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A Procedure for the Determination of Po-210 in Water Samples by Alpha Spectrometry

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A PROCEDURE FOR THE DETERMINATION OF Po-210 IN WATER SAMPLES BY ALPHA SPECTROMETRY

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FOREWORD

Reliable, comparable and ‘fit for purpose’ results are an essential requirement for any decision based on analytical measurements. For the analyst, the availability of tested and validated analytical procedures is a extremely important tool for production of such analytical measurements. For maximum utility, such procedures should be comprehensive, clearly formulated, and readily available to both the analyst and the customer for reference.

Since 2004 the Environment programme of the IAEA has included activities aimed at the development of a set of procedures for the determination of radionuclides in terrestrial environmental samples. In the case of ^{210}Po, this started with the collection and review of about 130 papers from the scientific literature. Based on this review, two candidate methods for the chemical separation of ^{210}Po from water samples were selected for testing, refinement and validation in accordance with ISO guidelines. A comprehensive methodology for calculation of results including quantification of measurement uncertainty was also developed. This report presents the final procedure which was developed based on that work.

The IAEA wishes to thank all the participants for their valuable contributions to develop the procedure and review the document. The IAEA officer responsible for this publication was C.-K. Kim of the Agency’s Laboratories, Seibersdorf, Austria.

CONTENTS

1. INTRODUCTION

Polonium-210 is a naturally occurring alpha emitter and exists in the environment as a result of the ^{210}Pb decay within the ^{238}U decay chain (Fig. 1). Polonium has 25 known radioactive isotopes with mass numbers of 192 to 218, of which only the 208, 209 and 210 isotopes have half-lives longer than 1 day (Table 1). Of these three, it is ^{210}Po which is of most interest from an environmental impact viewpoint, and its measurement is the subject of this report.

^{210}Po is considered to be one of the most toxic naturally occurring radionuclides [1], and one of the most important environmental radionuclides due to its wide distribution and potential for human radiation exposure through ingestion and inhalation [2-3]. In addition, for many sample types (for example, soils and sediments) determination of ^{210}Po is commonly used as a means of determining its progenitor ^{210}Pb.

The results of a recent proficiency test organized by the IAEA demonstrated that a number of laboratories are experiencing difficulties in obtaining reliable results for ^{210}Po determination in water samples [4]. This is most likely due in part to the limited number of methods available for its determination, the most commonly used being alpha-particle spectrometry [5]. In addition, there is a general lack of suitable, recently characterized reference materials for use in quality control.

As a part of its activities to support its Member State laboratories, the IAEA is developing a set of procedures for determination of selected radionuclides in environmental samples. In the case of ^{210}Po, this started with the collection and review of about 130 papers from the scientific literature [5]. Based on this review, two candidate methods for determination of ^{210}Po in water samples were selected for testing, refinement and validation. The results of this work are the subject of this report.

For determination of a low level activity concentration of ^{210}Po in a water sample, it is necessary to handle a large volume of sample. Direct evaporation of water samples has been used for reducing a small volume as a simple process [6-7]. However, this method is very time consuming for a large amount of a sample (> 1 L). Therefore, co-precipitation with iron hydroxide [8-9], or MnO_2 [10-12] have commonly been used to preconcentrate Po from water samples. When iron hydroxide is used for preconcentration, it is necessary to remove Fe from the sample solution using a solvent extraction step with an extractant such as di-isopropyl ether [8], because bulk iron may interfere with the purification of Po using solvent extraction or extraction chromatography as well as in the auto-deposition of Po. In the case that MnO_2 is used, manganese can be removed easily from Po by several alternative chemical separation procedures. Therefore, in this work MnO_2 co-precipitation was selected as the method for preconcentration.

Two Po separation procedures, based on diethylammonium diethyldithiocarbamate (DDTC) solvent extraction (DDTC-SE) [12] and extraction chromatography using Sr-resin (Sr-EC) [13], were selected for testing. Reports in the literature indicate that both methods have been successfully used for determination of ^{210}Po in a variety of sample matrices [7, 14-16]. The method validation of both methods was carried out in terms of trueness, repeatability and reproducibility with tap water spiked with a known amount of ^{210}Po.

Fig. 1. The uranium decay series.

TABLE 1. RELEVANT DECAY DATA FOR ^{208}Po, ^{209}Po AND ^{210}Po

Radionuclides	Half-life[1] ($T_{1/2}$)	Disintegration modes	E_α (MeV)	Intensity (%)	Reference
^{208}Po	2.898 y (2)	α : 99.99777%	5.1149(14)	99.9956(4)	[20]
			4.220(15)	2.4×10^{-4}(7)	
		ε : 4.0E-3%			
^{209}Po	102 y (5)	α : 99.52%	4.885(2)	19.7(20)	[20]
			4.883(2)	79(8)	
			4.622(5)	0.551(6)	
			4.310(15)	1.5×10^{-4}(4)	
			4.110(15)	5.6×10^{-4}(4)	
		ε : 0.48%			
^{210}Po	138.376 d (2)	α : 100%	5.40746(7)	99.99876(4)	[23]
			4.60436(9)	1.24×10^{-3}(4)	

α: α-decay, ε: electron capture,

[1] The number in brackets gives the uncertainty of the last digit.

2. SCOPE

This report describes a method for measuring ^{210}Po in freshwater by alpha-particle spectrometry using chemical separation techniques. The method validation was carried out using spiked tap water which has similar components as given in Table 2. The detection limit of the method is 2 mBq L^{-1}, assuming the counting efficiency is 25%, the counting time is 250,000 s and 0.5 L of sample is analysed. Sample volumes up to 10 L can be processed by this method. The method was validated in an activity range of 10 to1200 mBq L^{-1} of ^{210}Po.

TABLE 2. SEMI-QUANTITATIVE ANALYTICAL RESULT OF ELEMENTS IN THE TAP WATER OF IAEA'S LABORATORIES, SEIBERSDORF, AUSTRIA BY ICP-MS

Elements	Concentration (µg L^{-1})
Ca	93.1 x 10^{3}
Mg	26.0 x 10^{3}
Na	9.51 x 10^{3}
K	7.04 x 10^{3}
Al	2.80
As	0.59
Ba	54.4
Br	103
Cr	15.5
Cu	5.16
Mn	1.20
Pb	0.15
Se	2.01
Si	1.08 x 10^{3}
Sr	493
U	11.3
Zn	56.4

3. NORMATIVE REFERENCES

- ISO 18589-4 (Draft), 2007. Measurement of radioactivity in the environment – Soil – Part 4: Measurement of plutonium isotopes (plutonium 238 and plutonium 239+240) by alpha spectrometry.
- ISO 11929-7, 2005. Determination of the detection limit and decision threshold for ionizing radiation measurement – Part 7: Fundamentals and general applications.
- ISO/TS 21748, 2004. Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation
- ISO/IEC Guide 98-3:2008. Guide to the expression of uncertainty in measurement.
- ISO 5725-2, 1994. Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method
- ISO 5725-1, 1994. Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions
- ISO 5725-4, 1994. Accuracy (trueness and precision) of measurement methods and results - Part 4: Basic methods for the determination of the trueness of a standard measurement method
- ISO 7870, 1993. Control charts – General guide and introduction
- ISO 7966, 1993. Acceptance control charts

4. RESPONSIBILITIES

It is the responsibility of the analyst to follow this procedure and to establish appropriate safety and health practices. Compliance with national and internal rules for chemical and radiological safety is obligatory.

