
**Water quality — Strontium 90 and
strontium 89 — Test methods using
liquid scintillation counting or
proportional counting**

*Qualité de l'eau — Strontium 90 et strontium 89 — Méthodes d'essai
par comptage des scintillations en milieu liquide ou par comptage
proportionnel*

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Contents

Page

Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Principle	2
4.1 General.....	2
4.2 Chemical separation.....	3
4.3 Detection.....	3
5 Chemical reagents and equipment	3
6 Procedure	4
6.1 Test sample preparation.....	4
6.2 Chemical separation.....	4
6.2.1 General.....	4
6.2.2 Precipitation techniques.....	5
6.2.3 Liquid-liquid extraction technique.....	6
6.2.4 Chromatographic technique.....	6
6.3 Preparation of the source for test.....	6
6.3.1 Source preparation for liquid scintillation counter.....	6
6.3.2 Source preparation for proportional counter.....	6
6.4 Measurement.....	7
6.4.1 General.....	7
6.4.2 Liquid scintillation counter.....	7
6.4.3 Proportional counter.....	7
6.4.4 Efficiency calculation.....	8
6.4.5 Determination of the chemical yield.....	8
7 Expression of results	9
7.1 Determination of ^{90}Sr in equilibrium with ^{90}Y	9
7.1.1 Calculation of the activity concentration.....	9
7.1.2 Standard uncertainty.....	9
7.1.3 Decision threshold.....	10
7.1.4 Detection limit.....	10
7.2 Determination of ^{90}Sr from separated ^{90}Y	10
7.2.1 Calculation of the activity concentration.....	10
7.2.2 Standard uncertainty.....	11
7.2.3 Decision threshold.....	12
7.2.4 Detection limit.....	12
7.3 Determination of ^{90}Sr and ^{89}Sr utilizing $^{90}\text{Sr}/^{90}\text{Y}$ equilibrium.....	12
7.3.1 Calculation of the activity concentration.....	12
7.3.2 Standard uncertainty.....	13
7.3.3 Decision threshold.....	14
7.3.4 Detection limit.....	15
8 Limits of the coverage intervals	15
8.1 Limits of the of the probabilistically symmetric coverage interval.....	15
8.2 Limits of the shortest coverage interval.....	16
9 Quality control	16
10 Test report	16
Annex A (informative) Determination of ^{89}Sr and ^{90}Sr by precipitation and proportional counting	18

Annex B (informative) Determination of ^{89}Sr and ^{90}Sr by precipitation and liquid scintillation counting	22
Annex C (informative) Determination of ^{90}Sr from its decay progeny ^{90}Y at equilibrium by organic extraction and liquid scintillation counting	26
Annex D (informative) Determination of ^{90}Sr after ionic exchange separation by proportional counting	29
Annex E (informative) Determination of ^{90}Sr after separation on a crown ether specific resin and liquid scintillation counting	32
Annex F (informative) Determination of ^{90}Sr from its decay progeny ^{90}Y at equilibrium by organic extraction and proportional counting	34
Annex G (informative) Correction factor for ^{90}Sr purity using proportional counting	38
Bibliography	41

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 13160:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

- The way standard the uncertainty, decision threshold and detection limit are calculated has been updated in conformance with ISO 11929-1:2019.

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

Radioactivity from several naturally-occurring and anthropogenic sources is present throughout the environment. Thus, water bodies (e.g. surface waters, ground waters, sea waters) can contain radionuclides of natural, human-made, or both origins.

- Natural radionuclides, including ^{40}K , ^3H , ^{14}C , and those originating from the thorium and uranium decay series, in particular ^{226}Ra , ^{228}Ra , ^{234}U , ^{238}U , and ^{210}Pb , can be found in water for natural reasons (e.g. desorption from the soil and washoff by rain water) or can be released from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or phosphate fertilizer production and use).
- Human-made radionuclides, such as transuranium elements (americium, plutonium, neptunium, curium), ^3H , ^{14}C , ^{90}Sr , and gamma emitting radionuclides can also be found in natural waters. Small quantities of these radionuclides are discharged from nuclear fuel cycle facilities into the environment as the result of authorized routine releases. Some of these radionuclides used for medical and industrial applications are also released into the environment after use. Anthropogenic radionuclides are also found in waters as a result of past fallout contaminations resulting from the explosion in the atmosphere of nuclear devices and accidents such as those that occurred in Chernobyl and Fukushima.

