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Effects of the composition of standard reference material on the accuracy of determinations of ^{210}Pb and ^{137}Cs in soils with gamma spectrometry

Y. Li ^{a,b,*}, X.C. Geng ^b, H.Q. Yu ^b, G.J. Wan ^c

^a State Key Laboratory of Dryland Farming and Soil Erosion in the Loess Plateau, Institute of Soil and Water Conservation, CAS and MWR, Yangling 712100, China

^b Institute of Environment and Sustainable Development in Agriculture, CAAS, No. 12 Zhongguancun South Street, Beijing 100081, China

^c State Key Laboratory of Environmental Geochemistry, Institute of Geochemistry, Chinese Academy of Sciences, Guiyang, China

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ABSTRACT

The effect of the composition of the used standard reference material (SRM) on results of determination of fallout radionuclides in soil samples was studied. Using five soil types as SRMs, we measured the specific activity of ^{210}Pb and ^{137}Cs in six target samples of Chestnut soil. It was observed that the determination of the ^{210}Pb activity in the samples depended on the chemical composition of SRMs used to create the efficiency curves. Thus, using SRMs similar in chemical composition to the target samples should improve accuracy in the determination of ^{210}Pb in environmental samples.

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1. Introduction

To make better use of environmental fallout radionuclides (FRNs) in assessing soil erosion and solving associated environmental problems (Zapata, 2002), a number of proficiency tests designed to evaluate the accuracy of their determination have been performed (IAEA, 2006; Shakhshiro et al., 2008). In 2006, the International Atomic Energy Agency (IAEA) organized such a proficiency test, which involved 22 national laboratories in a coordinated research project (CRP D1.50.08). Approximately 82% of the laboratories reported 'acceptable' accuracy for ^{137}Cs quantification, but only 33% of the laboratories were able to demonstrate acceptable accuracy for ^{210}Pb . In the same year, IAEA organized another proficiency test with 38 national laboratories from 29 countries in the IAEA ALMERA Network (Shakhshiro et al., 2008). In the latter test, the results of ^{137}Cs determinations were comparable to the IAEA target values, but only 45% of all reported ^{210}Pb results were in agreement with the target values. Therefore, the discrepancies in the ^{210}Pb determination results clearly require further examination.

* Corresponding author at: State Key Laboratory of Dryland Farming and Soil Erosion in the Loess Plateau, Institute of Soil and Water Conservation, CAS and MWR, Yangling 712100, China; and Institute of Environment and Sustainable Development in Agriculture, CAAS, No. 12 Zhongguancun South Street, Beijing 100081, China. Tel./fax: +86 10 82106016.

E-mail address: yongli32@hotmail.com (Y. Li).

Some studies attributed the large discrepancies in the ^{210}Pb results from participating laboratories to two main factors: (i) inappropriate determination of the efficiency of the gamma-ray detector in the low energy range and (ii) overestimation or underestimation of the effects of self-absorption (Shakhshiro et al., 2008). The detector full-energy-peak (FEP) efficiency is defined as the ratio of the count rate in the peak corresponding to a gamma ray of energy E (from a standard) to its known emission rate. Measurements of detector efficiency as a function of energy are required for the quantification of the radionuclide content of unknown samples. The detector efficiency depends primarily on the type of detector, the composition of the standard reference materials (SRMs), the extent of sample self-absorption, the geometry of the sample, and its position with respect to the detector (Zapata, 2002). Sample self-absorption refers to the reduction in counts as a result of the interaction between emitted gamma rays and atoms in soil samples, particularly for 'thick' samples (Li and Geng, 2010). Self-absorption depends not only on the mass of the sample, but also on its chemical composition. Therefore, we studied the effects of differences in chemical composition between the test samples and the SRMs selected to measure detector full-energy-peak efficiency on the determination of sample activity.

We hypothesized that the poor accuracy of ^{210}Pb determination in environmental samples could be attributed to an inappropriate selection of SRMs for the efficiency curve calibration determination. We tested this hypothesis by comparing the differences between specific activities of ^{210}Pb and ^{137}Cs determined in Chestnut soil (*Kastanozems*) with efficiency calibration curves

constructed using curves of four different types, and the activities determined with curves constructed from the same soil. The four soil types were Cinnamon soil (*Lixisols*), Yellow soil (*Ferralsols*), Loessial soil (*Calcisols*), and Purple soil (*Cambisols*). The objectives of our study were: (i) to investigate the effects of different SRMs on uncertainty of measurements of FRN activities, (ii) to analyze a correlation of measured specific activities of ^{210}Pb and ^{137}Cs with chemical composition of different SRMs, and (iii) to identify factors that affect uncertainty in the measurements of FRNs activities, particularly ^{210}Pb .

