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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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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*.

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.

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}
$c^{\triangleleft}_{A,i}$, $c^{\triangleright}_{A,i}$	lower and upper limits of the confidence interval of radionuclide i	Bq l^{-1}

$R_{c,i}$	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 $^{90}Sr/^{90}Y$ ($t = 0$) 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 $k = 1, 2 ...$	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		$^{90}Sr+^{90}Y$				$^{90}Sr+^{90}Y$ ^{89}Sr	
Separation	Element	Sr		Y^a		Sr	
	Method	Chromatography[b]	Precipitation	Extraction	Precipitation	Chromatography[b]	Precipitation
	Product	^{90}Sr		^{90}Y		$^{90}Sr+^{89}Sr$	
	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 Cherenkov)		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

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, $^{90}Sr + {}^{90}Y$ 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

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

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}Sr + ^{90}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, $R_{c,Sr}$, 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:

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

where

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

$m_{c,Sr}$ 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.

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

where

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

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

The chemical yield of the yttrium, $R_{c,Y}$, is calculated from the yttrium carrier by a procedure similar to that presented for the chemical yield of the 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 reassessed until the daughter nuclide ^{90}Y has grown back in and is in 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.

To apply this method, neglect the ^{89}Sr contained in the test sample.

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

The activity concentration of ^{90}Sr plus ^{90}Y, $c_{A,Sr+Y}$, is calculated using Formula (3):

$$c_{A,Sr+Y} = \frac{r_g - r_0}{V\ R_{c,Sr}\ \varepsilon_{Sr+Y}} \tag{3}$$

and, the activity concentration of ^{90}Sr, is

$$c_{A,^{90}Sr} = \frac{c_{A,Sr+Y}}{2} = \frac{r_g - r_0}{2 \times V\ R_{c,Sr}\ \varepsilon_{Sr+Y}} = \left(r_g - r_0\right) w_{^{90}Sr} \tag{4}$$

with

$$w_{^{90}Sr} = \frac{1}{2 \times V R_{c,Sr} \varepsilon_{Sr+Y}}$$

7.1.2 Standard uncertainty

According to ISO/IEC Guide 98-3,[7] the standard uncertainty of $c_{A,^{90}Sr}$ is calculated by:

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

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

$$u_{rel}^2\left(w_{^{90}Sr}\right) = u_{rel}^2\left(R_{c,Sr}\right) + u_{rel}^2\left(V\right) + u_{rel}^2\left(\varepsilon_{Sr+Y}\right) \tag{6}$$

where the relative standard uncertainty of ε_{Sr+Y} is calculated using Formula (7):

$$u_{rel}^2\left(\varepsilon_{Sr+Y}\right) = u_{rel}^2\left(r_s - r_0\right) + u_{rel}^2\left(A_{Sr+Y}\right) = \left(\frac{r_s}{t_s} + \frac{r_0}{t_0}\right) \Big/ \left(r_s - r_0\right)^2 + u_{rel}^2\left(A_{Sr+Y}\right) \tag{7}$$

in which

$u_{rel}(A_{Sr+Y})$ includes all the uncertainties related to the calibration source, i.e. in the standard solution and the preparation of the calibration source;

$u_{rel}(R_{c,Sr})$ 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, $\tilde{u}\left(\tilde{c}_{\mathrm{A},^{90}\mathrm{Sr}}\right)$, i.e the standard uncertainty of $c_{\mathrm{A},^{90}\mathrm{Sr}}$ as a function of its true value, is required, calculated by Formula (8):

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

7.1.3 Decision threshold

In accordance with ISO 11929, 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, the detection limit, $c^{\#}_{\mathrm{A},^{90}\mathrm{Sr}}$, is calculated by

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

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

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

When taking $\alpha=\beta$, then $k_{1-\alpha}=k_{1-\beta}=k$ and 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 by ingrowth of ^{90}Y