5. TERMS AND DEFINITIONS, SYMBOLS AND ABBREVIATIONS

In the following, $u(x)$ indicates the uncertainty in quantity x:

$c_{Po,0}$ and $u(c_{Po,0})$: activity concentration of ^{210}Po on the sampling date [Bq L^{-1}]
$c_{Pb,0}$ and $u(c_{Pb,0})$: activity concentration of ^{210}Pb on the sampling date [Bq L^{-1}]
$c_{Po,1}$ and $u(c_{Po,1})$: activity concentration of ^{210}Po on the separation date [Bq L^{-1}]
a_T and $u(a_T)$: certified specific activity of ^{209}Po tracer on the reference date [Bq g^{-1}]
a^*: decision threshold [Bq L^{-1}]
$a^\#$: detection limit [Bq L^{-1}]

c: certified activity concentration of ^{210}Po in the spiked sample [Bq L^{-1}]

f_T and $u(f_T)$: correction factor for decay of ^{209}Po tracer during elapsed time between the reference date of the tracer and the separation date of Po

$f_{2\text{-}Po}$ and $u(f_{2\text{-}Po})$: correction factor for decay of ^{210}Po during elapsed time between the separation date of ^{210}Po and the beginning of the measurement

$f_{3\text{-}Po}$ and $u(f_{3\text{-}Po})$: correction factor for decay of ^{210}Po over the counting time

m_T and $u(m_T)$: mass of ^{209}Po tracer solution added [g]

V_S and $u(V_S)$: sample volume [L]

R and $u(R)$: chemical recovery of Po

R_L: Reproducibility limit [Bq L^{-1}]

r_L: Repeatability limit [Bq L^{-1}]

ROI: region of interest

r_n and $u(r_n)$: net count rates in ROIs of ^{210}Po [s^{-1}]

r_{nt} and $u(r_{nt})$: net count rates in ROIs of ^{209}Po [s^{-1}]

r_g and $u(r_g)$: gross count rates in ROIs of ^{210}Po [s^{-1}]

r_{gt} and $u(r_{gt})$: gross count rates in ROIs of ^{209}Po [s^{-1}]

r_0 and $u(r_0)$: count rates of blank in ROIs of ^{210}Po [s^{-1}]

r_{0t} and $u(r_{0t})$: count rates of blank in ROIs of ^{209}Po tracer [s^{-1}]

S_r : standard deviation of repeatability [Bq L^{-1}]

S_R : standard deviation of reproducibility [Bq L^{-1}]

t_0 : counting time of the blank [s]

t_T : time interval between the reference date for ^{209}Po tracer and the separation date [d]

$t_{1\text{-}Po}$: time interval between the sampling date and the separation date [d]

$t_{2\text{-}Po}$: time interval between the separation date of ^{210}Po and the start of the measurement [d]

$t_{3\text{-}Po}$: counting time of the sample [d]

$\overline{X}_r$: mean of measurement values in the repeatability test [Bq L^{-1}]

$\overline{X}_R$: mean of measurement values in the reproducibility test [Bq L^{-1}]

X_i: individual measurement value of the spiked sample [Bq L^{-1}]

λ_{Pb} and $u(\lambda_{Pb})$: decay constant of ^{210}Pb [d^{-1}]

λ_T and $u(\lambda_T)$: decay constant of ^{209}Po tracer [d^{-1}]

λ_{Po} and $u(\lambda_{Po})$: decay constant of ^{210}Po [d^{-1}]

δ: relative bias of the test method [%]

ε: counting efficiency of the α-particle detector

6. PRINCIPLE

The activity concentration of ^{210}Po is determined by alpha-particle spectrometry. The procedure involves the following major steps:

1. Sample preparation, including co-precipitation with MnO_2 in order to concentrate the Po from the bulk sample.
2. Chemical separation and purification of the polonium in order to avoid interference from natural or artificial alpha emitters and stable elements in the test sample.
3. Source preparation by auto-deposition of Po onto a silver disk.
4. Measurement in an alpha spectrometer.
5. Calculation of the analytical result and combined uncertainty.

The chemical separation of polonium is carried out by one of two alternative methods: (a) DDTC (Diethylammonium diethyldithiocarbamate) solvent extraction [12], or (b) Sr-resin extraction chromatography [13]. The chemical separation step improves the reliability of the procedure in terms of both Po recovery and spectrum peak resolution by removing interfering elements present in the sample.

Auto-deposition onto silver disks is the most commonly used form of source preparation for determination of ^{210}Po by alpha-particle spectrometry as it is simple and ensures separation of Po from other alpha-emitting radionuclides and matrix elements which may not have been completely separated in the chemical separation step. The auto-deposition of Po is also possible onto disks of copper, stainless steel, or nickel but with a drop in deposition efficiency of about 7-8% under the same conditions [17].

7. CHEMICAL REAGENTS AND EQUIPMENT

7.1. Tracers

The two tracers commonly used for ^{210}Po determination are ^{208}Po and ^{209}Po. ^{209}Po has a clear advantage over ^{208}Po in energy separation from the ^{210}Po peak (Table 1) and is the preferred tracer, if available. It should also be noted that ^{208}Po solutions normally contain trace activities of ^{209}Po. Unfortunately the long half-life of ^{209}Po has a disadvantage in relation to detector contamination. Due to its volatility, atoms of polonium readily leave the source disk in the vacuum of the counting chamber, thereby a significant fraction can drift to the detector. Otherwise, ^{208}Po solution normally contains trace activity of ^{209}Po.

7.2. Equipment

- Alpha spectrometer (PIPS detector)
- Vacuum pump
- Auto-deposition system
- Analytical balances with accuracies of (a) 0.1 mg and (b) 0.01 g
- Hot plate
- Centrifuge
- Silver disk (Ø: 17 mm)
- Water bath with temperature controller

7.3. Chemical reagents

- 0.2 mol L^{-1} $KMnO_4$
- 25 % NH_4OH
- 6 mol L^{-1} NaOH
- 0.3 mol L^{-1} $MnCl_2$
- H_2O_2 (30 %)
- 1 % H_2O_2 in 2 mol L^{-1} HCl
- 1 % H_2O_2 in 5 mol L^{-1} HCl
- HCl (32 %)
- 5 mol L^{-1} HCl
- 2 mol L^{-1} HCl
- 0.5 mol L^{-1} HCl
- HNO_3 (65 %)
- 1 mol L^{-1} HNO_3
- 0.1 % DDTC (diethylammonium diethyldithiocarbamate) in CH_3CCl_3
- ^{208}Po or ^{209}Po tracer
- ^{210}Po certified standard solution
- Ascorbic acid

The preparation of chemical reagents is described in Annex. All the chemical reagents needed to carry out this procedure must be analytical grade.

8. PRECAUTIONS

The time delay between sample collection and analysis should be kept as short as possible. In the case that the sample ^{210}Po/^{210}Pb activity ratio is of the order of 0.1, the separation of ^{210}Po from ^{210}Pb should be carried out within at most 15 days after sampling in order to achieve a 10 % relative standard uncertainty (for explanation see Section 10.2.).

