
Kakovost vode - Stroncij Sr-90 in Sr-89 - Preskusne metode s štetjem s tekočinskim scintilatorjem ali proporcionalnim štetjem

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

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

[SIST ISO 13160:2013](https://standards.iteh.ai/catalog/standards/sist/d87cad81-9131-4982-8a6b-111a4ca50f5b/sist-iso-13160-2013)

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

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Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Symbols, definitions, and units	1
4 Principle	2
4.1 General	2
4.2 Chemical separation	2
4.3 Detection	3
5 Chemical reagents and equipment	3
6 Procedure	3
6.1 Test sample preparation	3
6.2 Chemical separation	3
6.3 Preparation of the source for test	5
6.4 Measurement	6
7 Expression of results	8
7.1 Determination of ^{90}Sr in equilibrium with ^{90}Y	8
7.2 Determination of ^{90}Sr by ingrowth of ^{90}Y	9
7.3 Determination of ^{90}Sr in presence of ^{89}Sr when ^{90}Sr is in equilibrium with ^{90}Y	11
7.4 Confidence limits	14
8 Quality control	14
9 Test report	15
Annex A (informative) Determination of ^{89}Sr and ^{90}Sr by precipitation and proportional counting	16
Annex B (informative) Determination of ^{89}Sr and ^{90}Sr by precipitation and liquid scintillation counting	20
Annex C (informative) Determination of ^{90}Sr from its daughter product ^{90}Y at equilibrium by organic extraction and liquid scintillation counting	24
Annex D (informative) Determination of ^{90}Sr after ionic exchange separation by proportional counting	26
Annex E (informative) Determination of ^{90}Sr after separation on a crown ether specific resin and liquid scintillation counting	29
Annex F (informative) Determination of ^{90}Sr from its daughter product ^{90}Y at equilibrium by organic extraction by proportional counting	31
Annex G (informative) Correction factor for purity control using proportional counting	35
Bibliography	38

ISO 13160:2012(E)

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 13160 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radiological methods*.

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

WARNING — Persons using this International Standard 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 ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this International Standard be carried out by suitably qualified staff.

1 Scope

This International Standard specifies the test methods and their associated principles for the measurement of the activity of ^{90}Sr in equilibrium with ^{90}Y , and ^{89}Sr , pure beta-emitting radionuclides, in water samples. Different chemical separation methods are presented to produce strontium and yttrium sources, the activity of which is determined using a proportional counter (PC) or liquid scintillation counter (LSC). The selection of the test method depends on the origin of the contamination, the characteristics of the water to be analysed, the required accuracy of test results and the available resources of the laboratories.

These test methods are used for water monitoring following, past or present, accidental or routine, liquid or gaseous discharges. It also covers the monitoring of contamination caused by global fallout.

When fallout occurs immediately following a nuclear accident, the contribution of ^{89}Sr to the total amount of strontium activity is not negligible. This International Standard provides the test methods to determine the activity concentration of ^{90}Sr in presence of ^{89}Sr .

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2 Normative references

The following referenced documents are indispensable for the application 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 11929, *Determination of the characteristic limits (decision threshold, detection limit and limits of the confidence interval) for measurements of ionizing radiation — Fundamentals and application*

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

3 Symbols, definitions, and units

For the purposes of this document, the definitions, symbols, and abbreviated terms defined in ISO 11929 and ISO 80000-10 and the following apply.

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 confidence interval of radionuclide i	Bq l^{-1}

ISO 13160:2012(E)

$R_{c,i}$	chemical yield of the extraction of radionuclide i	1
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 $^{90}\text{Sr}/^{90}\text{Y}$ ($t = 0$) and 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
ε_i	counting efficiency for radionuclide i	1
λ_i	decay constant of radionuclide i	1

4 Principle

4.1 General

^{90}Sr , ^{90}Y and ^{89}Sr are pure beta-emitter radionuclides. 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

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	28,79 a	2,67 d	50,5 d

^{90}Sr can be directly measured or estimated through the measurement of its daughter product ^{90}Y . All the test methods are based on a chemical separation step followed by beta-counting of the element using PC or LSC. See Table 2.

