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Water quality — Strontium 90 — Test method using ICP-MS

Qualité de l'eau — Strontium 90 — 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 documents 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 Technical Committee 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 production and use of phosphate fertilisers).

— Anthropogenic radionuclides, such as ^{55}Fe, ^{59}Ni, ^{63}Ni, ^{90}Sr and ^{99}Tc, transuranic elements (e.g. Np, Pu, Am, 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 into 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 Chernobyl 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 ^{90}Sr in drinking water is 10 $\text{Bq}\cdot\text{l}^{-1}$[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[5].

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 [6] and [7].

NOTE 2 The guidance level calculated in Reference [4] is the activity concentration that results in an effective dose of 0,1 $\text{mSv}\cdot\text{a}^{-1}$ to members of the public for an intake of 2 $\text{l}\cdot\text{d}^{-1}$ 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 ^{90}Sr in water samples. The method(s) described in this document can be used for various types of waters (see Clause 1). For radiometric methods, 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. For ICP-MS methods, minor modifications to, for example, the sample pre-concentration volume and the interference separation, can be made if needed to ensure that the limit of detection, limit of quantification 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 — Strontium 90 — Test method using ICP-MS

WARNING — Persons using this document should be familiar with normal laboratory practices. 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 and to determine the applicability of any other restrictions.

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 strontium-90 (^{90}Sr) by inductively coupled plasma mass spectrometry (ICP-MS). The mass concentrations obtained can be converted into activity concentrations.

The method described in this document is applicable to test samples of supply water, drinking water, rainwater, surface and ground water, as well as cooling water, industrial water, domestic and industrial wastewater after proper sampling and handling and test sample preparation.

The limit of detection depends on the sample volume, the instrument used, the background count rate, the detection efficiency and the chemical yield. In this document, the limit of detection of the method using currently available apparatus and chemical pre-concentration is approximately 5 Bq·l^{-1}, which is lower than the WHO criteria for safe consumption of drinking water (10 Bq·l^{-1})[4].

The method described in this document covers the measurement of ^{90}Sr in water at activity concentrations up to 1 000 Bq·l^{-1}. Samples with higher activity concentrations than 1 000 Bq·l^{-1} can be measured if a dilution is performed.

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

Filtration of the test sample is necessary for the method described in this document. The analysis of ^{90}Sr 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/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/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 17294-1:2024, *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*

3 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 terminological 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/

4 Symbols

a	Regression line slope	$s^{-1} \cdot g^{-1} \cdot kg^{-1}$
b	Coordinate at the origin of the regression line	s^{-1}
C	Mass activity corresponding to the mass concentration, ρ, measured for a given radionuclide	$Bq \cdot kg^{-1}$
c_{int}	Internal standard correction factor	—
c_S	Specific activity corresponding to one gram of the radionuclide	$Bq \cdot g^{-1}$
I_{84}	Isotopic abundance of ^{84}Sr: 0,005 6 (natural isotopic abundance is assumed)	—
I_{86}	Isotopic abundance of ^{86}Sr: 0,098 6 (natural isotopic abundance is assumed)	—
k	Coverage facto used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty	—
L_D	Limit of detection, the lowest mass concentration that can be detected with statistical uncertainty	$g \cdot g^{-1}$
L_Q	Limit of quantification, the lowest mass concentration that can be quantified with statistical uncertainty	$g \cdot g^{-1}$
M	Isotope mass number	—
m	Mass of the sample	kg
Δm	Mass difference	—
m_c	Mass of the calibration standard tracer solution added to a standard	g
m_{cs}	Mass of the calibration standard solution added to a standard	g
m_{IS}	Mass of the internal standard added to a blank and a sample	g
m_{ISS}	Mass of the internal standard solution added to a blank or a sample	g
m_{SP}	Mass of the enriched spike solution	g

m/z	Mass-to-charge ratio measured by ICP-MS	—
N	Number of counts per second measured by ICP-MS of a sample at a given mass-to-charge ratio	counts·s^{-1}
N_0	Number of counts per second measured by ICP-MS of a blank sample at a given mass-to-charge ratio	counts·s^{-1}
$\overline{N_0}$	Average number of counts per second for several blank samples measured by ICP-MS at a given mass-to-charge ratio	counts·s^{-1}
N_{net}	Net number of counts per second, $N-N_0$	counts·s^{-1}
r	Measured isotopic ratio	—
R	Corrected isotopic ratio	—
S_{N_0}	Standard deviation obtained by measurement of 10 test portions of the blank sample	counts·s^{-1}
U	Expanded uncertainty and the coverage factor k with $k = 1, 2, ..., U = k \cdot u$	Bq·kg^{-1}
u	Uncertainty such as counts per second, mass added etc.	—
$u(C)$	Standard uncertainty of the mass activity result	Bq·kg^{-1}
u_{cal}	Uncertainty associated with the calibration	g·g^{-1}
$u(I_{84})$	Uncertainty on isotopic abundance of ^{84}Sr: 0,000 2 (natural isotopic abundance is assumed)	—
$u(I_{86})$	Uncertainty on isotopic abundance of ^{86}Sr: 0,002 0 (natural isotopic abundance is assumed)	—
$u(\rho)$	Standard uncertainty associated with the measurement result	g·kg^{-1}
V	Volume of the sample	l
α	Measurement bias constant which allows a correction for the signal intensity bias between the tracer and the analyte	—
ρ	Mass concentration of the analyte for a given radionuclide per sample unit mass	g·kg^{-1}
ρ_{c}	Mass concentration of the calibration standard solution	g·g^{-1}
ρ_{IS}	Mass concentration of the internal standard	g·g^{-1}
$\rho_{(\text{Sr})}$	Mass concentration of stable Sr	g·kg^{-1}
ρ_{v}	Mass concentration of a given radionuclide per sample unit volume	g·l^{-1}
ρ_{SP}	Mass concentration of the spike	g·g^{-1}