9. PROCEDURE

9.1. Sample preparation using MnO_2 co-precipitation *(see Fig. 2)*

The MnO_2 precipitation method given here is that of Bojanowski et al [18].

a) Filter sample with a membrane filter (pore size: 0.45 μm) and acidify a known aliquot of sample with 32 % HCl to pH≤2.
b) Add the tracer, vigorously stir for 2 hours to ensure tracer equilibrium.
c) Add 1 mL of 0.2 mol L^{-1} $KMnO_4$ and 1 mL of 0.3 mol L^{-1} $MnCl_2$ when the sample volume is 0.5 – 2 L or 0.2 mL of each reagent, if smaller volumes are analyzed (0.1 – 0.5 L). Sample volumes up to 10 L can be processed when the amounts of reagents are scaled up accordingly and stirred for one hour.
d) Adjust the pH to 8 – 9 with 25 % NH_4OH.

e) Allow the precipitate to settle (overnight).
f) Remove the supernatant by suction or decantation.
g) Collect the precipitate by centrifuging or filtration.
h) Dissolve the precipitate with 20 mL of 1 % H_2O_2 in
    (a) 5 mol L^{-1} HCl for DDTC solvent extraction, or
    (b) 2 mol L^{-1} HCl for Sr-resin extraction chromatography.
i) Gently heat the sample solution covered with a watch glass on a hot plate for 30 min to decompose H_2O_2.
j) Prepare at least one blank for each batch of samples with ^{210}Po free deionized water. The blank should be treated by the procedure described in steps a) to i).
k) Proceed to step 9.2. Chemical separation.

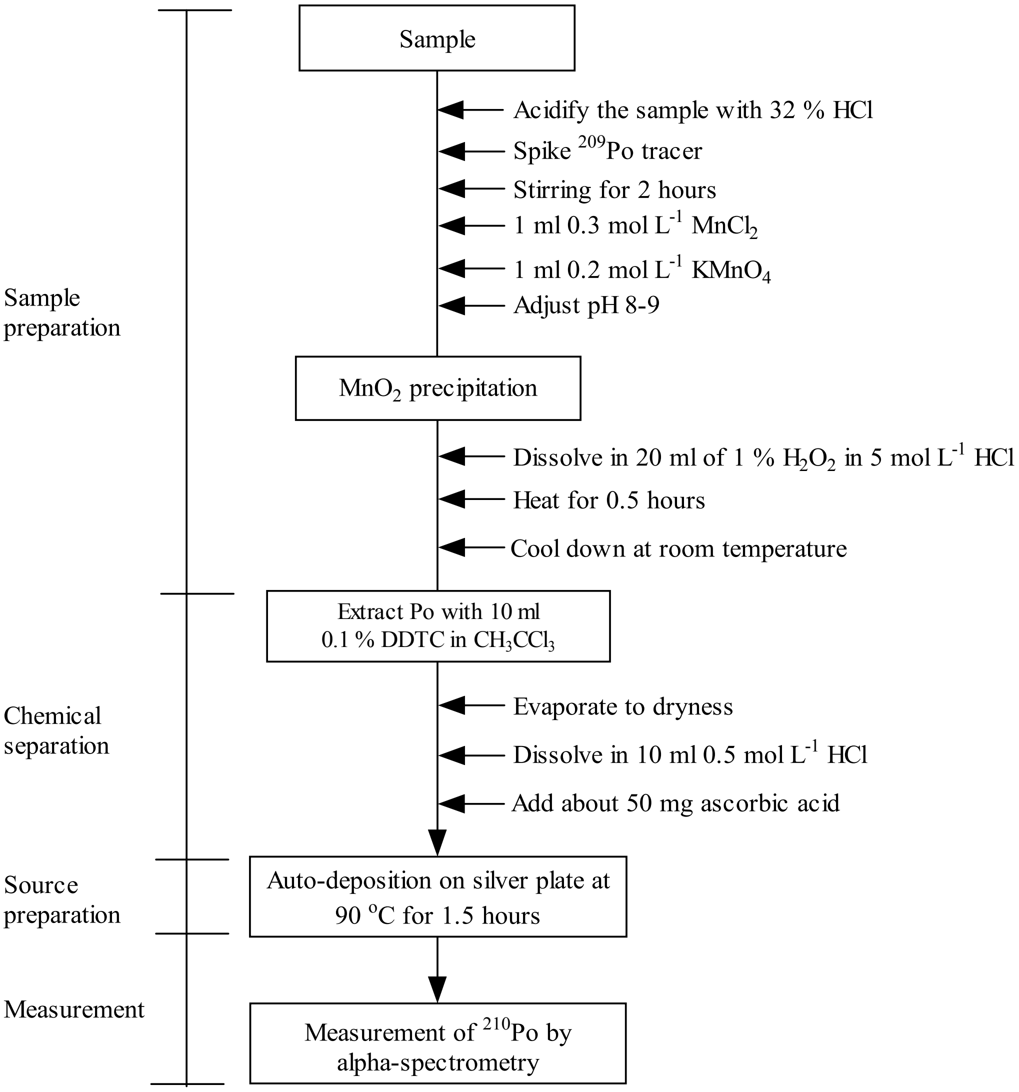

Fig. 2. A flow chart for chemical separation of Po by DDTC solvent extraction.

9.2. Chemical separation

Chemical separation involves use of one of the two following alternative procedures. The date of separation must be recorded as it is the date of separation of ^{210}Po from its parents ^{210}Pb and ^{210}Bi.

9.2.1. Solvent (DDTC) extraction (see Fig. 2)

a) Transfer the sample solution obtained from step 9.1. into a separate funnel and wash the beaker with 2 x 5 mL of 5 mol L^{-1} HCl to give a total volume of 30 mL.
b) Add a small amount (10-50 mg) of ascorbic acid to the sample solution until the disappearance of the yellow color (reduce Fe^{3+} to Fe^{2+}), if the sample solution is yellow colored.
c) Extract Po with 10 mL of 0.1 % DDTC in CH_3CCl_3 by shaking for 2 minutes.
d) Extract twice with 20 mL of 0.1 % DDTC in CH_3CCl_3. If the organic phase is colored, extract with further aliquots until it is colorless.
e) Combine the DDTC/ CH_3CCl_3 portions.
f) Evaporate to dryness at a temperature less than 100 °C.
g) Record the separation date of Po from Bi and Pb.
h) Radioisotopes of Pb, Ra, Th, U, and Ac may be determined in the remaining aqueous phase.
i) Carefully add 2 mL of 65 % HNO_3.
j) Heat the solution covered with a watch glass for ~15 min and then evaporate to dryness.
k) Dissolve the residue in 10 mL of 0.5 mol L^{-1} HCl.
l) Proceed to step 9.3. Source preparation.

9.2.2. Extraction chromatography (Sr-resin) (see Fig. 3)

a) Soak 3 g of Sr-resin (100 – 150 µm) in distilled water for 1 – 2 h, then fill into a chromatographic column (inner diameter 10 mm x 10 cm length) and wash with 100 mL of 1 mol L^{-1} HNO_3. The column is preconditioned with 100 mL of 2 mol L^{-1} HCl. To regenerate a used column, wash the column with 100 mL of distilled water, 100 mL of EDTA (pH adjusted to 7 with NH_4OH) and 100 mL of distilled water before preconditioning with 100 mL of 2 mol L^{-1} HCl.
b) Load the sample solution obtained from step 9.1. on the Sr column preconditioned in advance and rinse the beaker with 50 mL of 2 mol L^{-1} HCl and pass the washing solution through the Sr column.
c) Wash the column with 50 mL of 2 mol L^{-1} HCl.
d) Elute sequentially Po with 60 mL of 6 mol L^{-1} HNO_3 and Pb with 60 ml of 6 mol L^{-1} HCl.
e) Record the separation date of Po from Bi and Pb.
f) Evaporate the Po fraction nearly to dryness at about 120 °C.
g) Add 1 mL of 65 % HNO_3, and a few drops of 30 % H_2O_2, leave to dryness.
h) Add 1 mL of 65 % HNO_3 and 1 mL of 32 % HCl and then evaporate to dryness.
i) Add 1 mL of 65 % HNO_3 and then evaporate to dryness.
j) Repeat the steps g) – i) until a small amount of organic material disappears.
k) Dissolve the residue in 10 mL of 0.5 mol L^{-1} HCl.
l) Proceed to step 9.3. Source preparation.