4.2 Chemical separation

Strontium is isolated from the water using precipitation, ion chromatography or specific chromatographic separation using crown ether resin. Yttrium can be isolated by precipitation or liquid-liquid extraction.

The separation step should maximize the extraction of the pure element. The method chosen shall be selective with a high chemical yield. When thorium, lead or bismuth radioisotopes are present at high activity levels,

they may interfere with ^{90}Sr , ^{90}Y or ^{89}Sr emission during the detection step. Other matrix constituents such as alkaline earth metals and in particular calcium for strontium, or transuranic and lanthanide elements for yttrium, reduce the chemical yield of the extraction.

The radiochemical separation yield is calculated using a carrier such as stable strontium or yttrium, or a radioactive tracer such as ^{85}Sr . Techniques like 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, essentially 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 must be known to avoid the overestimation of the radiochemical separation yield.

4.3 Detection

The use of LSC, which provides spectra and permits 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 be checked by following the change over an appropriate time of the ^{90}Y or ^{89}Sr activity, even though this method 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.^[1]

6 Procedure

6.1 Test sample preparation

Strontium is determined from the water test sample.

If filtration is required, add the tracer or carrier after this step of the procedure and allow sufficient time to attain chemical equilibrium before starting the test sample preparation.

If stable strontium is added as carrier, the original concentration shall be determined in the test sample in this step of the procedure before the addition of the carrier.

6.2 Chemical separation

6.2.1 General

There are several routine analyses of ^{89}Sr and ^{90}Sr involved in the separation and purification of strontium: 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

Origin		Old contamination				Fresh contamination	
Radionuclide		$^{90}\text{Sr}+^{90}\text{Y}$				$^{90}\text{Sr}+^{90}\text{Y}$ ^{89}Sr	
Separation	Element	Sr		$\rightarrow$ Y^{a}		Sr	
	Method	Chromatography ^b	Precipitation	Extraction	Precipitation	Chromatography ^b	Precipitation
	Product	^{90}Sr			^{90}Y	$^{90}\text{Sr}+^{89}\text{Sr}$	
	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 Cherenkov)		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
a Y separation is performed following the $^{90}\text{Sr}-^{90}\text{Y}$ equilibrium in the test sample. b Liquid chromatography or specific chromatography using crown ether resin 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).							

6.2.2 Precipitation techniques

The precipitation technique is suitable for the separation of all 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 yttrium to reach equilibrium limit its use.

The addition of 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 procedures lead to a SrCO_3 precipitate.

For the test method with ^{90}Sr and ^{90}Y at equilibrium, either the global contribution of yttrium and strontium is directly measured in the precipitate or the yttrium activity is measured after a last 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 equilibrium.

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 using an organic solvent of ^{90}Y at equilibrium with its radioactive parent ^{90}Sr . The chemical separation is fast and requires few technical resources. A provisional result may be achieved after 3 d (approximately one yttrium 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.

This test method is suitable for all samples with low activity of beta-emitting radionuclide.

^{90}Y 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 F.

After the source preparation, the ^{90}Y is measured by PC (Annex F) or by measuring the Cherenkov radiation from the ^{90}Y with 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 a 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. The chemical separation is fast and suitable for inspection and monitoring of the environment. 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

LSC measures directly the photons produced by the scintillations in the liquid as a result of the excitation caused by the beta-emissions from the source.

A strontium or yttrium precipitate is dissolved and mixed with the liquid scintillator. When the strontium or yttrium is in solution, it is mixed directly with the liquid scintillator. The volume depends on the equipment (vial size) and the specific scintillator 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

The PC measures directly the beta-emission from the source prepared from a thin layer deposit to minimize the self-absorption effects.

ISO 13160:2012(E)

The 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 or D).

The 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.

6.4 Measurement

6.4.1 General

The same equipment conditions should be used for the sample, the background and the calibration source measurements.

The blank source should be prepared following the method chosen starting with laboratory water. Measure the background using a blank source prepared for the method chosen.

The counting time used depends on the sample and background count rates and also on the detection limit and decision threshold required.

