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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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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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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, ^{3}H, ^{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), ^{3}H, ^{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 l d^{-1} of drinking water for one year that results in an effective dose of 0,1 mSv 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.

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*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

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
$c_{\mathrm{A},i}$	activity concentration of radionuclide *i*	Bq l^{-1}
$c^{*}_{\mathrm{A},i}$	decision threshold of radionuclide *i*	Bq l^{-1}
$c^{\#}_{\mathrm{A},i}$	detection limit of radionuclide *i*	Bq l^{-1}
$c^{\triangleleft}_{\mathrm{A},i}, c^{\triangleright}_{\mathrm{A},i}$	lower and upper limits of the probabilistically symmetric coverage interval of radionuclide *i*	Bq l^{-1}
$c^{<}_{\mathrm{A},i}, c^{>}_{\mathrm{A},i}$	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 $1-\gamma/2$)	
$R_{\mathrm{c},i}$	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}
$r_{\mathrm{g}j}$	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 $^{90}\mathrm{Sr}/^{90}\mathrm{Y}$ ($t = 0$) 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 $U = ku(c_\mathrm{A})$ with $k = 1, 2 \ldots$	Bq l^{-1}
$u(c_\mathrm{A})$	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 $^{90}\mathrm{Sr}$, $^{90}\mathrm{Y}$ and $^{89}\mathrm{Sr}$ 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 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 ^{89}Sr and ^{90}Sr 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

	Origin	**Old contamination**				**Fresh contamination**	
	Radionuclide	^{90}Sr+^{90}Y				^{90}Sr+^{90}Y ^{89}Sr	
Separation	Element	Sr		Y[a]		Sr	
	Method	Chromatography[b]	Precipitation	Extraction	Precipitation	Chromatography[b]	Precipitation
	Product	^{90}Sr		^{90}Y		^{90}Sr,^{90}Y, ^{89}Sr	
	Carrier or Tracer[c]	^{85}Sr or stable Sr		Stable Y		^{85}Sr or stable Sr	
Measurement(s)	Equilibrium ^{90}Sr+^{90}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}Sr+^{90}Y	^{90}Sr ^{90}Y	^{90}Y		^{90}Sr+^{90}Y ^{89}Sr	^{90}Sr ^{90}Y ^{89}Sr

[a] Y separation is performed following the ^{90}Sr-^{90}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

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 F.

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}Sr + ^{90}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}Sr + ^{90}Y or ^{90}Y) with the same geometry and chemical composition as the source to be measured.

A blank source shall be prepared 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 counting time 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). The 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 19361:2017[15]). 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 is 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 measurement can be affected by chemiluminescence phenomena or quench due to chemical entities and to the presence of other radionuclides than ^{90}Y. 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:

- If the presence of ^{89}Sr can be neglected, the relevant contribution of ^{90}Y in equilibrium with ^{90}Sr can be assessed using LSC;
- If 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 Cerenkov radiation from the ^{90}Y, as there is negligible interference from ^{90}Sr.

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.

6.4.3 Proportional counter

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

Dual-window alpha/beta discrimination allows the presence of alpha-emitter contaminants in the source to be monitored. If other short radioactive half-life beta-emitters are present, they can be detected by performing successive measurements of the source over time.

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 (in the same chemical and physical form as the samples);
- calculate the counting efficiency of the counter by dividing the count rate measured by the activity of the calibration source given in Formula (1):

$$\varepsilon_i = \frac{r_s - r_0}{A_i} \tag{1}$$

6.4.5 Determination of the chemical yield

The chemical yield of the strontium, $R_{c,Sr}$, 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 at the start of the procedure, i.e. the mass of strontium in the test sample plus the mass of strontium in the added carrier given in Formula (2):

$$R_{c,Sr} = \frac{m_{c,p}}{m_{c,Sr}} \tag{2}$$

where

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

$m_{c,Sr}$ is the mass of strontium in the processed aliquot, i.e. the mass of Sr in the test sample plus the mass of Sr in the added carrier;

b) chemical yield calculated as the ratio of the activity of the ^{85}Sr collected, measured by gamma-spectrometry, over the activity of the ^{85}Sr added as a tracer at the start of the procedure. See Formula (3):

$$R_{c,Sr} = \frac{A_{^{85}\mathrm{Sr},M}}{A_{^{85}\mathrm{Sr},T}} \tag{3}$$

where

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

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

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

7 Expression of results

7.1 Determination of ^{90}Sr in equilibrium with ^{90}Y

7.1.1 Calculation of the activity concentration

The activity per unit mass in source samples where the ^{90}Y has been completely separated from the parent radionuclide ^{90}Sr cannot be assessed until the decay progeny ^{90}Y has grown back in and is in secular equilibrium with the parent nuclide ^{90}Sr. This occurs 20 d after $t = 0$, where $t = 0$ is the time at which all the ^{90}Y had been removed from the sample.

The result of the measurement gives the gross number of counts from the ^{90}Sr plus ^{90}Y. Dividing the gross counts by the counting time gives the gross count rate, r_g.

This method is only applicable to test samples known to contain negligible amounts of ^{89}Sr, low enough not to interfere with the ^{90}Sr analysis.

The background count rate, r_0, is obtained from the measurement of a blank source.

The activity concentration of ^{90}Sr, $c_{\mathrm{A},{}^{90}\mathrm{Sr}}$ is calculated using Formula (4):

$$c_{\mathrm{A},{}^{90}\mathrm{Sr}} = \frac{r_g - r_0}{V\, R_{\mathrm{c,Sr}}\,\varepsilon_{90\mathrm{Sr}}} = \left(r_g - r_0\right) w_{{}^{90}\mathrm{Sr}} \qquad (4)$$

where

$$w_{{}^{90}\mathrm{Sr}} = \frac{1}{V\, R_{\mathrm{c,Sr}}\,\varepsilon_{\mathrm{SR+Y}}}$$

For rapid measurements, i.e. in emergency situations, ^{90}Sr could be measured before reaching secular equilibrium with ^{90}Y as described in reference [16].

7.1.2 Standard uncertainty

According to ISO/IEC Guide 98-3, the standard uncertainty of $c_{\mathrm{A},{}^{90}\mathrm{Sr}}$ is calculated using Formula (5):

$$u\left(c_{\mathrm{A},{}^{90}\mathrm{Sr}}\right) = \sqrt{w^2_{{}^{90}\mathrm{Sr}}\left[u^2\left(r_g\right) + u^2\left(r_0\right)\right] + c^2_{\mathrm{A},{}^{90}\mathrm{Sr}}\, u^2_{\mathrm{rel}}\left(w_{{}^{90}\mathrm{Sr}}\right)} = \sqrt{w^2_{{}^{90}\mathrm{Sr}}\left(\frac{r_g}{t_g} + \frac{r_0}{t_0}\right) + c^2_{\mathrm{A},90_{\mathrm{Sr}}}\, u^2_{\mathrm{rel}_{\mathrm{Sr}}}\left(w_{{}^{90}\mathrm{Sr}}\right)} \qquad (5)$$

where the uncertainties of the sample and background counting times are neglected, and the relative standard uncertainty of w is calculated using Formula (6):

$$u^2_{\mathrm{rel}}\left(w_{{}^{90}\mathrm{Sr}}\right) = u^2_{\mathrm{rel}}\left(R_{\mathrm{c,Sr}}\right) + u^2_{\mathrm{rel}}\left(V\right) + u^2_{\mathrm{rel}}\left(\varepsilon_{\mathrm{Sr+Y}}\right) \qquad (6)$$

where the relative standard uncertainty of $\varepsilon_{\mathrm{Sr}}$ is calculated using Formula (7):

$$u^2_{\mathrm{rel}}\left(\varepsilon_{\mathrm{Sr+Y}}\right) = u^2_{\mathrm{rel}}\left(r_s - r_0\right) + u^2_{\mathrm{rel}}\left(A_{\mathrm{Sr}}\right) = \frac{\left(\frac{r_s}{t_s} + \frac{r_0}{t_0}\right)}{\left(r_s - r_0\right)^2} + u^2_{\mathrm{rel}}\left(A_{\mathrm{Sr}}\right) \qquad (7)$$

in which

$u_{\mathrm{rel}}(A_{\mathrm{Sr}})$	includes all the uncertainties related to the calibration source, i.e. in the standard solution and the preparation of the calibration source;
$u_{\mathrm{rel}}(R_{\mathrm{c,Sr}})$	is the uncertainty related to the chemical yield and depends on its method of evaluation.