5 Principle

The principle of measurement of analysis using ICP-MS is described in ISO 17294-1 and ISO 17294-2.

ICP-MS has been successfully used to measure the mass concentration of ^{90}Sr in water samples [8]-[14].

The results can be converted in activity concentrations using the specific activity as a conversion factor, which is given in Table 1.

Table 1 — Half-life and specific activity of ^{90}Sr[15]

Isotope	Half-life years	Specific activity Bq·g^{-1}
^{90}Sr	28,80 (7)	5,112 (12)·10^{12}

When performing the analysis of the pure beta-emitting radionuclide ^{90}Sr using radiation measurement, it is imperative to eliminate interfering nuclides prior to measurement. Nevertheless, ICP-MS, by discerning analyte ions through their mass-to-charge ratio, streamlines the pretreatment process before radiation measurement, thereby shortening the overall analysis time.

The limit of detection of direct measurement by ICP-MS is higher than the WHO criteria for safe consumption of drinking water (10 Bq·l^{-1})[4]. Therefore, preconcentration of water samples is essential prior to ICP-MS when demonstration of meeting this particular criterion is the aim.

An example of the limit of detection that can be obtained with ICP-MS with preconcentration and chemical separation is given in Table 2.

Table 2 — Example of the limit of detection

Isotope	Limit of detection µg·l^{-1}	Limit of detection Bq·l^{-1}
^{90}Sr	2·10^{-6}	10

The measurement of analysis of ^{90}Sr by ICP-MS is affected by several interferences shown in Table 3.

Table 3 — Interference of ^{90}Sr affecting ICP-MS measurement[8],[9],[10],[16]

Type of interference	Description	^{90}Sr interference
Isobaric	Isotopes with a similar mass to the analyte	^{90}Zr
Polyatomic	Isotopes combining in the plasma to form an ion with a similar mass to the analyte	^{50}Cr^{40}Ar, ^{50}Ti^{40}Ar, ^{74}Ge^{16}O, ^{74}Se^{16}O, ^{89}YH
Tailing interference	Isotopes of one or two mass units on either side of the analyte with a relatively high abundance (>10^{6}) relative to the analyte	^{88}Sr

It is important to ensure that all potential interferences have been removed prior to measurement. The most significant interference affecting ^{90}Sr measurement by ICP-MS is stable isobaric ^{90}Zr. The other interferences that shall be considered are polyatomic, most notably ^{74}Ge^{16}O and ^{74}Se^{16}O, and tailing from stable ^{88}Sr.

A chemical separation is performed to remove most of the potential interferences.

It is important to know the interference decontamination factor achievable by chemical separation. This can initially be assessed by running stable element standards at increasing concentrations to monitor the impact at $m / z = 90$.

An aliquot of a water sample can be directly measured by ICP-MS to determine the stable element composition. Samples with elevated levels of dissolved salts can need to be diluted to a greater extent before this measurement, depending on the sample introduction system of the instrument used. Some designs offer online aerosol dilution capability that can run high matrix samples such as seawater without prior dilution.

If some potential interferences are still present in the sample after chemical separation, it will result in an invalid result unless a correction is performed. The interference from ^{90}Zr can be monitored by measuring the ^{91}Zr isotope at $m / z = 91$ and correcting for ^{90}Zr assuming natural isotopic ratios. The same approach can be used to correct for ^{74}Ge^{16}O by monitoring Ge at $m / z = 72$. Selenium can be monitored at $m / z = 78$, and yttrium can be monitored at $m / z = 89$. This type of correction should only be used if absolutely necessary, as it increases the measurement uncertainty and affects the detection limit and measurement precision.

Chemical separation is required to remove interferences, sample matrix and pre-concentrate ^{90}Sr prior to measurement. As described in the ISO 17294 series, a chemical yield tracer is needed to evaluate the chemical recovery.

