

## RAPID METHOD FOR DETERMINING NATURAL RADIONUCLIDES IN DRINKING WATER

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**ABSTRACT.** Radionuclides from the natural decay series are ubiquitous in Earth's crust, and because of direct recoil, diffusion, migration, and dissolution, these radionuclides infiltrate drinking water through the water cycle. Extensive data exists on <sup>222</sup>Rn and <sup>226</sup>Ra in drinking water in Austria; however, little is known about the <sup>232</sup>Th decay products like <sup>228</sup>Ra, the <sup>222</sup>Rn progenies <sup>210</sup>Pb and <sup>210</sup>Po, and uranium. This paper presents a combination of 2 rapid methods for determining lead and radium in drinking water via liquid scintillation counting (LSC) and inductively coupled plasma mass spectrometer (ICP-MS) analyses for uranium and thorium. Measurements were performed as part of a project on drinking water radiometry in Upper Austria, which was partly funded by the federal state of Upper Austria. The Council of the European Union (1998, draft of 20 April 2005) on the quality of water intended for human consumption states a total indicative dose (TID) as a parametric value of 0.1 mSv/yr. The TID excludes tritium, <sup>40</sup>K, and <sup>222</sup>Rn and its progenies. However, reference values for <sup>222</sup>Rn and its progenies are proposed by the Commission of the European Communities (2001). Due to the high effective dose per unit intake via ingestion (Sv/Bq) of <sup>228</sup>Ra and <sup>210</sup>Po ( $3 \times 10^{-5}$  and  $2.6 \times 10^{-5}$ , respectively) for infants compared to <sup>226</sup>Ra ( $4.7 \times 10^{-6}$ ), it is essential to develop nuclide-specific methods to estimate the risk of these nuclides. One liter of stabilized drinking water (0.15M HNO<sub>3</sub>) is concentrated to 10% by evaporation, and the lead and its decay products are separated by sulfide precipitation with Na<sub>2</sub>S, dissolved in HNO<sub>3</sub>, and converted to the chloride form. <sup>210</sup>Bi (in equilibrium with <sup>210</sup>Pb) and <sup>210</sup>Po were selectively extracted using the POLEX™ (ETRAC, USA) extractive cocktail and measured via LSC. In the residual solution, radium was coprecipitated with BaSO<sub>4</sub>, dissolved in alkaline EDTA, and measured with OptiPhase HiSafe 3. The lower limit of detection (LLD) for <sup>228</sup>Ra is below 10 mBq/L. Uranium and thorium were measured directly from the water samples by ICP-MS; <sup>226</sup>Ra and <sup>222</sup>Rn were measured using a mineral oil scintillator. About 200 samples of drinking water from all areas of Upper Austria were analyzed in a screening program for <sup>226</sup>Ra, <sup>222</sup>Rn, gross alpha, gross beta, and <sup>3</sup>H, and about 100 samples were analyzed for <sup>228</sup>Ra, <sup>210</sup>Po, <sup>210</sup>Pb, and thorium.

### INTRODUCTION

In Council Directive 98/83/EC (Council of the European Union 1998), gross alpha and beta measurements are recommended for screening methods. If the gross alpha and beta activities are less than 0.1 Bq/L and 1.0 Bq/L, respectively, it can be assumed that the total indicative dose (TID) is less than the parametric indicator value of 0.1 mSv/yr. The TID is the effective dose for 1 yr of intake resulting from all the radionuclides detected in the water supply, both of natural and artificial origin, excluding tritium, <sup>40</sup>K, and radon and its decay products. If the gross alpha activity exceeds 0.1 Bq/L or the gross beta activity exceeds 1.0 Bq/L, analysis for specific radionuclides are required. For tritium, a separate parametric value of 100 Bq/L has been set.