For the calculation of the characteristic limits according to ISO 11929-1, $\tilde{u}\left(\tilde{c}_{\mathrm{A},{}^{90}\mathrm{Sr}}\right)$, i.e. the standard uncertainty of $c_{\mathrm{A},{}^{90}\mathrm{Sr}}$ as a function of its true value, is required and can be calculated by Formula (8):

$$\tilde{u}\left(\tilde{c}_{\mathrm{A},{}^{90}\mathrm{Sr}}\right)=\sqrt{w_{{}^{90}\mathrm{Sr}}^{2}\left[\left(\frac{\tilde{c}_{\mathrm{A},{}^{90}\mathrm{Sr}}}{w_{{}^{90}\mathrm{Sr}}}+r_0\right)/t_\mathrm{g}+\frac{r_0}{t_0}\right]+\tilde{c}_{\mathrm{A},{}^{90}\mathrm{Sr}}^{2}\,u_\mathrm{rel}^{2}\left(w_{{}^{90}\mathrm{Sr}}\right)} \tag{8}$$

7.1.3 Decision threshold

In accordance with ISO 11929-1, for $\tilde{c}_{\mathrm{A},{}^{90}\mathrm{Sr}}=0$, the decision threshold, $c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{*}$, is obtained from Formula (8). This yields:

$$c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{*}=k_{1-\alpha}\tilde{u}(0)=k_{1-\alpha}w_{{}^{90}\mathrm{Sr}}\sqrt{\frac{r_0}{t_\mathrm{g}}+\frac{r_0}{t_0}} \tag{9}$$

$\alpha = 0{,}05$ with $k_{1-\alpha} = 1{,}65$ is often chosen by default.

7.1.4 Detection limit

In accordance with ISO 11929-1, the detection limit, $c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{\#}$, is calculated by Formula (10):

$$c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{\#}=c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{*}+k_{1-\beta}\tilde{u}\left(c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{\#}\right)=c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{*}+k_{1-\beta}\sqrt{w_{{}^{90}\mathrm{Sr}}^{2}\left[\left(\frac{c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{\#}}{w_{{}^{90}\mathrm{Sr}}}+r_0\right)/t_\mathrm{g}+\frac{r_0}{t_0}\right]+c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{\#}\,u_\mathrm{rel}^{2}\left(w_{{}^{90}\mathrm{Sr}}\right)} \tag{10}$$

$\beta = 0{,}05$ with $k_{1-\beta} = 1{,}65$ is often chosen by default.

The detection limit can be calculated by solving Formula (10) for $c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{\#}$, more simply, by iteration with a starting approximation $c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{\#}=2c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{*}$.

When taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$ the solution of Formula (10) is given by Formula (11):

$$c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{\#}=\frac{2c_{\mathrm{A},{}^{90}\mathrm{Sr}}^{\#}+\left(k^{2}w_{{}^{90}\mathrm{Sr}}\right)/t_\mathrm{g}}{1-k^{2}u_\mathrm{rel}^{2}\left(w_{{}^{90}\mathrm{Sr}}\right)} \tag{11}$$

7.2 Determination of ^{90}Sr from separated ^{90}Y

7.2.1 Calculation of the activity concentration

Yttrium-90 is measured immediately after its separation from the test sample when strontium and yttrium are in equilibrium. The time when the ^{90}Y is separated from the ^{90}Sr is taken as $t = 0$.

The result of the measurement is the gross number of counts from the ^{90}Y, divided by the counting time, to give the gross count rate, r_g.

The gross count rate should be corrected by background count rate, r_0, obtained from the measurement of a blank source.

The activity concentration of ^{90}Y, $c_{\mathrm{A,Y}}$, is calculated at time $t = 0$, using Formula (12):

$$c_{\mathrm{A,Y}} = \frac{r_{\mathrm{g}} t_{\mathrm{g}} - r_0 t_{\mathrm{g}}}{\varepsilon_{\mathrm{Y}} V \, R_{\mathrm{c}} \int_{t_{\mathrm{d}}}^{t_{\mathrm{f}}} \exp\left(-\lambda_y t\right) \cdot dt} = \left(r_{\mathrm{g}} - r_0\right) w_{\mathrm{Y}} \tag{12}$$

where

$$w_{\mathrm{Y}} = \frac{\lambda_{\mathrm{Y}} t_{\mathrm{g}}}{\left[\exp\left(-\lambda_{\mathrm{Y}} t_{\mathrm{d}}\right) - \exp\left(-\lambda_{\mathrm{Y}} t_{\mathrm{f}}\right)\right]} \times \frac{1}{\varepsilon_{\mathrm{Y}} V \, R_{\mathrm{c}}}$$

and

$$R_{\mathrm{c}} = R_{\mathrm{c,Sr}} \cdot R_{\mathrm{c,Y}}$$

The integral allows the activity of the decay of ^{90}Y during the counting time to be corrected, $t_{\mathrm{g}} = t_{\mathrm{f}} - t_{\mathrm{d}}$, and the activity per unit of mass of ^{90}Sr, is calculated using Formula (13):

$$c_{\mathrm{A},^{90}\mathrm{Sr}} = c_{\mathrm{A,Y}} = \left(r_{\mathrm{g}} - r_0\right) w_{\mathrm{Y}} \tag{13}$$

7.2.2 Standard uncertainty

According to ISO/IEC Guide 98-3, the standard uncertainty of $c_{\mathrm{A}^{90}\mathrm{Sr}}$ is calculated by Formula (14):

$$u(c_{\mathrm{A},^{90}\mathrm{Sr}}) = \sqrt{w_{\mathrm{Y}}^2 \cdot (u^2(r_{\mathrm{g}}) + u^2(r_0)) + c_{\mathrm{A},^{90}\mathrm{Sr}}^2 \cdot u_{\mathrm{rel}}^2(w_{\mathrm{Y}})} = \sqrt{w_{\mathrm{Y}}^2 \cdot (r_{\mathrm{g}}/t_{\mathrm{g}} + r_0/t_0) + c_{\mathrm{A},^{90}\mathrm{Sr}}^2 \cdot u_{\mathrm{rel}}^2(w_{\mathrm{Y}})} \tag{14}$$

where the uncertainties of the sample and background counting times are neglected and the relative standard uncertainty of w_{Y} is calculated using Formula (15):

$$u_{\mathrm{rel}}^2(w_{\mathrm{Y}}) = u_{\mathrm{rel}}^2(R_{\mathrm{c}}) + u_{\mathrm{rel}}^2(V) + u_{\mathrm{rel}}^2(\varepsilon_{\mathrm{Y}}) \tag{15}$$

