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# Measurement of radioactivity in the environment — Soil —

## Part 5: Measurement of strontium 90

*Mesurage de la radioactivité dans l'environnement — Sol —*

*Partie 5: Mesurage du strontium 90*

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# Foreword

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

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.

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ISO 18589-5 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 2, *Radiation protection*.

ISO 18589 consists of the following parts, under the general title *Measurement of radioactivity in the environment — Soil*:

- *Part 1: General guidelines and definitions*
- *Part 2 : Guidance for the selection of the sampling strategy, sampling and pre-treatment of samples*
- *Part 3: Measurements of gamma-emitting radionuclides*
- *Part 4: Measurement of plutonium isotopes (plutonium 238 and plutonium 239 + 240) by alpha spectrometry*
- *Part 5: Measurement of strontium 90*
- *Part 6: Measurement of gross alpha and gross beta activities*

# Introduction

ISO 18589 is published in several parts for use jointly or separately according to needs. Parts 1 to 6 concerning the measurements of radioactivity in the soil have been prepared simultaneously. These parts are complementary and are addressed to those responsible for determining the radioactivity present in soil. The first two parts are general in nature. Parts 3 to 5 deal with nuclide-specific measurements and Part 6 with non-specific measurements of gross alpha or gross beta activities.

Additional parts can be added to ISO 18589 in the future if the standardization of the measurement of other radionuclides becomes necessary.

# Measurement of radioactivity in the environment — Soil —

## Part 5:
## Measurement of strontium 90

## 1 Scope

This part of ISO 18589 describes the principles for the measurement of the activity of $^{90}$Sr in equilibrium with $^{90}$Y, and $^{89}$Sr, pure beta-emitting radionuclides, in soil samples. Different chemical separation methods are presented to produce strontium and yttrium sources, the activity of which is determined using proportional counter (PC) or liquid scintillation counter (LSC). The selection of the measuring method depends on the origin of the contamination, the characteristics of the soil being analysed, the required accuracy of measurement and the resources of the available laboratories.

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

In the case of recent fallout immediately following a nuclear accident, the contribution of $^{89}$Sr to the total amount of strontium activity is not negligible. This part of ISO 18589 provides the measurement method to determine the activity of $^{90}$Sr in the 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 31-9, *Quantities and units — Part 9: Atomic and nuclear physics*

ISO 11074, *Soil quality — Vocabulary*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 18589-1, *Measurement of radioactivity in the environment — Soil — Part 1: General guidelines and definitions*

ISO 18589-2, *Measurement of radioactivity in the environment — Soil — Part 2: Guidance for the selection of the sampling strategy, sampling and pre-treatment of samples*

## 3 Terms, definitions and symbols

For the purposes of this document, the terms, definitions and symbols given in ISO 18589-1, ISO 11074 and ISO 31-9 and the following symbols apply.

| | |
|---|---|
| $m$ | Mass of the test portion, expressed in kilograms |
| $a_i$ | Activity per unit of mass, of radionuclide $i$, expressed in becquerel per kilogram |
| $A_{i,\mathrm{s}}$ | Calibration source activity of radionuclide $i$, at the calibration time, expressed in becquerel |
| $A_i$ | Sample source activity of radionuclide $i$, at time $t = 0$, expressed in becquerel |
| $t_\mathrm{g}$ | Sample counting time, expressed in seconds |
| $t_0$ | Background counting time, expressed in seconds, |
| $t_\mathrm{s}$ | Calibration source counting time |
| $r_\mathrm{g}$ | Gross count rate per second |
| $r_0$ | Background count rate per second |
| $r_\mathrm{s}$ | Calibration source count rate per second |
| $R_{\mathrm{c},i}$ | Chemical yield of the extraction of radionuclide $i$ |
| $\varepsilon_i$ | Counting efficiency of radionuclide $i$ |
| $\lambda_i$ | Decay constant of radionuclide $i$ |
| $t_\mathrm{d}$, $t_\mathrm{f}$ | Start and finish time respectively of the measurement referred to $t = 0$, expressed in seconds |
| $r_{\mathrm{g}j}$ | Gross count rate per second, for measurement $j$ |
| $r_{0j}$ | Background count rate per second, for measurement $j$ |
| $r_j$ | Net count rate per second, for measurement $j$ |
| $t_j$ | Start time of measurement $j$, referred to $t = 0$ |
| $u(a_i)$ | Standard uncertainty associated with the measurement result, expressed in becquerel per kilogram |
| $U$ | Expanded uncertainty, calculated by $U = k \cdot u\left(a_i\right)$ with $k = 1, 2, \ldots$, expressed in becquerel per kilogram |
| $a_i^*$ | Decision threshold of radionuclide $i$, expressed in becquerel per kilogram |
| $a_i^\#$ | Detection limit of radionuclide $i$, expressed in becquerel per kilogram |
| $a_i^\triangleleft$, $a_i^\triangleright$ | Lower and upper limits respectively of the confidence interval of radionuclide $i$, expressed in becquerel per kilogram |

## 4 Principle

$^{90}Sr$, $^{90}Y$ and $^{89}Sr$ are pure beta-emitter radionuclides. Their beta-emission energies and half-lives are given in Table 1 [1].

**Table 1 — Average beta-emission energies and half-lives of $^{90}Sr$, $^{90}Y$ and $^{89}Sr$**

| | $^{90}$**Sr** | $^{90}$**Y** | $^{89}$**Sr** |
|---|---|---|---|
| **Beta energy,** keV | 196 | 926,7 | 584,6 |
| **Half-life** | 28,8 a | 2,67 d | 50,6 d |

Strontium can be measured directly or estimated through the measurement of its daughter product, yttrium. All the measurements are based on a chemical separation step followed by beta counting of the element using PC or LSC. (See Table 2 and References [1], [2] and [3].)

### 4.1 Chemical separation

Following the desorption step of the soil test portion, isolate the strontium from the soil solution using precipitation or specific chromatographic resin separation such as a "crown ether" resin. Yttrium can be isolated by precipitation or liquid-liquid extraction.

The separation step should maximize the extraction of the pure element. Choose the method that is selective with a high chemical yield. As thorium, lead, and bismuth radioisotopes are present in soil, it is necessary that they be removed from the sample because they can interfere with $^{90}Sr$ or $^{90}Y$ or $^{89}Sr$ emissions during the detection step. It is necessary that other matrix constituents that can interfere, such as the alkaline earth elements, calcium for strontium, or the transuranic and lanthanide elements for yttrium, also be removed as they reduce the chemical yield of the extraction.

The radiochemical separation yield is calculated using a carrier such as stable Sr or Y, or a radioactive tracer such as $^{85}Sr$. Techniques, such atomic absorption spectroscopy (AAS) or 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 Sr.

When stable strontium is added as a carrier, it is necessary that its original concentration in the test sample be known to avoid the overestimation of the radiochemical separation yield.

### 4.2 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 is checked by following the change over an appropriate time of the $^{90}Y$ or $^{89}Sr$ activity, even though this method is time consuming.

A method of Sr/Y desorption of the soil test portion is given in Annex A. Three measurement methods are presented in Annexes B, C and D.

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[1] Source: BIPM (Bureau International des Poids et Mesures).

