International Standard

ISO 4685

Water quality — Radium 226 — Test method using ICP-MS

Qualité de l'eau — Radium 226 — Méthode d'essai par ICP-MS

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Contents

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Radionuclides are present throughout the environment; thus, water bodies (e.g. surface waters, ground waters, sea waters) contain radionuclides, which can be of either natural or anthropogenic origin:

— Naturally-occurring radionuclides, including ^{3}H, ^{14}C, ^{40}K and those originating from the thorium and uranium decay series, in particular ^{210}Pb, ^{210}Po, ^{222}Rn, ^{226}Ra, ^{228}Ra, ^{227}Ac, ^{232}Th, ^{231}Pa, ^{234}U, and ^{238}U, can be found in water bodies due to either natural processes (e.g. desorption from the soil and runoff by rain water) or released from technological processes involving naturally occurring radioactive materials (e.g. mining, mineral processing, oil, gas, and coal production, water treatment and the production and use of phosphate fertilisers).

— Anthropogenic radionuclides such as ^{55}Fe, ^{59}Ni, ^{63}Ni, ^{90}Sr, ^{99}Tc, transuranic elements (e.g. Np, Pu, Am, and Cm), and some gamma emitting radionuclides such as ^{60}Co and ^{137}Cs can also be found in natural waters. Small quantities of anthropogenic radionuclides can be discharged from nuclear facilities to the environment as a result of authorized routine releases. The radionuclides present in liquid effluents are usually controlled before being discharged to the environment[1] and water bodies. Anthropogenic radionuclides used for medical and industrial applications can be released to the environment after use. Anthropogenic radionuclides are also found in waters due to contamination from fallout resulting from above-ground nuclear detonations and accidents such as those that have occurred at the Chornobyl and Fukushima nuclear facilities.

Radionuclide activity concentrations in water bodies can vary according to local geological characteristics and climatic conditions and can be locally and temporally enhanced by releases from nuclear facilities during planned, existing, and emergency exposure situations.[2][3] Some drinking water sources can thus contain radionuclides at activity concentrations that can present a human health risk. The World Health Organization (WHO) recommends to routinely monitor radioactivity in drinking waters[4] and to take proper actions when needed to minimize the health risk.

National regulations usually specify the activity concentration limits that are authorized in drinking waters, water bodies and liquid effluents to be discharged to the environment. These limits can vary for planned, existing, and emergency exposure situations. As an example, during either a planned or existing situation, the WHO guidance level for ^{226}Ra in drinking water is 1 Bq·l^{-1},[4] see NOTES 1 and 2. Compliance with these limits is assessed by measuring radioactivity in water samples and by comparing the results obtained, with their associated uncertainties, as specified by ISO/IEC Guide 98-3 and ISO 5667-20.[6]

NOTE 1 If the value is not specified in Annex 6 of Reference [4], the value has been calculated using the formula provided in Reference [4] and the dose coefficient data from References [7] and [8].

NOTE 2 The guidance level calculated in Reference [4] is the activity concentration that results in an effective dose of 0,1 mSv·a^{-1} to members of the public for an intake of 2 l·d^{-1} of drinking water for one year. This is an effective dose that represents a very low level of risk to human health and which is not expected to give rise to any detectable adverse health effects.[4]

This document contains method(s) to support laboratories, which need to determine ^{226}Ra in water samples. The method(s) described in this document can be used for various types of waters. Minor modifications such as sample volume and counting time can be made if needed to ensure that the decision threshold, detection limit, and uncertainties are below the required limits. This can be done for several reasons such as emergency situations, lower national guidance limits and operational requirements.

Water quality — Radium 226 — Test method using ICP-MS

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices

IMPORTANT — It is essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies methods to determine ^{226}Ra concentration by inductively coupled plasma mass spectrometry (ICP-MS). The mass concentrations obtained can be converted into activity concentrations.

The method is applicable to test samples of drinking water, rainwater, surface and ground water, after proper sampling and handling, and test sample preparation.