The relative standard uncertainty of ε_{Y} is calculated by Formula (16):

$$u_{\mathrm{rel}}^2(\varepsilon_{\mathrm{Y}}) = u_{\mathrm{rel}}^2(r_{\mathrm{s}} - r_0) + u_{\mathrm{rel}}^2(A_{\mathrm{Y}}) = \frac{\left(\dfrac{r_{\mathrm{s}}}{t_{\mathrm{s}}} + \dfrac{r_0}{t_0}\right)}{\left(r_{\mathrm{s}} - r_0\right)^2} + u_{\mathrm{rel}}^2(A_{\mathrm{Y}}) \tag{16}$$

in which $u_{\mathrm{rel}}(A_{\mathrm{Y}})$ includes all the uncertainties related to the calibration source, i.e. in the standard solution and the preparation of the calibration source; and $u_{\mathrm{rel}}(R_{\mathrm{c}})$ is the uncertainty related with the chemical yield. It can be calculated from Formula (17):

$$u_{\mathrm{rel}}^2(R_{\mathrm{c}}) = u_{\mathrm{rel}}^2(R_{\mathrm{c,Sr}}) + u_{\mathrm{rel}}^2(R_{\mathrm{c,Y}}) \tag{17}$$

where $u_{\mathrm{rel}}^2(R_{\mathrm{c,Sr}})$, $u_{rel}^2(R_{\mathrm{c,Y}})$ are the squared relative uncertainties of the chemical yields of strontium and yttrium, respectively, and depends on their method of evaluation.

For the calculation of the characteristic limits according to ISO 11929-1, $\tilde{u}(\tilde{c}_{\mathrm{A},^{90}\mathrm{Sr}})$, i.e the standard uncertainty of $c_{\mathrm{A},^{90}\mathrm{Sr}}$ as a function of its true value, is required, calculated by Formula (18):

$$\tilde{u}\left(\tilde{c}_{\mathrm{A},^{90}\mathrm{Sr}}\right) = \sqrt{w_{\mathrm{Y}}^2 \left[\left(\frac{\tilde{c}_{\mathrm{A},^{90}\mathrm{Sr}}}{w_{\mathrm{Y}}} + r_0\right) / t_{\mathrm{g}} + \frac{r_0}{t_0}\right] + \tilde{c}_{\mathrm{A},^{90}\mathrm{Sr}}^2 \, u_{\mathrm{rel}}^2(w_{\mathrm{Y}})} \tag{18}$$

7.2.3 Decision threshold

In accordance with ISO 11929-1, for $\tilde{c}_{\mathrm{A},{}^{90}\mathrm{Sr}}=0$, the decision threshold, $c^{*}_{\mathrm{A},{}^{90}\mathrm{Sr}}$ is obtained from Formula (18). This yields Formula (19):

$$c^{*}_{\mathrm{A},{}^{90}\mathrm{Sr}}=k_{1-\alpha}\cdot\tilde{u}(0)=k_{1-\alpha}w_{\mathrm{Y}}\sqrt{\frac{r_0}{t_{\mathrm{g}}}+\frac{r_0}{t_0}} \tag{19}$$

α = 0,05 with $k_{1-\alpha}$ = 1,65 is often chosen by default.

7.2.4 Detection limit

In accordance with ISO 11929-1, the detection limit, $c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}}$ is calculated by Formula (20):

$$c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}}=c^{*}_{\mathrm{A},{}^{90}\mathrm{Sr}}+k_{1-\beta}\tilde{u}(c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}})=c^{*}_{\mathrm{A},{}^{90}\mathrm{Sr}}+k_{1-\beta}\sqrt{w_{\mathrm{Y}}^{2}\left[\left(\frac{c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}}}{w_{\mathrm{Y}}}+r_0\right)/t_{\mathrm{g}}+\frac{r_0}{t_0}\right]+c^{\#2}_{\mathrm{A},{}^{90}\mathrm{Sr}}\,u_{\mathrm{rel}}^{2}(w_{\mathrm{Y}})} \tag{20}$$

β = 0,05 with $k_{1-\beta}$ = 1,65 is often chosen by default.

The detection limit can be calculated by solving Formula (20) for $c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}}$, or, more simply, by iteration with starting approximations $c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}}=2\cdot c^{*}_{\mathrm{A},{}^{90}\mathrm{Sr}}$.

When taking $\alpha=\beta$ then $k_{1-\alpha}=k_{1-\beta}=k$ and the solution of Formula (20) is given by Formula (21):

$$c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}}=\frac{2\cdot c^{*}_{\mathrm{A},{}^{90}\mathrm{Sr}}+\left(k^{2}\cdot w_{\mathrm{Y}}\right)/t_{\mathrm{g}}}{1-k^{2}\cdot u_{\mathrm{rel}}^{2}(w_{\mathrm{Y}})} \tag{21}$$

7.3 Determination of ^{90}Sr and ^{89}Sr utilizing ^{90}Sr/^{90}Y equilibrium

7.3.1 Calculation of the activity concentration

This method is based on making two measurements of the same source at two different times t_1 and t_2 after the time of separation of the yttrium from the test sample. It is recommended that the same counting time, t_{g}, is used for both measurements. The net count rates, r_j, of these measurements can be calculated from the gross count rates, $r_{\mathrm{g}j}$, and the background count rates, r_{0j}, as $r_j=r_{\mathrm{g}j}-r_{0j}$.

If the measurements are made when equilibrium between the ^{90}Sr and ^{90}Y has been reached, then the net count rates can be calculated using the Formula (22) below, considering that the ^{90}Sr and ^{89}Sr activities are constant during the counting time, and the appropriate decay constants,

$$\begin{aligned} r_1 &= A_{^{90}\text{Sr}}\varepsilon_{^{90}\text{Sr}} + \varepsilon_{^{89}\text{Sr}} A_{^{89}\text{Sr}} \exp\left(-\lambda_{^{89}\text{Sr}} t_1\right) \\ r_2 &= A_{^{90}\text{Sr}}\varepsilon_{^{90}\text{Sr}} + \varepsilon_{^{89}\text{Sr}} A_{^{89}\text{Sr}} \exp\left(-\lambda_{^{89}\text{Sr}} t_2\right) \end{aligned} \tag{22}$$

From Formula (23):

$$\begin{aligned} A_{^{90}\text{Sr}} &= \frac{r_2 - r_1 \exp\left[-\lambda_{^{89}\text{Sr}}(t_2 - t_1)\right]}{\varepsilon_{^{90}\text{Sr}}\left\{1 - \exp\left[-\lambda_{^{89}\text{Sr}}(t_2 - t_1)\right]\right\}} \\ A_{^{89}\text{Sr}} &= \frac{(r_1 - r_2)\exp\left(+\lambda_{^{89}\text{Sr}} t_1\right)}{\varepsilon_{^{89}\text{Sr}}\left\{1 - \exp\left[-\lambda_{^{89}\text{Sr}}(t_2 - t_1)\right]\right\}} \end{aligned} \tag{23}$$

The activity concentration, $c_{\text{A},i}$, of the radionuclide i is calculated using Formula (24):

$$c_{\text{A},i} = \frac{A_i}{V\,R_{\text{c,Sr}}} \tag{24}$$

and Formula (25):

$$c_{\text{A},^{90}\text{Sr}} = w_{90}\left(r_2 - c r_1\right) \tag{25}$$

where

$$w_{90} = \frac{1}{V\,R_{\text{c,Sr}} \times 2 \times \varepsilon_{^{90}\text{Sr}}(1 - c)}$$

$$c = \exp\left[-\lambda_{89_{\text{Sr}}}(t_2 - t_1)\right]$$

and Formula (26):

$$c_{\text{A},^{89}\text{Sr}} = w_{89} \cdot (r_2 - r_1) \tag{26}$$

where

$$w_{89} = \frac{\exp\left(+\lambda_{^{89}\text{Sr}} t_1\right)}{V\,R_{\text{c,Sr}}\,\varepsilon_{^{89}\text{Sr}}(c - 1)}$$

$$c = \exp\left[-\lambda_{89_{Sr}}(t_2 - t_1)\right]$$

7.3.2 Standard uncertainty

When the measurements are made in equilibrium conditions and according to ISO/IEC Guide 98-3, the standard uncertainty of $c_{\text{A},i}$ is calculated by Formula (27):

$$\begin{aligned} u(c_{\text{A},^{90}\text{Sr}}) &= \sqrt{w_{90}^{\;2}\left[u^2(r_2) + c^2 u^2(r_1)\right] + c^2_{\text{A},^{90}\text{Sr}} u^2_{\text{rel}}(w_{90})} \\ u(c_{\text{A},^{89}\text{Sr}}) &= \sqrt{w_{89}^{\;2}\left[u^2(r_1) + u^2(r_2)\right] + c^2_{\text{A},^{89}\text{Sr}} u^2_{\text{rel}}(w_{89})} \end{aligned} \tag{27}$$

assuming that $u^2(c)$ = 0.