6.4.2 Liquid scintillation counter

The scintillation phenomenon results from interactions of ionizing radiations with solvents and compounds having fluorescent properties (scintillators). Both solvents and scintillators constitute the scintillation cocktail. The scintillation mixture is achieved by adding the scintillation cocktail to the test sample in order to obtain a homogeneous mixture.

The scintillation cocktail is chosen according to the characteristics of the sample to be analysed and according to the properties of the detection equipment (see ISO 18589-5^[6]). It is recommended that a hydrophilic scintillation cocktail be used, especially for the measurement of natural water.

The characteristics of the scintillation cocktail shall allow the mixture to be homogeneous and stable.

It is recommended that the scintillation cocktail be stored in the dark and, particularly just before use, exposure to direct sunlight or fluorescent light avoided in order to prevent interfering luminescence and to comply with the storage conditions specified by the scintillation cocktail supplier.

The mixtures (scintillation cocktail and test sample) should be disposed of as chemical waste, and, depending on the radioactivity, may require disposal as radioactive waste.

The measurement can be affected by chemiluminescence phenomena or quench due to chemical entities and to the presence of other radionuclides than yttrium. It is then necessary to take into account the characteristics of the water sample.

When assessing the ^{90}Sr activity by its measurement with ^{90}Y in equilibrium, two cases arise:

- the presence of ^{89}Sr can be neglected, the relevant contribution of ^{90}Y in equilibrium with ^{90}Sr can be assessed using LSC;
- the presence of ^{89}Sr cannot be neglected, it is necessary to measure the strontium at two different times, to estimate the ^{89}Sr activity through its decay.

When assessing ^{90}Sr activity by ^{90}Y measurement, if the presence of small amounts of ^{90}Sr cannot be excluded, then it is preferable to measure the Cherenkov radiation from the ^{90}Y , as it is negligible for ^{90}Sr .

6.4.3 Proportional counter

A PC measures directly the beta-radiation, without energy discrimination, from the source usually prepared as a thin layer deposit.

The use of double window (alpha and beta) in this type of counter allows the presence of alpha-emitter contaminants in the source to be checked. If other short radioactive half-life beta-emitters are present, they can be detected by performing successive measurements of the source at given times.

6.4.4 Efficiency calculation

The procedure to calibrate the counters is as follows:

- select t_s to collect at least 10^4 counts;
- determine the beta-count rate of the calibration source (^{90}Sr in equilibrium with ^{90}Y);
- calculate the counting efficiency of the counter by dividing the count rate measured by the activity of the calibration source:

$$\varepsilon_i = \frac{r_s - r_0}{A_i}$$

6.4.5 Determination of the chemical yield

The chemical yield of the strontium, $R_{\text{c,Sr}}$, is calculated from strontium carrier or tracer by one of the following procedures:

- a) chemical yield calculated as the ratio of the mass of the collected strontium to the mass of the strontium added as a carrier at the start of the procedure:

$$R_{\text{c,Sr}} = \frac{m_{\text{c,p}}}{m_{\text{c,Sr}}} \quad (1)$$

where

$m_{\text{c,p}}$ is the mass of the strontium collected, determined by an appropriate method (AA, ICP–AES or ICP–MS);

$m_{\text{c,Sr}}$ is the mass of the strontium carrier added;

- b) chemical yield calculated as the ratio of the activity of the ^{85}Sr collected, measured by gamma- spectrometry, over the theoretical activity of the equivalent ^{85}Sr added as a tracer at the start of the procedure.

$$R_{\text{c,Sr}} = \frac{A_{^{85}\text{Sr,M}}}{A_{^{85}\text{Sr,T}}} \quad (2)$$

where

$A_{^{85}\text{Sr,M}}$ is the activity of ^{85}Sr measured by gamma-spectrometry taking into account the ^{85}Sr decay from the start of procedure;

$A_{^{85}\text{Sr,T}}$ is the theoretical activity of ^{85}Sr added at the start of the procedure.

The chemical yield of the yttrium, $R_{\text{c,Y}}$, is calculated from the yttrium carrier by a procedure similar to that presented for the chemical yield of the strontium.