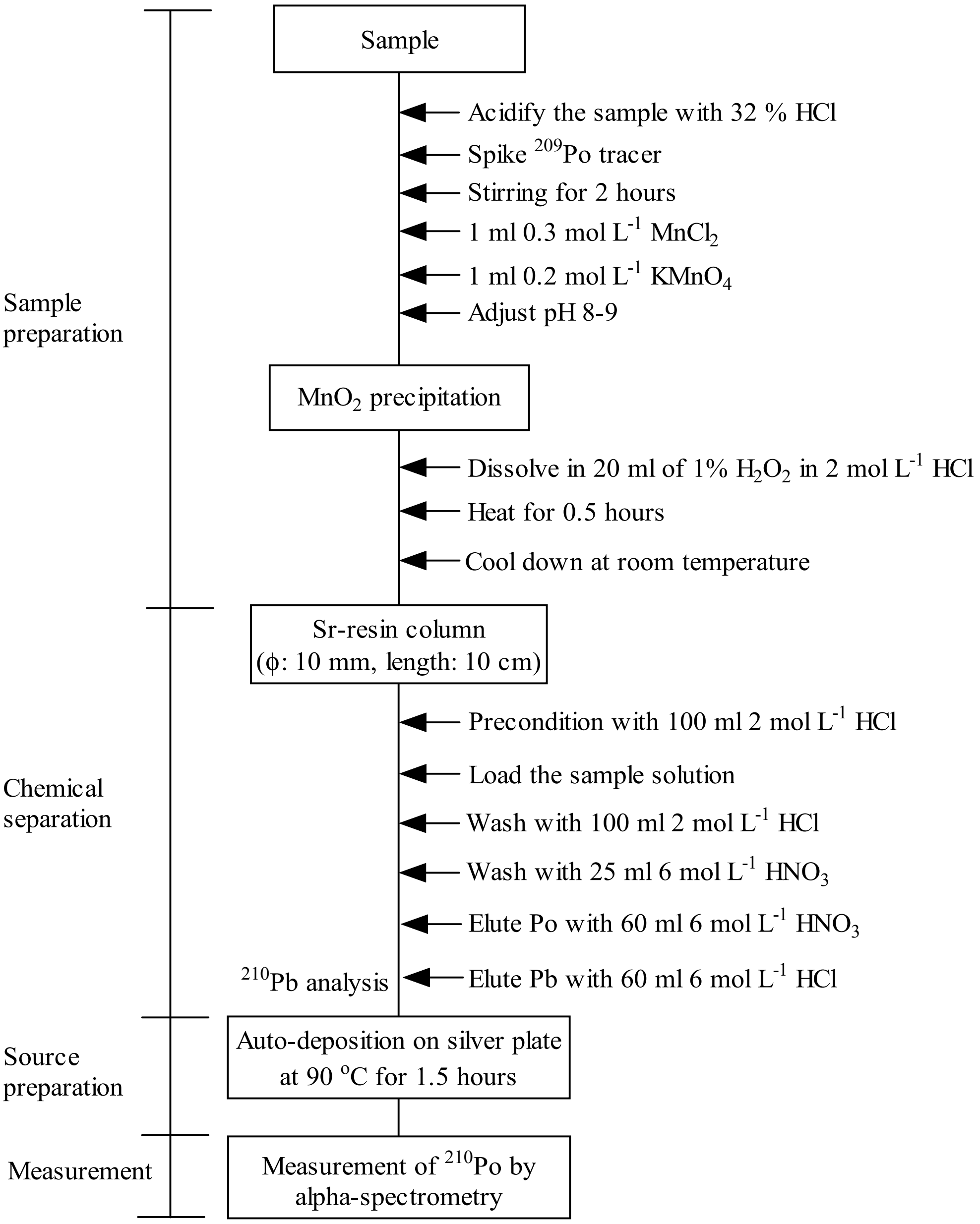

Fig. 3. A flow chart for chemical separation of Po by Sr-resin extraction chromatography.

9.3. Source preparation

a) Place the clean and degreased silver disk (active area: 133 mm^2) into the cap assembly of the deposition cell (Teflon vial) *(see Fig. 4).*
b) Screw the Teflon vial with the cap assembly.
c) Fill the deposition cell with water to check for leaks.
d) Empty the deposition cell.
e) Add ~ 50 mg of ascorbic acid to the solution obtained from step 9.2.1. or 9.2.2. to reduce Fe^{3+} to Fe^{2+}.
f) Transfer the solution to the deposition cell.
g) Rinse the beaker with 2 x 2 mL of 0.5 mol L^{-1} HCl.
h) Adjust the pH to 1 – 2 with 5 mol L^{-1} HCl and 6 mol L^{-1} NaOH.
i) Place the deposition cell into a water bath at 90 °C for 90 min with mechanical stirring or air bubbling.

j) Remove the silver disk, wash with distilled water and dry.
k) Proceed to step 9.4. Measurement.

9.4. Measurement

The ^{210}Po activity concentration is calculated by counting the sample source for an appropriate time. The counting time depends on the sample and blank count rates and also the detection limit and decision threshold required. The alpha spectra should be inspected to confirm good peak resolution and no interfering peaks.

a) Introduce the source into the vacuum chamber and pump until a vacuum of about 0.5 Pa (5 x 10^{-3} mbar) is obtained. Then switch on the bias voltage to the detector.
b) Record the file name, sample information (e.g. code number, sample name, sample mass, etc.).
c) Integrate the counts in the ROIs of tracer and ^{210}Po. The alpha spectrum should be similar to the one illustrated in Fig. 5. The FWHM of both peaks shall be typically in the range of 25-30 keV for a 450 mm^2 area detector.
d) Integrate the counts in the same ROIs of the sample spectrum in the blank spectrum.

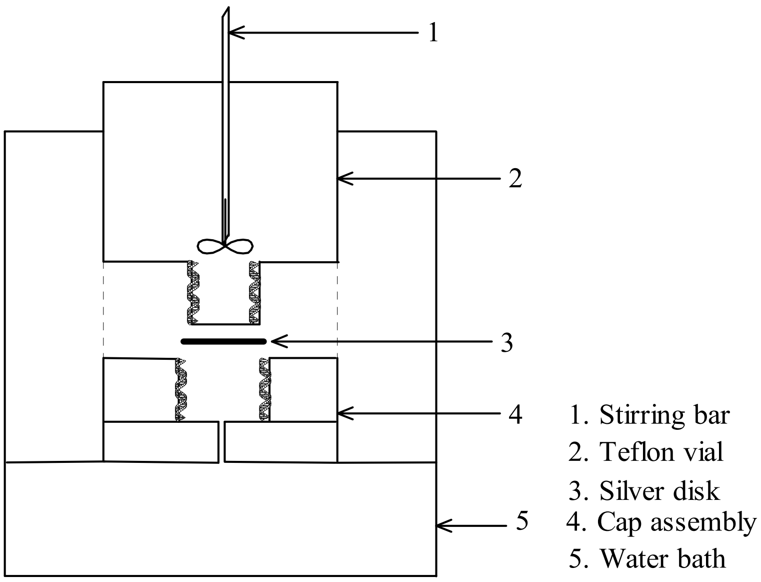

Fig. 4. Diagram of an auto-deposition cell.