Radionuclide activity concentration in water bodies can vary according to local geological characteristics and climatic conditions and can be locally and temporally enhanced by releases from nuclear installation during planned, existing and emergency exposure situations^[1]. Drinking water may thus contain radionuclides at activity concentrations which could present a risk to human health.

The radionuclides present in liquid effluents are usually controlled before being discharged into the environment^[2]. Water bodies and drinking waters are monitored for their radioactivity content as recommended by the World Health Organization (WHO)^[3] so that proper actions can be taken to ensure that there is no adverse health effect to the public. Following these international recommendations, national regulations usually specify radionuclide authorized concentration limits for liquid effluent discharged to the environment and radionuclide guidance levels for water bodies and drinking waters for planned, existing and emergency exposure situations. Compliance with these limits can be assessed using measurement results with their associated uncertainties as specified by ISO/IEC Guide 98-3 and ISO 5667-20^[4].

Depending on the exposure situation, there are different limits and guidance levels that would result in an action to reduce health risk. As an example, during a planned or existing situation, the WHO guidelines for guidance level in drinking water is $100 \text{ Bq}\cdot\text{l}^{-1}$ for ^{89}Sr activity concentration and $10 \text{ Bq}\cdot\text{l}^{-1}$ for ^{90}Sr activity concentration^[3].

NOTE 1 The guidance level is the activity concentration with an intake of $2 \text{ l}\cdot\text{d}^{-1}$ of drinking water for one year that results in an effective dose of $0,1 \text{ mSv}\cdot\text{a}^{-1}$ for members of the public. This is an effective dose that represents a very low level of risk and which is not expected to give rise to any detectable adverse health effects^[3].

In the event of a nuclear emergency, the WHO Codex guideline levels^[5] specifies that the activity concentration might not be greater than $1\,000 \text{ Bq}\cdot\text{kg}^{-1}$ for ^{89}Sr or $100 \text{ Bq}\cdot\text{kg}^{-1}$ for ^{90}Sr for infant food and $1\,000 \text{ Bq}\cdot\text{kg}^{-1}$ for ^{89}Sr or $100 \text{ Bq}\cdot\text{kg}^{-1}$ for ^{90}Sr for food other than infant food.

NOTE 2 The Codex guidelines levels (GLs) apply to radionuclides contained in food destined for human consumption and traded internationally, which have been contaminated following a nuclear or radiological emergency. These GLs apply to food after reconstitution or as prepared for consumption, i.e. not to dried or concentrated food, and are based on an intervention exemption level of 1 mSv in a year for members of the public (infant and adult)^[5].

Thus, the test method can be adapted so that the characteristic limits, decision threshold, detection limit and uncertainties ensure that the radionuclide activity concentrations test results can be verified to be below the guidance levels required by a national authority for either planned/existing situations or for an emergency situation^[6]^[7].

Usually, the test methods can be adjusted to measure the activity concentration of the radionuclide(s) in either wastewaters before storage or in liquid effluents before discharge to the environment. The test results will enable the plant/installation operator to verify that, before their discharge, wastewaters/liquid effluent radioactive activity concentrations do not exceed authorized limits.

The test method described in this document may be used during planned, existing and emergency exposure situations as well as for wastewaters and liquid effluents with specific modifications that could increase the overall uncertainty, detection limit, and threshold.

The test method may be used for water samples after proper sampling, sample handling, and test sample preparation (see the relevant part of the ISO 5667 series [8],[9],[10]).

This document has been developed to answer the need of test laboratories carrying out these measurements, that are sometimes required by national authorities, as they may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

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Water quality — Strontium 90 and strontium 89 — Test methods using liquid scintillation counting or proportional counting

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 and to determine the applicability of any other restrictions.

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

1 Scope

This document specifies conditions for the determination of ^{90}Sr and ^{89}Sr activity concentration in samples of environmental water using liquid scintillation counting (LSC) or proportional counting (PC).

The method is applicable to test samples of drinking water, rainwater, surface and ground water, marine water, as well as cooling water, industrial water, domestic, and industrial wastewater after proper sampling and handling, and test sample preparation. Filtration of the test sample and a chemical separation are required to separate and purify strontium from a test portion of the sample.