The TID excludes <sup>222</sup>Rn and its progenies. There are circumstances under which <sup>210</sup>Pb and <sup>210</sup>Po in drinking water could pose a comparable or a higher radiation risk than other natural radionuclides in drinking water. Accordingly, <sup>210</sup>Pb and <sup>210</sup>Po should not be disregarded when monitoring samples nor when taking action to reduce exposures. Reference values for these radionuclides are proposed by the Commission of the European Communities (2001), with the reference values 0.2 Bq/L and 0.1 Bq/L for <sup>210</sup>Pb and <sup>210</sup>Po, respectively. Above a concentration of 100 Bq/L, member states should set a reference level for radon to be used when considering whether remedial action is needed to protect human health. A level higher than 100 Bq/L may be adopted if national surveys show that this is necessary for implementing a practical radon program. For concentrations in excess of 1000 Bq/L, action is justified for radiological protection (Commission of the European Communities 2001).

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In Austria, mostly spring and groundwater is used as drinking water; very little surface water is used. In case of the release of artificial radionuclides like  $^{137}\text{Cs}$  or  $^{90}\text{Sr}$ , these radionuclides would be adsorbed (e.g. on soil or bedrock), and only a small amount would seep into the groundwater. Therefore, special attention is paid to natural radionuclides in drinking water.

Previous research in Lower Austria showed that a mean additional dose between 0.26 and 1.27 mSv is received by the population in the most affected areas every year via the liberation of  $^{222}\text{Rn}$  from water. The additional average dose for the Austrian population in the first year after the Chernobyl accident was approximately 0.57 mSv (Schönhofer 1992). In northwest Lower Austria,  $^{226}\text{Ra}$  concentrations in drinking water have been found that exceed the concentration limit of 0.122 Bq/L set by the former Austrian Radiation Protection Regulation (Schönhofer 1990). Due to geological conditions (Geologische Bundesanstalt 1987), further measurements of the nuclides of the uranium decay series are necessary in Austria. For this purpose and according to the requirements of the Drinking Water Ordinance, as well as the Austrian Standards for determination and evaluation of the total dose due to radionuclides in drinking water (Austrian Standardisation Institute 2005), a fast and easy method for specific radionuclide determination was developed in our laboratory.

This article presents simple procedures for determining various radionuclides like tritium, uranium, thorium,  $^{222}\text{Rn}$ ,  $^{226}\text{Ra}$ ,  $^{228}\text{Ra}$ ,  $^{210}\text{Pb}$ , and  $^{210}\text{Po}$  using 1–2 L of water sample. Uranium and thorium are measured by an inductively coupled plasma mass spectrometer (ICP-MS), while tritium,  $^{222}\text{Rn}$ ,  $^{226}\text{Ra}$ ,  $^{228}\text{Ra}$ ,  $^{210}\text{Pb}$ , and  $^{210}\text{Po}$  are measured by liquid scintillation counting (LSC) (Salonen and Hukkanen 1997). Numerous methods exist for determining  $^{228}\text{Ra}$  in drinking water.  $^{228}\text{Ra}$  can be measured by gamma counting via  $^{228}\text{Ac}$  (Petrow and Allen 1961; Percival and Martin 1974) or directly by LSC after a quick separation (Schönhofer and Wallner 2001), as well as directly by LSC after a more complex separation method in which  $^{228}\text{Ra}$  and  $^{210}\text{Pb}$  are separated (Chalupnik and Lebecka 1993).

The method we used for determining  $^{228}\text{Ra}$ ,  $^{210}\text{Pb}$ , and  $^{210}\text{Po}$  is very good and reproducible and is especially suitable for routine analyses because LSC spectrum interpretation is relatively easy after the chemical separation of  $^{228}\text{Ra}$  and  $^{210}\text{Pb}$ . Results from this project together with interpretation and evaluation of former data will be published with the project associates.

## METHODS

### Determination of Uranium and $^{232}\text{Th}$ Using ICP-MS

This method is based on direct introduction of the acidified samples (0.1M  $\text{HNO}_3$ ), without any chemical pretreatment, into an ICP-MS (PerkinElmer Elan DRC II). Uranium was determined at the mass number 238 using Rh as the internal standard. The method provides a limit of detection well below 1 ng/L, and resulted in good repeatability for tests at 3 different concentrations. Recovery percentage values found for the determination of uranium in synthetic water samples varied less than 2% (98–102%). Results obtained for the certified content of a reference material (TM-DWS from NRWI, Canada) are within the same range, indicating the accuracy of the ICP-MS procedure without the need for isotope dilution.  $^{232}\text{Th}$  screening measurements were performed in the same way via uranium calibration.