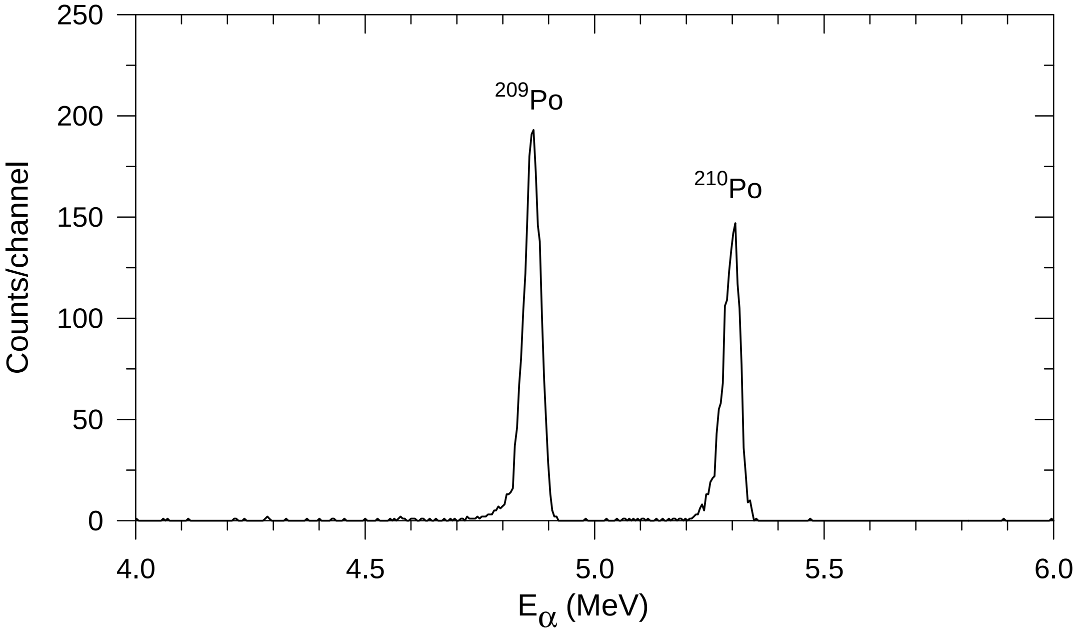

Fig. 5. Typical polonium alpha spectrum with ^{209}Po yield tracer.

10. EXPRESSION OF RESULTS

10.1. Calculation of the activity concentration and the combined uncertainty of ^{210}Po on the separation date

The ratio of the net count rates of the ^{209}Po and ^{210}Po peaks is used to calculate the activity concentration of ^{210}Po in the sample on the date of chemical separation, taking into account the specific activity of the tracer solution, the volume of the sample and the mass of the tracer solution used, the decay of ^{210}Po between separation and counting, and the decay of the tracer between its calibration date and counting.

As shown in Figure 6, two decay correction factors, $f_{2\text{-}Po}$ and $f_{3\text{-}Po}$, are considered for the calculation of the activity concentration of ^{210}Po on the separation date.

$$c_{Po,1} = \frac{a_T \cdot m_T \cdot r_n}{V_S \cdot r_{nt}} \cdot f_T \cdot f_{2-Po} \cdot f_{3-Po} \qquad (1)$$

$$r_n = r_g - r_0 \qquad (2)$$

$$r_{nt} = r_{gt} - r_{0t} \qquad (3)$$

The decay correction factors are calculated as follows:

$$f_T = \exp\,(-\lambda_{\mathrm{T}} \cdot t_T) \qquad (4)$$

$$f_{2\text{-}Po} = \exp\,(\lambda_{\mathrm{Po}} \cdot t_{2\text{-}Po}) \qquad (5)$$

$$f_{3\text{-}Po} = \lambda_{\mathrm{Po}} \cdot t_{3\text{-}Po} \,/\, (1 - \exp(-\lambda_{\mathrm{Po}} \cdot t_{3\text{-}Po})) \qquad (6)$$

According to the Guide for the expression of Uncertainty in Measurement [19] the combined uncertainty of the ^{210}Po activity concentration on the separation date $u(c_{Po,\,1})$ is calculated according equation (7):

$$u(c_{Po,1}) = c_{Po,1} \cdot \sqrt{\frac{u^2(a_T)}{a_T^2} + \frac{u^2(r_n)}{r_n^2} + \frac{u^2(r_{nt})}{r_{nt}^2} + \frac{u^2(V_S)}{V_S^2} + \frac{u^2(m_T)}{m_T^2} + \frac{u^2(f_{2-Po})}{f_{2-Po}^2} + \frac{u^2(f_T)}{f_T^2} + \frac{u^2(f_{3-Po})}{f_{3-Po}^2}} \qquad (7)$$

The uncertainties associated to the components of $u(c_{Po,\,1})$ can be calculated as follows:

$$u\,(r_n) \;=\; \sqrt{\frac{r_g}{t_{3-Po}} + \frac{r_0}{t_0}} \qquad (8)$$

$$u\,(r_{nt}) \;=\; \sqrt{\frac{r_{gt}}{t_{3-Po}} + \frac{r_{0t}}{t_0}} \qquad (9)$$

$$u\,(f_T) = f_T \cdot t_T \cdot u\,(\lambda_T) \qquad (10)$$

$$u(f_{2-Po}) = f_{2-Po} \cdot t_{2-Po} \cdot u(\lambda_{Po}) \qquad (11)$$

$$u(f_{3-Po}) = f_{3-Po} \cdot \left\{ 1 - \lambda_{Po} \cdot t_{3-Po} \cdot \frac{\exp(-\lambda_{Po} \cdot t_{3-Po})}{1 - \exp(-\lambda_{Po} \cdot t_{3-Po})} \right\} \cdot \frac{u(\lambda_{Po})}{\lambda_{Po}} \qquad (12)$$

As an example, the measurement uncertainty of ^{210}Po in spiked water sample on separation date is summarized in Table 3.

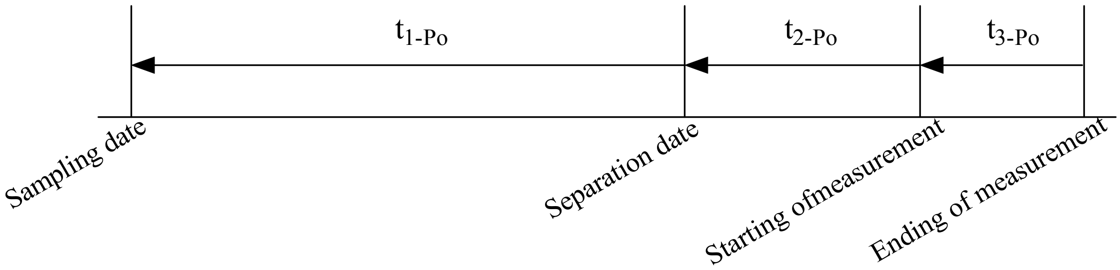

Fig. 6. Time intervals between sampling date, separation date and measurement date.

10.2. Calculation of the activity concentration and estimation of the combined uncertainty of ^{210}Po on the sampling date

In this section, the calculation of results back to the sampling date is discussed.

In order to determine the activity concentration of ^{210}Po in an environmental sample on the sampling date ($c_{Po,\,0}$), it is necessary to carry out the analysis within a limited time period after sampling.

