of Nuclear Physics and Its Application at the University of Silesia, radium isotopes in mineral water are measured using a WinSpectral 1414  $\alpha/\beta$  liquid scintillation (LS) counter (Wallac).

Radium isotopes in mineral water can be determined using different radiochemical methods. One common procedure for chemical treatment is based on the coprecipitation of radium with barium into  $\text{Ba}(\text{Ra})\text{SO}_4$ . The precipitate with purified radium is transferred into a scintillation vial and mixed with water and a gelling scintillator. This method allows one to extract radium and remove other radioisotopes from the mixture of radioactive substances found in the water samples. The chemical procedure is based on the Polish Norm PN-89 Z-70072 (1989).

The aim of this work is to find the optimum conditions for determining the  $\beta$ -radioactive isotope  $^{228}\text{Ra}$  in water samples directly via the low-energy  $\beta$ -spectrum component ( $E_{\beta\text{max}} = 38.9$  keV) (Firestone et al. 1996). Measurement via liquid scintillation counting (LSC) should be performed as soon as possible following the sample preparation. The most common procedure for determining radium isotope concentration in water samples requires both  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  isotopes, a measurement that lasts about a month (Chau et al. 1997; Hetman et al. 1998; Kozłowska et al. 1999). The  $^{226}\text{Ra}$  content is calculated from the  $\alpha$  component of the spectrum.  $^{228}\text{Ra}$  activity is determined from the high-energy  $\beta$  component originating from its daughter  $^{228}\text{Ac}$  after being in radioactive equilibrium with  $^{228}\text{Ra}$  after 40 hr. This method is accurate and precise since the measurement of the  $\alpha$  particles and high-energy  $\beta$  particles with LSC is assumed to be 100% efficient. The theoretical calculations are based on Bateman's (1910) equations for each radium isotope series. The disadvantage of this method is that a long time is needed to obtain the result. Thus, it was of interest to check whether it was possible to obtain the  $^{228}\text{Ra}$  activity concentration value from the first measurement of the low-

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energy  $\beta$  spectrum component ( $E_{\beta\text{max}} = 38.9$  keV) without waiting for radioactive equilibrium. Very few publications exist concerning such attempts (Chafupnik and Lebecka 1993; Wallner 2002).

### **$^{228}\text{Ra}$ and $^{224}\text{Ra}$**

Based on the Bateman (1910) equations, one can obtain the relative activities of given radioisotopes from each radium series. From the point when  $t = 0$ , there is a very insignificant decrease in the relative activity of  $^{228}\text{Ra}$  connected with its radioactive disintegration. However, it is also possible to observe a rapid growth of the relative activity of  $^{228}\text{Ac}$ , which after 40 hr achieves a relative activity equal to  $A(t)/A_0 = 0.989$ . Therefore, this radionuclide is often used to determine  $^{228}\text{Ra}$  activity concentration in water samples (Chau et al. 1997; Hetman et al. 1998; Kozłowska et al. 1999b).

The  $\alpha$ -radioactive isotope  $^{224}\text{Ra}$  is a daughter of  $^{228}\text{Ra}$ , present in the radioactive chain. Since  $^{228}\text{Ra}$  can be present in the investigated water samples, one should take this isotope under consideration and trace its possible effect on  $^{228}\text{Ra}$  determination.

From the point when  $t = 0$ , the relative activity of  $^{224}\text{Ra}$  decreases. For the daughter isotopes  $^{220}\text{Rn}$  and  $^{216}\text{Po}$  emitting  $\alpha$  particles, there is a rapid growth from the first hour of the relative activity until the activity reaches  $A(t)/A_0 = 0.99$ , and then a rapid decrease. In case of the  $\beta$ -radioactive  $^{212}\text{Pb}$  isotope, one can observe a fast growth of relative activity, achieving its maximum  $A(t)/A_0 = 0.75$  in the 37th hr; after this point a decrease in relative activity occurs. A similar process of changes in the relative activity is observed for the  $\beta$ -radioactive isotope  $^{208}\text{Tl}$ ;  $\alpha$ -radioactive isotope  $^{212}\text{Po}$ ; and  $^{212}\text{Bi}$ , which emits both  $\alpha$  and  $\beta$  particles.