7.2.1 Calculation of the activity concentration

The ^{90}Y is measured immediately after its separation in the test sample when strontium and yttrium are in equilibrium. The time when the ^{90}Y is separated from the ^{90}Sr and starts to decay with a half-life of 2,7 d 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, $c_{A,Y}$, is calculated at time $t = 0$, using Formula (12):

$$c_{A,Y} = \frac{r_g t_g - r_0 t_g}{\varepsilon_Y V R_c \int_{t_d}^{t_f} \exp(-\lambda_Y t).dt} = (r_g - r_0) w_Y \quad (12)$$

where

$$w_Y = \frac{\lambda_Y t_g}{\left[\exp(-\lambda_Y t_d) - \exp(-\lambda_Y t_f)\right]} \times \frac{1}{\varepsilon_Y V R_c}$$

$$R_c = R_{c,Sr} R_{c,Y}$$

The integral allows the activity of the decay of ^{90}Y during the counting time to be corrected, $t_g = t_f - t_d$, and the activity per unit of mass of ^{90}Sr, is

$$c_{A,^{90}Sr} = c_{A,Y} = (r_g - r_0) w_Y \quad (13)$$

7.2.2 Standard uncertainty

According to ISO/IEC Guide 98-3,[7] the standard uncertainty of $c_{A,^{90}Sr}$ is calculated by:

$$u(c_{A,^{90}Sr}) = \sqrt{w_Y^2 \left[u^2(r_g) + u^2(r_0)\right] + c^2_{A,^{90}Sr} u^2_{rel}(w_Y)} = \sqrt{w_Y^2 \left(\frac{r_g}{t_g} + \frac{r_0}{t_0}\right) + c^2_{A,^{90}Sr} u^2_{rel}(w_Y)} \quad (14)$$

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):

$$u^2_{rel}(w_Y) = u^2_{rel}(R_c) + u^2_{rel}(V) + u^2_{rel}(\varepsilon_Y) \quad (15)$$

the relative standard uncertainty of ε_Y is calculated by

$$u^2_{rel}(\varepsilon_Y) = u^2_{rel}(r_s - r_0) + u^2_{rel}(A_Y) = \left(\frac{r_s}{t_s} + \frac{r_0}{t_0}\right) \Big/ (r_s - r_0)^2 + u^2_{rel}(A_Y) \quad (16)$$

in which $u_{rel}(A_Y)$ includes all the uncertainties related to the calibration source, i.e. in the standard solution and the preparation of the calibration source; and $u_{rel}(R_c)$ is the uncertainty related with the chemical yield. It can be calculated from

$$u^2_{rel}(R_c) = u^2_{rel}(R_{c,Sr}) + u^2_{rel}(R_{c,Y}) \quad (17)$$

where $u^2_{rel}(R_{c,Sr})$, $u^2_{rel}(R_{c,Y})$ 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, $\tilde{u}(\tilde{c}_{A,^{90}Sr})$, i.e the standard uncertainty of $c_{A,^{90}Sr}$ as a function of its true value, is required, calculated by:

$$\tilde{u}(\tilde{c}_{A,^{90}Sr}) = \sqrt{w_Y^2 \left[\left(\frac{\tilde{c}_{A,^{90}Sr}}{w_Y} + r_0\right) \Big/ t_g + \frac{r_0}{t_0}\right] + \tilde{c}^2_{A,^{90}Sr} u^2_{rel}(w_Y)} \quad (18)$$

7.2.3 Decision threshold

In accordance with ISO 11929, 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:

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

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

7.2.4 Detection limit

In accordance with ISO 11929, the detection limit, $c^{\#}_{\mathrm{A},^{90}\mathrm{Sr}}$, is calculated by

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

$\beta = 0{,}05$ 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 $c^{\#}_{\mathrm{A},^{90}\mathrm{Sr}} = 2c^{*}_{\mathrm{A},^{90}\mathrm{Sr}}$.

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

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

7.3 Determination of ^{90}Sr in presence of ^{89}Sr when ^{90}Sr is in equilibrium with ^{90}Y

7.3.1 Calculation of the activity concentration

This method is based in the realization of two measurements of the same source at two different times t_1 and t_2 after the time $t = 0$ of the separation of the yttrium present in the test sample. It is suggested 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 equations below, considering that the ^{90}Sr and ^{89}Sr activities are constant during the counting time, and the appropriate decay constants,

$$\begin{aligned} r_1 &= 2A_{^{90}\mathrm{Sr}}\varepsilon_{^{90}\mathrm{Sr+Y}} + \varepsilon_{^{89}\mathrm{Sr}} A_{^{89}\mathrm{Sr}} \exp\left(-\lambda_{^{89}\mathrm{Sr}} t_1\right) \\ r_2 &= 2A_{^{90}\mathrm{Sr}}\varepsilon_{^{90}\mathrm{Sr+Y}} + \varepsilon_{^{89}\mathrm{Sr}} A_{^{89}\mathrm{Sr}} \exp\left(-\lambda_{^{89}\mathrm{Sr}} t_2\right) \end{aligned} \tag{22}$$