**Table 2 — Determination procedures for strontium depending on its origin**

| | **Origin** | **Old fallout** | | | | **Fresh fallout** | |
|---|---|---|---|---|---|---|---|
| | **Radionuclide contents** | $^{90}Sr+^{90}Y$ | | | | $^{90}Sr+^{90}Y$<br>$^{89}Sr$ | |
| **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 $^{90}Sr+^{90}Y$** | Yes<br>15 to 20 days (recommended) | No | No | | Yes<br>15 to 20 days (recommended) | No |
| | **Number** | One | | One | | Two or more | |
| | **Emissions** | $^{90}Sr$<br>$^{90}Y$ | | $^{90}Y$ | | $^{90}Sr$<br>$^{90}Y$<br>$^{89}Sr$ | |
| | **Equipment** | PC or LSC (total) | | PC or LSC (total or Cerenkov) | | PC or LSC (total) | |
| | **Calibration of sources** | $^{90}Sr+^{90}Y$ | $^{90}Sr$<br>$^{90}Y$ | $^{90}Y$ | | $^{90}Sr+^{90}Y$<br>$^{89}Sr$ | $^{90}Sr$<br>$^{90}Y$<br>$^{89}Sr$ |

[a] Y separation is performed following the $^{90}Sr$ –$^{90}Y$ equilibrium in the test sample.

[b] Specific chromatography using crown-ether resin.

[c] Carrier or tracer measurements are done using gamma spectrometry for $^{85}Sr$ and gravimetry, atomic absorption (AAS) or mass spectrometry (MS) for Sr and Y.

## 5 Chemical reagents and equipment

The necessary chemical reagents and equipment for each strontium measurement method are specified in Annexes A, B, C and D.

All the chemical reagents required to carry out this procedure shall be of analytical grade.

## 6 Procedure

### 6.1 Strontium desorption

Strontium is desorbed from the soil test portion by acid treatment and remains in solution in the leachate fraction.

The tracer or carrier is added at the start of this step of the procedure; time is allowed, usually up to one day, to obtain equilibrium before starting the strontium desorption.

If stable strontium is added as a carrier, it is necessary that the original concentration of Sr be determined in the test sample in this step of the procedure before the addition of the carrier.

A strontium desorption method for soil samples is given in Annex A.

## 6.2 Chemical separation

There are three usual techniques for the chemical separation: precipitation [4], [5] liquid-liquid extraction and chromatography extraction using selective crown-ether resin [6], [7], [8]. Annexes B, C and D give a procedure for each of these techniques.

### 6.2.1 Precipitation

The precipitation technique is suited to separate all mineral elements, including strontium, in soils 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 requirement to wait for the yttrium to reach equilibrium limit its use.

The addition of nitric acid leads to a strontium precipitate containing other interfering elements. Successive cycles of dissolution-precipitations 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 and are presented in Annex B.

For the measurement procedure with $^{90}Sr$ and $^{90}Y$ at equilibrium, either the global contribution of yttrium and strontium is measured directly in the precipitate or the yttrium activity can be measured after a last separation from the strontium. In this latter case, the chemical yield is estimated by the addition of a yttrium carrier to the source before the yttrium separation. The final product is a yttrium precipitate, usually under the form of an oxalate precipitate. A method is presented in Annex B.

In 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 the presence of $^{89}Sr$ in the test portion cannot be neglected, it is necessary to choose this technique giving direct measurement of strontium at two different times.

### 6.2.2 Liquid-liquid extraction

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 can be achieved after three days (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 can occur, making it difficult to determine very low levels of activity.

This method is well suited to emergency situations and generally to all samples with low $\beta$-emitting radionuclide contents.

$^{90}Y$ is extracted from the leachate fraction using an organic solvent in the form of an yttrium precipitate. A method is presented in Annex C.

After the source preparation, the $^{90}Y$ should be measured by PC or by measuring the Cerenkov radiation from the $^{90}Y$, by LSC.

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.

### 6.2.3 Chromatography separation on “crown ether” resin

This technique is based on the selective chromatographic separation of strontium using a specific resin with crown ether. The chemical separation is fast and well suited to inspection and monitoring of the environment.

The final source to be measured is a strontium precipitate. A method is given in Annex D.

For the measurement procedure with $^{90}Sr$ and $^{90}Y$ at equilibrium, either the global contribution of yttrium and strontium is measured directly in the precipitate, or the yttrium activity can be measured after the final separation from strontium. In this latter case, the chemical recovery yield is estimated by the addition of a yttrium carrier to the source before the yttrium separation. The final product is a yttrium precipitate, usually in the form of an oxalate precipitate. A method is given in Annex B.

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 the presence of $^{89}$Sr in the test portion cannot be neglected, this technique giving direct measurement of strontium at two different times must be chosen.

## 6.3 Preparation of the source for measurement

### 6.3.1 Source preparation for liquid scintillation counter

A liquid scintillation counter (LSC) directly measures the photons produced by the scintillations in the liquid as a result of the excitation caused by the β-emissions from the source.

The Sr or Y precipitate is dissolved and mixed with the liquid scintillation solution. The solution volume depends on the equipment (vial size) and the specific liquid scintillation solution used. (See one method in Annex C.)

It is necessary that the calibration source 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 being measured. Methods that allow the calibration with a different radionuclide are also available [9].

The blank source should be prepared following the method chosen starting with a test portion without $^{90}$Sr (or directly with distilled water).

### 6.3.2 Source preparation for proportional counter

The proportional counter measures directly the β-emission from the source prepared from a thin-layer deposit to minimize the self-absorption effects.

The Sr or Y precipitate is deposited on a filter by filtration or in a stainless steel dish by direct evaporation (see Annex C).

The diameter of the filter or dish should be similar to that of the detector size.

It is necessary that the calibration source 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.

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

## 6.4 Background determination

Measure the background using a blank source prepared for the method chosen.

## 6.5 Measurement

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

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

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

### 6.5.1 Liquid scintillation counter

LSC measures directly the photons produced further to the excitement of the liquid scintillator by the β-particles. The advantage of this method is the possibility of discriminating β-particle energies, allowing one to check the presence of other beta contaminants in the source.

When assessing the $^{90}$Sr activity by the $^{90}$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 it is negligible for $^{90}$Sr.

### 6.5.2 Gas-flow proportional counter

A gas-flow proportional counter measures directly the β-radiation, without energy discrimination, from the source, usually prepared as a thin-layer deposit.

The use of a double window (α and β) in this type of counter allows checking for the presence of alpha-emitter contaminants in the source being measured. If other short-half-life β-emitters are present, they can be detected by performing successive measurements of the source at given times.

When assessing the $^{90}$Sr activity by the $^{90}$Y measurement, if the presence of small amounts of $^{90}$Sr cannot be excluded, then its presence can be detected by performing successive measurements.

## 6.6 Calibration

The procedure to calibrate the counters is as follows.

- Select $t_{\mathrm{s}}$ to collect at least $10^4$ counts.
- Determine the beta count rate of the calibration source 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 being measured.
- Calculate the counting efficiency of the counter by dividing the count rate measured with the activity of the calibration source, as given in Equation (1):

$$\varepsilon_i = (r_{\mathrm{s}} - r_0)/A_i \qquad (1)$$

# 7 Expression of results

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

### 7.1.1 Calculation of the activity per unit of mass

The activity per unit of mass in source samples where the $^{90}$Y has been completely separated from the parent nuclide $^{90}$Sr cannot be reassessed until the daughter nuclide $^{90}$Y has grown back and is in equilibrium with the parent nuclide, $^{90}$Sr. This occurs 15 days to 20 days after $t = 0$, where $t = 0$ is the time at which all the $^{90}$Y was removed from the sample.