Stable strontium is usually used to determine the chemical yield. Isotope dilution with an enriched strontium isotope spike is an alternative solution to correct for ^{90}Sr losses.

It is also important to evaluate the mass bias and to correct it.

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

6 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 the different types of water in References [17] to [23]. It is important that the laboratory receives a sample that is truly representative and has neither been damaged nor modified during either transportation or storage.

The sample is filtered to remove suspended matter using a 0,45 µm filter (e.g. 0,45 µm PTFE membrane). A smaller pore size filter can also be used, but the filtration can be more time consuming. The sample shall be acidified after filtration to a pH of less than 2 using HNO_3.

Minimising contamination and losses is of primary concern. Dust in the laboratory and impurities in the reagents and on the laboratory equipment, which are in contact with the samples, can be potential sources of stable element contamination that increases the background at $m / z = 90$. The sample containers can lead to either a positive or a negative bias in the determination of trace elements by superficial desorption or adsorption.

7 Chemical reagents and equipment

7.1 General

Impurities in reagents and gases can increase the detection limit by increasing the background and decreasing sensitivity of the analyte. 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.

The chemical reagents and equipment used for chemical treatment and preparation of the samples are described in Annexes A, B and C.

7.2 Chemical reagents

7.2.1 Ultrapure water, with a resistivity of 18,2 MΩ·cm at 25 °C and total organic carbon of less than 1 $\mu g \cdot l^{-1}$.

Unless otherwise stated, water refers to ultrapure water.

7.2.2 Instrument blank, for example 0,3 $mol \cdot l^{-1}$ nitric acid used to determine the background count rate of the instrument at selected mass-to-charge ratios.

7.2.3 Strontium-90 solution, used to prepare calibration standards to calculate the concentration in the sample.

7.2.4 Internal standard solution, prepared with a stable element. The choice of element for the reference-element solution depends on the analytical problem. Solutions of these elements should cover the mass range of interest. The concentrations of these elements in the sample should be negligibly low. If not, their concentrations shall be taken into account. For example, In at m / z = 115 has been found to be suitable for this purpose.

7.2.5 Artificially enriched stable strontium isotope spike solution, used for isotope dilution. ^{84}Sr and ^{86}Sr enriched spike are commercially available (e.g. U.S. Department of Energy and National Isotope Development Center[1]).

7.2.6 Argon gas, for plasma generation in ICP-MS. The user should consult the instrument's manufacturer on the minimum gas purity required.

7.2.7 Cell gases, such as helium, oxygen and hydrogen, of a volume fraction of at least 99,999 % pure, for collision and/or reaction cell instruments.

7.3 Apparatus

Usual laboratory apparatus and in particular the following:

7.3.1 ICP-MS and associated software, quadrupole (with or without collision or reaction cell capability), **tandem, sector field or multi-collector**. Follow the manufacturer's instruction for laboratory setup and instrument operation.

7.3.2 Argon supply, equipped with pressure control and, suitable extract and gas regulation system.

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

7.3.4 Pipette, suitable for the accurate transfer of calibration standard, spike and internal standard solution with a total precision within ±2 % (k = 2).

7.3.5 Balance, capable of achieving ±0,1 mg precision.

8 Chemical separation

It is the user's responsibility to ensure that all potential interferences have been appropriately minimized. The removal of potential interferences is limited by the decontamination factor of the method. Suggested chemical separation options are outlined in Annexes A, B and C.

Alternative chemical separation procedures that are outside the scope of this document can be used (such as those in References [24], [25] and [26]).

9 Quality control

9.1 General

Measurement 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 either a reference or spiked material. Acceptance limits should be within the limits specified by the laboratory.

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

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[1] www.isotopes.gov

9.2 Variables that can influence the measurement

Special care shall be taken in order to limit 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, and instrument, can require corrective factors to be applied to the measured results.

9.3 Instrument verification

Follow the instructions provided by the instrument manufacturer and the steps described in ISO 17294-1:2024, Clauses 7 and 9 and ISO 17294-2:2023, Clauses 8 to 11.

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 laboratory measurement limits (e.g. sensitivity, stability and uncertainty), follow the recommendations of the instrument manufacturer and perform the optimization of parameters again.

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

If a dual-mode detector or similar is used, then cross calibration of detectors can be required depending on the mass concentration 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):

$$m_c = \rho_c \cdot m_{cs} \tag{1}$$

The uncertainty on m_c can be calculated using Formula (2):

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

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):

$$y = D \cdot x + E \tag{3}$$

where

D is the gradient of the calibration line;

y is the counts per second for the analyte in the sample;

x is the analyte mass concentration;

E is the intercept for the y-axis when $x = 0$.

A linear calibration line gives values for D and E.

If a calibration curve is used to determine the mass concentration of the measurand, the sample matrix effects on the instrument sensitivity are not always accounted for. The determination of the mass concentration using a recovery tracer is a more robust approach.