### Tritium, Gross Alpha, and Gross Beta Analysis

Tritium was determined during the gross alpha and gross beta screening. Seven mL of water were mixed with 13 mL of OptiPhase HiSafe 3 and measured via a Quantulus 1220<sup>TM</sup> with a pulse-shape analysis (PSA) value of 120. Pulse-shape analysis allows for the identification of the particle and

enables simultaneous recording of pure alpha and beta spectra and counting of very small alpha activity in the presence of high beta activity.

### **<sup>222</sup>Rn and <sup>226</sup>Ra Analysis**

Water samples of 1–2 L are collected in a gastight bottle prefilled with 20 mL of concentrated HNO<sub>3</sub> for stabilization. It is necessary to use bottles of appropriate size to avoid <sup>222</sup>Rn diffusion into the free space between the sample and the neck of the bottle. For <sup>222</sup>Rn determination, 10 mL of the water sample is mixed with 10 mL of PerkinElmer high-efficiency mineral oil scintillator, agitated, and measured in equilibrium with <sup>218</sup>Po and <sup>214</sup>Po by LSC (Schönhofer 1990). From these data, the ingrowth of <sup>210</sup>Pb from <sup>222</sup>Rn can be calculated and used for correction in the <sup>210</sup>Pb analysis. <sup>222</sup>Rn (half-life of 3.82 d) is the short-lived mother nuclide of <sup>210</sup>Pb (half-life of 22.3 yr). <sup>222</sup>Rn and <sup>210</sup>Pb cannot achieve radioactive equilibrium, but the ingrowth of <sup>210</sup>Pb can be calculated using the ratio of the half-lives  $T_{1/2}({}^{222}\text{Rn}) / T_{1/2}({}^{210}\text{Pb}) = 0.47 \times 10^{-3}$  (see Equation 1 below; Katzlberger 2001):

$$A_{({}^{210}\text{Pb})} \approx \frac{T_{1/2}({}^{222}\text{Rn})}{T_{1/2}({}^{210}\text{Pb})} A_0({}^{222}\text{Rn}) \left( 1 - e^{-\frac{\ln 2}{3.82} t} \right) \quad (1)$$

After 2 weeks of storage, the result will be 0.43 mBq of <sup>210</sup>Pb from 1 Bq of <sup>222</sup>Rn. For this reason, the <sup>222</sup>Rn should be measured shortly after sampling and storage time, and any further analyses for <sup>210</sup>Pb and <sup>210</sup>Po should be done shortly thereafter.

From another 10 mL of the sample, <sup>226</sup>Ra is determined using the same method as the one used for measuring <sup>222</sup>Rn. Before measurement, however, the sample is stored until unsupported <sup>222</sup>Rn is decayed below the lower limit of detection (LLD) and <sup>226</sup>Ra is in equilibrium with its short-lived progenies (<sup>222</sup>Rn, <sup>218</sup>Po, and <sup>214</sup>Po are counted in the alpha spectrum). Depending on the <sup>222</sup>Rn concentration measured, the decay period can be calculated.

Due to the little difference of the energy of beta radiation between <sup>228</sup>Ra and <sup>210</sup>Pb, <sup>228</sup>Ra can only be measured by LSC after separation from lead. Therefore, 2 methods are combined to get 1 fraction of <sup>228</sup>Ra and 1 fraction of <sup>210</sup>Pb and <sup>210</sup>Po from 1 sample.

### **<sup>210</sup>Pb and <sup>210</sup>Po Analysis**

One mL of Pb(NO<sub>3</sub>)<sub>2</sub> solution (1.6 mg Pb<sup>2+</sup>/mL) is added to 1 L of water sample, and the sample is evaporated to ~80 mL. These 80 mL are evaporated to near dryness and repeatedly fumed with HCl. The residue is dissolved in 15 mL 1M HCl, and precipitation is done at pH 1.5 with 1M Na<sub>2</sub>S. After centrifugation and filtration through a blue ribbon filter, the filtrate is used for <sup>228</sup>Ra analysis, and the precipitate is dissolved in concentrated HNO<sub>3</sub> and fumed off with concentrated HNO<sub>3</sub> and HCl. The sample is evaporated to near dryness, dissolved in 2 mL 1M HCl and 12 mL 7.5M H<sub>3</sub>PO<sub>4</sub>, and stored in a refrigerator until the <sup>210</sup>Bi is in equilibrium with <sup>210</sup>Pb. After 2 weeks, <sup>210</sup>Bi and <sup>210</sup>Po are selectively extracted with POLEX™ (ETRAC, USA) and measured by LSC (see Figures 1 and 2). In order to determine the recovery of the lead precipitation, 3.8 mL of the aqueous phase are diluted to 50 mL with 1% HNO<sub>3</sub> and measured with an atomic absorption spectrophotometer (AAS) (Katzlberger 2001).