The result of the measurement gives the gross number of counts from the $^{90}$Sr+$^{90}$Y. Dividing the gross counts by the counting time gives the gross count rate, $r_{\mathrm{g}}$.

NOTE To apply this method, it is necessary to 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 per unit of mass of $^{90}$Sr+$^{90}$Y, $a_{\text{Sr+Y}}$, can be calculated as given in Equation (2):

$$a_{\text{Sr+Y}} = \frac{r_\text{g} - r_0}{m \cdot R_{\text{c,Sr}} \cdot \varepsilon_{\text{Sr+Y}}} \tag{2}$$

The activity per unit of mass of $^{90}$Sr, can be calculated as given in Equation (3):

$$a_{^{90}\text{Sr}} = \frac{a_{\text{Sr+Y}}}{2} = \frac{r_\text{g} - r_0}{2 \cdot m \cdot R_{\text{c,Sr}} \cdot \varepsilon_{\text{Sr+Y}}} = \left(r_\text{g} - r_0\right) \cdot w_{^{90}\text{Sr}} \quad \text{with} \quad w_{^{90}\text{Sr}} = \frac{1}{2 \cdot m \cdot R_{\text{c,Sr}} \cdot \varepsilon_{\text{Sr+Y}}} \tag{3}$$

### 7.1.2 Standard uncertainty

According to ISO/IEC Guide 98-3 [13], also known as the *GUM*, the standard uncertainty of $a_{^{90}\text{Sr}}$ is calculated as given in Equation (4):

$$\begin{aligned} u(a_{^{90}\text{Sr}}) &= \sqrt{w_{^{90}\text{Sr}}^2 \cdot \left[u^2(r_\text{g}) + u^2(r_0)\right] + a_{^{90}\text{Sr}}^2 \cdot u_\text{rel}^2\left(w_{^{90}\text{Sr}}\right)} \\ &= \sqrt{w_{^{90}\text{Sr}}^2 \cdot \left(r_\text{g}/t_\text{g} + r_0/t_0\right) + a_{^{90}\text{Sr}}^2 \cdot u_\text{rel}^2\left(w_{^{90}\text{Sr}}\right)} \end{aligned} \tag{4}$$

where the uncertainties of the sample and background counting times are neglected.

The relative standard uncertainty of $w$ is calculated as given in Equation (5):

$$u_\text{rel}^2\left(w_{^{90}\text{Sr}}\right) = u_\text{rel}^2\left(R_{\text{c,Sr}}\right) + u_\text{rel}^2(m) + u_\text{rel}^2\left(\varepsilon_{\text{Sr+Y}}\right) \tag{5}$$

The relative standard uncertainty of $\varepsilon_{\text{Sr+Y}}$ is calculated as given in Equation (6):

$$u_\text{rel}^2\left(\varepsilon_{\text{Sr+Y}}\right) = u_\text{rel}^2\left(r_\text{s} - r_0\right) + u_\text{rel}^2\left(A_{\text{Sr+Y}}\right) = \left(r_\text{s}/t_\text{s} + r_0/t_0\right)/\left(r_\text{s} - r_0\right)^2 + u_\text{rel}^2\left(A_{\text{Sr+Y}}\right) \tag{6}$$

$u_\text{rel}\left(A_{\text{Sr+Y}}\right)$ includes all the uncertainties related to the calibration source, that is, those of the standard solution and the preparation of the calibration source.

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

The calculation of the characteristic limits (see ISO 11929 [12]) requires the calculation of $\tilde{u}\left(\tilde{a}_{^{90}\text{Sr}}\right)$, i.e., the standard uncertainty of $a_{^{90}\text{Sr}}$ as a function of its true value, calculated [10] as given in Equation (7):

$$\tilde{u}\left(\tilde{a}_{^{90}\text{Sr}}\right) = \sqrt{w_{^{90}\text{Sr}}^2 \cdot \left[\left(\tilde{a}_{^{90}\text{Sr}}/w_{^{90}\text{Sr}} + r_0\right)/t_\text{g} + r_0/t_0\right] + \tilde{a}_{^{90}\text{Sr}}^2 \cdot u_\text{rel}^2\left(w_{^{90}\text{Sr}}\right)} \tag{7}$$

### 7.1.3 Decision threshold

The decision threshold, $a_{^{90}\text{Sr}}^*$, is obtained from Equation (7) for $\tilde{a}_{^{90}\text{Sr}} = 0$ (see ISO 11929), to yield Equation (8):

$$a_{^{90}\text{Sr}}^* = k_{1-\alpha} \cdot \tilde{u}(0) = k_{1-\alpha} \cdot w_{^{90}\text{Sr}} \cdot \sqrt{r_0/t_\text{g} + r_0/t_0} \tag{8}$$

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

### 7.1.4 Detection limit

The detection limit, $a^{\#}_{{}^{90}\mathrm{Sr}}$, is calculated as given in Equation (9) (see ISO 11929):

$$\begin{aligned} a^{\#}_{{}^{90}\mathrm{Sr}} &= a^{*}_{{}^{90}\mathrm{Sr}} + k_{1-\beta} \cdot \tilde{u}\left(a^{\#}_{{}^{90}\mathrm{Sr}}\right) \\ &= a^{*}_{{}^{90}\mathrm{Sr}} + k_{1-\beta} \cdot \sqrt{w^{2}_{{}^{90}\mathrm{Sr}} \cdot \left[\left(a^{\#}_{{}^{90}\mathrm{Sr}} \middle/ w_{{}^{90}\mathrm{Sr}} + r_0\right)\middle/ t_g + r_0/t_0\right] + a^{\#2}_{{}^{90}\mathrm{Sr}} \cdot u^{2}_{\mathrm{rel}}\left(w_{{}^{90}\mathrm{Sr}}\right)} \end{aligned} \tag{9}$$

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

The detection limit can be calculated by solving Equation (9) for $a^{\#}_{{}^{90}\mathrm{Sr}}$ or, more simply, by iteration with a starting approximation of $a^{\#}_{{}^{90}\mathrm{Sr}} = 2 \cdot a^{*}_{{}^{90}\mathrm{Sr}}$.

By taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$, and the solution of Equation (9) is given by Equation (10):

$$a^{\#}_{{}^{90}\mathrm{Sr}} = \frac{2 \cdot a^{*}_{{}^{90}\mathrm{Sr}} + \left(k^{2} \cdot w_{{}^{90}\mathrm{Sr}}\right)\big/ t_g}{1 - k^{2} \cdot u^{2}_{\mathrm{rel}}\left(w_{{}^{90}\mathrm{Sr}}\right)} \tag{10}$$

## 7.2 Determination of $^{90}$Sr from $^{90}$Y

### 7.2.1 Calculation of the activity per unit of mass

The $^{90}$Y is measured immediately after its separation in the test portion when Sr and Y are in equilibrium, $t = 0$ being the time when the $^{90}$Y is separated from the $^{90}$Sr and starts to decay with a half-life of 2,7 days.