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.

An internal standard shall be prepared to monitor and correct for any change in the instrument response during a run, using an element which has a similar mass and ionisation energy to ^{90}Sr and that is not present in the sample being measured, for example ^{115}In.

9.4 Method verification

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

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

10 Expression of results

10.1 General

A chemical separation of ^{90}Sr is needed prior to its measurement by ICP-MS to remove potential interferences. The losses that occur during the separation can be accounted for using either stable strontium as a recovery tracer or by using the isotope dilution method.

The analytical procedure using natural strontium isotopes as a yield tracer consists of three steps: direct measurement of stable strontium isotopes, chemical separation and measurement of isotope ratio of ^{90}Sr to stable strontium isotope. A ^{90}Sr concentration can be evaluated based on the product of stable strontium concentration and the ^{90}Sr-to-stable strontium isotope ratio.

The analytical procedure using the isotope dilution method consists of adding a known amount of artificially enriched strontium isotope to the sample solution prior to the chemical separation. The isotopic ratios are maintained through the preconcentration, chemical separation and mass spectrometry measurement steps.

Follow the instructions provided by the instrument manufacturer and the steps described in ISO 17294-1:2024, Clauses 7 and 9 and ISO 17294-2:2023, Clauses 8 to 11.

The sensitivity, the instrumental detection limit and the precision should be established for each analysis performed with the instrument.

The result is expressed as an estimate of the “true” value, to which an uncertainty is associated, itself a combination of elementary uncertainties.

The results, with their associated uncertainties, are expressed in mass concentration. The coverage factor is specified in the presentation of the results.

10.2 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 are carried out, apply the appropriate factor to the values of the sample.

10.3 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 diluted HNO_3. A blank solution shall be measured at regular intervals to verify that all remaining ^{90}Sr and interferences are removed from the system by returning to the baseline.

Depending on the radionuclide and interfering element chemistry and concentration, more than one rinsing solution of different concentrations can be required to return the count rate 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 a built-in background correction capability. The user shall take care that only one background correction is applied.

10.4 Mass bias evaluation

The mass bias is a fundamental notion in mass spectrometry and the user shall take it into account during calculations to obtain a good accuracy of measurement. This is a systematic error produced by the instrument. However, the mass bias can be relatively small for heavy ions as they are better focused through the skimmer cone after the sampling cone and can potentially be neglected.

This fractionation coefficient deviation can be defined as a function of the different masses studied. The true isotope ratio, R, of isotopes A and B can be expressed from the measured isotope ratio, r, by different relations called linear law, power law, kinetic law, equilibrium law or generalized power law.

There are four stable isotopes of strontium: ^{84}Sr (0,56 atom%), ^{86}Sr (9,86 atom%), ^{87}Sr (7,00 atom%) and ^{88}Sr (82,58 atom%). It should be noted that ^{88}Sr can exceed the range of the detector after preconcentration.

The bias per unit mass, α, is determined measuring stable strontium isotopes in samples.

The linear law is commonly used, as shown by Formula (4):

$$\frac{r}{R}=1+\alpha\cdot\Delta m \tag{4}$$

where α is the fractionation factor, which is evaluated by measurement of two stable strontium isotopes (e.g. A: ^{86}Sr, B: ^{87}Sr) using Formula (5):

$$\alpha=\left(\frac{N_{\mathrm{net(A)}}}{N_{\mathrm{net\,(B)}}}\cdot\frac{1}{R_{\mathrm{(A/B)}}}-1\right)\cdot\frac{1}{\left(M_{\mathrm{(A)}}-M_{\mathrm{(B)}}\right)} \tag{5}$$

10.5 Use of naturally occurring stable strontium as chemical yield tracer

10.5.1 Internal standard

An internal standard shall be added to the samples before measurement, including a blank sample. The signal for internal standard monitors for changes in the 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 partially blocked during a run, reducing sample transmission.

A known mass of internal standard, m_{IS}, shall be added to the sample. For this purpose, a solution of known mass 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}, can be calculated using Formula (6):

$$m_{\mathrm{IS}} = \rho_{\mathrm{IS}} \cdot m_{\mathrm{ISS}} \tag{6}$$

The uncertainty on m_{IS} can be calculated using Formula (7):

$$u(m_{\mathrm{IS}}) = m_{\mathrm{IS}} \cdot \sqrt{u_{\mathrm{rel}}^2(\rho_{\mathrm{IS}}) + u_{\mathrm{rel}}^2(m_{\mathrm{ISS}})} \tag{7}$$

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

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

10.5.2 Determination of stable strontium concentration

The calibration curve is established from a series of working solutions of known and increasing concentrations, including the expected concentrations (at least six points that can be five different mass concentrations and the blank) and is expressed with Formula (8):

$$N_{\mathrm{net}} = a \cdot C + b \tag{8}$$

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

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

The calibration curve coefficient values are determined by the least-squares method.