2. Materials and methods

2.1. Target soil and accumulation of gamma spectra

The Chestnut soil (*Kastanozems*) was selected as the target soil because it is distributed worldwide (ISSS/ISIC/FAO, 1998). Six different samples of Chestnut soil were used (referred to as Target samples no. 1–6 below). They exhibited a wide range of activities of ^{210}Pb (27–175 Bq kg $^{-1}$) and ^{137}Cs (7–29 Bq kg $^{-1}$). The samples were randomly collected from the Inner Mongolian Steppe in the Xilinhot River Basin in northern China. After weeds and litter were removed from the surface, the soil samples were taken out from a depth of 0–15 cm with a hand core sampler (6.7 cm in diameter) driven by a hammer. The samples were then placed in plastic bags. In the laboratory, the target samples were air-dried, weighed, and passed through a 2 mm sieve. The fraction below 2 mm was placed in a Plexiglass cylinder (101 mm in diameter and 25 mm in height). The sample weight was 250 g, and its thickness was 25 mm; the cylinders were sealed with a plastic tape. The gamma spectra of FRNs, including ^{210}Pb and ^{137}Cs , in the soil samples were determined using a broad-energy HPGe detector with a relative detection efficiency of 50.9% (BE5030, Canberra, USA). The crystal was 80 mm in diameter and 30 mm in length; the end-cap material was aluminum (1.5-mm-thick). The 46.5 and 662 keV lines were used for ^{210}Pb and ^{137}Cs , respectively. The spectra were accumulated for 24,946–56,342 s, which resulted in analytical precision of $\pm 12\%$ for ^{210}Pb and $\pm 6\%$ for ^{137}Cs .

2.2. Standard reference materials (SRMs) and efficiency calibration

We used Laboratory Sourceless Calibration Software (LabSOCS, <<http://www.canberra.com/products/839.asp>>) to construct efficiency calibration curves. The main advantage of LabSOCS technique is that no radioactive sources are needed for accurate efficiency calibrations in the laboratory and, hence, one can construct the efficiency calibration curve based on the chemical composition and geometry of the target soil sample that acts as an SRM.

To investigate the effects of the composition of SRMs on ^{210}Pb and ^{137}Cs determinations in soils, we selected the following five soil types that acted as SRMs in this study because they not only represent different climatic zones (temperate zone, warm temperate zone, and subtropical zone), but are also widespread all over the world (ISSS/ISIC/FAO, 1998).

The Chestnut soil (*Kastanozems*) is developed under steppe vegetation in temperate semi-arid regions; it can be found in the Eurasian Continental region and in the western part of North America.

The Cinnamon soil (*Lixisols*) is developed in semi-arid forest-shrub regions in the Mediterranean and warm temperate monsoon climatic zones; it is abundant in western Eurasian Continental regions, northern Africa, southwestern America, and northern Mexico, as well as in the southern part of South America, Russia, and Australia.

The Yellow soil (*Ferralsols*) is developed under evergreen broad-leaved forests in subtropical humid mountain areas and in plateaus. It occurs in the eastern Eurasian Continental region, eastern

North America, and in the southern part of the Tropic of Capricorn in South America.

The Loessial soil (*Calcisols*) represents the continental-phase yellow silt deposits formed in the Quaternary period, and is widespread in arid and semi-arid regions in the middle-latitude area of the northern hemisphere.

The Purple soil (*Cambisols*) is developed from calcareous purple sandy shale parent materials in subtropical areas and located mainly in southwestern China.

Input parameters for the efficiency calibrations using LabSOCS included: (i) chemical composition of the five soil types (that acted as SRMs) as listed in Table 1, (ii) characteristics of the sample containers, namely, Plexiglass cylinders with 5-mm-thick walls, 1-mm-thick bottoms, and 101 mm inner diameters (density 1.2 g/cm 3) and the Plexiglass chemical composition (8.05% H, 59.99% C, and 31.96% O), (iii) source-to-detector distance 2 mm, and (iv) sample density 1.25 g/cm 3 with sample thickness 25 mm.