### **<sup>228</sup>Ra Analysis**

The filtrate from <sup>210</sup>Pb and <sup>210</sup>Po analysis is diluted with 35 mL of water and brought to pH 2. One mL of 1M H<sub>2</sub>SO<sub>4</sub> is added, and radium is coprecipitated with BaSO<sub>4</sub> by adding 2 mL of Ba(NO<sub>3</sub>)<sub>2</sub>

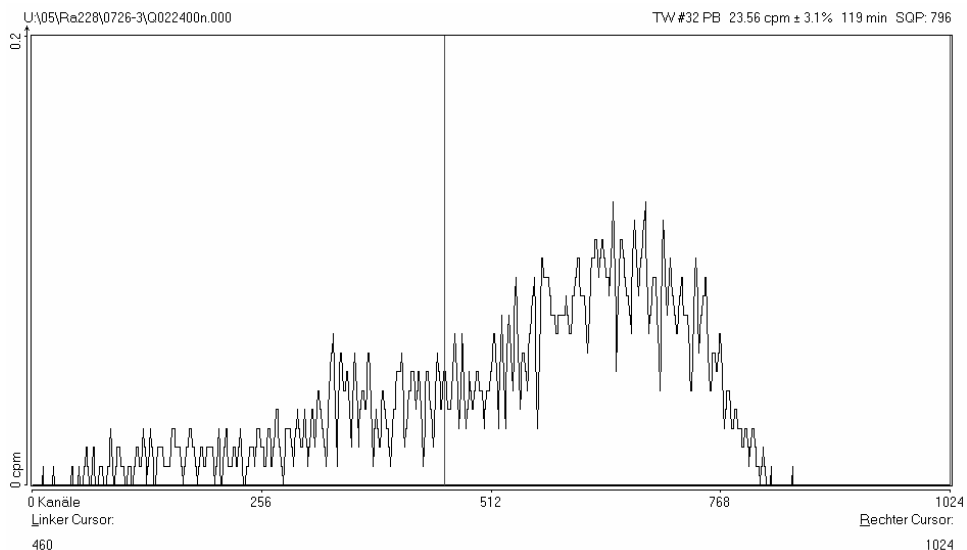


Figure 1 Spectrum of  $^{210}\text{Bi}$ , extracted with POLEX, after reaching radioactive equilibrium with  $^{210}\text{Pb}$ . The vertical line defines the selected range for the analysis.