The detection limit depends on the sample volume, the instrument used, the background count rate, the detection efficiency, the counting time and the chemical yield. The detection limit of the method described in this document, using currently available equipment, is approximately 10 mBq·l^{-1}, which is better than the WHO criteria for safe consumption of drinking water (1 Bq·l^{-1}).[4]

This method covers the measurement of ^{226}Ra in water at activity concentrations between 0,001 Bq·l^{-1} and 100 Bq·l^{-1}. Samples with concentrations higher than 1 Bq·l^{-1} can be measured if a dilution is performed.[9]

The method described in this document is applicable in the event of an emergency situation.

In this method, filtration of the test sample is necessary. The analysis of ^{226}Ra adsorbed to suspended matter is not covered by this method. The analysis of the insoluble fraction requires a mineralization step that is not covered by this document. In this case, the measurement is made on the different phases obtained.

It is the user's responsibility to ensure the validity of this test method for the water samples tested.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste water*

ISO 17294-1:2004, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines*

ISO 17294-2:2023, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of selected elements including uranium isotopes*

ISO 80000-10, *Quantities and units — Part 10: Atomic and nuclear physics*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

3 Term, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/IEC Guide 98-3, ISO/IEC Guide 99 and ISO 80000-10 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.2 Symbols

Table 1 — Symbols

Symbol	Description	Unit
c_s	Specific activity corresponding to one gram of the radionuclide	Bq·g^{-1}
L_D	Detection limit in mass concentration, the lowest mass concentration that can be considered statistically different from a blank sample	Bq·kg^{-1}
L_Q	Limit of quantification, the lowest mass concentration that can be quantified with statistical uncertainty	g·kg^{-1}
f	Correction factor	
m	Mass of sample	kg
m/z	Mass to charge ratio measured by ICP-MS	—
m_c	Mass of the calibration standard tracer added to sample	g
m_{cs}	Mass of calibration standard solution added to standard	g
m_{IS}	Mass of the internal standard added to blank and sample	g
m_{ISS}	Mass of the internal standard solution added to a blank or a sample	g
N_0	Counts per second measured when performing ICP-MS measurement of a blank sample at a given mass-to-charge ratio	Counts·s^{-1}
$\bar{N}_0$	Average number of counts per second for several blank samples measured by ICP-MS at a given mass-to-charge ratio	Counts·s^{-1}
U	Expanded uncertainty and the coverage factor k with k = 1,2,..., $U=k \cdot u$	—
u	Uncertainty such as counts per second, mass added etc.	—
$u(c)$	Standard uncertainty of the activity concentration result	Bq·kg^{-1}
$u(\rho)$	Standard uncertainty associated with the measurement result	—
$u(\rho_V)$	Standard uncertainty associated with conversion from mass to volume units of the calibration standard solution	g·g^{-1}
ρ	Analyte mass for a given radionuclide per sample unit mass	g·g^{-1}
ρ_c	Mass concentration of the calibration standard solution	g·g^{-1}
ρ_{IS}	Mass of internal standard element or isotope per unit mass of the internal standard solution.	g·g^{-1}

Table 1 *(continued)*

Symbol	Description	Unit
ρ_V	Conversion from mass to volume units	g·l^{-1}
V	Volume of sample	l
z	Electric charge of an ion	

4 Principle

The principles of analysis using ICP-MS are described in ISO 17294-1 and ISO 17294-2.

^{226}Ra is natural in origin, occurring in the ^{238}U decay chain.

ICP-MS has been successfully used to measure the concentration of ^{226}Ra in water samples.[9],[10]

The results can be converted to activity concentrations using the specific activity as a conversion factor given in Table 2.

Table 2 — ^{226}Ra half-life and specific activity[11]

Isotope	Half-life years	Specific activity Bq·g^{-1}
Radium-226	1,600·(7)·10^{3}	3,658·(16)·10^{10}

An example of the limit of detection that can be obtained with ICP-MS is given in Table 3, with a typical measurement time of several minutes per sample, including sample uptake, analysis time and washout before the next sample.

Table 3 — Examples of detection limit[9],[10]

Isotope	Limit of detection µg·l^{-1}	Limit of detection Bq·l^{-1}
Radium-226	2,70·10^{-7}	0,01

Measurement by ICP-MS is affected by several interferences, which are shown in Table 4.