### **EFFICIENCY MEASUREMENT OF THE LOW-ENERGY $^{228}\text{Ra}$ $\beta$ COMPONENT**

$^{228}\text{Ra}$  decays in time with the emission of  $\beta$  particles (maximum energy of 38.9 keV), and  $^{228}\text{Ra}$  activity is fairly constant. Hence, the low-energy  $\beta$  component can be used to determine the content of this isotope in water samples. Regarding the very low energy of the emitted  $\beta$  particles and the large contribution of quenching effects at this energy region, one must determine the measurement efficiency for this part of the  $\beta$  spectrum. The efficiency of the low-energy  $\beta$  component of the spectrum was determined using the standard  $^{228}\text{Ra}$  isotope (with activity 1.06 Bq/g) obtained from the International Atomic Energy Agency (IAEA) in Vienna. A set of 4 standard samples was prepared with  $^{228}\text{Ra}$  activities of 3, 5, 8, 10 Bq. Figure 1 shows the  $\alpha$  and  $\beta$  spectra for Sample 4 eleven hr after sample preparation.

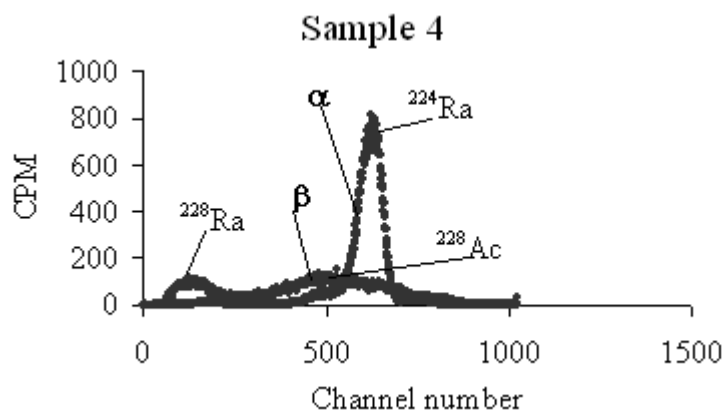


Figure 1  $\alpha$  and  $\beta$  spectra of Sample 4, 11 hr after  $t = 0$  (cpm = counts per minute)

Figure 2 presents the change in time of the count rate ( $N$ ; black dots) corresponding to the low-energy  $\beta$  component of the Sample 4 spectrum. The error bars are within the size of the points. It is clear that the count rate ( $N$ ) is not constant with measurement time, which confirms the earlier assumption that the <sup>224</sup>Ra series contributes to the low-energy  $\beta$ -spectrum component.

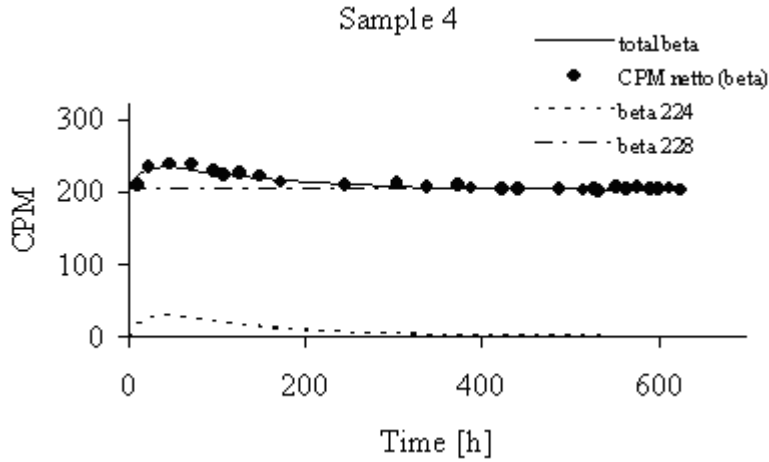


Figure 2 Change in time of the relative activities of  $\beta$  component for the test Sample 4