2.3. Determinations of specific activities

Spectrum data were converted into specific activities of ^{210}Pb and ^{137}Cs in the target samples (Bq kg $^{-1}$) with established efficiency curves derived from different SRMs using Genie-2000 spectrum analysis software.

A precise determination of low levels of FRNs requires a highly stable HPGe detector and a standard source material containing the FRNs in order to calibrate energy and efficiency. Moreover, an intercomparison of results obtained from several international laboratories is essential. This was achieved by cooperation between our laboratory (Analytical Laboratory for Measurement of Environmental Radioactivity, ALMERA, of the Chinese Academy of Agricultural Science, CAAS) and the State Key Laboratory of Environmental Geochemistry, Institute of Geochemistry, Chinese Academy of Sciences (SKLEG/IGC, CAS). We quantified FRNs in parallel and, as a result, corrected our HPGe γ -spectrometry system efficiency. The standard reference source material containing FRNs (Catalog No. 7500, Source No 586-26-6) used by SKLEG/IGC, CAS was produced by the Isotope Products Laboratories, USA. CAAS-ALMERA Laboratory achieved a high score in the ^{137}Cs and ^{210}Pb determination proficiency test organized by IAEA in 2006 (Table 2).

2.4. Data analysis

We analyzed results of the radioactivity measurements in the target soil (Chestnut soil) using different efficiency calibration curves constructed from five soil types that served as the SRMs (Chestnut soil, Cinnamon soil, Yellow soil, Loessial soil, and Purple soil). A partial correlation analysis was used to relate the measured

Table 1
Soils used as standard reference materials (SRMs) and their chemical composition.

Soil type ^a	Element concentration (%)								
	O	Si	Al	Fe	Ca	K	Na	Mg	Ti
Chestnut soil (<i>Kastanozems</i>)	50.02	34.69	5.93	1.68	1.00	2.47	1.56	0.25	0.16
Cinnamon soil (<i>Lixisols</i>)	49.08	32.26	7.21	3.89	2.29	2.02	1.43	1.36	0.46
Yellow soil (<i>Ferralsols</i>)	49.16	31.73	11.27	3.73	0.24	2.51	0.41	0.47	0.49
Loessial soil (<i>Calcisols</i>)	51.35	27.52	6.58	3.26	5.37	1.76	1.39	1.33	0.37
Purple soil (<i>Cambisols</i>)	48.22	32.22	8.68	4.47	1.34	2.15	1.32	1.10	0.41

^a Names in parentheses are soil types according to the WRB soil classification system (ISSS/ISIC/FAO, 1998).

Table 2
Proficiency test on the determination of ^{137}Cs and ^{210}Pb in spiked soil (IAEA-CU-2006-02).

Code	Radionuclide	IAEA		ALMERA-CAAS			Acceptance criteria				
		Value	Uncertainty	Value	Uncertainty	Relative bias	Trueness			Precision	
		(Bq kg ⁻¹)	(Bq kg ⁻¹)	(Bq kg ⁻¹)	(Bq kg ⁻¹)	(%)	A1	A2	Score ^a	P (%)	Score ^a
1	^{137}Cs	20.30	0.50	20.21	1.02	0	0.09	2.39	A	5.62	A
	^{210}Pb	289.00	7.00	284.76	29.23	-1	4.24	77.55	A	10.55	A
2	^{137}Cs	38.40	0.80	40.23	1.97	5	1.83	5.49	A	5.32	A
	^{210}Pb	530.00	11.30	513.67	52.63	-3	116.33	138.88	A	10.47	A
3	^{137}Cs	2.60	0.20	2.40	0.20	-8	0.20	0.73	A	11.34	A
	^{210}Pb	48.00	1.50	56.53	6.24	18	8.53	16.56	A	11.47	A
4	^{137}Cs	38.40	0.80	41.84	2.05	9	3.44	5.68	A	5.32	A
	^{210}Pb	530.00	11.30	541.91	55.61	2	11.91	146.41	A	10.48	A
5	^{137}Cs	20.30	0.50	20.32	0.97	0	0.02	2.82	A	5.37	A
	^{210}Pb	289.00	7.00	289.14	29.45	0	0.14	78.10	A	10.47	A

^a A means the acceptable results.