The relative standard uncertainty of r_j is calculated by Formula (28):

$$u^2(r_j)=\frac{r_{gj}}{t_g}+\frac{r_{0j}}{t_0} \tag{28}$$

The relative standard uncertainties of w_{90} and w_{89} are calculated by Formula (29):

$$\begin{aligned} u_{\text{rel}}^2(w_{90})&=u_{\text{rel}}^2(R_{\text{c,Sr}})+u_{\text{rel}}^2(V)+u_{\text{rel}}^2(\varepsilon_{90_{\text{Sr}}})\\ u_{\text{rel}}^2(w_{89})&=u_{\text{rel}}^2(R_{\text{c,Sr}})+u_{\text{rel}}^2(V)+u_{\text{rel}}^2(\varepsilon_{89_{\text{Sr}}}) \end{aligned} \tag{29}$$

The relative standard uncertainty of ε_i is calculated by Formula (30):

$$u_{\text{rel}}^2(\varepsilon_i)=u_{\text{rel}}^2(r_s-r_0)+u_{\text{rel}}^2(A_i)=\left(\frac{r_s}{t_s}+\frac{r_0}{t_0}\right)/(r_s-r_0)^2+u_{\text{rel}}^2(A_i) \tag{30}$$

where

$u_{\text{rel}}(A_i)$ includes all the uncertainties related to the calibration source, i.e. in the standard solution and the preparation of the calibration source;

$u_{\text{rel}}(R_{\text{c, Sr}})$ is the uncertainty related to the chemical yield, and depends on its method of evaluation.

For the calculation of the characteristic limits according to ISO 11929-1, $\tilde{u}(\tilde{c}_{\text{A},i})$, i.e the standard uncertainty of A_i as a function of its true value is required and calculated by Formula (31) and Formula (32):

$$\tilde{u}(\tilde{c}_{\text{A},90_{\text{Sr}}})=\sqrt{w_{90}^2\left[(r_{02}+c^2r_{01})\left(\frac{1}{t_0}+\frac{1}{t_g}\right)+\frac{1+c^2}{t_g(1-c)}\left(\frac{\tilde{c}_{\text{A},90_{\text{Sr}}}}{w_{90}}-\frac{c_{\text{A},89_{\text{Sr}}}}{w_{89}}\right)+\frac{c_{\text{A},89_{\text{Sr}}}}{w_{89}t_g}\right]+\tilde{c}_{\text{A},90_{\text{Sr}}}^2u_{\text{rel}}^2(w_{90})} \tag{31}$$

$$\tilde{u}(\tilde{c}_{\text{A},89_{\text{Sr}}})=\sqrt{w_{89}^2\left[(r_{01}+r_{02})\left(\frac{1}{t_0}+\frac{1}{t_g}\right)+\frac{2}{t_g(1-c)}\left(\frac{\tilde{c}_{\text{A},90_{\text{Sr}}}}{w_{90}}-\frac{\tilde{c}_{\text{A},89_{\text{Sr}}}}{w_{89}}\right)+\frac{\tilde{c}_{\text{A},89_{\text{Sr}}}}{t_gw_{89}}\right]+\tilde{c}_{\text{A},89_{\text{Sr}}}^2u_{\text{rel}}^2(w_{89})} \tag{32}$$

7.3.3 Decision threshold

In accordance with ISO 11929-1, the decision thresholds, $c_{\text{A},i}^*$, are obtained from the Formula (32) for $\tilde{c}_{\text{A,i}}=0$. This yields Formula (33):

$$\begin{aligned} c_{\text{A},90_{\text{Sr}}}^*&=k_{1-\alpha}\tilde{u}(0)=k_{1-\alpha}w_{90}\sqrt{(r_{02}+c^2r_{01})\left(\frac{1}{t_0}+\frac{1}{t_g}\right)+\frac{c(c+1)}{(c-1)}\frac{c_{\text{A},89_{\text{Sr}}}}{t_gw_{89}}}\\ c_{\text{A},89_{\text{Sr}}}^*&=k_{1-\alpha}\tilde{u}(0)=k_{1-\alpha}w_{89}\sqrt{(r_{01}+r_{02})\left(\frac{1}{t_0}+\frac{1}{t_g}\right)+\frac{2}{t_g(1-c)}\frac{c_{\text{A},90_{\text{Sr}}}}{w_{90}}} \end{aligned} \tag{33}$$

α = 0,05 with $k_{1-\alpha}$ = 1,65 is often chosen by default.

7.3.4 Detection limit

In accordance with ISO 11929-1, the detection limits, $c^{\#}_{\mathrm{A},i}$, are calculated by Formula (34):

$$\begin{aligned}
&c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}} = c^{*}_{\mathrm{A},{}^{90}\mathrm{Sr}} + k_{1-\beta}\,\tilde{u}(c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}}) = \\
&= c^{*}_{\mathrm{A},{}^{90}\mathrm{Sr}} + k_{1-\beta}\sqrt{{w_{90}}^{2}\left[(r_{02}+c^{2}r_{01})\left(\frac{1}{t_0}+\frac{1}{t_g}\right)+\frac{(1+c^{2})}{t_g(1-c)}\left[\frac{c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}}}{w_{90}}-\frac{c_{\mathrm{A},{}^{89}\mathrm{Sr}}}{w_{89}}\right]+\frac{c_{\mathrm{A},{}^{89}\mathrm{Sr}}}{w_{89}t_{\mathrm{g}}}\right]+{c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}}}^{2}u^{2}_{\mathrm{rel}}(w_{90})} \\
&c^{\#}_{\mathrm{A},{}^{89}\mathrm{Sr}} = c^{*}_{\mathrm{A},{}^{89}\mathrm{Sr}} + k_{1-\beta}\,\tilde{u}\left(c^{\#}_{\mathrm{A},{}^{89}\mathrm{Sr}}\right) = \\
&= c^{*}_{\mathrm{A},{}^{89}\mathrm{Sr}} + k_{1-\beta}\sqrt{{w_{89}}^{2}\left[(r_{01}+r_{02})\left(\frac{1}{t_0}+\frac{1}{t_g}\right)+\frac{2}{t_g(1-c)}\left[\frac{c_{\mathrm{A},{}^{90}Sr}}{w_{90}}-\frac{c^{\#}_{\mathrm{A},{}^{89}\mathrm{Sr}}}{w_{89}}\right]+\frac{c^{\#}_{\mathrm{A},{}^{89}\mathrm{Sr}}}{t_{\mathrm{g}}w_{89}}\right]+{c^{\#}_{\mathrm{A},{}^{89}\mathrm{Sr}}}^{2}u^{2}_{\mathrm{rel}}(w_{89})}
\end{aligned} \tag{34}$$

$\beta = 0{,}05$ with $k_{1-\beta} = 1{,}65$ is often chosen by default.