From earlier analyses of changes in time of the relative activities for the <sup>224</sup>Ra series, it appears that after about 400 hr this isotope and its daughters disintegrate almost completely. After  $t > 400$  hr, the <sup>228</sup>Ra isotope is responsible for the total count rate in the samples studied in this energy window. Fitting a disintegration curve of <sup>228</sup>Ra to the points (dot-dash line in Figure 2), the  $\beta$  count rate coming directly from <sup>228</sup>Ra was adjusted. The “excess amount” in count rate for  $t < 400$  hr comes from the <sup>224</sup>Ra  $\beta$  component, which was calculated using the Bateman (1910) equations (dashed line in Figure 2). After adding up both  $\beta$  components from <sup>224</sup>Ra and <sup>228</sup>Ra, a full line results that perfectly fits the experimental points.

The calculated count rates from the <sup>228</sup>Ra isotope ( $E_{\beta\max} = 38.9$  keV), together with set activities ( $A$ ) for 4 test samples, are presented in Table 1.

Table 1 Activity and obtained count rate for test samples.

Sample #	Activity ( $A \pm \Delta A$ ) <sup>228</sup> Ra (Bq)	Count rate ( $N \pm \Delta N$ ) <sup>228</sup> Ra (cps) (from low-energy component)
1	$3.00 \pm 0.05$	$1.00 \pm 0.02$
2	$5.00 \pm 0.05$	$1.66 \pm 0.02$
3	$8.00 \pm 0.05$	$2.68 \pm 0.03$
4	$10.00 \pm 0.05$	$3.33 \pm 0.03$

The statistical uncertainty ( $\Delta N$ ) of the count rate is calculated using the formula:

$$\Delta N = \sqrt{\frac{N}{t}}$$

where  $N$  is the netto count rate in cps (counts per second) at a chosen window, and  $t$  is the measurement time of the sample (in seconds).

The measurement efficiency ( $\eta$ ) for the low-energy  $\beta$  spectrum component was calculated using the equation:

$$\eta = \frac{N}{A} \cdot 100 \%$$

Using the formula above, the counting efficiency at the low-energy region was calculated for 4 test samples (see Table 2). The uncertainties ( $\Delta\eta$ ) of particular results were estimated by taking into account the maximum contribution of the  $^{224}\text{Ra}$  radioactive chain  $\beta$  component to the activity of the test samples. The average counting efficiency ( $\eta_{av} \pm \Delta\eta_{av}$ ) was obtained as the weighted mean of the 4 results.

Table 2 Detection efficiency of particles calculated for 4 test samples.

Sample #	Efficiency $\eta \pm \Delta\eta$ (%)	Average efficiency $\eta_{av} \pm \Delta\eta_{av}$ (%)
1	$33.2 \pm 1.0$	$33 \pm 5$
2	$33.2 \pm 0.7$	
3	$33.4 \pm 0.6$	
4	$33.3 \pm 0.5$	

#### $^{228}\text{Ra}$ DETERMINATION FROM THE LOW-ENERGY SPECTRUM COMPONENT

The activity of  $^{228}\text{Ra}$  isotopes can be estimated directly from the netto count rate ( $N$ ) at the low-energy  $\beta$  window. One must take into account the chemical recovery ( $\varepsilon$ ) and counting efficiency ( $\eta_{av}$ ) of the low-energy  $\beta$  particles, as well as the initial sample volume ( $V$ ). Chemical recovery ( $\varepsilon$ ) was calculated from the measurement of a set of 4 standard  $^{226}\text{Ra}$  samples;  $\varepsilon$  is equal to  $97 \pm 1.1\%$  (see Bliźnicka 2004). The activity (per unit of volume) is thus calculated using the formula:

$$A = \frac{N(t) - N_0}{V \cdot \varepsilon \cdot \eta_{av}} = \frac{N}{V \cdot \varepsilon \cdot \eta_{av}}$$

where  $A$  is the activity concentration of a radioactive substance in Bq/L;  $N(t)$  is the count rate in seconds at the chosen window;  $N_0$  is the count rate in seconds at the chosen window for background;  $N$  is the netto count rate in seconds at the chosen window;  $V$  is the initial sample volume;  $\eta_{av}$  is the detection efficiency of  $\beta$  particles measured via LSC at the low-energy region; and  $\varepsilon$  is the chemical recovery. The uncertainty  $\Delta A$  was calculated as the square root of the sum of uncertainties in all quantities in quadrature.