TABLE 3. AN EXAMPLE OF QUANTIFYING UNCERTAINTIES IN THE MEASUREMENT OF ^{210}Po IN SPIKED WATER SAMPLE BY ALPHA-PARTICLE SPECTROMETRY

Symbol, unit	Value of variable	uncertainty	Conversion factor to standard uncertainty	Standard uncertainty (u)	Percent contribution to $(u_{aT})^2$
r_{gt}, s^{-1}	$1.2\text{x}10^{-3}$	$1.0\text{x}10^{-4}$	1.0	$1.0\text{x}10^{-4}$	35.9
r_{0t}, s^{-1}	$1.3\text{x}10^{-5}$	$2.0\text{x}10^{-6}$	1.0	$2.0\text{x}10^{-6}$	0.03
r_g, s^{-1}	$9.8\text{x}10^{-4}$	$1.0\text{x}10^{-4}$	1.0	$1.0\text{x}10^{-4}$	58.4
r_0, s^{-1}	$8.5\text{x}10^{-5}$	$2.0\text{x}10^{-5}$	1.0	$2.0\text{x}10^{-5}$	5.11
a_T, Bq g^{-1}	0.942	$4.0\text{x}10^{-3}$	1.0	$4.0\text{x}10^{-3}$	0.21
m_T, g	0.01005	$1.0\text{x}10^{-4}$	$\sqrt{3}$	$5.8\text{x}10^{-5}$	0.39
V_s, L	0.5	$1.0\text{x}10^{-4}$	$\sqrt{6}$	$4.1\text{x}10^{-5}$	0.00001
λ_T, d^{-1}	$1.9\text{x}10^{-5}$	$1.3\text{x}10^{-5}$	1.0	$1.3\text{x}10^{-5}$	negligible
λ_{Po}, d^{-1}	$5.0\text{x}10^{-3}$	$7.2\text{x}10^{-9}$	1.0	$7.2\text{x}10^{-9}$	negligible
f_T	0.91254	$2.0\text{x}10^{-5}$	1.0	$2.0\text{x}10^{-4}$	negligible
$f_{2\text{-}Po}$	1.005	$7.3\text{x}10^{-9}$	1.0	$7.3\text{x}10^{-9}$	negligible
$f_{3\text{-}Po}$	1.0055	$1.8\text{x}10^{-8}$	1.0	$1.0\text{x}10^{-8}$	negligible
$t_{2\text{-}Po}$, d	1.00				negligible
$t_{3\text{-}Po}$, d	2.18				negligible
$c_{Po,\,1}$, mBq L^{-1}	13.1	1.2			

In environmental samples the ^{210}Po is often not in equilibrium with its ^{210}Pb parent. If the initial activity of ^{210}Pb is much higher than the activity of ^{210}Po, or the time period between sampling and Po separation is long, the ^{210}Po activity changes considerably from its initial value at the sampling date due to the production of ^{210}Po from the ^{210}Pb decay and the decay of the ^{210}Po itself. This has to be considered in the calculation of decay correction of the ^{210}Po activity to the sampling date.

The uncertainty of ^{210}Po calculated to the sampling date can be more significantly affected by the time interval between the separation date and the sampling date in comparison with that of ^{210}Pb, due to the short half life of ^{210}Po (138.38 d). Therefore we discuss here how the relative standard uncertainty of ^{210}Po on the sampling date varies with the time interval between the sampling date and the separation date of Po under some assumptions.

As an example, it was assumed that the specific activity of ^{210}Pb in the sample on the sampling date was determined to be 130 mBq L^{-1} with a 10 % relative standard uncertainty ($u(c_{Pb,0})/c_{Pb,0}$), while the relative standard uncertainty ($u(c_{Po,1})/c_{Po,1}$) of ^{210}Po specific activity on the separation date was 3 %. In this assumption only the activity contribution of ^{210}Pb was considered for the calculation of ingrowth of ^{210}Po between sampling and separation, because in most cases the major contributor to ingrowth over the delay period between sampling and separation is ^{210}Pb. ^{210}Bi was assumed to be in secular equilibrium with ^{210}Pb in the sample.

On the basis of these assumptions, the activity concentration of ^{210}Po can be decay corrected to the sampling date by use of the following equation:

$$c_{Po,0} = \left\{ c_{Po,1} - \frac{\lambda_{Po}}{\lambda_{Po} - \lambda_{Pb}} \cdot c_{Pb,0} \cdot \exp(\lambda_{Pb} t_{1-Po}) \cdot \left[\exp(-\lambda_{Pb} t_{1-Po}) - \exp(-\lambda_{Po} t_{1-Po}) \right] \right\} \cdot \exp(\lambda_{Po} t_{1-Po}) \tag{13}$$

The activity concentration of ^{210}Pb $c_{Pb,0}$ can be determined by processing the aqueous solution from 9.2.1 (d)/(h) or the eluate from 9.2.2. (d), respectively, e.g. according the references [12], [13].

Differentiating equation (13) each partial derivative can be calculated as follows:

$$\frac{\partial\, c_{Po,0}}{\partial\, c_{Po,1}} = I \tag{14}$$

$$\frac{\partial\, c_{Po,0}}{\partial\, c_{Pb,0}} = -\lambda_{Po} \cdot y \tag{15}$$

$$\frac{\partial\, c_{Po,0}}{\partial\, \lambda_{Pb}} = J \cdot \left(t_{1-Po} \cdot \exp(\lambda_{Pb} \cdot t_{1-Po}) - y \right) \tag{16}$$

$$\frac{\partial\, c_{Po,0}}{\partial\, \lambda_{Po}} = c_{Po,1} \cdot t_{1-Po} \cdot I - J \left(t_{1-Po} \cdot I - \frac{\lambda_{Pb}}{\lambda_{Po}} \cdot y \right) \tag{17}$$

where

$$I = \exp(\lambda_{Po} \cdot t_{1-Po}) \tag{18}$$

$$J = c_{Pb,0} \cdot \frac{\lambda_{Po}}{\lambda_{Po} - \lambda_{Pb}} \tag{19}$$

$$y = \frac{1}{\lambda_{Po} - \lambda_{Pb}} \cdot \left[I - \exp(\lambda_{Pb} \cdot t_{1-Po}) \right]. \tag{20}$$

Therefore, the combined uncertainty of ^{210}Po activity concentration on the sampling date $u(c_{Po,0})$ can be calculated by the following formula:

$$u(c_{Po,0}) = \sqrt{I^2 \cdot u^2(c_{Po,1}) + (\lambda_{Po} y)^2 \cdot u^2(c_{Pb,0}) + J^2 (t_{1-Po} \exp(\lambda_{Pb} t_{1-Po}) - y)^2 \cdot u^2(\lambda_{Pb}) + \left[c_{Po,1} \cdot t_{1-Po} \cdot I - J(t_{1-Po} \cdot I - \frac{\lambda_{Pb}}{\lambda_{Po}} \cdot y \right]^2 \cdot u^2(\lambda_{Po})} \qquad (21)$$

Figure 7 shows how the relative standard uncertainty for ^{210}Po on the sampling date changes depending on the time interval between the sampling date and the Po separation date, for the example case detailed above, the relative standard uncertainty increases exponentially with the delay time interval. If the ^{210}Po/^{210}Pb activity ratio is 1, then the relative standard uncertainty for ^{210}Po corrected to the sampling date reaches 10 % for a sampling–separation delay period of 120 days. However, in the case that the ratio is 0.1 (a typical ratio in rain water [21, 22]), a 10 % relative standard uncertainty for ^{210}Po is reached after only 16 days, while in the case the ratio is 3, the relevant time period is 250 days from the sampling date (Fig. 7).

As mentioned above, in these calculations ^{210}Bi was assumed to be in secular equilibrium with ^{210}Pb in the sample. This is not always the case, for example in rainwater the ^{210}Bi/^{210}Pb activity ratio is normally less than 1. In most cases this assumption causes only small errors. However, it should be kept in mind that for some sample types where extreme disequilibria may occur (for example, process waters from the mining industry) additional correction factors may be required. In any case, the introduction of both quantifiable and unquantifiable uncertainties may be minimised by keeping the time delay between sample collection and analysis as short as possible.