In cases where an internal standard is added to the calibration solutions, a correction factor is introduced. This correction factor can be applied by the ICP-MS software or any validated in-house calculation sheet or software.

The mass concentration of the stable strontium, expressed in g·kg^{-1}, is equal to Formulae (9) and (10):

$$\rho(\mathrm{Sr}) = \frac{N_{\mathrm{net}}(\mathrm{Sr}) - b}{a} \tag{9}$$

$$N_{\mathrm{net}}(\mathrm{Sr}) = c_{\mathrm{int}} \cdot N(\mathrm{Sr}) - N_0(\mathrm{Sr}) \tag{10}$$

The associated uncertainty shall be determined. The measurement uncertainty is expressed in relation to the uncertainty associated with the calibration, u_{cal}, and the uncertainty associated with the measurement, as given in Formula (11):

$$u(\rho(\mathrm{Sr})) = \sqrt{u_{\mathrm{cal}}^2 + u^2(N_{\mathrm{net}}(\mathrm{Sr}))} \tag{11}$$

with Formula (12):

$$u(N_{\mathrm{net}}(\mathrm{Sr})) = \sqrt{(N(\mathrm{Sr}))^2 \cdot u^2(c_{\mathrm{int}}) + c_{\mathrm{int}}^2 \cdot u^2(N(\mathrm{Sr})) + u^2(N_0(\mathrm{Sr}))} \tag{12}$$

10.5.3 Mass bias evaluation

In Formula (13), the raw isotopic ratio is corrected to the mass bias, if needed.

$$R_{\left({}^{90}\mathrm{Sr}/{}^{\mathrm{A}}\mathrm{Sr}\right)}=\frac{N_{\mathrm{net}\,(90)}}{N_{\mathrm{net}\,(\mathrm{A})}}\cdot\frac{1}{1+\left(M_{(90)}-M_{(\mathrm{A})}\right)\cdot\alpha} \tag{13}$$

The associated uncertainty shall be determined, as shown by Formula (14):

$$u_{\mathrm{rel}}\left(R_{\left({}^{90}\mathrm{Sr}/{}^{\mathrm{A}}\mathrm{Sr}\right)}\right)=\sqrt{u_{\mathrm{rel}}^{2}\left(\frac{N_{\mathrm{net}(90)}}{N_{\mathrm{net}(\mathrm{A})}}\right)+u_{\mathrm{rel}}^{2}\left(\frac{1}{1+\left(M_{(90)}-M_{(\mathrm{A})}\right)\cdot\alpha}\right)} \tag{14}$$

10.5.4 Sample mass concentration

The mass concentration, ρ, of ^{90}Sr in the sample tested is calculated using Formula (15):

$$\rho=\rho_{(\mathrm{Sr})}\cdot I_{(\mathrm{A})}\cdot\frac{M_{(90)}}{M_{(\mathrm{A})}}\cdot R_{\left({}^{90}\mathrm{Sr}/{}^{\mathrm{A}}\mathrm{Sr}\right)} \tag{15}$$

where $I_{(\mathrm{A})}$ is the isotopic abundance of stable strontium isotope A.

The uncertainty associated with the mass concentration of ^{90}Sr is expressed by Formula (16):

$$u(\rho)=\rho\cdot\sqrt{u_{\mathrm{rel}}^{2}\left(\rho_{(\mathrm{Sr})}\right)+u_{\mathrm{rel}}^{2}\left(I_{(\mathrm{A})}\right)+u_{\mathrm{rel}}^{2}\left(R_{\left({}^{90}\mathrm{Sr}/{}^{\mathrm{A}}\mathrm{Sr}\right)}\right)} \tag{16}$$

10.5.5 Limit of detection

The limit of detection, L_{D}, corresponds to the equivalent mass concentration of three times the standard deviation of the measurement of 10 test portions of a blank sample, $S_{N(90)}$. The blank sample shall have passed through all steps of the method. The standard deviation of the measured counts per second can be converted to grams per kilogram of sample using Formula (17):

$$L_{\mathrm{D}}=\rho_{(\mathrm{Sr})}\cdot I_{(\mathrm{A})}\cdot\frac{M_{(90)}}{M_{(\mathrm{A})}}\cdot\frac{3\cdot S_{N(90)}}{N_{\mathrm{net}\,(\mathrm{A})}}\cdot\frac{1}{1+\left(M_{(90)}-M_{(\mathrm{A})}\right)\cdot\alpha} \tag{17}$$

10.5.6 Limit of quantification

The limit of quantification, L_{Q}, is 10 times the standard deviation of the measurement of 10 test portions of the blank sample. This can be calculated using Formula (18):

$$L_{\mathrm{Q}}=\rho_{(\mathrm{Sr})}\cdot I_{(\mathrm{A})}\cdot\frac{M_{(90)}}{M_{(\mathrm{A})}}\cdot\frac{10\cdot S_{N(90)}}{N_{\mathrm{net}\,(\mathrm{A})}}\cdot\frac{1}{1+\left(M_{(90)}-M_{(\mathrm{A})}\right)\cdot\alpha} \tag{18}$$

10.6 Isotope dilution method using an enriched isotope spike

10.6.1 General

The enriched stable isotope spike (e.g. ^{84}Sr) account for any loss of analyte during sample preparation.