From these equations

$$\begin{aligned} A_{^{90}\mathrm{Sr}} &= \frac{r_2 - r_1 \exp\left[-\lambda_{^{89}\mathrm{Sr}}(t_2 - t_1)\right]}{2\varepsilon_{^{90}\mathrm{Sr+Y}}\left\{1 - \exp\left[-\lambda_{^{89}\mathrm{Sr}}(t_2 - t_1)\right]\right\}} \\ A_{^{89}\mathrm{Sr}} &= \frac{(r_1 - r_2)\exp\left(+\lambda_{^{89}\mathrm{Sr}} t_1\right)}{\varepsilon_{^{89}\mathrm{Sr}}\left\{1 - \exp\left[-\lambda_{^{89}\mathrm{Sr}}(t_2 - t_1)\right]\right\}} \end{aligned} \tag{23}$$

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

$$c_{A,i} = \frac{A_i}{VR_{c,Sr}} \tag{24}$$

and

$$c_{A,^{90}Sr} = w_{90}\left(r_2 - cr_1\right) \tag{25}$$

where

$$w_{90} = \frac{1}{VR_{c,Sr} \times 2 \times \varepsilon_{^{90}Sr+Y}(1-c)}$$

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

and

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

where

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

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

7.3.2 Standard uncertainty

When the measurements are made in equilibrium conditions and according to ISO/IEC Guide 98-3,[7] the standard uncertainty of $c_{A,i}$ is calculated by:

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

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

The relative standard uncertainty of r_j is calculated by:

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

The relative standard uncertainties of w_{90} and w_{89} are calculated by

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

The relative standard uncertainty of ε_i is calculated by

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

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, $\tilde{u}(\tilde{c}_{A,i})$, i.e the standard uncertainty of A_i as a function of its true value, is required, calculated by:

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

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

7.3.3 Decision threshold

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

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

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

7.3.4 Detection limit

In accordance with ISO 11929, the detection limits, $c^{\#}_{\mathrm{A,i}}$, are calculated by

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

$\beta = 0{,}05$ 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 $c^{\#}_{\mathrm{A,i}} = 2c^{*}_{\mathrm{A,i}}$.

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

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

7.4 Confidence limits

Confidence limits can be calculated in accordance with ISO 11929. The values of lower limit, $c^{\triangleleft}_{\mathrm{A,i}}$, and upper limit, $c^{\triangleright}_{\mathrm{A,i}}$, are calculated using Formulae (36) and (37):

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

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

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:

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

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

8 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[5]).

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

9 Test report

The test report shall contain at least the following information:

a) the test method used, together with reference to this International Standard (ISO 13160:2012);

b) identification of the sample;

c) units in which the results are expressed;

d) test result, $c_{A,i} \pm u(c_{A,i})$ or $c_{A,i} \pm U$, with the associated k value;

e) complementary information can be provided such as:

— probabilities α, β, and $1 - \gamma$;

— decision threshold and the detection limit;

f) depending on customer requirements, there are different ways to present the result:

— when the activity concentration $c_{A,i}$ is compared with the decision threshold, in accordance with ISO 11929, the result of the measurement should be expressed as $\leq c^{*}_{A,i}$ when the result is below the decision threshold,

— when the activity concentration $c_{A,i}$ is compared with the detection limit, the result of the measurement can be expressed as $\leq c^{\#}_{A,i}$ when the result is below the detection limit

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

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

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 about 10 Bq m^{-3} and 2 Bq m^{-3} 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) or ^{85}Sr solution.

A.2.1.2 Ammonia, NH_4OH, 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 Nitric acid, HNO_3, concentrated, 690 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}.

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

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 fibreglass 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 **atomic emission spectrometer** (ICP–AES or 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 **Plastics 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 in known quantity [40 mg ml^{-1} Sr(II)] if chemical yield strontium extraction is determined by AAS, ICP–AES or ICP–MS, and add a well known activity of ^{85}Sr, if this yield is established by gamma-spectrometry.

A.3.2 Separation of alkaline metals

Heat the water sample, stirring without boiling for 30 min, 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 and centrifuge, then rinse the precipitate using 0,1 mol l^{-1} ammonia solution.

Dry the precipitate in a sand bath.

A.3.3 Separation of calcium

The optimum nitric acid concentration for the calcium and strontium separation is between 650 g l^{-1} and 750 g l^{-1}.

Allow the precipitate to cool in an ice bath and add 45 ml of concentrated nitric acid while stirring.

Leave for 30 min in the ice bath.