The detection limit depends on the sample volume, the instrument used, the sample count time, the background count rate, the detection efficiency and the chemical yield. The method described in this document, using currently available LSC counters, has a detection limit of approximately 10 mBq l^{-1} and 2 mBq l^{-1} for ^{89}Sr and ^{90}Sr , respectively, which is lower than the WHO criteria for safe consumption of drinking water (100 Bq l^{-1} for ^{89}Sr and 10 Bq l^{-1} for ^{90}Sr)^[3]. These values can be achieved with a counting time of 1 000 min for a sample volume of 2 l.

The methods described in this document are applicable in the event of an emergency situation. When fallout occurs following a nuclear accident, the contribution of ^{89}Sr to the total amount of radioactive strontium is not negligible. This document provides test methods to determine the activity concentration of ^{90}Sr in presence of ^{89}Sr .

The analysis of ^{90}Sr and ^{89}Sr adsorbed to suspended matter is not covered by this method.

It is the user's responsibility to ensure the validity of this test method selected 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 for 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 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 11929-1, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 1: Elementary applications*

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

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 11929-1 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 <http://www.electropedia.org/>

Symbol	Definition	Unit
A_i	calibration source activity of radionuclide i , at the time of calibration	Bq
$c_{A,i}$	activity concentration of radionuclide i	Bq l ⁻¹
$c_{A,i}^*$	decision threshold of radionuclide i	Bq l ⁻¹
$c_{A,i}^\#$	detection limit of radionuclide i	Bq l ⁻¹
$c_{A,i}^<, c_{A,i}^>$	lower and upper limits of the probabilistically symmetric coverage interval of radionuclide i	Bq l ⁻¹
$c_{A,i}^{<}, c_{A,i}^{>}$	lower and upper limits of the shortest coverage interval of radionuclide i	Bq l ⁻¹
k_p	quantile of the standardized normal distribution for the probability p (for instance $p=1-\alpha$, $1-\beta$ or $1-\gamma/2$)	
$R_{c,i}$	chemical yield of the extraction of radionuclide i	
r_0	background count rate	s ⁻¹
r_{0j}	background count rate for measurement j	s ⁻¹
r_g	gross count rate	s ⁻¹
r_{gj}	gross count rate for measurement j	s ⁻¹
r_j	net count rate for measurement j	s ⁻¹
r_s	calibration source count rate	s ⁻¹
t	time elapsed between separation of ⁹⁰ Sr/ ⁹⁰ Y ($t = 0$) and mid-point of counting	s
t_0	background counting time	s
t_d, t_f	start and finish time respectively of the measurement, referred to $t = 0$	s
t_g	sample counting time	s
t_j	start time of the measurement j , referred to $t = 0$	s
t_s	calibration source counting time	s
U	expanded uncertainty, calculated by $U = ku(c_A)$ with $k = 1, 2 \dots$	Bq l ⁻¹
$u(c_A)$	standard uncertainty associated with the measurement result	Bq l ⁻¹
V	volume of the test sample	l
α, β	probability of a false positive and false negative decision, respectively	
ε_i	counting efficiency for radionuclide i	
λ_i	decay constant of radionuclide i	

4 Principle

4.1 General

The radionuclides ⁹⁰Sr, ⁹⁰Y and ⁸⁹Sr are all pure beta-particle emitters. Their beta-emission energies and half-lives are given in [Table 1](#).

Table 1 — Half-lives, maximum energies, and average energies of ^{90}Sr , ^{90}Y , and ^{89}Sr [11]

Parameter	^{90}Sr	^{90}Y	^{89}Sr
Maximum energy	546,0 keV	2 283,9 keV	1 491,0 keV
Average energy	196,4 keV	935,3 keV	586,3 keV
Half-life	28,80 (7) a	2,6684 (13) d	50,57 (3) d

Strontium-90 can either be measured directly or estimated through the measurement of its decay progeny ^{90}Y . All the test methods are based on a chemical separation step followed by beta-counting using proportional counting (PC) or liquid scintillation counting (LSC). See [Table 2](#) which contains guidance over method selection.

4.2 Chemical separation

Strontium is isolated from the water using precipitation, ion exchange or specific chromatographic separation by crown ether resin^[12]. Yttrium can then be isolated by precipitation or liquid-liquid extraction.

The method chosen shall be selective with a high chemical yield. When certain radionuclides such as thorium, lead or bismuth radioisotopes are present at high activity levels, they may interfere with ^{90}Sr , ^{90}Y or ^{89}Sr detection. Other matrix constituents, such as other alkaline earth metals, particularly calcium which interferes with strontium separation; or transuranic and lanthanide elements which interfere with yttrium separation, reduce the chemical yield of the various extraction steps.