10.6.2 Isotope spike solution

A known quantity of spike is added to the samples. The spike solution concentration and isotopic composition should be known with high precision if possible. A defined mass concentration of spike, ρ_{SP}, and added mass,

m_{SP}, of the enriched stable spike solution are recorded. The amount of $^{84}\mathrm{Sr}$ added, $m_{\left(^{84}\mathrm{Sr,SP}\right)}$, can be calculated with Formula (19):

$$m_{\left(^{84}\mathrm{Sr,SP}\right)} = \rho_{\mathrm{SP}} \cdot m_{\mathrm{SP}} \cdot I_{\left(^{84}\mathrm{Sr,SP}\right)} \tag{19}$$

where $I_{\left(^{84}\mathrm{Sr,SP}\right)}$ is the isotopic abundance of $^{84}\mathrm{Sr}$ of the spike.

The uncertainty on $m_{\left(^{84}\mathrm{Sr,SP}\right)}$ can be calculated using Formula (20):

$$u\left(m_{\left(^{84}\mathrm{Sr,SP}\right)}\right) = m_{\left(^{84}\mathrm{Sr,SP}\right)} \cdot \sqrt{u_{\mathrm{rel}}^2\left(\rho_{\mathrm{SP}}\right) + u_{\mathrm{rel}}^2\left(m_{\mathrm{SP}}\right) + u_{\mathrm{rel}}^2\left(I_{\left(^{84}\mathrm{Sr,SP}\right)}\right)} \tag{20}$$

10.6.3 Mass bias evaluation

In Formulae (21) and (22), the raw isotopic ratio is corrected to the mass bias, if needed.

$$R_{\left(^{90}\mathrm{Sr}/^{84}\mathrm{Sr}\right)_{\mathrm{sample}}} = \frac{N_{\mathrm{net}\,(90)}}{N_{\mathrm{net}\,(84)}} \cdot \frac{1}{1+\left(M_{(90)} - M_{(84)}\right)\cdot\alpha} = \frac{N_{\mathrm{net}\,(90)}}{N_{\mathrm{net}\,(84)}} \cdot \frac{1}{1+6\cdot\alpha} \tag{21}$$

$$R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)_{\mathrm{sample}}} = \frac{N_{\mathrm{net}\,(86)}}{N_{\mathrm{net}\,(84)}} \cdot \frac{1}{1+\left(M_{(86)} - M_{(84)}\right)\cdot\alpha} = \frac{N_{\mathrm{net}\,(86)}}{N_{\mathrm{net}\,(84)}} \cdot \frac{1}{1+2\cdot\alpha} \tag{22}$$

The associated uncertainty shall be determined, as shown by Formulae (23) and (24):

$$u_{\mathrm{rel}}\left(R_{\left(^{90}\mathrm{Sr}/^{84}\mathrm{Sr}\right)_{\mathrm{sample}}}\right) = \sqrt{u_{\mathrm{rel}}^2\left(\frac{N_{\mathrm{net}(90)}}{N_{\mathrm{net}(84)}}\right) + u_{\mathrm{rel}}^2\left(\frac{1}{1+6\cdot\alpha}\right)} \tag{23}$$

$$u_{\mathrm{rel}}\left(R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)_{\mathrm{sample}}}\right) = \sqrt{u_{\mathrm{rel}}^2\left(\frac{N_{\mathrm{net}(86)}}{N_{\mathrm{net}(84)}}\right) + u_{\mathrm{rel}}^2\left(\frac{1}{1+2\cdot\alpha}\right)} \tag{24}$$

10.6.4 Sample mass concentration

The mass concentration, ρ, of $^{90}\mathrm{Sr}$ in the sample tested is calculated using Formula (25):

$$\rho = R_{\left(^{90}\mathrm{Sr}/^{84}\mathrm{Sr}\right)_{\mathrm{sample}}} \cdot \left[\frac{R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)\mathrm{SP}} - R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)\mathrm{sample}}}{R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)\mathrm{sample}} - R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)N}} + 1\right] \cdot \frac{m_{\left(^{84}\mathrm{Sr,SP}\right)}}{m} \tag{25}$$

The uncertainty associated with the mass concentration of $^{90}\mathrm{Sr}$ is expressed by Formula (26):

$$u(\rho) = \rho \cdot \sqrt{u_{\mathrm{rel}}^2\left(R_{\left(^{90}\mathrm{Sr}/^{84}\mathrm{Sr}\right)\mathrm{sample}}\right) + u_{\mathrm{rel}}^2\left(m_{\left(^{84}\mathrm{Sr,SP}\right)}\right) + u_{\mathrm{rel}}^2(m)} \tag{26}$$