Table 4 — ^{226}Ra interferences affecting ICP-MS measurement

Interference name	Description	^{226}Ra interference
Isobaric	An isotope with a similar mass to the analyte	None
Polyatomic	Isotopes combining in the plasma to form an ion with a similar mass to the analyte	$^{86}Sr\ ^{140}Ce$ $^{87}Sr\ ^{139}La$ $^{88}Sr\ ^{138}Ba$ $^{40}Ca_2{}^{146}Nd$ $^{209}Bi\ ^{16}O\ ^{1}H$ $^{208}Pb\ ^{18}O$ $^{186}Os\ ^{40}Ar$ $^{188}Os\ ^{38}Ar$ $^{190}Os\ ^{36}Ar$ $^{186}W\ ^{40}Ar$ $^{203}Tl\ ^{23}Na$
Tailing	Isotopes with one or two mass units superior or inferior than the analyte to measure with a relatively high abundance (>10^6) relative to analyte	None

It is important to ensure that all potential interferences have been removed prior to measurement. Polyatomic interference is the most significant type of interference affecting ^{226}Ra measurement by ICP-MS.

The half-life of ^{226}Ra for ICP-MS measurement means direct measurement without chemical separation is possible. A pre-concentration and chemical separation can be required prior to measurement if removal of elements that can form polyatomic interferences is required. The test method is based on chemical separation of ^{226}Ra from interfering elements by ion-exchange and extraction chromatography followed by ICP-MS analysis.

It is important to know the interference decontamination factor. This can initially be assessed by running stable element standards at increasing concentrations to monitor the impact at m/z= 226.

If any interference has an impact on the ^{226}Ra result that cannot be corrected for, then the result cannot be considered to be valid.

The sample can be measured directly, if necessary, after filtration (at 0,45 µm porosity), without chemical separation of radium. In this case, it is necessary to use a matrix effect correction with an internal standard, as described in ISO 17294-1. An internal standard, for example ^{205}Tl or ^{195}Pt, is added to correct the matrix effects.

To quantify any potential interference coming from the reagents, a blank sample is prepared in the same way as the test sample. This blank sample is prepared using laboratory water.

5 Sampling and sample storage

Sampling, handling and storage of the water shall be done as specified in ISO 5667-1, ISO 5667-3 and ISO 5667-10 and guidance is given for different types of water in References [12] to [19]. It is important that the laboratory receives a sample that is truly representative and has not been damaged nor modified during either transportation or storage.

The sample is filtered to remove suspended matter using a 0,45 µm filter. A smaller pore size filter can also be used, but the filtration can be more tedious and time consuming. The sample shall be acidified after filtration to 1 % HNO_3.

6 Chemical reagents and equipment

6.1 General

The chemical reagents and equipment used for chemical treatment and preparation of the samples are described in Annex A.

Reagents of an appropriate purity should be used depending on the detection limit required, impact of interferences on the measurement and the ICP-MS instrument design used for measurement.

6.2 Chemical reagents

6.2.1 Laboratory water, used as a blank, as free as possible of chemical or radioactive impurities, with a resistivity of more than 18,2 MΩ·cm at 25 °C and total organic carbon less than 1 $\mu g \cdot l^{-1}$.

Unless otherwise stated, water refers to laboratory water.

6.2.2 An instrument blank, approximately 0,3 $mol \cdot l^{-1}$ nitric acid, is used to determine the background count rate of the instrument at selected mass to charge ratios.

The same reagent is also used to prepare the calibration standards and final samples for measurement.

6.2.3 A ^{226}Ra standard solution can be used to prepare calibration standards to calculate the sample activity concentration.

6.2.4 An internal standard solution prepared with a stable element. For example, ^{205}Tl and ^{195}Pt have been found suitable for this purpose. Their typical concentrations can be $c(^{205}Tl) = 50\ \mu g \cdot l^{-1}$ and $c(^{195}Pt) = 0{,}25\ \mu g \cdot l^{-1}$.

6.2.5 Argon gas for the plasma in the ICP-MS. The user should consult the instrument manufacturer on the minimum gas purity required.

6.3 Equipment

Usual laboratory equipment and in particular the following:

6.3.1 ICP-MS (quadrupole (with or without collision or reaction cell capability), tandem, sector field or multi-collector) and associated software. Operation at constant temperature is recommended. Follow the manufacturers instruction for laboratory setup and instrument operation.