#### MEASUREMENT OF $^{228}\text{Ra}$ IN WATER SAMPLES

Before analyzing the environmental samples, test samples with known activities were measured. Sample 1 contained a mixture of standard isotopes  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$ , with equal  $^{228}\text{Ra}$  and  $^{226}\text{Ra}$  activities of  $3.00 \pm 0.05$  Bq. The sample was prepared in such a way that its composition was similar to that of natural mineral waters, where all radium isotopes may be present. The aim of this work was to test different methods to determine  $^{228}\text{Ra}$  from the low-energy  $\beta$  component when a sample contains all 3 radium isotopes.

The content of the <sup>228</sup>Ra isotope in Sample 1 was first determined from the fit of the total activity theoretical curve with a count rate at the low-energy component of the β spectrum. Figure 3 presents the fit of the theoretical relative activities of the β component from the <sup>228</sup>Ra isotope, as well as from the <sup>224</sup>Ra and <sup>226</sup>Ra series with the experimental points. It is clear that a contribution from the <sup>226</sup>Ra radioactive chain is significant (long-dashed line). The <sup>228</sup>Ra activity was determined from the contribution of <sup>228</sup>Ra to the β overall activity (see Table 3). The counting efficiency of low-energy β particles and the chemical recovery were also taken into account.

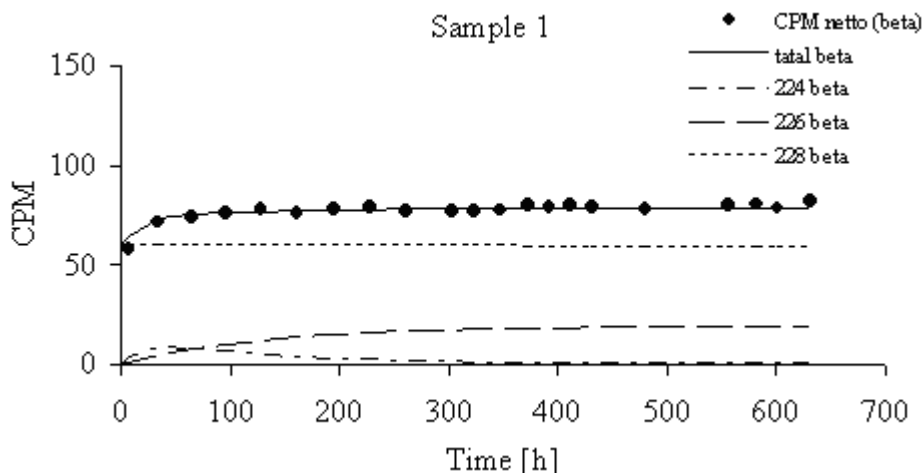


Figure 3 Change of the relative activity of β components in time (Sample 1)

Table 3 <sup>228</sup>Ra activity in Sample 1 obtained from the low-energy β window.

From the fit	From the first measurement
3.0 ± 0.5	3.0 ± 0.5

Figure 3 shows that for a sample composed of 2 standards, <sup>228</sup>Ra and <sup>226</sup>Ra, the contribution in activity at the low-energy β window comes from 3 isotopes: <sup>228</sup>Ra, <sup>224</sup>Ra, and <sup>226</sup>Ra. Analysis of the uranium-radium radioactive chain indicates that the <sup>226</sup>Ra series present in β activity in this energy window may come from <sup>214</sup>Pb and <sup>214</sup>Bi.

Figure 4 presents 2 graphs showing the change in measurement time of the relative activity of <sup>214</sup>Pb and <sup>214</sup>Bi isotopes from the <sup>226</sup>Ra series. The contribution in β activity coming from these isotopes is significant; therefore, one must measure a sample as soon as possible after its preparation, i.e. between 4 and 8 hr after radium precipitation. One must, however, take under consideration the possibility of the chemiluminescence effect. To eliminate this effect, samples should be kept in the dark (avoiding exposure to direct sunlight) for 1 or 2 hr in the counter and cooled down. If chemiluminescence is suspected, samples should be counted repeatedly at 1-hr intervals until a stable reading is obtained (Barnett et al. 1996; National Diagnostics 2004, 2005a,b; University of Wisconsin-Milwaukee 2005). The activity of the <sup>228</sup>Ra isotope in Sample 1 was calculated using the method described above (see Table 3).