10.3. Decision threshold

In accordance with ISO 11929-7, the decision threshold, a*, is obtained from the following equation:

$$a^* = \frac{k_{1-\alpha}}{V_S \cdot \varepsilon \cdot R} \sqrt{\frac{r_0}{t_{3-Po}} + \frac{r_0}{t_0}} \qquad (22)$$

The quantil $k_{1-\alpha} = 1.65$ is often chosen by default for an error probability of the first kind of 0.05.

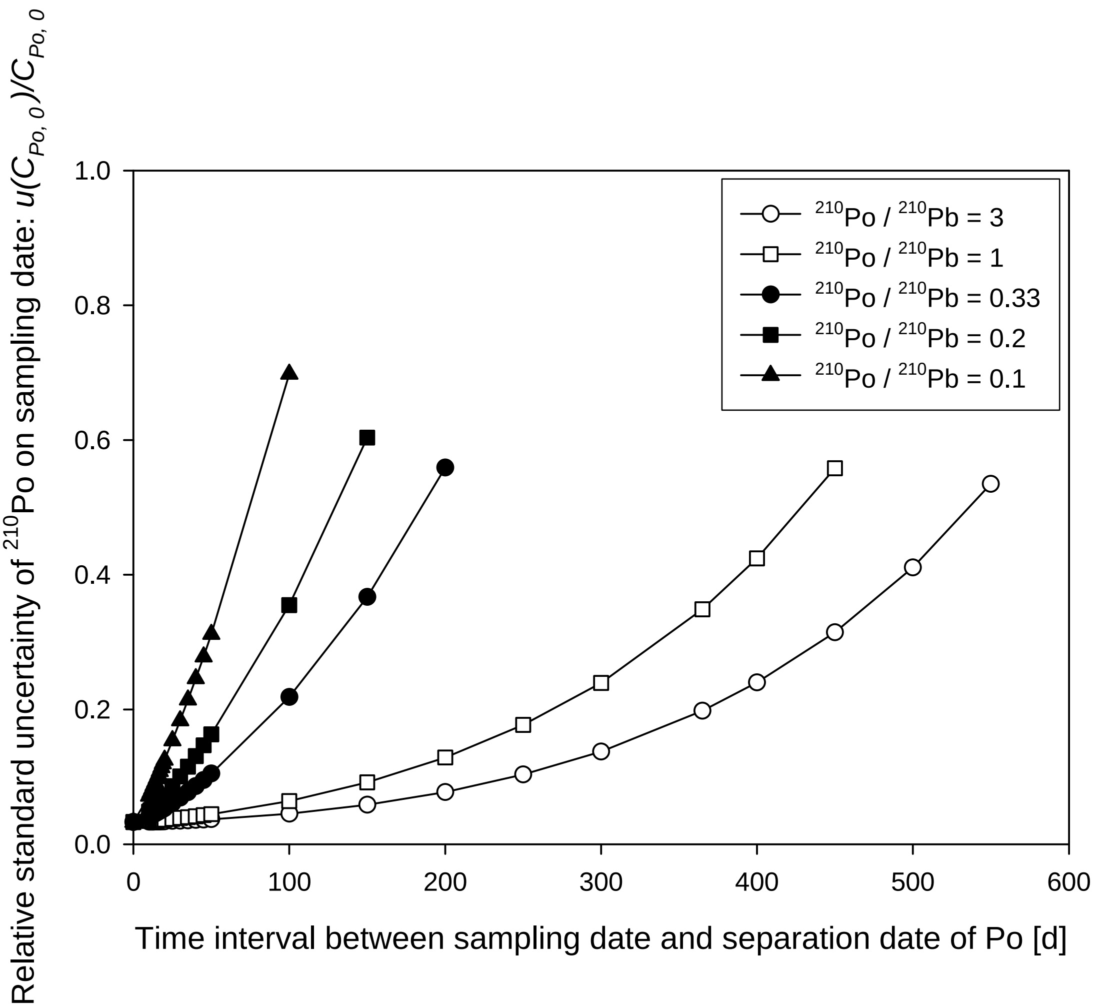

Fig. 7. The variation of relative standard uncertainty ($u(c_{Po,0})/c_{Po,0}$) in the measurement of ^{210}Po in a spiked water sample on sampling date with the time interval between the sampling date and the separation time of Po (It is assumed that the ^{210}Pb activity concentration is 130 mBq L^{-1}, a 10% relative standard uncertainty of ^{210}Pb on the sampling date, and a 3% relative standard uncertainty of ^{210}Po on the separation date)

10.4. Detection limit

In accordance with ISO 11929-7, the detection limit, $a^{\#}$, is calculated by the following equation:

Assuming $\alpha = \beta$ then $k_{1-\alpha} = k_{1-\beta} = k$,

$$a^{\#} = \frac{2 \cdot a^{*} + (k^{2} \cdot w)/t_{3-Po}}{1 - k^{2} \cdot u^{2}(w)} \qquad (23)$$

$$w = \frac{1}{V_S \cdot \varepsilon \cdot R} \qquad (24)$$

where the uncertainty of the counting time is neglected and the relative standard uncertainty of w is calculated using the following equation:

$$\left(\frac{u(w)}{w}\right)^2 = \left(\frac{u(R)}{R}\right)^2 + \left(\frac{u(V_S)}{V_S}\right)^2 + \left(\frac{u(\varepsilon)}{\varepsilon}\right)^2 \tag{25}$$

the relative standard uncertainty of R and ε are calculated by

$$\left(\frac{u(R)}{R}\right)^2 = \left(\frac{u(r_{nt})}{r_{nt}}\right)^2 + \left(\frac{u(a_T)}{a_T}\right)^2 + \left(\frac{u(\varepsilon)}{\varepsilon}\right)^2 + \left(\frac{u(m_T)}{m_T}\right)^2 \tag{26}$$

$$\left(\frac{u(\varepsilon)}{\varepsilon}\right)^2 = \left(\frac{u(r_{nt})}{r_{nt}}\right)^2 + \left(\frac{u(a_T)}{a_T}\right)^2 + \left(\frac{u(R)}{R}\right)^2 + \left(\frac{u(m_T)}{m_T}\right)^2 + \left(\frac{u(\lambda_{Po})}{\lambda_{Po}}\right)^2 \tag{27}$$

where $u(a_T)$ includes all the uncertainties related to the tracer activity: that is the standard solution and preparation of the tracer solution. The detection limit of the procedures is described in Table 4.

TABLE 4. THE RESULT OF REPEATABILITY AND REPRODUCIBILITY TESTS

Validation parameter	Method	
	DDTC solvent extraction	Sr-resin chromatography
Repeatability		
Mean ($\overline{X_r}$) in repeatability test, Bq L^{-1}	1.55	1.58
Repeatability standard deviation (S_r), Bq L^{-1}	0.06	0.05
Repeatability limit (r_L), Bq L^{-1}	0.17	0.14
Relative bias (δ), %	0.64	1.28
Reproducibility		
Mean ($\overline{X}_R$) in reproducibility test, mBq L^{-1}	12.7	13.1
Reproducibility standard deviation (S_R), mBq L^{-1}	0.46	0.57
Reproducibility limit (R_L), mBq L^{-1}	1.29	1.60
Relative bias (δ), %	1.55	1.55
Chemical recovery (R), %	54 - 97	50 - 94
Detection limit ($a^{\#}$), mBq L^{-1}	2*	2*

* Counting efficiency ε, 25 %; counting time $t_{3\text{-}Po}$, 250,000 s; sample volume, 500 mL.