6.3.2 Argon supply, equipped with pressure control and suitable extract system.

6.3.3 Autosampler if available, and compatible tubing for running multiple samples automatically.

6.3.4 Pipette, suitable for the accurate transfer of calibration standard and internal standard solution with a total precision within ±1 %.

7 Separation

It is the user's responsibility to ensure that all potential interferences have been removed. The removal of potential interferences is limited by the decontamination factor of the method. Suggested chemical separation options are outlined in Annex A.

8 Quality control

8.1 General

Measurement methods shall be performed by suitably skilled staff under a quality assurance program, such as the one that is described in ISO/IEC 17025.

If an analyst has not performed this procedure before, a precision and bias test should be performed by running a duplicate measurement of a reference or spiked material. Acceptance limits should be within limits specified by the laboratory.

A similar evaluation should be performed by the analyst who routinely applies this procedure, with a periodicity defined by the laboratory. Acceptance limits should be within limits specified by the laboratory.

8.2 Variables that can influence the measurement

Special care shall be taken in order to limit as much as possible the influence of parameters that can bias the measurement and lead to a non-representative result. Failure to take sufficient precautions during the different steps of the measurement process such as sampling, transportation and storage, reagents, transfer, instrument can require corrective factors to be applied to the measured results.

8.3 Instrument verification

Follow the instructions provided by the instrument manufacturer and the steps described in ISO 17294-1:2004, Clauses 7 and 9 in particular and ISO 17294-2:2023, Clauses 8 to 11 in particular.

The instrument sensitivity, detection limit and measurement precision should be determined for every analysis performed on the instrument.

Before any sample measurement, measure a quality control solution. Ensure that the measured value of the concentration does not deviate from the expected value (within measurement limits). If the deviation exceeds the established measurement limits (e.g. sensitivity, stability, uncertainty), follow the recommendations of the instrument manufacturer and perform the optimization of parameters again.

The instrument sensitivity can be determined from ^{226}Ra calibration standards run by ICP-MS prior to samples measurement. The concentration of calibration standard solutions should be known with high precision, using a certified standard if possible.

If the detector is used in dual-mode detector (or similar), then detector cross calibration can be required depending on the activity range of calibration standards measured.

A known amount of standard (m_{cs}) at a known concentration (ρ_c) shall be added for each standard, with the mass of calibration standard (m_c) calculated using Formula (1):

$$m_c = \rho_c \cdot m_{cs} \quad (1)$$

The uncertainty on m_c is calculated using Formula (2):

$$u(m_c) = m_c \sqrt{u_{rel}^2(\rho_c) + u_{rel}^2(m_{cs})} \quad (2)$$

A calibration plot can be produced using either the ICP-MS instrument software or a spreadsheet. The calibration standard concentration can be plotted against the counts per second. A linear calibration line gives the instrument sensitivity based on Formula (3):

$$y = D \cdot x + E \quad (3)$$

where

D	is the gradient of the calibration line;
y	is the count per second for the analyte in the sample;
x	is the analyte mass concentration or activity concentration;
E	is the intercept for the y-axis when $x = 0$.

A linear calibration line gives values for D and E.

Using the calibration curve to determine the concentration should account for the impact of sample matrix on instrument sensitivity. Assessment of activity using a recovery tracer is a more robust approach.

An internal standard shall be prepared to monitor and correct for any change in instrument response during a run, using an element at a similar mass and ionisation energy to ^{226}Ra that is not present in the sample being measured, for example ^{205}Tl or ^{195}Pt.

Equipment quality control solutions shall also be measured at regular intervals during the procedure to verify that the measurement equipment is performing within agreed limits.

8.4 Method verification

The method should be validated periodically through replicate measurements of appropriate samples such as spiked samples, reference materials, or participation in inter-comparison exercises.

The repeatability of the method should be verified (e.g. by replicate measurements).

9 Expression of results

9.1 Data analysis

The output from the instrument is typically in counts per second. Gross count rates for samples are corrected for background, and change in instrument response using the internal standard. A correction for chemical recovery is also required.

If dilutions were carried out, apply the appropriate factor to the values of the sample.