The detection limit can be calculated by solving Formula (34) for $c^{\#}_{\mathrm{A},i}$ or, more simply, by iteration with starting approximations $c^{\#}_{\mathrm{A},i} = 2 \cdot c^{*}_{\mathrm{A},i}$.

When taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of Formula (34) is given by the following Formulae (35):

$$\begin{aligned}
c^{\#}_{\mathrm{A},{}^{90}\mathrm{Sr}} &= \frac{2c^{*}_{\mathrm{A},{}^{90}\mathrm{Sr}} + k^{2}w_{90}\,(1+c^{2})/t_{\mathrm{g}}(1-c)}{1-k^{2}u^{2}_{\mathrm{rel}}(w_{90})} \\
c^{\#}_{\mathrm{A},{}^{89}\mathrm{Sr}} &= \frac{2c^{*}_{\mathrm{A},{}^{89}\mathrm{Sr}} + k^{2}w_{89}\,(1+c)/(c-1)t_{\mathrm{g}}}{1-k^{2}u^{2}_{\mathrm{rel}}(w_{89})}
\end{aligned} \tag{35}$$

8 Limits of the coverage intervals

8.1 Limits of the of the probabilistically symmetric coverage interval

The lower, $c^{\triangleleft}_{\mathrm{A},i}$, and upper, $c^{\triangleright}_{\mathrm{A},i}$, coverage limits are calculated using Formulae (36) and (37) in accordance to ISO 11929-1:

$$c^{\triangleleft}_{\mathrm{A},i} = c_{\mathrm{A},i} - k_{\mathrm{p}}\,u\left(c_{\mathrm{A},i}\right) \qquad p = \omega\left(1-\frac{\gamma}{2}\right) \tag{36}$$

$$c^{\triangleright}_{\mathrm{A},i} = c_{\mathrm{A},i} + k_{\mathrm{q}}\,u\left(c_{\mathrm{A},i}\right) \qquad q = 1-\frac{\omega\gamma}{2} \tag{37}$$

where $\omega = \Phi[y/u(y)]$, Φ being the distribution function of the standardized normal distribution.

The value of ω can be set to 1, if $c_{\mathrm{A},i} \geq 4u(c_{\mathrm{A},i})$. In this case Formula (38):

$$c^{\triangleleft,\triangleright}_{\mathrm{A},i} = c_{\mathrm{A},i} \pm k_{1-\gamma/2}\,u\left(c_{\mathrm{A},i}\right) \tag{38}$$

$\gamma = 0{,}05$ with $k_{1-\gamma/2} = 1{,}96$ is often chosen by default.

8.2 Limits of the shortest coverage interval

As described in detail in ISO 11929-1, the lower limit of the shortest coverage interval, $c_A^{<}$, and the upper limit of the shortest coverage interval, $c_A^{>}$, are calculated from a primary measurement result, c_A, of the measurand and the standard uncertainty, $u(c_A)$, associated with c_A, either by Formula (39):

$$c_A^{<}, c_A^{>} = c_A \pm k_p u(c_A); p = (1 + \omega(1 - \gamma))/2 \tag{39}$$

or if $c_A^{<} < 0$ then the result is given by Formula (40)

$$c_A^{<} = 0; c_A^{>} = c_A + k_p u(c_A); q = 1 - \omega\gamma \tag{40}$$

where $\omega = \Phi\left[\frac{y}{u(y)}\right]$, Φ being the distribution function of the standardized normal distribution;

The relations $0 \le c_A^{<} < c_A^{>}$ apply and the approximation of Formula (38) is valid.

9 Quality control

Measurement methods shall be selected and associated procedures performed by suitably skilled staff under a quality assurance programme and control.

Confidence in the measurement results is maintained by regular use of certified reference materials and the participation in suitable programme of interlaboratory comparisons or proficiency testing (e.g. in compliance with ISO/IEC 17025).

Laboratory procedures shall ensure that laboratory and equipment contamination as well as cross-sample contamination is avoided.

10 Test report

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

a) the test method used, with reference to this document (ISO 13160:2021);

b) identification of the sample;

c) units in which the results are expressed;

d) the test result:

1) when the activity concentration, c_A, is compared with the decision threshold (see ISO 11929-1);

- if the result is less than the decision threshold, the result of the measurement is expressed as $\le c_A^*$,

- if the result is greater than the decision threshold, the result of the measurement is expressed as $c_A \pm u_c(c_A)$ or $c_A \pm U$ with the associated *k* value,

2) when the activity concentration, c_A, is compared with the detection limit;

- if the result is less than the detection limit, the result of the measurement is expressed as $\le c_A^{\#}$,

- if the result is greater than the detection limit, the result of the measurement is expressed as $c_A \pm u_c(c_A)$ or $c_A \pm U$ with the associated *k* value.

Complementary information can be provided such as:

e) the uncertainty can also be expressed as the limits of the probabilistically symmetric coverage interval $c_{\mathrm{A},}^{\triangleleft} c_{\mathrm{A}}^{\triangleright}$ and/or the limits of the shortest coverage interval $c_{\mathrm{A},}^{<} c_{\mathrm{A}}^{>}$;

f) probabilities α, β and $(1 - \gamma)$;

g) decision threshold and the detection limit;

h) if the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose;

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

NOTE Occasionally, it is requested by the customer or regulator to compare the primary measurement result with the detection limit, in order to decide whether the physical effect is recognized or not. Such stipulations are not in accordance with the ISO 11929 series. They have the consequence that it is decided too frequently that the physical effect is absent when in fact it is not absent.

Annex A
(informative)

Determination of ^{89}Sr and ^{90}Sr by precipitation and proportional counting

A.1 Principle

Strontium is precipitated by adding nitric acid. Yttrium and other interfering elements are eliminated by precipitating the hydroxides, followed by precipitation with barium chromate. The final product is a strontium precipitate, in the form of $SrCO_3$, which is measured by PC.

The mass of the test sample shall take into account the presumed activity of the sample and the desired detection limit. The procedure presented in the following is given for samples of 2 l.

The detection limit is approximately 10 mBq l^{-1} and 2 mBq l^{-1} for ^{89}Sr and ^{90}Sr, respectively, for volumes of 2 l and measuring time of 60 000 s.

A.2 Chemical reagents and equipment

A.2.1 Chemical reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

A.2.1.1 Strontium carrier solution, 40 mg ml^{-1} Sr(II)

A.2.1.2 Ammonia solution, 250 g l^{-1}.

A.2.1.3 Ammonia solution, 4 mol l^{-1}.

A.2.1.4 Ammonia solution, 0,1 mol l^{-1}.

A.2.1.5 Fuming nitric acid, HNO_3, >860 g l^{-1}.

A.2.1.6 Nitric acid, HNO_3, 4 mol l^{-1}.

A.2.1.7 Sodium chromate, Na_2CrO_4; 1,5 mol l^{-1}.

A.2.1.8 Sodium carbonate, Na_2CO_3.

A.2.1.9 Sodium carbonate solution, Na_2CO_3, saturated.

A.2.1.10 Fe(III) carrier solution, 5 mg ml^{-1} of Fe(III) in the carrier.

A.2.1.11 Ba(II) carrier solution, 10 mg ml^{-1} of Ba(II) in the carrier.

A.2.1.12 Ammonium acetate solution, CH_3COONH_4, 5 g l^{-1}.

A.2.1.13 Sodium acetate and acetic acid buffer, pH 5,2.

Mix 2,75 mol of sodium acetate and 1 mol of acetic acid and make up to 1 l with laboratory water.

A.2.1.14 ^{85}Sr solution, for tracer if used for chemical yield determination.

A.2.1.15 Hydrogen peroxide, 60 g l^{-1}.