The radiochemical separation yield is calculated using a carrier such as stable strontium or yttrium, or a radioactive tracer such as ^{85}Sr . Techniques such as atomic absorption spectroscopy (AAS), inductively coupled plasma-atomic emission spectroscopy (ICP-AES) or inductively coupled plasma-mass spectrometry (ICP-MS) to measure the carrier, and gamma-spectrometry to measure ^{85}Sr , are recommended. A carrier can also be measured by gravimetric methods, but the presence of inactive elements, notably alkaline earth elements, in the leaching solutions can lead to an overestimation of the radiochemical separation yields, particularly for the measurement of strontium.

When stable strontium is added as a carrier, the original strontium concentration in the test sample shall be known to determine the chemical yield.

4.3 Detection

The use of LSC, which provides spectra and may allow the detection of interference from unwanted radionuclides, is recommended in preference to PC, which does not distinguish between emissions from different beta-emitters. When PC is used, it is recommended that the purity of the precipitate is checked by following the change over an appropriate time of the ^{90}Y or ^{89}Sr activity, even though this is time consuming.

Six test methods are presented in [Annexes A, B, C, D, E and F](#).

5 Chemical reagents and equipment

The necessary chemical reagents and equipment for each strontium measurement method are specified in [Annexes A, B, C, D, E and F](#).

During the analyses, unless otherwise stated, use only reagents of recognized analytical grade and laboratory water such as distilled or demineralized water or water of equivalent purity as specified in ISO 3696.

6 Procedure

6.1 Test sample preparation

Strontium-89/90 activity concentration is determined from the water test sample after appropriate sampling procedures^{[13][14]}.

Filtration should be done prior to the addition of the tracer or carrier and sufficient time should be allowed to attain chemical equilibrium before starting the test sample preparation.

When stable strontium is added as a carrier, the original strontium concentration in the test sample shall be known in order to determine the chemical yield. In the case of separation based on extraction chromatography, the total strontium content shall be below the sorption capacity of the resin to avoid saturation of the resin.

6.2 Chemical separation

6.2.1 General

There are several approaches to the routine analysis of ⁸⁹Sr and ⁹⁰Sr involving the separation and purification of strontium or yttrium: precipitation, liquid-liquid extraction or chromatographic techniques (ion exchange or chromatographic extraction). [Annexes A, B, C, D, E and F](#) describe a test method for each of these techniques.

Table 2 — Determination procedures for strontium depending on its origin

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Origin		Old contamination				Fresh contamination	
Radionuclide		$^{90}\text{Sr}+^{90}\text{Y}$				$^{90}\text{Sr}+^{90}\text{Y}$ ^{89}Sr	
Separation	Element	Sr		Y ^a		Sr	
	Method	Chromatography ^b	Precipitation	Extraction	Precipitation	Chromatography ^b	Precipitation
	Product	^{90}Sr		^{90}Y		$^{90}\text{Sr}, ^{90}\text{Y}, ^{89}\text{Sr}$	
	Carrier or Tracer ^c	^{85}Sr or stable Sr		Stable Y		^{85}Sr or stable Sr	
Measurement(s)	Equilibrium $^{90}\text{Sr}+^{90}\text{Y}$ 20 d	Yes (recommended)	No	No		Yes	No
	Number	One		One		Two or more	
	Emissions	^{90}Sr ^{90}Y		^{90}Y		^{90}Sr ^{90}Y ^{89}Sr	
	Equipment	PC or LSC (total)		PC or LSC (total or Cerenkov)		PC or LSC (total)	
	Calibration sources	$^{90}\text{Sr}+^{90}\text{Y}$	^{90}Sr ^{90}Y	^{90}Y		$^{90}\text{Sr}+^{90}\text{Y}$ ^{89}Sr	^{90}Sr ^{90}Y ^{89}Sr
<p>^a Y separation is performed following the $^{90}\text{Sr}-^{90}\text{Y}$ equilibrium in the test sample.</p> <p>^b Liquid chromatography or specific chromatography using crown ether resin.</p> <p>^c Carrier or tracer element measurements can be taken using gamma-spectrometry for ^{85}Sr (tracer), by gravimetry, atomic absorption spectrometry (AAS), inductively coupled plasma (ICP) or mass spectrometry (MS) for Sr and Y (tracer and/or carrier).</p>							

6.2.2 Precipitation techniques

A precipitation step is suitable for the separation of most mineral elements, including strontium, in water samples with high mineral salt contents. This technique is very efficient, but not selective for strontium. The use of large quantities of nitric acid and the need to wait for the ^{90}Y to reach equilibrium limit its use.