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

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

The activity per unit of mass of $^{90}$Y, $a_Y$, is calculated at time $t = 0$, as given in Equation (11):

$$a_Y = \frac{r_g \cdot t_g - r_0 \cdot t_g}{\varepsilon_Y \cdot m \cdot R_c \cdot \int_{t_d}^{t_f} e^{-\lambda_Y t} \cdot dt} = \left(r_g - r_0\right) \cdot w_Y \quad \text{with} \quad w_Y = \frac{\lambda_Y \cdot t_g}{\left(e^{-\lambda_Y t_d} - e^{-\lambda_Y t_f}\right)} \cdot \frac{1}{\varepsilon_Y \cdot m \cdot R_c} \tag{11}$$

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

The integral allows a correction for the activity of the decay of $^{90}$Y during the counting time ($t_g = t_f - t_d$) and the activity per unit of mass of $^{90}$Sr is given by Equation (12):

$$a_{{}^{90}\mathrm{Sr}} = a_Y = \left(r_g - r_0\right) \cdot w_Y \tag{12}$$

### 7.2.2 Standard uncertainty

According to ISO/IEC Guide 98-3, the standard uncertainty of $a_{{}^{90}\mathrm{Sr}}$ is calculated as given in Equation (13):

$$u\left(a_{{}^{90}\mathrm{Sr}}\right) = \sqrt{w_Y{}^{2} \cdot \left[u^{2}\left(r_g\right) + u^{2}\left(r_0\right)\right] + a^{2}_{{}^{90}\mathrm{Sr}} \cdot u^{2}_{\mathrm{rel}}\left(w_Y\right)} = \sqrt{w_Y{}^{2} \cdot \left(r_g/t_g + r_0/t_0\right) + a^{2}_{{}^{90}\mathrm{Sr}} \cdot u^{2}_{\mathrm{rel}}\left(w_Y\right)} \tag{13}$$

where the uncertainties of the sample and background counting times are neglected.

The relative standard uncertainty of $w$ is calculated as given in Equation (14):

$$u_{\text{rel}}^2(w_{\text{Y}}) = u_{\text{rel}}^2(R_{\text{c}}) + u_{\text{rel}}^2(m) + u_{\text{rel}}^2(\varepsilon_{\text{Y}}) \tag{14}$$

The relative standard uncertainty of $\varepsilon_{\text{Y}}$ is calculated as given in Equation (15):

$$u_{\text{rel}}^2(\varepsilon_{\text{Y}}) = u_{\text{rel}}^2(r_{\text{s}} - r_0) + u_{\text{rel}}^2(A_{\text{Y}}) = (r_{\text{s}}/t_{\text{s}} + r_0/t_0)\Big/(r_{\text{s}} - r_0)^2 + u_{\text{rel}}^2(A_{\text{Y}}) \tag{15}$$

$u_{\text{rel}}(A_{\text{Y}})$ includes all the uncertainties related to the calibration source, that is, those of the standard solution and the preparation of the calibration source.

$u_{\text{rel}}(R_{\text{c}})$ is the uncertainty related with the chemical yield, which can be calculated as given in Equation (16):

$$u_{\text{rel}}^2(R_{\text{c}}) = u_{\text{rel}}^2(R_{\text{c,Sr}}) + u_{\text{rel}}^2(R_{\text{c,Y}}) \tag{16}$$

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

The calculation of the characteristic limits (see ISO 11929), requires the calculation of $\tilde{u}(\tilde{a}_{^{90}\text{Sr}})$, i.e., the standard uncertainty of $a_{^{90}\text{Sr}}$ as a function of its true value, calculated [10] as given in Equation (17):

$$\tilde{u}(\tilde{a}_{^{90}\text{Sr}}) = \sqrt{w_{\text{Y}}^2 \cdot \left[\left(\tilde{a}_{^{90}\text{Sr}}/w_{\text{Y}} + r_0\right)\Big/t_{\text{g}} + r_0/t_0\right] + \tilde{a}_{^{90}\text{Sr}}^2 \cdot u_{\text{rel}}^2(w_{\text{Y}})} \tag{17}$$

### 7.2.3 Decision threshold

The decision threshold, $a_{^{90}\text{Sr}}^*$, is obtained from Equation (17) for $\tilde{a}_{^{90}\text{Sr}} = 0$ (see ISO 11929) to yield Equation (18):

$$a_{^{90}\text{Sr}}^* = k_{1-\alpha} \cdot \tilde{u}(0) = k_{1-\alpha} \cdot w_{\text{Y}} \cdot \sqrt{r_0/t_{\text{g}} + r_0/t_0} \tag{18}$$

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

### 7.2.4 Detection limit

The detection limit, $a_{^{90}\text{Sr}}^\#$, is calculated as given in Equation (19) (see ISO 11929):

$$a_{^{90}\text{Sr}}^\# = a_{^{90}\text{Sr}}^* + k_{1-\beta} \cdot \tilde{u}\left(a_{^{90}\text{Sr}}^\#\right) = a_{^{90}\text{Sr}}^* + k_{1-\beta} \cdot \sqrt{w_{\text{Y}}^2 \cdot \left[\left(a_{^{90}\text{Sr}}^\#/w_{\text{Y}} + r_0\right)\Big/t_{\text{g}} + r_0/t_0\right] + a_{^{90}\text{Sr}}^{\#2} \cdot u_{\text{rel}}^2(w_{\text{Y}})} \tag{19}$$

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

The detection limit can be calculated by solving Equation (19) for $a_{^{90}\text{Sr}}^\#$ or, more simply, by iteration with a starting approximation $a_{^{90}\text{Sr}}^\# = 2 \cdot a_{^{90}\text{Sr}}^*$.

By taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of Equation (19) is given by Equation (20):

$$a_{^{90}\text{Sr}}^\# = \frac{2 \cdot a_{^{90}\text{Sr}}^* + \left(k^2 \cdot w_{\text{Y}}\right)\Big/t_{\text{g}}}{1 - k^2 \cdot u_{\text{rel}}^2(w_{\text{Y}})} \tag{20}$$

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

### 7.3.1 Calculation of the activity per unit of mass

This method is based on the realization of two measurements of the same source at two different times, $t_1$ and $t_2$, after the time $t = 0$, the moment of separation of the Yttrium present in the test portion. It is suggested that the same counting time, $t_\mathrm{g}$, 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 given in Equation (21):

$$r_j = r_{\mathrm{g}j} - r_{0j} \tag{21}$$

If the measurements are made when equilibrium between the $^{90}$Sr and $^{90}$Y has been reached, then the net counting rates can be calculated using the Equations (22) and (23), considering that the $^{90}$Sr and $^{89}$Sr activities are constant during the counting time, and using the appropriate decay constants:

$$r_1 = 2 \cdot A_{^{90}\mathrm{Sr}} \cdot \varepsilon_{^{90}\mathrm{Sr+Y}} + \varepsilon_{^{89}\mathrm{Sr}} A_{^{89}\mathrm{Sr}} \mathrm{e}^{-\lambda_{^{89}\mathrm{Sr}} t_1} \tag{22}$$

$$r_2 = 2 \cdot A_{^{90}\mathrm{Sr}} \cdot \varepsilon_{^{90}\mathrm{Sr+Y}} + \varepsilon_{^{89}\mathrm{Sr}} A_{^{89}\mathrm{Sr}} \mathrm{e}^{-\lambda_{^{89}\mathrm{Sr}} t_2} \tag{23}$$