Table 3
Specific activities (mean \pm standard error of the mean) of FRNs in the target samples of Chestnut soil obtained with different SRMs and their deviations from the values obtained with Chestnut soil as a SRM ("reference results").

Target sample no.	SRM	^{210}Pb		^{137}Cs	
		Activity (Bq kg ⁻¹)	Deviation from the reference result (%)	Activity (Bq kg ⁻¹)	Deviation from the reference result (%)
1	Chestnut soil (<i>Kastanozems</i>)	27.22 \pm 2.25	0	7.29 \pm 0.28	0
	Cinnamon soil (<i>Lixisols</i>)	29.13 \pm 2.41	7.02	7.29 \pm 0.28	0.00
	Yellow soil (<i>Ferralsols</i>)	27.54 \pm 3.03	1.18	7.35 \pm 0.37	0.82
	Loessial soil (<i>Calcisols</i>)	27.65 \pm 3.08	1.58	7.32 \pm 0.37	0.41
	Purple soil (<i>Cambisols</i>)	29.37 \pm 2.43	7.90	7.29 \pm 0.28	0.00
2	Chestnut soil (<i>Kastanozems</i>)	66.37 \pm 4.55	0	12.80 \pm 0.43	0
	Cinnamon soil (<i>Lixisols</i>)	71.03 \pm 4.87	7.02	12.79 \pm 0.43	-0.08
	Yellow soil (<i>Ferralsols</i>)	67.16 \pm 6.71	1.19	12.90 \pm 0.61	0.78
	Loessial soil (<i>Calcisols</i>)	67.43 \pm 6.82	1.60	12.85 \pm 0.60	0.39
	Purple soil (<i>Cambisols</i>)	71.62 \pm 4.91	7.91	12.79 \pm 0.43	-0.08
3	Chestnut soil (<i>Kastanozems</i>)	68.61 \pm 4.82	0	37.40 \pm 1.16	0
	Cinnamon soil (<i>Lixisols</i>)	73.43 \pm 5.16	7.03	37.37 \pm 1.16	-0.08
	Yellow soil (<i>Ferralsols</i>)	69.43 \pm 7.01	1.20	37.67 \pm 1.70	0.72
	Loessial soil (<i>Calcisols</i>)	69.71 \pm 7.13	1.60	37.53 \pm 1.69	0.35
	Purple soil (<i>Cambisols</i>)	74.04 \pm 5.20	7.91	37.37 \pm 1.16	-0.08
4	Chestnut soil (<i>Kastanozems</i>)	111.68 \pm 7.77	0	34.71 \pm 1.13	0
	Cinnamon soil (<i>Lixisols</i>)	119.50 \pm 8.31	7.00	34.68 \pm 1.13	-0.09
	Yellow soil (<i>Ferralsols</i>)	112.99 \pm 11.34	1.17	34.96 \pm 1.61	0.72
	Loessial soil (<i>Calcisols</i>)	113.45 \pm 11.53	1.58	34.83 \pm 1.61	0.35
	Purple soil (<i>Cambisols</i>)	120.49 \pm 8.38	7.89	34.68 \pm 1.13	-0.09
5	Chestnut soil (<i>Kastanozems</i>)	150.34 \pm 9.95	0	36.47 \pm 1.13	0
	Cinnamon soil (<i>Lixisols</i>)	160.89 \pm 10.64	7.02	36.44 \pm 1.13	-0.08
	Yellow soil (<i>Ferralsols</i>)	152.12 \pm 14.94	1.18	36.73 \pm 1.66	0.71
	Loessial soil (<i>Calcisols</i>)	152.74 \pm 15.19	1.60	36.59 \pm 1.65	0.33
	Purple soil (<i>Cambisols</i>)	162.22 \pm 10.73	7.90	36.43 \pm 1.13	-0.11
6	Chestnut soil (<i>Kastanozems</i>)	174.62 \pm 11.45	0	28.65 \pm 0.91	0
	Cinnamon soil (<i>Lixisols</i>)	186.88 \pm 12.25	7.02	28.63 \pm 0.91	-0.07
	Yellow soil (<i>Ferralsols</i>)	176.69 \pm 17.29	1.19	28.86 \pm 1.31	0.73
	Loessial soil (<i>Calcisols</i>)	177.42 \pm 17.58	1.60	28.75 \pm 1.31	0.35
	Purple soil (<i>Cambisols</i>)	188.43 \pm 12.35	7.91	28.62 \pm 0.91	-0.10