9.2 Background

The blank solution is measured as a sample. The obtained value shall be subtracted from the measured sample values. A blank solution should also be measured regularly throughout the procedure.

A rinsing sequence is usually performed. The sample introduction system is rinsed between each sample using a solution of dilute HNO_3. A blank solution shall be measured at regular intervals to verify that all remaining ^{226}Ra and interferences are removed from the system by returning to the baseline.

Depending on the instrument software used, it is possible to set a threshold count rate that shall be reached during the rinsing sequence before moving on to the next sample.

The ICP-MS instrument software can have built-in background correction capability. The user shall take care that only one background correction is applied.

9.3 Internal standard

An internal standard shall be added to samples before measurement, including to the blank sample. This monitors for changes in instrument performance during a run. This can be due to small variations in (for example) plasma gas flow rate, or, for higher matrix samples such as seawater, internal components such as interface cones becoming be partially blocked during a run, reducing sample transmission.

A known amount of internal standard (m_{IS}) shall be added to the sample. For this purpose, a solution of known concentration of internal standard (ρ_{IS}), ideally with great precision, is needed. The mass of internal standard solution (m_{ISS}) added is recorded. The mass of internal standard added (m_{IS}) is calculated using Formula (4):

$$m_{IS} = \rho_{IS} \cdot m_{ISS} \tag{4}$$

The uncertainty on m_{IS} is calculated using Formula (5):

$$u(m_{IS}) = m_{IS}\sqrt{u_{rel}^2(\rho_{IS}) + u_{rel}^2(m_{ISS})} \tag{5}$$

Some ICP-MS instruments are equipped with online internal standard lines in the sample introduction that can measure a separate solution containing the internal standard. In such cases, the mass of internal standard added, and the associated uncertainty correction does not apply.

The net count number for the internal standard in each sample should be corrected based on the net count number in the first background sample, and this correction factor applied to each sample.

The ICP-MS instrument software can have built-in internal standard correction capability. The user shall take care that only one internal standard correction is applied.

9.4 Internal calibration

A calibration plot can be produced using either the ICP-MS instrument software or a spreadsheet. The calibration standard concentration can be plotted against the counts per second. A linear calibration line gives the instrument sensitivity based on Formula (6):

$$y = D \cdot x + E \tag{6}$$

It is recommended that the activity concentration in the sample is close to the centroid of the curve in order to minimize the standard uncertainty linked to the calibration curve.

The formula of the calibration curve is determined by the least squares method.

Additionally, the use of an internal standard is useful to correct the signal fluctuations. In cases where an internal standard is used, a correction factor f is introduced. This correction can be applied by the ICP software or any validated in-house calculation sheet or software. The mass concentration of ^{226}Ra, expressed in g·l^{-1}, is given by Formula (7):

$$x = (y - E)/D \tag{7}$$

9.5 Detection limit

The detection limit (L_D) corresponds to the equivalent concentration of three times the standard deviation of the measurement of 10 test portions of a blank sample. The blank sample shall have passed through all steps of the method. The detection limit is calculated using Formula (8):

$$L_D = \overline{N_0} + 3 \cdot S_{N_0} \tag{8}$$

9.6 Limit of quantification

The limit of quantification (L_Q) is ten times the standard deviation of the measurement of ten test portions of the blank. This is calculated using Formula (9):

$$L_Q = \overline{N_0} + 10 \cdot S_{N_0} \tag{9}$$

NOTE The calculation of the detection limit and limit of quantification using atom counting is different from that using decay counting techniques.

9.7 Conversion of mass concentration to activity concentration

The specific activity (c_S) of ^{226}Ra can be multiplied by the mass concentration (ρ_c), detection limit (L_D) and limit of quantification (L_Q) to convert mass concentrations to activity concentrations (c) using Formula (10):

$$c = \rho \cdot c_s \tag{10}$$

The uncertainty of the activity concentration is calculated using Formula (11):

$$u(c) = c\sqrt{u_{rel}^2(\rho) + u_{rel}^2(c_s)} \tag{11}$$

The L_D and L_Q are estimated values and do not require an uncertainty calculation.