10.6.5 Limit of detection

The limit of detection, 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 standard deviation of the measured counts per second can be converted to grams per kilogram of sample using Formula (27):

$$L_{\mathrm{D}} = \frac{3 \cdot S_{N(90)}}{N_{\mathrm{net}\,(84)}} \cdot \frac{1}{1+6\cdot\alpha} \cdot \left[\frac{R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)\mathrm{SP}} - R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)\mathrm{sample}}}{R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)\mathrm{sample}} - R_{\left(^{86}\mathrm{Sr}/^{84}\mathrm{Sr}\right)N}} + 1\right] \cdot \frac{m_{\left(^{84}\mathrm{Sr,SP}\right)}}{m} \tag{27}$$

10.6.6 Limit of quantification

The limit of quantification, L_Q, is 10 times the standard deviation of the measurement of 10 test portions of the blank sample. This can be calculated using Formula (28):

$$L_Q = \frac{10 \cdot S_{N(90)}}{N_{\text{net}\,(84)}} \cdot \frac{1}{1+6 \cdot \alpha} \cdot \left[\frac{R\left({}^{86}\text{Sr}/{}^{84}\text{Sr}\right)_{\text{SP}} - R\left({}^{86}\text{Sr}/{}^{84}\text{Sr}\right)_{\text{sample}}}{R\left({}^{86}\text{Sr}/{}^{84}\text{Sr}\right)_{\text{sample}} - R\left({}^{86}\text{Sr}/{}^{84}\text{Sr}\right)_{N}} + 1 \right] \cdot \frac{m\left({}^{84}\text{Sr,SP}\right)}{m} \tag{28}$$

10.7 Conversion of mass concentration to mass activity

The specific activity, c_S, of ^{90}Sr can be multiplied by the mass concentration, ρ, to convert mass concentrations to activity concentrations. The limit of detection, L_D, and limit of quantification, L_Q,, can be multiplied by the mass concentration, ρ, to be converted to mass activities. The conversion is carried out using Formula (29):

$$c = \rho \cdot c_S \tag{29}$$

The uncertainty of the activity concentration is calculated using Formula (30):

$$u(C) = C\sqrt{u_{\text{rel}}^2(\rho) + u_{\text{rel}}^2(c_S)} \tag{30}$$

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

10.8 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 concentration, ρ, mass, m, and volume, V, of the sample recorded during the procedure. The conversion is carried out using Formula (31):

$$\rho_v = \rho \cdot \frac{m}{V} \tag{31}$$

The uncertainty in this conversion can be calculated using Formula (32):

$$u(\rho_v) = \rho_v \cdot \sqrt{u_{\text{rel}}^2(\rho) + u_{\text{rel}}^2(m) + u_{\text{rel}}^2(V)} \tag{32}$$

11 Test report

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

a) a reference to this document (i.e. ISO 4721:2024);

b) identification of the sample;

c) the units in which the results are expressed;

d) the test result can be given according to either 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 ≤L_D,
    - if the result is between the limit of detection and the limit of quantification, the result of the measurement is expressed as ≤L_Q,

  - if the result is greater than 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 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 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:

e) the limit of application;

f) the detection limit and the limit of quantification;

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

Annex A
(informative)

Chemical separation of strontium by crown ether-based extraction chromatographic resin — Sample strontium solution in 4 mol·l^{-1} HNO_3

A.1 Principle

This technique gives options for the extraction chromatography separation and pre-concentration of strontium. The pre-concentration procedure is good for high concentration of interference element samples, whilst extraction chromatography chemical separation is fast and well suited for monitoring ^{90}Sr activity in waters by ICP-MS.

The method consists of acidification, chemical separation with a strontium specific extraction chromatographic resin and ICP-MS measurement.

A.2 Reagents and equipment

A.2.1 Reagents

A.2.1.1 Strontium specific extraction resin, functionalized by crown ether (4,4'(5')-di-*t*-butylcyclohexano 18-crown-6) in 1-octanol (e.g. Triskem Sr Resin[2]).

A.2.1.2 Ultrapure water.

Ultrapure water with a resistivity of 18,2 MΩ·cm or higher at 25 °C is recommended.

A.2.1.3 Nitric acid, HNO_3, concentrated, 15,2 mol·l^{-1}.

A.2.1.4 4 mol·l^{-1} nitric acid, dilute HNO_3 (A.2.1.3) with ultrapure water (A.2.1.2).

A.2.1.5 1 mol·l^{-1} nitric acid, dilute HNO_3 (A.2.1.3) with ultrapure water (A.2.1.2).

A.2.1.6 0,05 mol·l^{-1} nitric acid, dilute HNO_3 (A.2.1.3) with ultrapure water (A.2.1.2).