The <sup>228</sup>Ra low-energy line determination method was tested also on waters provided by the IAEA during the intercalibration experiment. The aim of intercalibration was to compare different methods of <sup>226</sup>Ra and <sup>228</sup>Ra determination by laboratories all over the world. Table 4 presents <sup>228</sup>Ra activi-

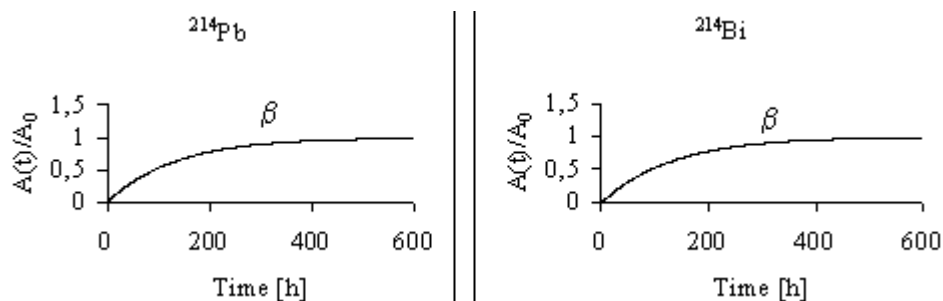


Figure 4 Change in time of the relative activity of the isotopes  $^{214}\text{Pb}$  and  $^{214}\text{Bi}$  from  $^{226}\text{Ra}$  series

ity concentrations of 3 samples from IAEA (codes 425, 426, 430) obtained from the low-energy  $\beta$  component from the first measurement and from the high-energy  $\beta$  component ( $^{228}\text{Ac}$ ). The average value, a weighted mean of all results sent by participants taking part in the intercalibration, is shown in Table 4, the last column of which shows the  $^{226}\text{Ra}$  isotope activity in the samples. Samples 425 (health spa water, low salinity) and 426 (mine discharge water, high salinity) were natural water samples, while Sample 430 (spiked, high salinity) was synthetic water.

Table 4 Comparison of activity concentrations of  $^{228}\text{Ra}$  isotope obtained by different methods with the average values determined by participants of the interlaboratory experiment for Samples 425, 426, and 430. The column on the far right shows the activity concentration of  $^{226}\text{Ra}$ .

Sample #	$^{228}\text{Ra}$ activity concentration (Bq/L)		Average value determined by all participants	Activity concentration $^{226}\text{Ra}$ (Bq/L)
	From high-energy $\beta$ component ( $^{228}\text{Ac}$ )	From low-energy $\beta$ component		
425	$0.43 \pm 0.04$	$0.57 \pm 0.09$	$0.50 \pm 0.18$	$0.31 \pm 0.02$
426	$7.68 \pm 0.17$	$9.21 \pm 1.40$	$8.2 \pm 3.0$	$7.40 \pm 0.30$
430	$7.71 \pm 0.26$	$8.60 \pm 1.31$	$8.2 \pm 0.15$	$3.25 \pm 0.15$

A low-energy line  $^{228}\text{Ra}$  determination method was also tested during analysis of environmental water samples collected from natural mineral water springs in health resorts in the Sudety and Carpathian mountains. Activity concentrations of  $^{228}\text{Ra}$  and  $^{226}\text{Ra}$  isotopes in 16 samples are presented in Table 5. In several examined environmental samples, the content of the  $^{228}\text{Ra}$  isotope obtained with the use of 2 analytical methods (i.e. from both the low- and high-energy parts of the spectrum) was below the lower limit of detection (LLD). The LLD (Currie 1968) for the  $^{228}\text{Ra}$  isotope determined from the high-energy  $\beta$  component is 0.06 Bq/L at 0.502 cps in the background, while the LLD for the low-energy  $\beta$  component is 0.1 Bq/L at 0.242 cps in the background. The activity concentrations obtained using the 2 methods overlap within the uncertainty value. Although the uncertainties for the low-energy line determination method are larger than for the high-energy line method, the results are obtained much faster. For quick screening, this method seems to be very convenient. The LLD for the  $^{226}\text{Ra}$  isotope is 0.03 Bq/L.