11. QUALITY CONTROL MEASUREMENT

For the quality control of the procedure, two kinds of test water samples, which have different activity concentrations of ^{210}Po, were prepared by spiking tap water collected at the IAEA's laboratories, Seibersdorf, Austria with a known activity of the certified ^{210}Po standard solution respectively, because there are no available reference materials of ^{210}Po with water matrix for quality control measurement. Prior to spike ^{210}Po to the tap water, some elements in the tap water were determined by ICP-MS (Elan 6000 Perkin-Elmer SCIEX instrument, equipped with a Scott-type spray chamber, a cross-flow nebuliser and a Gilson Minipuls 3 peristaltic pump) in the totalquant mode to screen the elemental composition. Therefore, the accuracy of the totalquant measurements in ICP-MS was not better than 20-30 %. The semi-quantitative results of the tap water sample are given in Table 2.

The results of test samples are evaluated in terms of relative bias, repeatability and reproducibility. The control chart of relative bias, chemical recovery, repeatability, reproducibility limits and linearity of the procedures are described in Table 4, Fig. 8 and Fig. 9.

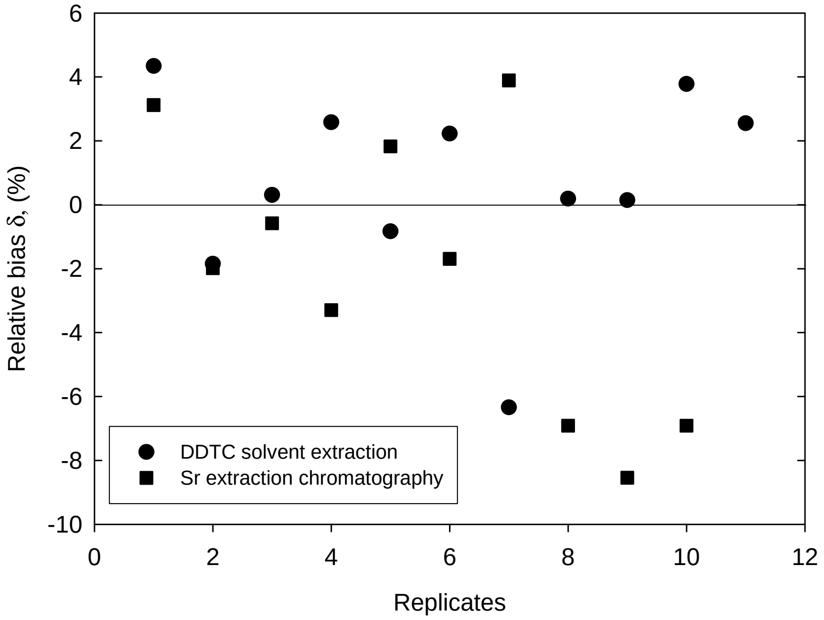

Fig. 8. Control chart for the relative bias δ of determination of ^{210}Po in spiked water samples using DDTC solvent extraction and Sr-resin extraction chromatography.

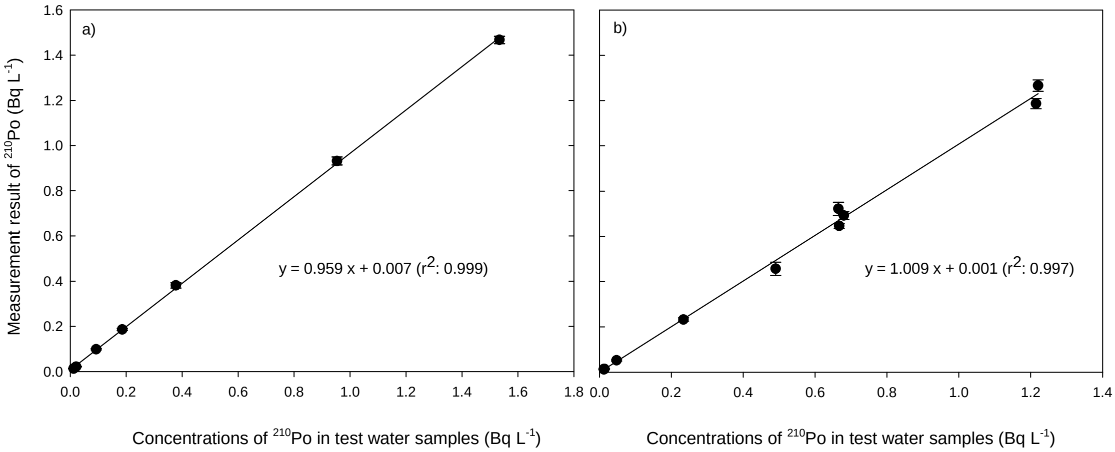

Fig. 9. Linearity of analytical results obtained by (a) DDTC solvent extraction and (b) Sr-resin extraction chromatography.

11.1. Relative bias of the method (trueness)

A spiked sample is analyzed with ten duplicates to evaluate the trueness. The relative bias of the method is calculated by the following formula:

$$\delta_i(\%) = \frac{c - X_i}{c} \cdot 100 \tag{28}$$

11.2. Repeatability limit (within–run precision)

The repeatability limit is estimated by analyzing ten duplicates of a spiked sample with a known activity concentration of ^{210}Po in repeatability conditions: (instrument, analyst, shortest time, etc.). The repeatability limit is calculated by the following formula:

$$r_L = S_r \times 2.8 \tag{29}$$

11.3. Reproducibility limit

The repeatability limit is also estimated by analyzing ten duplicates of a spiked sample with a known activity concentration of ^{210}Po, but with at least one parameter being changed in the analytical system (analyst, analysis date, instrument, etc.). The reproducibility limit is calculated by the following formula:

$$R_L = S_R \times 2.8 \tag{30}$$

12. TEST REPORT

The test report shall conform to ISO 17025 requirements and shall contain the following information:

- Sample code
- Sample description
- Sampling date (if known)
- ^{210}Po separation date
- ^{209}Po tracer solution calibration reference date
- Analysis request code
- Responsible person
- Reference to the measurement and evaluation procedure used
- Test result
- Units in which the test result is expressed
- Date to which the test result has been corrected.

Unless otherwise required, the test result should be stated together with the expanded uncertainty [19] calculated using a coverage factor k = 2. The following form is recommended:

$c \pm u\ (c)$, with the associated k value.

The date to which the test result has been corrected may be stated as “corrected to sampling date” or “corrected to ^{210}Po separation date” as appropriate.

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ANNEX: CHEMICAL REAGENTS AND EQUIPMENT

- 0.2 mol L^{-1} $KMnO_4$: Dissolve 3.161 g of $KMnO_4$ in 100 mL of deionized water. Filter through a dense glass fiber filter (Whatman GF/F) and keep in a dark and cool place.
- 0.3 mol L^{-1} $MnCl_2$: Dissolve 5.938 g of $MnCl_2 \cdot 4H_2O$ in 100 mL of deionized water.
- 0.1% DDTC in CH_3CCl_3 : Dissolve 0.1 g of DDTC in 100 mL of CH_3CCl_3.
- 1% H_2O_2 in 2 mol L^{-1} HCl : Take 20 mL of 32 % HCl in 100 mL volume of volumetric flask and add 79 mL of deionized water, and then add 1 mL of 30 % H_2O_2.
- 1% H_2O_2 in 5 mol L^{-1} HCl : Take 50mL of 32 % HCl in 100 mL volume of volumetric flask and, add 49 mL of deionized water, and then add 1 mL of 30 % H_2O_2.

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