Equations (22) and (23) can be rearranged to yield Equations (24) and (25):

$$A_{^{90}\mathrm{Sr}} = \frac{r_2 - r_1 \mathrm{e}^{-\lambda_{^{89}\mathrm{Sr}}(t_2 - t_1)}}{2\varepsilon_{^{90}\mathrm{Sr+Y}} \left[1 - \mathrm{e}^{-\lambda_{^{89}\mathrm{Sr}}(t_2 - t_1)}\right]} \tag{24}$$

$$A_{^{89}\mathrm{Sr}} = \frac{(r_1 - r_2)\mathrm{e}^{+\lambda_{^{89}\mathrm{Sr}} t_1}}{\varepsilon_{^{89}\mathrm{Sr}} \left[1 - \mathrm{e}^{-\lambda_{^{89}\mathrm{Sr}}(t_2 - t_1)}\right]} \tag{25}$$

The activity per unit of mass, $a_i$, of radionuclide $i$ is calculated using Equation (26):

$$a_i = A_i / m \cdot R_{\mathrm{c,Sr}} \tag{26}$$

Thus,

$$a_{^{90}\mathrm{Sr}} = w_{90} \cdot (r_2 - c r_1) \tag{27}$$

where

$$w_{90} = \frac{1}{m \cdot R_{\mathrm{c,Sr}} \cdot 2 \cdot \varepsilon_{90_{\mathrm{Sr+Y}}} \cdot (1 - c)}; \tag{28}$$

$$c = \mathrm{e}^{-\lambda_{^{89}\mathrm{Sr}}(t_2 - t_1)}. \tag{29}$$

And,

$$a_{^{89}\mathrm{Sr}} = w_{89} \cdot (r_2 - r_1) \tag{30}$$

where

$$w_{89} = \frac{\mathrm{e}^{+\lambda_{89_{\mathrm{Sr}}} t_1}}{m \cdot R_{\mathrm{c,Sr}} \cdot \varepsilon_{89_{\mathrm{Sr}}} \cdot (c-1)}; \tag{31}$$

$$c = \mathrm{e}^{-\lambda_{89_{\mathrm{Sr}}}(t_2 - t_1)}. \tag{32}$$

### 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 $a_i$ is calculated as given by Equations (33) and (34):

$$u\left(a_{90_{\mathrm{Sr}}}\right) = \sqrt{{w_{90}}^2 \cdot \left[u^2(r_2) + c^2 \cdot u^2(r_1)\right] + a_{90_{\mathrm{Sr}}}^2 \cdot u_{\mathrm{rel}}^2(w_{90})} \tag{33}$$

$$u\left(a_{89_{\mathrm{Sr}}}\right) = \sqrt{{w_{89}}^2 \cdot \left[u^2(r_1) + u^2(r_2)\right] + a_{89_{\mathrm{Sr}}}^2 \cdot u_{\mathrm{rel}}^2(w_{89})} \tag{34}$$

Assuming that $u^2(c) = 0$, the relative standard uncertainty of $r_j$ is calculated as given by Equation (35):

$$u^2\left(r_j\right) = r_{\mathrm{g}j}/t_{\mathrm{g}} + r_{0j}/t_0 \tag{35}$$

The relative standard uncertainties of $w_{90}$ and $w_{89}$ are calculated by Equations (36) and (37), respectively:

$$u_{\mathrm{rel}}^2(w_{90}) = u_{\mathrm{rel}}^2(R_{\mathrm{c,Sr}}) + u_{\mathrm{rel}}^2(m) + u_{\mathrm{rel}}^2\left(\varepsilon_{90_{\mathrm{Sr+Y}}}\right) \tag{36}$$

$$u_{\mathrm{rel}}^2(w_{89}) = u_{\mathrm{rel}}^2(R_{\mathrm{c,Sr}}) + u_{\mathrm{rel}}^2(m) + u_{\mathrm{rel}}^2\left(\varepsilon_{89_{\mathrm{Sr}}}\right) \tag{37}$$

The relative standard uncertainty of $\varepsilon_i$ is calculated by Equation (38):

$$u_{\mathrm{rel}}^2(\varepsilon_i) = u_{\mathrm{rel}}^2(r_{\mathrm{s}} - r_0) + u_{\mathrm{rel}}^2(A_i) = (r_{\mathrm{s}}/t_{\mathrm{s}} + r_0/t_0)\big/(r_{\mathrm{s}} - r_0)^2 + u_{\mathrm{rel}}^2(A_i) \tag{38}$$

$u_{\mathrm{rel}}(A_i)$ includes all the uncertainties related to the calibration source, that is, 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, which depends on its method of evaluation.

The calculation of the characteristic limits (see ISO 11929) requires a calculation of $\tilde{u}(\tilde{a}_i)$, i.e., the standard uncertainty of $a_i$ as a function of its true value, calculated [10] as given by Equations (39) and (40):

$$\tilde{u}\left(\tilde{a}_{90_{\mathrm{Sr}}}\right) = \sqrt{{w_{90}}^2 \cdot \left[\left(r_{02} + c^2 r_{01}\right)\left(\frac{1}{t_0} + \frac{1}{t_{\mathrm{g}}}\right) + \frac{1+c^2}{t_{\mathrm{g}}(1-c)}\left(\frac{\tilde{a}_{90_{\mathrm{Sr}}}}{w_{90}} - \frac{a_{89_{\mathrm{Sr}}}}{w_{89}}\right) + \frac{a_{89_{\mathrm{Sr}}}}{w_{89} t_{\mathrm{g}}}\right] + \tilde{a}_{90_{\mathrm{Sr}}}^2 \cdot u_{\mathrm{rel}}^2(w_{90})} \tag{39}$$

$$\tilde{u}\left(\tilde{a}_{89_{\mathrm{Sr}}}\right) = \sqrt{{w_{89}}^2 \cdot \left[\left(r_{01} + r_{02}\right)\left(\frac{1}{t_0} + \frac{1}{t_{\mathrm{g}}}\right) + \frac{2}{t_{\mathrm{g}}(1-c)}\left(\frac{a_{90_{\mathrm{Sr}}}}{w_{90}} - \frac{\tilde{a}_{89_{\mathrm{Sr}}}}{w_{89}}\right) + \frac{\tilde{a}_{89_{\mathrm{Sr}}}}{t_{\mathrm{g}} w_{89}}\right] + \tilde{a}_{89_{\mathrm{Sr}}}^2 \cdot u_{\mathrm{rel}}^2(w_{89})} \tag{40}$$

### 7.3.3 Decision threshold

The decision thresholds, $a_i^*$, are obtained from Equations (39) and (40) for $\tilde{a}_i = 0$ (see ISO 11929) to yield Equations (41) and (42):

$$a^*_{90_{\text{Sr}}} = k_{1-\alpha}\cdot\tilde{u}(0) = k_{1-\alpha}\cdot w_{90}\cdot\sqrt{\left(r_{02}+c^2 r_{01}\right)\left(\frac{1}{t_0}+\frac{1}{t_{\text{g}}}\right)+\frac{c\cdot(c+1)}{(c-1)}\frac{a_{89_{\text{Sr}}}}{t_{\text{g}} w_{89}}} \tag{41}$$

$$a^*_{89_{\text{Sr}}} = k_{1-\alpha}\cdot\tilde{u}(0) = k_{1-\alpha}\cdot w_{89}\cdot\sqrt{\left(r_{01}+r_{02}\right)\left(\frac{1}{t_0}+\frac{1}{t_{\text{g}}}\right)+\frac{2}{t_{\text{g}}(1-c)}\frac{a_{90_{\text{Sr}}}}{w_{90}}} \tag{42}$$