Centrifuge and discard the solution.

Add 45 ml of concentrated nitric acid to the precipitate and proceed as before.

A.3.4 Separation of barium, radium and lead

Dissolve the precipitate using 10 ml of laboratory 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.

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

Leave to cool (frost bath), filter using a fibreglass filter.

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

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 centrifugate. Discard the solution.

A.3.5 Iron 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, avoid 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 fibreglass filter.

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

Note the date and time of separation of $Sr(NO_3)_2$ 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

Place the filter carefully on the previously identified planchette.

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 recount of the planchette before 24 h after the preparation so that the descendants of radon decay radioactively, preferably between 24 h to 72 h after the separation.

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

Carry out the second measurement with the PC.

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 Bq m^{-3} and 12 Bq m^{-3} 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 Fuming nitric acid, HNO_3, >860 g l^{-1}.

B.2.1.3 Ammonia, NH_4OH, concentrated, 250 g l^{-1}.

B.2.1.4 Ammonia solution, 4 mol l^{-1}.

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

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

B.2.1.7 Nitric acid, HNO_3, 4 mol l^{-1}.

B.2.1.8 Nitric acid, HNO_3, 2,5 mol l^{-1}.

B.2.1.9 Sodium chromate, Na_2CrO_4, 1,5 mol l^{-1}.

B.2.1.10 Sodium carbonate, Na_2CO_3.

B.2.1.11 Ammonium carbonate, $(NH_4)_2CO_3$.

B.2.1.12 Fe^{3+} carrier solution, 5 mg g^{-1}.

B.2.1.13 Ba^{2+} carrier solution, 10 mg g^{-1}.

B.2.1.14 Ammonium acetate solution, CH_3COONH_4, 5 g l^{-1}.

B.2.1.15 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.2.1.16 Hydrogen peroxide, 330 g l^{-1}.

B.2.1.17 Hydrogen peroxide, 60 g l^{-1}.

B.2.1.18 Scintillation solution.

B.2.1.19 ^{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 fibreglass 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 **atomic emission spectrometer** (ICP–AES or 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, stirring without boiling, add 1 ml of concentrated ammonia and 20 g of ammonium carbonate.

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

Filter using a fibreglass 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

The optimum nitric acid concentration for the calcium and strontium separation is between 650 g l^{-1} and 750 g l^{-1}.

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 fibreglass 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 in a cool bath.

Filter using a fibreglass 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 fibreglass filter.

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

Discard the precipitate.

Add 1 ml of concentrated ammonia and two spatulafuls 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 without boiling for a few minutes. Leave to cool, then filter using a fibreglass filter.

Discard the solution.

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 fibreglass filter.

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

NOTE The date and time of separation of $Sr(NO_3)_2$ 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 two spatulafuls of sodium carbonate 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 fibreglass 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 Sources preparation to be measured by LSC

Dissolve the $SrCO_3$ precipitate using a volume of 35 ml to 40 ml of 2,5 mol l^{-1} nitric acid (final strontium solution). Keep the solution in a pre-weighed plastics flask and note the mass.

Vial A preparation (first measure), transfer 14 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.

Keep at 4 °C until measurement by LSC.

After 20 d of the yttrium separation, prepare the vial B (second measure), transfer 14 ml (or the optimized quantity) of the final strontium solution to a 20 ml pre-weighed polyethylene vial and note the mass.

Add the optimizer scintillation liquid volume, weigh, and stir until dissolved. This is the source to be measured.

Keep at 4 °C until measurement by LSC.

Annex C
(informative)

Determination of ^{90}Sr from its daughter product ^{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 m^{-3} 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.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.2.1.9 Yttrium oxide, Y_2O_3.

C.2.1.10 Yttrium carrier solution, 10 g l^{-1}.

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.

Stir vigorously 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 in 50 ml of 0,08 mol l^{-1} HCl, stir for 1 min, leave for 2 min and throw the aqueous phase away each time.

Extract the ^{90}Y from the organic phase using 50 ml of 3 mol l^{-1} HNO_3, stir for 1 min and leave for 2 min each time. Recover the aqueous phases.

Collect the aqueous phase and add 50 ml of water.

Add concentrated ammonia solution until $Y(OH)_3$ total precipitation (pH = 9 to 10) and boil for 2 min to achieve flocculation of the yttrium hydroxide.

Leave to cool.

C.3.2 Source preparation to be measured

Remove the supernatant and dissolve the residue by adding 1 ml of concentrated nitric acid, carried to volume.

Take an aliquot fraction of 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.

