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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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Foreword

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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 $Bq \cdot l^{-1}$[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 $mSv \cdot a^{-1}$ to members of the public for an intake of 2 $l \cdot d^{-1}$ 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.

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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}
$\rho_{(Sr)}$	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	Specific activity
	years	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	Limit of detection
	μg·l^{-1}	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 μg·l^{-1}.

Unless otherwise stated, water refers to ultrapure water.

7.2.2 Instrument blank, for example 0,3 mol·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,