A.2.2 Equipment

Usual laboratory equipment and in particular the following.

A.2.2.1 Filtration equipment.

The diameters of the filters shall correspond to the filtration device and the geometry of the counter used.

A.2.2.2 Cellulose and fiberglass filter.

A.2.2.3 Buchner funnel.

A.2.2.4 Analytical balance, accuracy 0,1 mg.

A.2.2.5 Hotplate, with temperature control and magnetic stirring.

A.2.2.6 pH meter.

A.2.2.7 Atomic absorption spectrometer (AAS) or Inductively coupled plasma atomic emission spectrometer (ICP–AES) or Inductively coupled plasma mass spectrometer (ICP-MS) or Gamma-spectrometer.

A.2.2.8 Proportional counter.

A.2.2.9 Desiccator.

A.2.2.10 Stainless steel test planchet, with a diameter compatible with the geometry of the counter.

A.2.2.11 Plastic flasks.

A.2.2.12 Vacuum pump.

A.3 Procedure

A.3.1 Introduction

Filter 2 l of water sample and add 1,5 ml of strontium carrier if the chemical yield of strontium extraction is to be determined by AAS, ICP–AES or ICP–MS. Alternatively, spike the sample with known amount of ^{85}Sr tracer solution if the chemical yield is going to be established by gamma-spectrometry.

A.3.2 Separation of alkaline metals

Heat the water sample to near boiling for 30 min with occasional stirring. Add 1 ml of concentrated ammonia and 20 g of sodium carbonate.

Allow to cool while stirring, adjust the pH to >9 and leave to stand for 12 h (minimum).

Decant the maximum volume of solution, centrifuge, discard the supernatant then rinse the precipitate using 0,1 mol l^{-1} ammonia solution.

Dry the centrifuge tube containing the precipitate in a sand bath or oven.

A.3.3 Separation of calcium

The use of fuming nitric acid (>860 g l^{-1}) is optimal for the separation of calcium and strontium.

Allow the centrifuge tube containing the precipitate to cool in an ice bath and add 45 ml of fuming nitric acid while stirring.

Leave for 30 min in the ice bath.

Centrifuge and discard the solution.

Add 45 ml of fuming nitric acid to the precipitate and repeat the separation procedure.

A.3.4 Separation of barium, radium and lead

Dissolve the precipitate using 10 ml of water.

Add 1 ml of barium carrier [10 mg ml^{-1} Ba(II)].

Add 4 mol l^{-1} ammonia solution to adjust to pH 5,0 to pH 5,5.

Add 1,5 ml of pH 5,2 buffer solution; check the pH to ensure it is between pH 5,0 and pH 5,5.

Boil the solution and add 0,5 ml of 1,5 mol l^{-1} sodium chromate solution.

Leave for 30 min to cool in a cold-water bath and filter using a fiberglass filter.

Rinse the precipitate twice using 5 ml of 5 g l^{-1} ammonium acetate solution.

Retain the filtered solution and discard the precipitate.

Add 1 ml of concentrated ammonia and 2 g to 4 g of sodium carbonate and allow the precipitate to settle out.

Heat gently and verify that precipitation is total by adding a few drops of saturated sodium carbonate solution to the solution. If precipitate appears, add more sodium carbonate.

Heat without boiling for a few minutes, leave to cool, and centrifuge. Discard the solution.

A.3.5 Yttrium separation

Dissolve the precipitate using 10 ml of 4 mol l^{-1} nitric acid, add 20 ml of water.

Add 1 ml of 60 g l^{-1} hydrogen peroxide and 0,5 ml of Fe(III) carrier solution (5 mg ml^{-1}).

Heat for 10 min to near boiling.

Adjust the pH using concentrated ammonia (about 15 ml) (optimum pH for the yttrium hydroxide precipitation is 10 to 10,3, if pH > 10,5, strontium can precipitate).

Allow to cool and filter using a fiberglass filter.

Rinse the precipitate with 20 ml of diluted ammonia solution (a few drops) and retain the solution.

Note the date and time of separation of ^{90}Sr from ^{90}Y as $t = 0$ or as time elapsed after precipitation of the yttrium present in the test sample.

Discard the yttrium precipitate.

A.3.6 Strontium purification and sources preparation to be measured by proportional counter

Add 2 g to 4 g of sodium carbonate until total precipitation of $SrCO_3$.

Heat and stir for 30 min without boiling.

Leave to cool.

Filter the $SrCO_3$ obtained using a membrane filter.

Note the date and time of precipitation as $t = 0$.

A.3.7 Sources preparation to be measured by proportional counter

Mark the back of a planchette with the sample reference number then place the filter carefully on the upper surface.

Dry the precipitate in an oven for 1 h at 105 °C to constant mass.

After weighing, place the source in the desiccator until the PC measurement.

Do not carry out the first count of the planchet before 24 h after the preparation to allow radon progeny to decay, (between 24 h to 72 h after the separation).

Keep the planchet in the desiccator for 20 d to obtain radioactive equilibrium between ^{90}Sr and ^{90}Y.

Annex B
(informative)

Determination of ^{89}Sr and ^{90}Sr by precipitation and liquid scintillation counting

B.1 Principle

The strontium is precipitated by adding fuming nitric acid. Yttrium and other interfering elements are eliminated by precipitating the hydroxides followed by precipitation with barium chromate. The final product, a strontium precipitate in the form of $SrCO_3$, is measured by LSC after being dissolved.

The detection limit is about 22 m Bq l^{-1} and 12 m Bq l^{-1} for ^{89}Sr and ^{90}Sr, respectively, for a volume of 1 l and measuring time of 86 400 s.

B.2 Chemical reagents and equipment

B.2.1 Chemical reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

B.2.1.1 Sr(II) carrier solution, 40 mg ml^{-1}.

B.2.1.2 Ammonia solution, 250 g l^{-1}.

B.2.1.3 Ammonia solution, 4 mol l^{-1}.

B.2.1.4 Fuming nitric acid, HNO_3, >860 g l^{-1}.

B.2.1.5 Nitric acid, HNO_3, 8 mol l^{-1}.

B.2.1.6 Nitric acid, HNO_3, 4 mol l^{-1}.

B.2.1.7 Nitric acid, HNO_3, 1 mol l^{-1}.

B.2.1.8 Sodium chromate, Na_2CrO_4, 1,5 mol l^{-1}.

B.2.1.9 Sodium carbonate, Na_2CO_3, 2 mol l^{-1}.

B.2.1.10 Ammonium carbonate, $(NH_4)_2CO_3$.

B.2.1.11 Fe(III)carrier solution, 5 mg g^{-1} of Fe(III) in the carrier.

B.2.1.12 Ba(II) carrier solution, 10 mg g^{-1} of Ba(II) in the carrier.

B.2.1.13 Ammonium acetate solution, CH_3COONH_4, 5 g l^{-1}.

B.2.1.14 Sodium acetate and acetic acid buffer, pH 5,2.

Mix 2,75 mol of sodium acetate and 1 mol of acetic acid and make up to 1 l with water.

B.2.1.15 Hydrogen peroxide, 330 g l^{-1}.

B.2.1.16 Hydrogen peroxide, 60 g l^{-1}.

B.2.1.17 Scintillation solution.

B.2.1.18 ^{85}Sr solution, for tracer, if used for chemical yield determination.

B.2.2 Equipment

Usual laboratory equipment and in particular the following.

B.2.2.1 Filtration equipment (the diameters of the filters shall correspond to the filtration device).

B.2.2.2 Cellulose and fiberglass filter.

B.2.2.3 Buchner funnel.

B.2.2.4 Desiccator.

B.2.2.5 Analytical balance, accuracy 0,1 mg.

B.2.2.6 Hotplate, with temperature control and magnetic stirring.