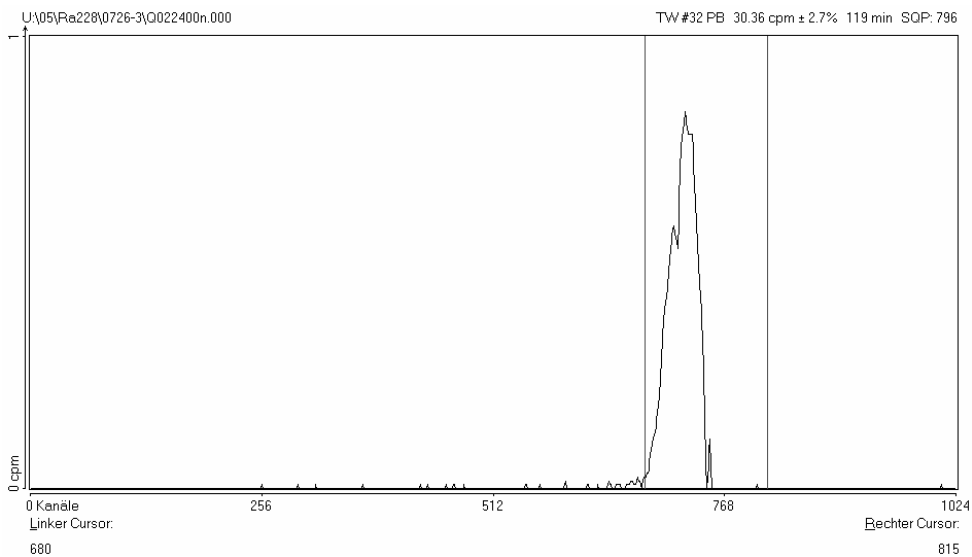


Figure 2 Spectrum of  $^{210}\text{Po}$ , extracted with POLEX. The 2 vertical lines define the selected range for the analysis.

solution while the solution is boiling. After cooling, the precipitate is centrifuged, rinsed with water, and dissolved while cooking with 5 mL of 0.25M EDTA. The solution is evaporated to a volume of 2–3 mL and measured by LSC with 18 mL OptiPhase HiSafe 3 (Steininger 2002; see Figure 3 below). For determining the chemical recovery via barium, the exact volume of the solution is weighed, and 20  $\mu\text{L}$  are diluted to 20 mL with 1%  $\text{HNO}_3$  and measured via an inductively coupled plasma optical emission spectrometer (ICP-OES).

Four different radium isotopes are included in the natural radioactive decay series:  $^{226}\text{Ra}$  in the uranium series,  $^{223}\text{Ra}$  in the actinium series, and  $^{228}\text{Ra}$  and  $^{224}\text{Ra}$  in the thorium series. Depending on the occurrence of uranium and thorium in bedrock, these Ra isotopes are present everywhere in Earth's crust in various concentrations. During the weathering processes, the nuclides of the radioactive decay series dissolve in water (Asikainen 1981). By using barium sulfate precipitation, all Ra isotopes are coprecipitated. Measurements are performed via a Quantulus 1220, and a PSA of 130. Thus,  $^{228}\text{Ra}$  (a beta emitter) can be identified in the beta window (see Figure 3). The other radium nuclides are alpha emitters and can be identified in the alpha window. After the separation, there is only 1 beta emitter in the beta window ( $^{228}\text{Ra}$ ); however,  $^{226}\text{Ra}$  is still present in the sample, so there can be interference due to the beta-emitting decay products of  $^{226}\text{Ra}$ . Ingrowth of these decay products starts after separation. To avoid this effect, the sample has to be measured immediately after separation.

First, experiments were performed with synthetic water samples containing 41 mg of sodium, 71 mg of calcium, and 107 mg of carbonate in 1 L of deionized water using the method described above. These samples were spiked with 0.5 mL of a  $^{232}\text{Th}$  standard solution of 1.06 Bq/mL and 0.3 mL of a  $^{210}\text{Pb}$  standard solution of 1.74 Bq/mL. Both standard solutions are in equilibrium with their daughter products. Figure 3 shows a spectrum of a mixed standard (dark upper curve) with the same amounts of  $^{232}\text{Th}$  standard solution and  $^{210}\text{Pb}$  standard solution as the spiked sample (lower curve). The darker upper curve represents the spectrum of the mixed standard nuclides, and the lighter lower curve represents the  $^{228}\text{Ra}$  spectrum of a spiked sample after chemical separation; the spectrum confirms the good quality of the separation. It is obvious that the  $^{210}\text{Pb}$ , which was present in the original solution (upper curve), is separated (not present in the lower curve). Some real water samples from Upper Austria have been spiked with different amounts of the  $^{232}\text{Th}$  and the  $^{210}\text{Pb}$  standard solutions. The chemical recovery for these experiments is estimated by LSC and finally determined by ICP-OES. Results are shown in Table 1.