## CONCLUSIONS

$^{228}\text{Ra}$  isotope determination from the low-energy  $\beta$  component is simple and fast. Measurement should be performed soon after the radiochemical preparation. To reduce the chemiluminescence effect, samples should be kept in the dark and cooled in the LS counter.

Table 5 Activity concentration of <sup>228</sup>Ra obtained with the use of 2 analytical methods and <sup>226</sup>Ra in natural mineral waters.

Sample #, Spa	Activity concentration <sup>228</sup> Ra [Bq/L]		Activity concentration <sup>226</sup> Ra (Bq/L)
	From low-energy β component	From high-energy β component ( <sup>228</sup> Ac)	
1, Zloty Stok.	<0.1	<0.06	0.064 ± 0.004
2, Jedlina.	0.21 ± 0.03	0.21 ± 0.02	0.168 ± 0.006
3, Stare Bobrowniki.	<0.1	<0.06	0.141 ± 0.006
4, Jedlina.	0.26 ± 0.04	0.24 ± 0.02	0.179 ± 0.008
5, Cieplice Slaskie Zdroj.	<0.1	<0.06	6.39 ± 0.21
6, Polanica Zdroj.	<0.1	<0.06	0.14 ± 0.02
7, Slotwinka.	0.19 ± 0.03	0.21 ± 0.01	0.208 ± 0.008
8, Ladek Zdroj.	<0.1	<0.06	0.038 ± 0.004
9, Swieradow Zdroj	0.60 ± 0.09	0.67 ± 0.04	1.355 ± 0.048
10, Ladek Zdroj.	<0.1	<0.06	0.021 ± 0.003
11, Ladek Zdroj.	<0.1	<0.06	0.017 ± 0.003
12, Jedlina.	0.16 ± 0.03	0.16 ± 0.01	0.141 ± 0.006
13, Muszyna.	0.49 ± 0.08	0.48 ± 0.02	0.461 ± 0.018
14, Muszyna.	0.20 ± 0.03	0.23 ± 0.02	0.158 ± 0.007
15, Nove Mesto.	0.41 ± 0.06	0.41 ± 0.02	0.399 ± 0.014
16, Krynica Zdroj.	0.31 ± 0.05	0.35 ± 0.02	0.304 ± 0.011

Growth of the β-activity series is due to <sup>212</sup>Pb isotope decay, which emits low-energy β particles. Moreover, by examining samples containing <sup>226</sup>Ra isotopes from the uranium series, one can see that the contribution at the low-energy β activity window additionally comes from the <sup>226</sup>Ra radioactive chain (mainly from <sup>214</sup>Pb and <sup>214</sup>Bi isotopes). In order to avoid a contribution of <sup>224</sup>Ra (mainly from the <sup>212</sup>Pb isotope) and <sup>226</sup>Ra series, one should perform the measurement immediately after the sample preparation, taking care to avoid chemiluminescence.

Even if a sample contains <sup>226</sup>Ra, one can obtain proper results using the low-energy β component (Tables 4 and 5). Some of the samples were measured almost 12 hr after sample preparation. Additionally, particular attention should be paid to the efficiency determination for the low-energy window. The largest uncertainty is obtained when the measurement is performed after 38 hr (after the purification of a sample from <sup>228</sup>Ra daughters), because this is the time when <sup>224</sup>Ra reaches its maximum β activity.

Sample measurement should be performed within 4 to 8 hr after the precipitation of radium isotopes. The uncertainty of the activity determination from the low-energy β component is considerably higher than from the high-energy β component, and also the lower limit of detection (LLD) is higher than for the high-energy β component coming from <sup>228</sup>Ac. Determining <sup>228</sup>Ra activity from the low-energy β component is fast but only provides an estimate. It can be used for screening or for determining water activities that are higher than the environmental levels, as in mine waters.

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