B.2.2.7 pH meter.

B.2.2.8 Atomic absorption spectrometer (AAS) or Inductively coupled plasma atomic emission spectrometer (ICP–AES) or Inductively coupled plasma mass spectrometer (ICP–MS) or gamma-spectrometer.

B.2.2.9 Liquid scintillation counter.

B.2.2.10 Polyethylene vials.

B.2.2.11 Plastic flasks.

B.2.2.12 Vacuum pump.

B.3 Procedure

B.3.1 Introduction

Filter 1 l of water sample and add 1,5 ml of strontium carrier solution.

B.3.2 Separation of alkaline metals

Heat the water sample to near boiling with occasional stirring, add 1 ml of concentrated ammonia to adjust the pH to >9 and 20 g of ammonium carbonate.

Cover with a watch glass and leave to cool while stirring for 1,5 h to 2 h.

Centrifuge or filter using a fiberglass filter.

Discard the solution.

Dissolve the precipitate using the minimum quantity of 8 mol l^{-1} nitric solution, clean the filter with an equal volume of water.

B.3.3 Separation of calcium

Reduce the solution volume to 20 ml, leave to cool and add 50 ml of fuming nitric acid (2,5 ml per millilitre of solution).

Leave 30 min in a cool bath.

Filter using a fiberglass filter (use a dried filter).

Discard the solution.

Dissolve the precipitate with 5 ml of hot water; if necessary, add 5 ml more.

Add fuming nitric acid (3 ml per millilitre of solution).

Leave for 30 min to cool in a cold-water bath.

Filter using a fiberglass filter (use a dried filter). Discard the solution.

Dissolve the precipitate using 30 ml of hot water.

B.3.4 Separation of barium, radium and lead

Add 0,5 ml of barium carrier.

Add 4 mol l^{-1} ammonia solution until the pH reaches 5,0 to 5,2.

Add 1,5 ml of pH 5,2 buffer solution; check the pH.

Boil the solution and add 0,5 ml of sodium chromate solution.

Leave to cool, filter using a fiberglass filter.

Rinse the precipitate twice using 5 ml of 5 g l^{-1} ammonium acetate solution.

Retain the filtered solution and discard the precipitate.

Add 1 ml of concentrated ammonia and 10 g of ammonium carbonate and allow the precipitate to settle out. Verify that precipitation is total by adding a few drops of saturated ammonium carbonate solution to the solution. If a precipitate appears, add more ammonium carbonate.

Heat to near boiling for a few minutes. Leave to cool, then filter using a fiberglass filter.

Discard the supernatant.

Dissolve the precipitate using 10 ml of 4 mol l^{-1} nitric acid and clean the filter using 20 ml of water.

From this point, the procedure should be carried out without interruption, due to the yttrium separation produced.

B.3.5 Separation of fission products and yttrium

Add 1 ml of 60 g l^{-1} hydrogen peroxide and 0,5 ml of Fe(III) carrier solution.

Heat for 10 min, avoid boiling.

Adjust the pH to 9 to 10 using concentrated ammonia (about 15 ml) (optimum pH for the yttrium hydroxide precipitation is 10 to 10,3, if pH > 10,5 strontium can precipitate).

Leave to cool and filter using a fiberglass filter.

Rinse the precipitate with 20 ml of dilute ammonia solution (a few drops).

Note the date and time of separation of ^{90}Sr from ^{90}Y as $t = 0$, the time elapsed since precipitation of the yttrium present in the test portion.

Discard yttrium precipitate.

B.3.6 Strontium purification

Add 50 ml of 2 mol l^{-1} sodium carbonate solution until total precipitation of $SrCO_3$.

Heat and stir for 30 min without boiling.

Leave to cool and filter the $SrCO_3$ obtained using the filter pump and a fiberglass filter.

Rinse the beaker and the precipitate using 10 ml of dilute ammonia and 10 ml of water.

Leave the precipitate to dry with the vacuum pump for at least 10 min.

B.3.7 LSC sources preparation

Dissolve the $SrCO_3$ precipitate in 10 ml of 1 mol l^{-1} nitric acid (final strontium solution). Keep the solution in a pre-weighed plastic vial and note the mass.

Vial A preparation: transfer 10 ml (or the optimized quantity) of the final strontium solution to a 20 ml pre-weighed polyethylene vial; note the mass.

Add the required volume of liquid scintillation cocktail solution and shake for complete dissolution; this is the source to be measured.

Laboratories shall use appropriate LSC cocktails and measurement settings depending on the type of samples (see ISO 19361:2017[15]).

Keep at 4 °C until measurement by LSC.

After 20 days of the yttrium separation, prepare the vial B (second measure), transfer 14 ml (or other optimum volume) of the final strontium solution to a 20 ml pre-weighed polyethylene vial and note the mass. Add the optimum volume of scintillation cocktail, weigh, and stir until dissolved. This is the source to be measured.

Keep at 4 °C until measurement by LSC.

Typical spectra of ^{89}Sr, ^{90}Sr and ^{90}Y are given in Reference [16].

Annex C
(informative)

Determination of ^{90}Sr from its decay progeny ^{90}Y at equilibrium by organic extraction and liquid scintillation counting

C.1 Principle

Yttrium is extracted from the sample solution using an organic solvent, HDEHP, with a pH of 1,0 to 1,2. After washing the organic phase in 0,08 mol l^{-1} HCl, the yttrium is re-extracted from the organic phase using 3 mol l^{-1} HNO_3. Finally, the yttrium is precipitated as its hydroxide and dissolved in 1 ml of concentrated nitric acid, before being measured by beta-counting using an LSC.

The detection limit is about 15 Bq l^{-1} for ^{90}Sr, for a volume of 1 l and measuring time of 86 400 s.

C.2 Chemical reagents and equipment

C.2.1 Chemical reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

C.2.1.1 Ammonia, NH_4OH, concentrated, 250 g l^{-1}.

C.2.1.2 Citric acid, $C_6H_8O_7$.

C.2.1.3 Hydrochloric acid, HCl, concentrated, 350 g l^{-1}.

C.2.1.4 Hydrochloric acid, 0,08 mol l^{-1}.

C.2.1.5 Nitric acid, HNO_3, concentrated, 690 g l^{-1}.

C.2.1.6 Nitric acid, HNO_3, 3 mol l^{-1}.

C.2.1.7 HDEHP solution [di-(2-ethylhexyl) hydrogenphosphoric acid], 100 g l^{-1} in toluene.

C.2.1.8 Toluene, C_7H_8.

C.2.1.9 Yttrium oxide, Y_2O_3.

C.2.1.10 Y(III) carrier solution, 10 mg ml^{-1} of Y(III) in the carrier.

C.2.2 Equipment

Usual laboratory equipment and in particular the following.

C.2.2.1 Analytical balance, accuracy 0,1 mg.

C.2.2.2 Hotplate, with temperature control and magnetic stirring.

C.2.2.3 pH meter.

C.2.2.4 Filtration equipment.

C.2.2.5 Membrane filter, pore size 0,45 µm.

C.2.2.6 Separating funnel, 250 ml.

C.2.2.7 Polyethylene vials.

C.2.2.8 Liquid scintillation counter.

C.3 Procedure

C.3.1 Chemical separation of yttrium

The tracer is added to the sample before the radiochemical separation. In principle, this leads to a nitric solution for which the organic separation procedure described in the following applies.

Concentrate a volume of water sample.

Add 1 ml of yttrium carrier and 2 g of citric acid.

Adjust the pH to 1,0 to 1,2 using 6 mol l^{-1} ammonia solution.

Transfer the sample into a 250 ml separation funnel.

Add 50 ml of the HDEHP solution in toluene.

Shake for 1 min, note the date and time of separation as $t = 0$ or the time from the separation of the yttrium present in the test sample.