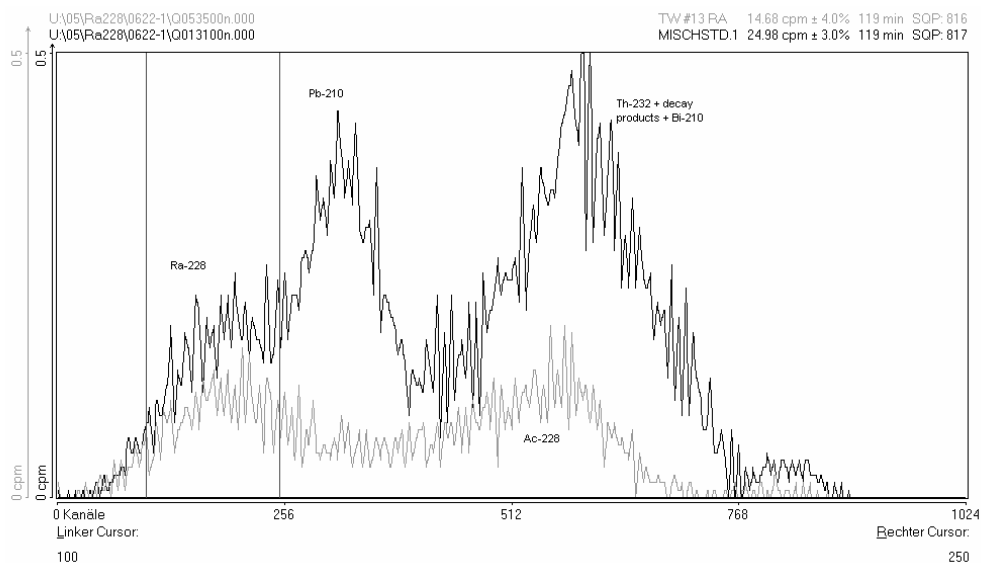


Figure 3 Spectrum of a mixed standard solution with 0.3 mL of a  $^{210}\text{Pb}$  standard solution with 1.74 Bq/mL and 0.5 mL of a  $^{232}\text{Th}$  standard solution of 1.06 Bq/mL (darker upper curve). The lower curve shows the spectrum of  $^{228}\text{Ra}$  of this mixed standard solution after separation. The 2 horizontal bars define the selected range for the analysis.

Table 1 Recovery of spiked water samples, synthetic water samples (IAEA) measured by LSC, and spiked water samples from Upper Austria measured by LSC and determined with an atomic absorption spectrometer (AAS) and inductively coupled plasma optical emission spectrometer (ICP-OES).

Radionuclide	Simulated water (LSC)	Simulated water (AAS)	IAEA water (LSC)	Upper Austria samples (LSC)	Upper Austria samples (AAS/ICP-OES)
$^{228}\text{Ra}$	75–96%	—	69–79%	78–96%	75–98%
$^{210}\text{Pb}$	65–93%	79–96%	—	75–79%	79–88%
$^{210}\text{Po}$	94–100%	—	—	97–100%	—

## RESULTS AND DISCUSSION

In ~70 water samples from Upper Austria,  $^{222}\text{Rn}$  and  $^{226}\text{Ra}$  were determined. Figures 4 and 5 show the distributions of the concentrations and the median values of  $^{222}\text{Rn}$  and  $^{226}\text{Ra}$ , respectively. To evaluate this method, 3 spiked water samples from an IAEA proficiency test with different known  $^{228}\text{Ra}$  activity concentrations were measured. The results and compositions are shown in detail in Table 2. Using these known activity concentrations and the measured  $^{228}\text{Ra}$ , the chemical recovery was estimated. The recovery is sufficient, but correction via the chemical yield of barium (determination as described above) should be done, since the recovery values for  $^{228}\text{Ra}$  estimated by LSC are in a range from ~70 to ~95%. Table 3 shows a selection of interesting results of radionuclide activity concentrations in water samples measured as part of a project on drinking water radiometry in Upper Austria.