A.2.2 Equipment

Usual laboratory equipment and, in particular, the following.

A.2.2.1 Analytical balance.

A.2.2.2 Hot plate.

A.2.2.3 Centrifuge tubes.

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[2] Triskem Sr Resin is an example of suitable extraction chromatography medium. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

A.3 Procedure

A.3.1 Chemical separation

A.3.1.1 HNO_3 addition

Add 8 ml of concentrated HNO_3 (A.2.1.3) to 20 ml of water samples.

A.3.1.2 Separation and purification by extraction chromatography

The separation and purification procedure is carried out with two main steps: extraction and elution of strontium.

- Prepare a pre-packed column or cartridge containing 2 ml of strontium-specific extraction resin (A.2.1.1) and condition with 10 ml of 4 $mol \cdot l^{-1}$ HNO_3 (A.2.1.4).
- Load the sample into the column and allow it to pass through.
- Rinse the column with 4 ml of 4 $mol \cdot l^{-1}$ HNO_3 (A.2.1.4).
- Rinse strontium specific extraction resin column with 4 ml of 1 $mol \cdot l^{-1}$ nitric acid (A.2.1.5) to remove any Zr and other possible interferences that can be present and discard the column eluate.
- Elute strontium from the column with 5 ml of 0,05 $mol \cdot l^{-1}$ HNO_3 (A.2.1.6) and collect the eluate in a clean plastic vial.

A.3.2 Measurement

Perform the ICP-MS measurement.

Determine the mass concentrations of the isotopes.

Calculate the corresponding activity concentrations.

Annex B
(informative)

Chemical separation of strontium by strontium-specific extraction chromatographic resin — Sample strontium solution in 0,01 mol·l^{-1} HNO_3

B.1 Principle

This technique gives options for extraction chromatography separation and pre-concentration of strontium. The procedure is good for low ^{90}Sr activity concentration samples in high volume (up to 1 l) samples, whilst chromatography chemical separation is fast and well suited for monitoring ^{90}Sr activity in waters by ICP-MS.

The mass of the test portion shall take into account the presumed activity of the sample and the desired detection limit. The procedure described in this annex applies to water samples of a volume of 0,1 l to 1 l.

The method consists of acidification, chemical separation with a strontium-specific extraction resin and ICP-MS measurement.

B.2 Reagents and equipment

B.2.1 Reagents

B.2.1.1 Crown-ether based extraction chromatography resin, functionalized by (Di-t-butyl dicyclohexyl-18-crown-6 and Di(2-ethyl-hexyl) phosphoric acid) (HDEHP) (e.g. Triskem International TK100 resin[3]).

B.2.1.2 Ultrapure water, with a resistivity of 18,2 MΩ·cm or higher at 25 °C is recommended.

B.2.1.3 Nitric acid, HNO_3, concentrated, 15,2 mol·l^{-1}.

B.2.1.4 Hydrochloric acid, HCl, concentrated, 12 mol·l^{-1}.

B.2.1.5 0,01 mol·l^{-1} nitric acid, dilute HNO_3 (B.2.1.3) with ultrapure water (B.2.1.2).

B.2.1.6 2 mol·l^{-1} hydrochloric acid, dilute HNO_3 (B.2.1.4) with ultrapure water (B.2.1.2).

B.2.2 Equipment

Usual laboratory equipment and, in particular, the following.

B.2.2.1 Analytical balance.

B.2.2.2 Hot plate.

B.2.2.3 Centrifuge tubes.

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[3] Triskem International TK100 resin is an example of suitable extraction chromatography medium. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

B.3 Procedure

B.3.1 HNO_3 addition

Add the concentrated HNO_3 (B.2.1.3) to achieve a nitric acid concentration of 0,01 $mol \cdot l^{-1}$ in the sample solution.

B.3.2 Chemical separation: Preconcentration and separation by extraction chromatography resin

This procedure is carried out with two main steps: retention and elution of strontium.

Prepare a pre-packed column or cartridge containing 2 ml of extraction chromatography resin (B.2.1.1) and condition with 10 ml of 0,01 $mol \cdot l^{-1}$ HNO_3 (B.2.1.5).

Load the sample into the column and allow it to pass through.

Elute strontium from the column with 20 ml of 2 $mol \cdot l^{-1}$ HCl (B.2.1.6) and collect the eluate in a clean glass beaker.

NOTE 1 Under 0,01 $mol \cdot l^{-1}$ HNO_3 and 2 $mol \cdot l^{-1}$ HCl conditions, Zr is well retained on the resin and will not be eluted.

NOTE 2 3 $mol \cdot l^{-1}$ HCl and 0,01 $mol \cdot l^{-1}$ EDTA have also been shown to effectively elute strontium from TK100 resin.

B.3.3 Measurement

Perform the ICP-MS measurement.

Determine the mass concentrations of the isotopes.

Calculate the corresponding activity concentrations.