specific activities of the FRNs in the target soil with percentages of chemical elements in the SRMs. In a multiple linear regression analysis, partial correlation analysis establishes a correlation between one independent variable and a dependent variable in the presence of several independent variables. A larger absolute value of the partial correlation coefficient suggests a stronger linear correlation between the two variables and vice versa. All statistical analyses were performed using Statistical Analysis System (SAS) software (SAS Institute, 1990).

3. Results

3.1. Variations of FRN radioactivity measurements obtained with different SRMs

The measured specific activities of ^{210}Pb and ^{137}Cs in the target soil samples (Chestnut soil) depended on the soil types used as SRMs (Table 3). The measured specific activities decreased in the following order of the standard soils: Purple soil > Cinnamon

Table 4
Partial correlation coefficients between the specific activities of ^{210}Pb and ^{137}Cs obtained with different SRMs and the concentrations of selected chemical elements in the SRMs.

Target sample no.	^{210}Pb	^{137}Cs				
		Si	Fe	Na		
1	<i>r</i>	0.975	0.997	0.989	0.982	−0.996
	<i>t</i> -value	4.364	12.420	6.828	7.393	15.384
	<i>p</i> -value	0.049	0.006	0.021	0.005	0.001
2	<i>r</i>	0.975	0.997	0.990	0.993	−0.998
	<i>t</i> -value	4.379	12.517	6.869	12.093	22.052
	<i>p</i> -value	0.048	0.006	0.021	0.001	0.000
3	<i>r</i>	0.975	0.997	0.990	0.994	−0.998
	<i>t</i> -value	4.370	12.512	6.863	12.985	24.005
	<i>p</i> -value	0.049	0.006	0.021	0.001	0.000
4	<i>r</i>	0.975	0.997	0.990	0.994	−0.998
	<i>t</i> -value	4.390	12.526	6.890	12.881	23.618
	<i>p</i> -value	0.048	0.006	0.020	0.001	0.000
5	<i>r</i>	0.975	0.997	0.990	0.998	−0.999
	<i>t</i> -value	4.360	12.469	6.851	22.253	40.633
	<i>p</i> -value	0.049	0.006	0.021	0.000	0.000
6	<i>r</i>	0.975	0.997	0.990	0.999	−1.000
	<i>t</i> -value	4.367	12.512	6.877	32.447	58.884
	<i>p</i> -value	0.049	0.006	0.020	0.000	0.000

soil > Loessial soil > Yellow soil > Chestnut soil for ^{210}Pb and Yellow soil > Loessial soil > Chestnut soil > Cinnamon soil > Purple soil for ^{137}Cs .

Changes in composition of SRMs had a greater effect on the ^{210}Pb results than on the ^{137}Cs results. Cinnamon soil and Purple soil used as SRMs produced 7% and 8% higher specific activities of ^{210}Pb , respectively, than the target soil used in that capacity. By contrast, both the Yellow soil and Loessial soil resulted in less than 2% higher ^{210}Pb activities. Unlike in the case of ^{210}Pb , the type of SRM did not affect the measured specific activities of ^{137}Cs . In fact, the differences in specific activities of ^{137}Cs obtained with different SRMs were less than 0.9% of the target values (Table 3).

3.2. Partial correlations between specific activities of FRNs obtained with different SRMs and the chemical composition of the SRMs

We calculated the partial correlation coefficients (significant at $p < 0.05$ to $p < 0.01$) between specific activities of ^{210}Pb and ^{137}Cs obtained with the efficiency curves based on different SRMs, and the chemical composition of the SRMs (Table 4). The specific activities of ^{210}Pb were positively correlated with the concentrations of Si and Na (significant at $p < 0.05$) and the concentration of Fe (significant at $p < 0.01$) (Table 4). In other words, the measured ^{210}Pb activity in the target samples increased with concentrations of Si, Na, and Fe in the SRMs used. By contrast, ^{137}Cs activities were not significantly correlated with concentrations of either Si or Fe in the soils (data not shown), but were positively correlated with the concentration of O (at $p < 0.01$ to $p < 0.001$) and negatively correlated with the concentration of Na (at $p < 0.001$).