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

### 7.3.4 Detection limit

The detection limits, $a_i^\#$, are calculated as given by Equation (43):

$$a^\#_{90_{\text{Sr}}} = a^*_{90_{\text{Sr}}} + k_{1-\beta}\cdot\tilde{u}\left(a^\#_{90_{\text{Sr}}}\right) = a^*_{90_{\text{Sr}}} + k_{1-\beta}\cdot\sqrt{w_{\text{Y}}^2\cdot\left[\left(a^\#_{90_{\text{Sr}}}/w_{\text{Y}}+r_0\right)\Big/t_{\text{g}}+r_0/t_0\right]+a^{\#2}_{90_{\text{Sr}}}\cdot u^2_{\text{rel}}\left(w_{\text{Y}}\right)} \tag{43}$$

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

The detection limit can be calculated by solving Equation (43) for $a_i^\#$ or, more simply, by iteration with a starting approximation of $a_i^\# = 2\cdot a_i^*$.

By taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$, and the solution of Equation (43) is given by Equations (44) and (45):

$$a^\#_{90_{\text{Sr}}} = \frac{2\cdot a^*_{90_{\text{Sr}}} + k^2\cdot w_{90}\cdot\left(1+c^2\right)\big/t_{\text{g}}\cdot(1-c)}{1-k^2\cdot u^2_{\text{rel}}\left(w_{90}\right)} \tag{44}$$

$$a^\#_{89_{\text{Sr}}} = \frac{2\cdot a^*_{89_{\text{Sr}}} + k^2\cdot w_{89}(1+c)/(c-1)\cdot t_{\text{g}}}{1-k^2\cdot u^2_{\text{rel}}\left(w_{89}\right)} \tag{45}$$

## 7.4 Confidence limits

The values of the lower confidence limit, $a_i^\triangleleft$, and the upper confidence limit, $a_i^\triangleright$, are calculated as given by Equations (46) and (47) respectively (see ISO 11929):

$$a_i^\triangleleft = a_i - k_p\cdot u\left(a_i\right);\quad p = \omega\cdot(1-\gamma/2) \tag{46}$$

$$a_i^\triangleright = a_i + k_q\cdot u\left(a_i\right);\quad q = 1-\omega\cdot\gamma/2 \tag{47}$$

where

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

$\omega$ may be set equal to 1 if $a_i \geqslant 4 \cdot u(a_i)$. In this case, Equation (48) holds:

$$a_i^{\triangleleft,\triangleright} = a_i \pm k_{1-\gamma/2} \cdot u(a_i) \tag{48}$$

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

## 8 Test report

The test report shall be in accordance with ISO/IEC 17025 and shall contain the following information:

a) reference to this part of ISO 18589;

b) identification of the sample;

c) units in which the results are expressed;

d) test result, $a_i \pm u(a_i)$ or $a_i \pm U$, with the associated $k$ value.

Complementary information can be provided, such as the following:

- probabilities $\alpha$, $\beta$ and (1 – $\gamma$);
- decision threshold and the detection limit; there are different ways to present the results depending on the customer's request:
    - when the activity per unit of mass, $a_i$, is compared with the decision threshold (see ISO 11929), the result of the measurement should be expressed as $\leqslant a_i^*$ when the result is below the decision threshold;
    - when the activity per unit of mass, $a_i$, is compared with the detection limit, the result of the measurement can be expressed as $\leqslant a_i^\#$ 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;
- any relevant information likely to affect the results.

# Annex A
(informative)

# Test portion preparation

## A.1 Principles

The strontium content in the test portion of soils can be extracted by a simple nitric acid digestion.

## A.2 Chemical reagents

**A.2.1** $Sr^{2+}$ **carrier**, $^{85}Sr$ **tracer** or Y **metal carrier**.

**A.2.2** **Nitric acid**, diluted, $c(HNO_3) = 8$ mol/l.

**A.2.3** **Hydrogen peroxide**, $w(H_2O_2) = 30$ %.

**A.2.4** **Sodium nitrite** ($NaNO_2$).

## A.3 Equipment

**A.3.1** **Analytical balance**, accurate to 0,1 mg.

**A.3.2** **Hot plate**.

**A.3.3** **Centrifuge** and **tubes**.

**A.3.4** **Furnace**.

## A.4 Procedure

This procedure, based on a cycle of acid leaching and dry evaporation, includes the following steps.

a) Weigh between 0,1 g and 50 g of the test sample prepared in compliance with ISO 18589-2. The mass of the test portion depends on the assumed activity of the sample, the desired detection limit and the method chosen.

b) Gradually add 10 ml of 8 molar $HNO_3$ (A.2.2) per gram of sample.

c) Add the tracer or carrier (A.2.1) and stir to homogenize.

d) Wait up to one day.

e) Digest on a hot plate (A.3.2) for at least 8 h with intermittent additions of $H_2O_2$ (A.2.3).

f) Transfer the sample to a centrifuge tube (A.3.3) and centrifuge it. Retain the supernatant.

NOTE It is possible to filter the sample, once it has cooled, using a fibreglass filter instead of centrifuging.

g) Rinse the beaker with 8 molar $HNO_3$ (A.2.2), add to the same tube, stir and centrifuge. Add the supernatant to that retained from step e). Repeat the process until the supernatant is clear.

h) Evaporate the supernatant to dryness.

i) Add 50 ml of 8 molar $HNO_3$ (A.2.2).

j) Add 0,6 g of $NaNO_2$ (A.2.4), heat to eliminate the nitrous fumes, then leave it to cool.

The sample is ready to undergo a radiochemical separation.

The dissolution may also be carried out using microwave digestion apparatus. Operating conditions for each system should be adjusted to the type of apparatus used and the nature of the soil to be digested.

# Annex B
(informative)

# Measurement of strontium by precipitation

## B.1 Principle

The 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, a strontium carbonate precipitate, can be measured by proportional counting or by liquid-scintillation counting after being dissolved. Another possibility is a separation of the yttrium, when it has reached equilibrium, from the strontium by precipitation in the form of an oxalate and subsequent measurement by PC or LSC.

The mass of the test portion shall take into account the presumed activity of the sample and the desired detection limit. The procedures presented in Clause B.4 are applicable to solid samples from 1 g to 50 g.