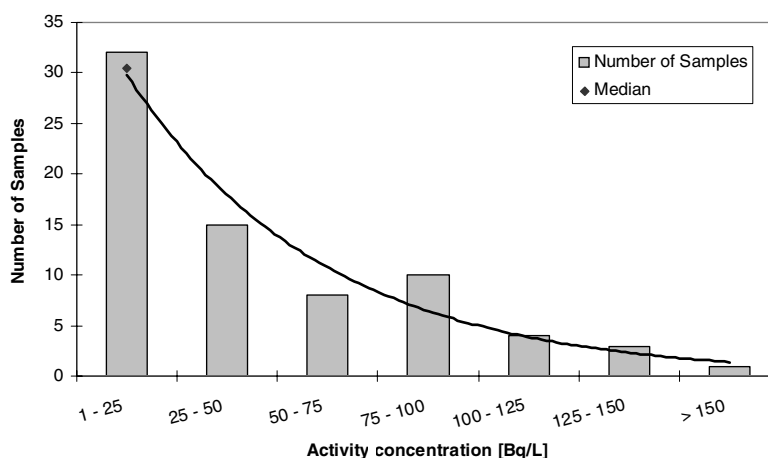


Figure 4 Distribution of the  $^{222}\text{Rn}$  concentration in Upper Austrian drinking water. The point upon the curve represents the median value of the samples.

The lower limit of detection (LLD) for routine screening of  $^{228}\text{Ra}$  should be 80 mBq/L, as discussed in a draft of Annex II of Council Directive 98/83/EC (Table 4). For a new water source for which it is plausible that  $^{228}\text{Ra}$  exceeds 20% of the reference concentration (0.2 Bq/L), the LLD for the first measurement should be 20 mBq/L. The LLD of our method for  $^{228}\text{Ra}$  is between 5.5 and 6.0 mBq/L, calculated using  $3\sigma$  of the background measurement, as described above using 1 L of sample and the efficiency and chemical recovery.  $^{228}\text{Ra}$  could also be determined by gamma counting of its

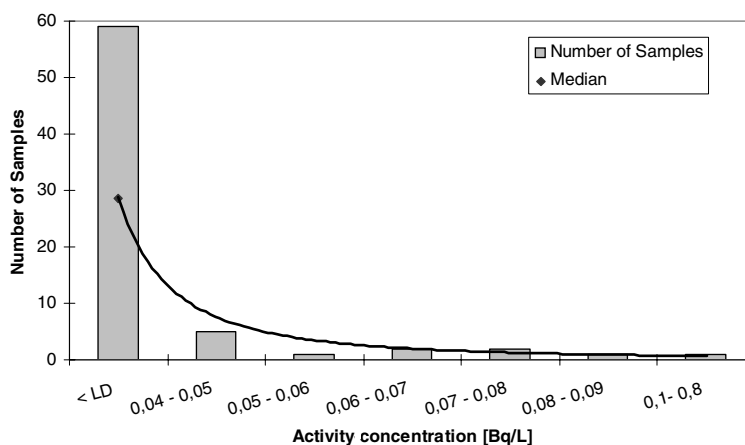


Figure 5 Distribution of the <sup>226</sup>Ra concentration in Upper Austrian drinking water. The point upon the curve represents the median value of the samples.

Table 2 Composition and results for chemical recovery of the spiked IAEA water samples.

Sample	<sup>228</sup> Ra (mBq/kg)	<sup>228</sup> Ra measured (mBq/kg)	Chemical recovery (%)
IAEA-419	2.6 ± 0.9	<5.5	—
IAEA-421	124 ± 2	84 ± 3	69
IAEA-423	2510 ± 44	1990 ± 5	79

Table 3 Selected results of samples from the Upper Austria project.

Sample	Tritium (Bq/L)	<sup>222</sup> Rn (Bq/L)	<sup>226</sup> Ra (mBq/L)	<sup>228</sup> Ra (mBq/L)	<sup>210</sup> Pb (mBq/L)	<sup>210</sup> Po (mBq/L)
Hellmonsödt 1	2 ± 1	21 ± 4	<42.1	8 ± 6	27 ± 4	11 ± 2
Hellmonsödt 2	2 ± 1	126 ± 14	78 ± 36	<5.5	65 ± 7	53 ± 6
Kopfing 1	2 ± 1	35 ± 3	<42.1	<5.5	13 ± 3	8 ± 2
Kopfing 2	2 ± 1	17 ± 2	<42.1	<5.5	42 ± 7 grown from Rn = 17 mBq/L	26 ± 3

daughter, <sup>228</sup>Ac. This method is not suitable for fast drinking water analyses; ~50 L of water would have to be evaporated to achieve a LD of 6.0 mBq/L. The lower limit of detection (LLD) for <sup>210</sup>Pb and <sup>210</sup>Po is between 3.5–4.6 and 1.1–2.8 mBq/L, respectively.