4. Discussion

The results of this study (Table 3) support our hypothesis that an inappropriate use of SRMs for constructing an effective calibration curve deteriorates the accuracy of measurements of ^{210}Pb activities in environmental samples. In addition to the effects of self-absorption due to the sample geometry reported by Li and Geng (2010), the differences in the chemical composition between the

target soil and the four other soil types used as SRMs (Table 1) could explain the variability in the measured ^{210}Pb activities. The target soil used in the study differed from the soils used as SRMs in the concentrations of several elements, such as Si, Na, and, particularly, Fe (94–166%, Table 1), which were positively correlated with the ^{210}Pb activities found in the corresponding SRMs (at $p < 0.05$ to $p < 0.01$ level; Table 4). In general, an element with a big atomic number may have a greater effect on the determination of gamma rays than that with a small atomic number. As ^{210}Pb is produced from soils and rocks (Zapata and Nguyen, 2010), the determination of ^{210}Pb may be more affected by Fe, than Si and Na in environmental samples. Not all of the possible chemical elements contained in soil samples were investigated in this study; further research is required to ensure the appropriate use of SRMs for the efficient use of gamma-ray detectors in the determination of ^{210}Pb activities in a wide range of environmental samples.

Unlike in the case of ^{210}Pb , variations of the SRMs used for the efficiency calibration had insignificant effects on the determined ^{137}Cs activities (Table 3). The small differences (Table 1) in concentrations of O and Na between the target soil and the four other soil types that served as the SRMs, which affected ^{137}Cs activities (Table 4), could explain these insignificant effects. These results can probably be attributed to the fact that, unlike ^{210}Pb , ^{137}Cs is an artificial radionuclide generated by global atomic bomb tests and absorbed by clay minerals (Zapata and Nguyen, 2010), and the accuracy of its determination is therefore unlikely to be affected by chemical composition of the SRMs. It is more likely that the geometry and bulk density of the SRMs, as well as sample thickness play a more significant role (Li et al., 2008; Li and Geng, 2010).

The significant differences in ^{210}Pb activities in the same soil measured with different SRMs illustrate the need to improve the procedure for measuring ^{210}Pb in environmental samples. For example, when preparing efficiency curves, one should try to choose SRMs of the same chemical composition and shape as the target samples. As there is a high cost in making SRMs similar to the target samples, it may be more efficient to apply the LabSOCS technique when comparing the results obtained with efficiency curves created from different SRMs (Li and Geng, 2010). With this consideration in mind, we have launched a program to create relative SRMs for the participating countries in the Asia-Pacific region with support from the IAEA (no. RAS5043-004-005E). The objective is to establish relative SRMs for the determination of ^{210}Pb that would be related to the main soil types in the participating countries through the use of the LabSOCS technique. This campaign requires each participating country to provide our laboratory with 10 samples of 5 soil types (2 replicate samples for each soil type) that are representatives of their countries. The soil type according to the US or ISSS/ISIC/FAO soil classification system and its chemical composition in terms of major elements should be also provided. We will return the soil samples to the participating countries along with the results of ^{210}Pb measurements analyzed with the LabSOCS technique. Participating countries in the Asia-Pacific region will then be able to use these samples as adequate reference materials for the determination of ^{210}Pb in their soil samples of the same type.

5. Conclusions

Variations in the type of soil used as SRM resulted in significant variations in the measured ^{210}Pb specific activities, but insignificant variations in the measured ^{137}Cs specific activities were due to the differences in concentrations of Si, Fe, and Na, which affect results of ^{210}Pb measurements, between the target soil and the four other soil types used as SRMs, which were much bigger than the differences in the concentrations of O and Na, which affect the results of ^{137}Cs measurements. Our results demonstrated that

accurate determination of ^{210}Pb and ^{137}Cs depends on a similarity in the concentrations of several elements in the SRMs and in the target soil. The significant variation in the measured ^{210}Pb activities in the same soil obtained using different SRMs shows that it is important to use SRMs similar in chemical composition to those of the targeted samples.

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