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 m^{-3} for ^{90}Sr, for a volume of 1 l to 6 l and a measuring time of 86 400 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 Nitric acid, HNO_3, 650 g 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, $Na_2(CO_3)$, 1,5 mol l^{-1} and 0,1 mol l^{-1}.

D.2.1.7 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.2.1.9 Reference ^{85}Sr solution, if used for chemical yield determination.

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.

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 **atomic emission spectrometer** (ICP–AES or ICP–MS) or **gamma-spectrometer**.

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 fixation

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

Add enough ammonium hydroxide to reach pH 3,8.

Stir the solution for 2 h and leave overnight.

Filter on a fibreglass filter and discard the solid.

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

Load the column (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 to pH 8 with ammonium hydroxide and add 10 ml 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 650g 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 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 planchette. Dry and weigh the residue for self-absorption correction.

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 FAAS, ICP–AES or ICP–MS. The beta-activity of ^{90}Sr is measured by LSC.

The detection limit is about 50 Bq m^{-3}, 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 Inactive strontium salt.

E.2.1.3 Reference ^{85}Sr 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 Scintillator 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 spectrophotometer, ICP–AES or ICP–MS.

E.2.2.3 pH meter.

E.2.2.4 Evaporation system.

E.2.2.5 Liquid scintillation counter.

E.3 Procedure

E.3.1 Concentration of the sample

The tracer (10 mg of inactive strontium) is added to the sample before evaporation.

Evaporate the water sample until almost dry.

Dissolve the residue in 10 ml to 20 ml of a solution of 3 mol l^{-1} HNO_3 and 0,5 mol l^{-1} $Al(NO_3)_3$.

E.3.2 Preparation of the column

Prepare the column (containing 2,8 g of specific strontium resin) by adding 20 ml of 3 mol l^{-1} HNO_3.

E.3.3 Strontium fixation

Load the column with the solution.

Rinse the column with 10 ml of 3 mol l^{-1} HNO_3 and 10 ml of 8 mol l^{-1} HNO_3.

Note the date and time after rinsing.

E.3.4 Strontium extraction

Elute the strontium with at least 20 ml of 0,05 mol l^{-1} HNO_3 solution.

E.3.5 Preparation of the sources to be measured

Take an aliquot fraction of 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 mixture stability and the nature of the sample.

Annex F
(informative)

Determination of ^{90}Sr from its daughter product ^{90}Y at equilibrium by organic extraction by proportional counting

F.1 Principle

Yttrium is extracted from the sample solution using an organic solvent, HDEHP, at pH ~1,4 after washing the organic phase in 1 mol l^{-1} HCl and re-extracting the yttrium from the same phase using 9 mol l^{-1} HCl. The solution is then purified using the TOM solution. Finally, yttrium oxalate is precipitated and calcined in an oven at 900 °C, before being measured by beta-counting using a PC.

The absence of other interfering beta-emitters is assessed during the decay of the ^{90}Y by monitoring the count rate decrease. After a complete decay, the residual activity level can be compared with the background activity.

The tracer used is stable yttrium in the form of metal powder or yttrium oxide. It is added to the sample during the initial phase in a mass of between 5 mg to 50 mg.

The mass of the test portion shall take into account the presumed activity of the sample and the desired detection limit. The procedure described in the following applies to clear or charged water (such as seawater) samples, from 0,1 l to 250 l volume.

The detection limit is about 15 Bq l^{-1} or 0,1 Bq l^{-1} for a 1 l or 200 l sample volume, respectively, and 6 000 s of measuring time.

F.2 Chemical reagents and equipment

F.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.

F.2.1.1 Hydrochloric acid, dilute, HCl, 9 mol l^{-1}.

F.2.1.2 Hydrochloric acid, dilute, HCl, 1 mol l^{-1}.

F.2.1.3 Heptane.

F.2.1.4 HDEHP solution [di-(2-ethylhexyl) hydrogenphosphoric acid]: 145 ml of HDEHP plus 855 ml of heptane.

This reagent should be purified by washing it in an equal volume of water whose pH after this operation shall be greater than 3.

F.2.1.5 Toluene.

F.2.1.6 TOM solution (trioctylmethylammonium chloride): 333 ml of TOM for 666 ml of toluene.

F.2.1.7 Ammonia, concentrated, NH_4OH, 280 g l^{-1}.

F.2.1.8 Oxalic acid, saturated solution, $H_2C_2O_4 \cdot 2H_2O$, 140 g l^{-1}.