## B.2 Chemical reagents

**B.2.1 Demineralized water.**

**B.2.2 Ammonia**, concentrated, $w(NH_4OH) = 25$ %.

**B.2.3 Nitric acid**, concentrated, $w(HNO_3) = 65$ %.

**B.2.4 Nitric acid**, diluted, $c(HNO_3) = 8$ mol/l.

**B.2.5 Nitric acid, diluted**, $c(HNO_3) = 2{,}5$ mol/l.

**B.2.6 Sodium chromate**, solution, $c(Na_2CrO_4) = 1$ mol/l.

**B.2.7 Sodium carbonate** ($Na_2CO_3$).

**B.2.8** $Fe^{3+}$ **carrier solution** (20 mg/ml), $w(FeCl_3) = 30$ %.

**B.2.9 Barium acetate** (BaAc), solution.

**B.2.10 Hydrochloric acid**, concentrated, $w(HCl) = 37$ %.

**B.2.11 Oxalic acid**, saturated solution ($H_2C_2O_4$ $2H_2O = 140$ g/l).

**B.2.12 Scintillation solution.**

**B.2.13 Yttrium carrier solution** (20 mg/ml in 0,1 mol/l $HNO_3$) (only in case of Y separation).

**B.2.14 Methanol or ethanol.**

**B.2.15 Sodium carbonate**, $c(Na_2CO_3) = 0{,}1$ mol/l.

**B.2.16 Ammonia**, diluted, 1 %.

## B.3 Equipment

**B.3.1 Standard laboratory equipment**.

**B.3.2 Analytical balance**, accurate to 0,1 mg.

**B.3.3 Oven**.

**B.3.4 Furnace**.

**B.3.5 Hot plate**, with temperature control and magnetic stirring.

**B.3.6 pH meter**.

**B.3.7 Centrifuge** and **tubes**.

**B.3.8 Atomic absorption spectroscope** (AAS) or **atomic emission spectroscope** (ICP-AES) or **mass spectrometer** (ICP-MS) or **gamma spectrometer**.

**B.3.9 Proportional** or **liquid-scintillation counter**.

**B.3.10 Filtration apparatus**, for fibreglass or cellulose filters with diameters, for example, of 5,5 cm and 2,8 cm.

The diameters of the filters are for information; they shall correspond to the filtration device and the geometry of the counter used).

**B.3.11 Filters**, cellulose and fibreglass.

**B.3.12 Funnel**, Buchner.

**B.3.13 Lamps**, infrared.

**B.3.14 Desiccator**.

**B.3.15 Test dish**, stainless steel, with a diameter compatible with the geometry of the counter or polyethylene vials.

**B.3.16 Flasks**, plastic.

## B.4 Procedure

### B.4.1 Separation of alkaline metals and calcium

This phase consists of the following stages.

a) Evaporate the nitric solution obtained after the test portion preparation until the volume is between 50 ml and 100 ml or until the salts appears.

b) Add 200 ml of concentrated $HNO_3$ (B.2.3) and evaporate until the volume is 100 ml or until salt appears. Cover with a watch glass and leave it to cool.

c) Add 50 ml of concentrated $HNO_3$ (B.2.3).

d) Stir for 30 min.

e) Filter the $Sr(NO_3)_2$ obtained using the filter pump and fibreglass filter apparatus (B.3.10).

f) Leave the precipitate to dry. Clean with concentrated $HNO_3$ (B.2.3). Throw away the filtrate.

g) Dissolve the precipitate in hot, demineralized water (B.2.1). Clean the filter to obtain 50 ml of solution.

### B.4.2 Separation of barium, radium and lead

This phase consists of the following stages.

a) Adjust the pH to 4 to 6 with concentrated ammonia (B.2.2).

b) Add 4 ml of barium acetate (B.2.9) and heat.

c) Add 1 ml of 1 molar sodium chromate (B.2.6) and stir.

d) Leave it to cool; filter using a filter pump and fibreglass filter apparatus (B.3.10) or Buchner funnel (B.3.12).

e) Wash the filter with demineralized water (B.2.1). Throw away the filter with the precipitate.

### B.4.3 Separation of fission products and yttrium

This phase consists of the following stages.

a) Transfer the filtrate to a 250 ml glass beaker and add 1 ml of the $Fe^{3+}$ carrier solution (B.2.8).

b) Adjust the pH to 9 using concentrated ammonia (B.2.2).

c) Heat for 10 min, avoid boiling.

d) Leave it to cool and filter using a fibreglass filter apparatus (B.3.10).

e) Wash the filter with 10 ml of demineralized water (B.2.1). Throw away the filter with the precipitate.

f) Record the date and time of precipitation of $Sr(NO_3)_2$ as $t = 0$ for the time from the separation of the yttrium present in the test portion.

### B.4.4 Strontium purification

This phase consists of the following stages.

a) Adjust the pH to 8 using ammonia (B.2.2), add 20 ml of the saturated sodium carbonate solution (B.2.15). Stir for 30 min.

b) Filter the $SrCO_3$ obtained using the filter pump and a fibreglass filter (B.3.11). Rinse the beaker and the precipitate with the 0,1 molar sodium carbonate solution (B.2.15) and then with demineralized water (B.2.1).

c) Throw away the filtrate, leaving the precipitate to dry for at least 10 min, with the vacuum pump from the filter system running.

d) Turn off the vacuum pump (B.3.10) and slowly pour 100 ml of concentrated $HNO_3$ (B.2.3) through the precipitate.

e) Turn on the vacuum pump and leave the precipitate to dry.

f) Dissolve the precipitate with water and collect the solution in a glass beaker; the use of hot water is advisable.

g) Adjust the pH to 8 to 9 using concentrated ammonia (B.2.2); heat for a few minutes, avoid boiling.

h) Add 10 ml of the saturated sodium carbonate solution (B.2.15). Stir for 30 min.

i) Filter the $SrCO_3$ obtained using the filter pump and a pre-weighed fibreglass or cellulose filter (B.3.11). Rinse the beaker and the precipitate with 1 % ammonia solution and then with demineralized water (B.2.1) or methanol (B.2.14).

#### B.4.4.1 Preparation of the source for measurement by PC

This procedure is carried out as follows.

a) Put the filter with the $SrCO_3$ precipitate directly into a pre-weighed test dish (B.3.15), which is the source that is measured.

b) Leave the source to dry in an oven (B.3.3) until a constant mass is obtained, then leave it to cool in a desiccator (B.3.14).

c) After weighing, put the source in the desiccator (B.3.14) until it is measured.

#### B.4.4.2 Preparation of the source for measurement by LSC

This procedure is carried out as follows.

a) Dissolve the $SrCO_3$ precipitate in a maximum of 35 ml of 2,5 molar nitric acid (B.2.5). Put the solution in a pre-weighed plastic flask (B.3.16) and record the mass.

b) Transfer 14 ml of this solution to a 20 ml pre-weighed polyethylene flask (B.3.16) and record the mass.

c) Add 6 ml of scintillation solution (B.2.12); weigh and stir until dissolved. This is the source that is measured.

d) Maintain the source at 4 ± 3 °C until it is measured.

### B.4.5 Yttrium extraction

This phase consists of the following stages.

a) Dissolve the $SrCO_3$ precipitate in a maximum volume of 35 ml of 2,5 molar nitric acid (B.2.5). Put the solution in a pre-weighed plastic flask (B.3.16) and record the mass.

b) Add the yttrium carrier (B.2.13).

c) Store for two weeks in order to reach an equilibrium of $^{90}Y$ of more than 95 %.

d) Pour the solution at equilibrium into a centrifuge tube (B.3.7). Adjust the pH to 8 using ammonia (B.2.2). Heat in the water bath to 90 °C. Cold centrifuge to separate the precipitate.

e) Collect in a beaker the supernatant, which contains the Sr carrier or tracer that allows the determination of the extraction yield of the strontium.

f) Record the date and time of the separation of the strontium.

g) Dissolve the yttrium precipitate in five drops of concentrated HCl (B.2.10) and 30 ml of demineralized water (B.2.1).

h) Add, whilst stirring, 5 ml of the saturated oxalic acid solution (B.2.11) and adjust the pH to 2 to 2,5 using ammonia (B.2.2). Heat in the water bath to 90 °C, then leave to cool for 15 min.

i) Filter the yttrium oxalate precipitate through the pre-weighed fibreglass filter (B.3.11). Rinse the centrifuge tube with demineralized water.