Leave for 30 min and remove the aqueous phase.

Wash the organic phase five times with 50 ml of 0,08 mol l^{-1} HCl. Each time shake for 1 min, leave for 2 min and discard the aqueous phase.

Extract the ^{90}Y from the organic phase using 50 ml of 3 mol l^{-1} HNO_3 by shaking for 1 min and leaving for 2 min. Recover the aqueous phases.

Repeat the extraction several times, combine the aqueous phases and add 50 ml of water.

Add concentrated ammonia solution until $Y(OH)_3$ precipitates completely (pH = 9 to 10). Boil for 2 min to achieve flocculation of the yttrium hydroxide.

Leave to cool.

C.3.2 Source preparation to be measured

Discard the supernatant and dissolve the residue by adding 1 ml of concentrated nitric acid. Make to a known volume.

Take a fraction of known volume from the solution to determine the chemical yield.

A known quantity of test sample and scintillation cocktail is introduced into the counting vial.

After closing, shake the vial thoroughly to homogenize the mixture.

The vial identification shall be written on the vial cap. The storage time depends upon the scintillation mixture, the stability of the mixture, and the nature of the sample.

Laboratories shall use appropriate LSC cocktails and measurement settings depending on the type of samples (see ISO 19361:2017 [15]).

Typical spectra of ^{89}Sr, ^{90}Sr and ^{90}Y are given in[16].

Annex D
(informative)

Determination of ^{90}Sr after ionic exchange separation by proportional counting

D.1 Principle

Water sample with carrier and ethylenediaminetetracetic acid (EDTA) is passed through a cationic exchange resin. At pH 3,8, alkali metals and most alkaline earths are adsorbed on to the cationic resin, and the Ca-EDTA complex passes through the column. The alkaline earth metals are eluted from the cationic resin with sodium chloride and strontium is then precipitated as carbonate at pH 8.

The detection limit is about 5 Bq l^{-1} for ^{90}Sr, for a volume of 1 l to 6 l and a measuring time of 14 400 s.

D.2 Chemical reagents and equipment

D.2.1 Chemical reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

D.2.1.1 Sodium acetate buffer solution, pH 4,66.

D.2.1.2 Concentrated nitric acid, HNO_3, 15,8 mol l^{-1}.

D.2.1.3 Ammonium hydroxide, NH_4OH, 250 g l^{-1}.

D.2.1.4 Disodium ethylenediaminetetraacetate, $Na_2EDTA \cdot 2H_2O$; 20 g l^{-1}.

D.2.1.5 Sodium chloride, 4 mol l^{-1}.

D.2.1.6 Sodium carbonate, Na_2CO_3, 1,5 mol l^{-1} and 0,1 mol l^{-1}.

D.2.1.7 Prepacked column containing 2mL of cationic exchange resin, e.g. Dowex 50W-X8,[1] 50 mesh to 100 mesh.

D.2.1.8 Carrier solution for strontium nitrate $Sr(NO_3)_2$, 20 mg ml^{-1} in 0,1 mol l^{-1} HNO_3.

D.2.2 Equipment

Usual laboratory equipment and in particular the following.

D.2.2.1 Filtration equipment.

The diameters of the filters shall correspond to the filtration device and the geometry of the counter used.

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[1] Dowex 50W-X8 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

D.2.2.2 Cellulose filters.

D.2.2.3 Buchner funnel.

D.2.2.4 Analytical balance, precision 0,1 mg.

D.2.2.5 Hotplate, with temperature control.

D.2.2.6 pH meter.

D.2.2.7 Stirrer.

D.2.2.8 Ion exchange column.

D.2.2.9 Atomic absorption spectrometer (AAS) or Inductively coupled plasma **atomic emission spectrometer (ICP–AES) or Inductively coupled plasma mass spectrometry (ICP–MS).**

D.2.2.10 Proportional counter.

D.2.2.11 Desiccator.

D.2.2.12 Oven.

D.2.2.13 Stainless steel test planchet, with a diameter compatible with the geometry of the counter.

D.3 Procedure

D.3.1 Strontium complexation

Add 40 g Na_2EDTA and 60 mg of strontium carrier to 1 l of water sample.

Add enough ammonia solution (D.2.1.3) to reach pH 3,8.

Stir the solution for 2 h and leave overnight.

Filter on a fiberglass filter and discard the solid.

Adjust the solution to between pH 4,4 and pH 4,6 by adding ammonium hydroxide, then add 20 ml of sodium acetate buffer solution. Adjust to pH 4,8.

Load the prepacked cationic exchange column at a flow rate of 10 ml min^{-1}.

D.3.2 Strontium extraction

Add 600 ml of 20 g l^{-1} Na_2EDTA (pH 5,1) at a flow rate of 20 ml min^{-1}, then add 250 ml water.

Elute the strontium with 400 ml of 4 mol l^{-1} sodium chloride.

D.3.3 Strontium precipitation

Adjust the eluate to pH 8 with ammonium hydroxide and add 10 ml of 1,5 mol l^{-1} sodium carbonate $Na_2(CO_3)$ while stirring for 30 min.

Filter and dry the precipitate in an oven at 50 °C to 60 °C. Note the date and time of the strontium precipitation.

D.3.4 Preparation of sources to be measured

Put the filter with the precipitate on the filtration device and add 25 ml of 15,8 mol l^{-1} nitric acid, HNO_3.

Dissolve the precipitate with water. Adjust to pH 8 with ammonium hydroxide and precipitate the strontium with 10 ml of 1,5 mol l^{-1} sodium carbonate $Na_2(CO_3)$, while stirring for 30 min.

Filter the strontium carbonate in a cellulose filter previously weighed in a planchet. Dry and weigh the residue for self-absorption correction and recovery calculation. Allow 20 days or longer to establish equilibrium with ^{90}Y prior to the counting.

Annex E
(informative)

Determination of ^{90}Sr after separation on a crown ether specific resin and liquid scintillation counting

E.1 Principle

The strontium is selectively extracted on a specific crown ether column in an HNO_3 medium and then eluted using 0,05 mol l^{-1} HNO_3. The separation yield is determined from the stable strontium added initially as a carrier and the strontium measured subsequently by AAS, ICP–AES or ICP–MS. The beta-activity of ^{90}Sr is measured by LSC.

The detection limit is about 50 mBq l^{-1}, for 1 l sample volume and 3 600 s of measuring time.

E.2 Chemical reagents and equipment

E.2.1 Chemical reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

E.2.1.1 Support soaked in crown ether, specific to the extraction of strontium, e.g. 1,0 mol l^{-1} 4,4'(5')-di-(*t*-butylcyclohexano)-18-crown-6 (crown ether) in 1-octanol.

E.2.1.2 Strontium salt (e.g. strontium nitrate, strontium chloride).

E.2.1.3 Sr carrier solution, if used for chemical yield determination.

E.2.1.4 Aluminium nitrate, $Al(NO_3)_3$, 0,5 mol l^{-1}, solution in a 3 mol l^{-1} nitric acid.

E.2.1.5 Nitric acid, HNO_3, 3 mol l^{-1}.

E.2.1.6 Nitric acid, HNO_3, 8 mol l^{-1}.

E.2.1.7 Nitric acid, HNO_3, 0,05 mol l^{-1}.

E.2.1.8 Scintillation cocktail.

E.2.2 Equipment

Usual laboratory equipment and in particular the following.

E.2.2.1 Analytical balance, accuracy 0,1 mg.

E.2.2.2 Atomic absorption spectrometer (AAS) or Inductively coupled plasma **atomic emission spectrometer (ICP–AES) or Inductively coupled plasma mass spectrometry (ICP–MS).**

E.2.2.3 pH meter.