The addition of fuming nitric acid leads to a strontium precipitate with other interfering elements. Successive dissolution-precipitation cycles concentrate strontium in the precipitate, while yttrium and other elements remain in the supernatant fraction. The most usual procedure leads to a SrCO_3 precipitate.

For the test method with ^{90}Sr and ^{90}Y in equilibrium, either the total concentration of yttrium and strontium is directly measured in the precipitate or the yttrium activity is measured after a separation from the strontium. In this latter case, the chemical yield is estimated by the addition of an yttrium carrier to the source before the yttrium separation. The final product is an yttrium precipitate, usually in the form of an oxalate.

In the absence of ^{89}Sr , ^{90}Sr is measured by counting the beta-emission of ^{90}Y or of ^{90}Y and ^{90}Sr . In the latter case, the sample can be counted at equilibrium or counted at any time if a mathematical correction for ^{90}Y ingrowth and ^{90}Sr decay is applied. When ^{89}Sr in the water test sample cannot be neglected, the direct measurement method of strontium at two different times shall be chosen.

Two precipitation methods are described: [Annex A](#) employs PC for ^{89}Sr and ^{90}Sr ; [Annex B](#) employs LSC for ^{89}Sr and ^{90}Sr .

6.2.3 Liquid-liquid extraction technique

This technique is based on the extraction of ^{90}Y in equilibrium with its radioactive parent ^{90}Sr using an organic solvent. The chemical separation is fast and requires few technical resources. A provisional result may be achieved after 3 d (approximately one ^{90}Y decay period). However, total selectivity of the extraction is not always possible. In the presence of high levels of natural radioactivity, interference may occur, making it difficult to determine very low levels of strontium activity.

Yttrium-90 is extracted from the water test sample fraction using an organic solvent, and then after re-extraction, recovered in the form of an yttrium precipitate. Test methods are presented in [Annexes C](#) and [E](#).

After the source preparation, the ^{90}Y is measured by PC ([Annex F](#)) or LSC ([Annex C](#)). The absence of other interfering beta-emitters is verified during the decay of ^{90}Y by measuring the decrease in count rate of the ^{90}Y and once the decay is complete, comparing it with the background level activity.

6.2.4 Chromatographic technique

6.2.4.1 Ion exchange resin

This technique is based on Sr(II) exchange on a cationic resin and is used for separation and purification of strontium in large volume samples. A method is presented in [Annex D](#) in which the measurement is carried out with PC.

6.2.4.2 Crown ether resin

This technique is based on the selective chromatographic separation of strontium using a specific crown ether resin. However, this method may have limited application for samples containing high amounts of stable Sr and Ca in amounts exceeding the resin sorption capacity. A method is presented in [Annex E](#) in which the measurement is carried out by LSC.

6.3 Preparation of the source for test

6.3.1 Source preparation for liquid scintillation counter

The strontium or yttrium precipitate is dissolved and mixed with a liquid scintillation cocktail. When the strontium or yttrium is already in solution, it is mixed directly with the liquid scintillator. The volume of the analysed aliquot depends on the equipment (vial size) and the specific scintillation cocktail used.

The calibration source shall be prepared from a known activity of tracer (^{90}Sr , ^{89}Sr , $^{90}\text{Sr} + ^{90}\text{Y}$ or ^{90}Y) with the same geometry and chemical composition as the source to be measured.

The blank source should be prepared following the method chosen starting with a clean test sample (or water).

6.3.2 Source preparation for proportional counter

A strontium or yttrium precipitate is deposited on a filter by filtration or on a stainless steel planchet by direct evaporation.

The filter or planchet size diameter should be similar to the detector size (see [Annex A](#), [D](#) and [G](#)).

A calibration source shall be prepared from a known amount of tracer (^{90}Sr , ^{89}Sr , $^{90}\text{Sr} + ^{90}\text{Y}$ or ^{90}Y) with the same geometry and chemical composition as the source to be measured.