About 130 water samples from different locations of Upper Austria measured by <sup>238</sup>U were also measured using ICP-MS. Figure 6 shows the distribution of the concentration and the median value. The values for the <sup>234</sup>U concentration were all below the LLD of 1 ng/L. For calculating the TID in these samples, it was assumed that the 2 isotopes were in equilibrium due to the natural uranium. Because of the low dose coefficients of uranium and the small contribution to TID in these concentration ranges (all of the measured values are <7 µg/L), the possible error resulting by the presence of possible non-equilibrium between the 2 uranium isotopes (<sup>234</sup>U and <sup>238</sup>U) is negligible. For water samples with higher (dose-relevant) <sup>238</sup>U concentrations (e.g. 20 µg/L or more), the <sup>234</sup>U mass con-

Table 4 Comparison between the lower limit of detection (LLD) of Council of the European Union (1998, Draft of 20 April 2005 to Annex II) and the achieved LLD.

Parameters	LLD Council Directive 98/83/EC (Bq/L)	LLD achieved (Bq/L)
Tritium	10	1.25
$^{238}\text{U}$	0.02	0.0006
$^{226}\text{Ra}$	0.04	0.04
$^{228}\text{Ra}^{\text{a}}$	0.08	0.006
$^{228}\text{Ra}^{\text{b}}$	0.02	

<sup>a</sup>For routine screening.

<sup>b</sup>A new water source for which it is plausible that  $^{228}\text{Ra}$  exceeds 20% of the reference concentration, 0.2 Bq/L.

concentrations would be above the LLD of 1 ng/L (the ratio of the mass concentrations of  $^{238}\text{U}$  and  $^{234}\text{U}$  in equilibrium is about  $5.5 \times 10^{-5}$ , the ratio of the half-lives of the 2 radionuclides), and, therefore, the measured values for the  $^{234}\text{U}$  concentration could be used for dose calculation.

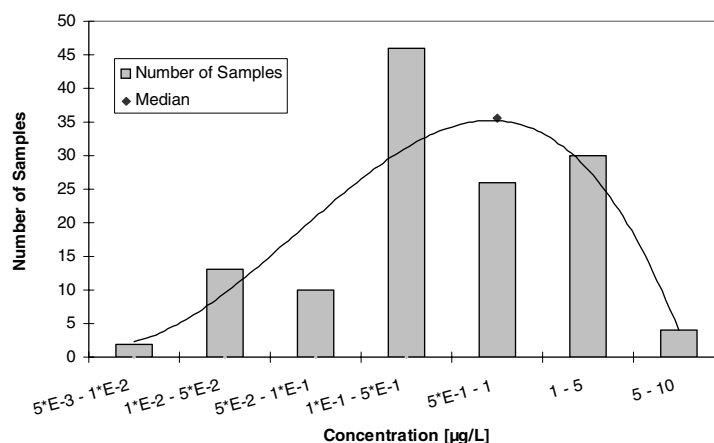


Figure 6 Distribution of the  $^{238}\text{U}$  concentration in Upper Austrian drinking water. The point upon the curve represents the median value of the samples.

The Austrian Standard for determination and evaluation of the total dose due to radionuclides in drinking water (ÖNORM ON-S5251, Austrian Standardisation Institute 2005) requires a LLD of 0.04 Bq/L for  $^{228}\text{Ra}$  and 0.1 Bq/L for  $^{226}\text{Ra}$ . The chemicals needed for measurement are inexpensive. These limits of detection are achieved with this method. For determining  $^{228}\text{Ra}$ , no correction for the  $^{210}\text{Pb}$  (same energy range in the spectrum) is needed. The method can be extended for the  $^{226}\text{Ra}$  determination and by applying the few steps given in the section “ $^{210}\text{Pb}$  and  $^{210}\text{Po}$  Analysis,” additional information about the  $^{210}\text{Pb}$  and  $^{210}\text{Po}$  activity concentration can be obtained. Due to the low limits of detection, the fast and easy measurement, and the small amounts of sample needed, this method is ideally suited for use in routine analyses of drinking water.

## ACKNOWLEDGMENTS

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