9.8 Conversion from mass to volume units

A conversion from mass (grams per kilogram) to volume (grams per litre) units can be achieved using the analyte mass (ρ), mass (m) and volume (V) of the sample recorded during the procedure. The conversion is carried out using Formula (12):

$$\rho_V = \frac{m \cdot \rho}{V} \tag{12}$$

The uncertainty in this conversion is calculated using Formula (13):

$$u(\rho_V) = \rho_V \sqrt{u_{rel}^2(\rho) + u_{rel}^2(m) + u_{rel}^2(V)} \tag{13}$$

10 Test report

The test report shall conform to ISO/IEC 17025 requirements. It shall contain the following information:

a) reference to this document, i.e. ISO 4685:2024;

b) identification of the sample;

c) units in which the results are expressed;

d) the test result can be given according to method 1 or method 2. The method used shall be clearly stated in the report.

Method 1)

— if the result is less than the limit of detection, the result of the measurement is expressed as $\leq L_D$,

— if the result is between the limit of detection and the limit of quantification, the result of the measurement is expressed as $\leq L_Q$,

— if the result is greater than the limit of quantification, the result of the measurement is expressed as ($\rho \pm k \cdot u(\rho)$ or $\rho \pm U(\rho)$) or as ($c \pm k \cdot u(c)$ or $c \pm U(c)$) with the associated k value.

If the limit of detection exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose.

Method 2)

— if the result is less than the limit of quantification, the result of the measurement is expressed as $\leq L_Q$,

— if the result is greater than the limit of quantification, the result of the measurement is expressed as ($\rho \pm k{\cdot}u(\rho)$ or $\rho \pm U$) or as ($c \pm k{\cdot}u(c)$ or $c \pm U$) with the associated k value.

If the limit of quantification exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose.

Complementary information can be provided such as:

a) limit of application;

b) the detection limit and limit of quantification;

c) mention of any relevant information likely to affect the results.

Annex A
(informative)

Chemical separation of ^{226}Ra using a cation exchange and a crown ether based extraction chromatography resin[20],[21],[22],[23]

A.1 Principle

The sample is prepared as described in Clause 5. Tracer is added. Depending on the salt content of the sample, ^{226}Ra can be first preconcentrated before undergoing purification from potential interferences by passing solution through a cation exchange resin followed by a crown-ether based extraction chromatography. ^{226}Ra is measured by ICP-MS.

An approximate detection limit of 0,001 $Bq \cdot l^{-1}$ is usually obtained using this method.

A.2 Technical resources

A.2.1 Reagents

Use only reagents of recognized analytical grade. It is recommended to use acids and bases of trace metal grade or equivalent (a better purity grade can also be employed). Reagent grade acids/bases usually do not have enough purity for ICP-MS measurement.

A.2.1.1 Nitric acid solution , $\rho(HNO_3)$ = 69 %.

A.2.1.2 Nitric acid solution, $c(HNO_3)$ = 1 $mol \cdot l^{-1}$.

A.2.1.3 Nitric acid solution, $c(HNO_3)$ = 3 $mol \cdot l^{-1}$.

A.2.1.4 Hydrochloric acid solution distilled, ρ (HCl) = 32 %.

A.2.1.5 Hydrochloric acid solution, $c(HCl)$ = 4 $mol \cdot l^{-1}$.

A.2.1.6 Hydrochloric acid solution, $c(HCl)$ = 3 $mol \cdot l^{-1}$.

A.2.1.7 Calcium nitrate solution, $c(Ca(NO_3)_2)$ = 1,25 $mol \cdot l^{-1}$.

A.2.1.8 Ammonia solution, $c(NH_4.OH)$ = 25 %.

A.2.1.9 Hydrogen peroxide, $\rho(H_2O_2)$ = 30 %.

A.2.1.10 Hydrochloric acid/hydrogen peroxyde solution, $c(HCl/H_2O_2)$ = 4 $mol \cdot l^{-1}$/1,5 %.

A.2.1.11 Hydroxylamine solution, $c(NH_2OH \cdot HCl)$ = 1 $mol \cdot l^{-1}$.

A.2.1.12 MnO_2 based resin (1,5 g).

A.2.1.13 Strong cation exchange resin (e.g. AG50Wx8) – 1 ml to 10 ml resin.