#### B.4.5.1 Preparation of the source for measurement by PC

This procedure is carried out as follows.

a) Put the filter with the yttrium oxalate precipitate directly into a pre-weighed test dish (B.3.15). This is the source that is measured.

b) Leave the source to dry in an oven (B.3.3) until a constant mass is obtained, then leave it to cool in a desiccator (B.3.14).

c) After weighing, put the source in the desiccator (B.3.14) until it is measured.

#### B.4.5.2 Preparation of the source for measurement by LSC

This procedure is carried out as follows.

a) Dissolve the yttrium oxalate precipitate in a maximum volume of 35 ml of 2,5 molar nitric acid (B.2.5). Put the solution in a pre-weighed plastic flask (B.3.16) and record the mass.

b) Transfer 14 ml of this solution to a 20 ml pre-weighed polyethylene flask (B.3.16) and weigh.

c) Add 6 ml of scintillation solution (B.2.12), weigh and stir until dissolution. This is the source that is measured.

d) Maintain at (4 ± 3) °C until it is measured.

### B.4.6 Determination of the chemical yields

The chemical yield is determined as follows.

a) The chemical yield of the yttrium, $R_{\mathrm{c,Y}}$, is calculated from the ratio of the mass of the collected oxalate precipitate to the mass of the equivalent yttrium oxalate added as a carrier in the middle of the procedure, as given by Equation (B.1):

$$R_{\mathrm{c,Y}} = \frac{m_{\mathrm{o,p}}}{m_{\mathrm{o,Y}}} \tag{B.1}$$

where

$m_{\mathrm{o,p}}$ is the mass of the oxalate precipitate collected;

$m_{\mathrm{o,Y}}$ is the mass of the oxalate calculated from the quantity of yttrium carrier added.

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

1) The chemical yield is calculated from the ratio of the mass of the collected carbonate precipitate to the mass of the equivalent strontium carbonate added as a carrier at the start of the procedure, as given by Equation (B.2):

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

where

$m_{\mathrm{c,p}}$ is the mass of the carbonate precipitate collected;

$m_{\mathrm{c,Sr}}$ is the mass of the carbonate calculated from the quantity of strontium carrier added.

2) The chemical yield is calculated from the ratio of the activity of the collected carbonate precipitate, measured by gamma spectrometry, to the theoretical activity of the equivalent $^{85}$Sr added as a tracer at the start of the procedure, as given by Equation (B.3):

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

where

$A_{^{85}\mathrm{Sr,M}}$ is the activity of $^{85}$Sr measured by gamma spectrometry;

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

# Annex C
(informative)

# Measurement of strontium 90 from its daughter product yttrium 90 at equilibrium by organic extraction

## C.1 Principle

Yttrium is extracted from the sample solution using an organic solvent, HDEHP, at a pH of 1,4. After washing the organic phase in 1 molar HCl and re-extracting the yttrium from the same phase using 9 molar HCl, the solution is again purified using the TOM solution. Finally, the yttrium is precipitated as an oxalate and calcined in an oven at 900 °C, before being measured by β-counting using a proportional counter.

In the absence of other interfering β-emitters, as assessed during the decay of the $^{90}$Y by monitoring the count rate decrease, and once decay is complete, the residual activity level is compared with the background activity.

The tracer used is stable yttrium in the form of metal powder. It is added to the sample during the digestion phase at a ratio of between 5 mg and 10 mg per gram of test sample.

The mass of the test portion shall take into account the presumed activity of the sample and the desired detection limit. The procedure described below applies to solid samples weighing 5 g to 10 g.

## C.2 Chemical reagents

**C.2.1** **Water**, demineralized.

**C.2.2** **Hydrochloric acid**, diluted, $c(\text{HCl}) = 9$ mol/l.

**C.2.3** **Hydrochloric acid**, diluted, $c(\text{HCl}) = 1$ mol/l.

**C.2.4** **Heptane**.

**C.2.5** **HDEHP solution** [di-(2-ethylhexyl)phosphoric acid]: 145 ml of HDEHP for 855 ml of heptane.

Ensure the reagent is pure by washing it in an equal volume of water whose pH after this operation shall be greater than 3.

**C.2.6** **Toluene**.

**C.2.7** **TOM solution** (trioctyl methyl ammonium chloride): 333 ml of TOM for 666 ml of toluene.

**C.2.8** **Ammonia**, concentrated, $w(\text{NH}_4\text{OH}) = 28$ % minimum.

**C.2.9** **Oxalic acid**, saturated solution ($\text{H}_2\text{C}_2\text{O}_4{,}2\text{H}_2\text{O} = 140$ g/l).

**C.2.10** **Yttrium**, metallic, powder form.

**C.2.11** **Nitric acid**, diluted, $c(\text{HNO}_3) = 2$ mol/l.

**C.2.12** **Nitric acid**, diluted, $c(\text{HNO}_3) = 2{,}5$ mol/l.

## C.3 Equipment

**C.3.1 Balance**, analytical, accurate to 0,1 mg.

**C.3.2 Muffle furnace**, with programmable temperature control.

**C.3.3 Hot plate**, with temperature controls.

**C.3.4 Centrifuge**, refrigerated.

**C.3.5 pH meter**.

**C.3.6 Filtration apparatus**, for ashless filter paper.

**C.3.7 Gas-flow proportional counter** or **liquid scintillation counter**.

**C.3.8 Membrane filter** or **ashless filter paper**, with an average porosity of 0,45 µm.

**C.3.9 Sepatory funne**l, 250 ml.

**C.3.10 Test dish**, stainless steel, with edges, having a diameter compatible with the geometry of the counter or polyethylene vials.

**C.3.11 Centrifuge tubes**, 250 ml.

**C.3.12 Crucible**, silica.

## C.4 Procedure

The sample is dissolved according to the method described in Annex A. The tracer [5 mg to 10 mg of metal yttrium (C.2.10) per gram of test material] is added to the sample during this digestion phase. In principle, this leads to a nitric solution for which the organic separation procedure described below applies.

### C.4.1 Chemical separation of yttrium

The chemical separation phase of yttrium consists of the following stages.

a) Evaporate the nitric solution obtained following the dissolution of the sample until it is almost dry.

b) Add 100 ml of 1 molar HCl (C.2.3). Adjust the pH to 1,4 with the diluted ammonia.

c) Pour the sample into a 250 ml sepatory funnel (C.3.9) and add 50 ml of HDEHP solution (C.2.5.).

d) Stir vigorously for several minutes, record the date and time of separation as $t = 0$ for the time from the separation of the yttrium present in the test portion and leave it to settle for 30 min. Remove the aqueous phase.

e) Wash the organic phase five times in 20 ml of 1 molar HCl (C.2.3). Stir for 1 min, leave to settle for 2 min, each time discarding the aqueous phase.

f) Extract the $^{90}$Y from the organic phase five times using 10 ml of 9 molar HCl (C.2.2). Stir for 1 min and leave to settle for 2 min, each time recovering the aqueous phases.

g) Transfer the aqueous phases to another 250 ml sepatory funnel.

h) Add 50 ml of TOM solution (C.2.7), stir vigorously for several minutes and leave it to settle for 15 min.