### INTERNATIONAL STANDARD

## ISO 18589-5

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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 —

iTeh STPartie 5: Mesurage du strontium 90 W

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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.

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 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: (standards.iteh.ai)

- Part 1: General guidelines and definitions ISO 18589-5:2009
- 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.

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### 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 <sup>90</sup>Sr in equilibrium with <sup>90</sup>Y, and <sup>89</sup>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 <sup>89</sup>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 <sup>90</sup>Sr in the presence of <sup>89</sup>Sr.

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#### 2 Normative references 680

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.

т	Mass of the test portion, expressed in kilograms
a <sub>i</sub>	Activity per unit of mass, of radionuclide <i>i</i> , expressed in becquerel per kilogram
$A_{i,\mathbf{S}}$	Calibration source activity of radionuclide <i>i</i> , at the calibration time, expressed in becquerel
$A_i$	Sample source activity of radionuclide $i$ , at time $t = 0$ , expressed in becquerel
t <sub>g</sub>	Sample counting time, expressed in seconds
t <sub>0</sub>	Background counting time, expressed in seconds,
t <sub>s</sub>	Calibration source counting time
r <sub>g</sub>	Gross count rate per second
r <sub>0</sub>	Background count rate per second
r <sub>s</sub>	Calibration source count rate per second
R <sub>c,i</sub>	Chemical yield of the extraction of radionuclide iD PREVIEW
$\varepsilon_{i}$	Counting efficiency of radionuclide ndards.iteh.ai)
$\lambda_i$	Decay constant of radionuclide $i$ ISO 18589-5:2009
<i>t</i> d, <i>t</i> f	https://standards.iteh.ai/catalog/standards/sist/26dc82d1-299d-4c13-8619- Start and finish time respectively of the measurement referred to $t = 0$ , expressed in seconds
r <sub>gj</sub>	Gross count rate per second, for measurement <i>j</i>
r <sub>0j</sub>	Background count rate per second, for measurement <i>j</i>
$r_j$	Net count rate per second, for measurement <i>j</i>
$t_j$	Start time of measurement <i>j</i> , referred to $t = 0$
<i>u</i> ( <i>a<sub>i</sub></i> )	Standard uncertainty associated with the measurement result, expressed in becquerel per kilogram
U	Expanded uncertainty, calculated by $U = k \cdot u(a_i)$ with $k = 1, 2,,$ expressed in becquerel per kilogram
$a_i^*$	Decision threshold of radionuclide <i>i</i> , expressed in becquerel per kilogram
$a_i^{\#}$	Detection limit of radionuclide <i>i</i> , expressed in becquerel per kilogram
$a_i^{\triangleleft}, a_i^{\triangleright}$	Lower and upper limits respectively of the confidence interval of radionuclide <i>i</i> , expressed in becquerel per kilogram

#### 4 Principle

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

	<sup>90</sup> Sr	<sup>90</sup> Y	<sup>89</sup> Sr
Beta energy, keV	196	926,7	584,6
Half-life	28,8 a	2,67 d	50,6 d

Table 1 — Average beta-emission energies and half-lives of <sup>90</sup>Sr, <sup>90</sup>Y and <sup>89</sup>Sr

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, fead, and bismuth radioisotopes are present in soil, it is necessary that they be removed from the sample because they can interfere with <sup>90</sup>Sr or <sup>90</sup>Y or <sup>89</sup>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 18589-5:2009

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The radiochemical separation yield is calculated using a carrier such as stable Sr or Y, or a radioactive tracer such as <sup>85</sup>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 <sup>85</sup>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 <sup>90</sup>Y or <sup>89</sup>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.

<sup>1)</sup> Source: BIPM (Bureau International des Poids et Mesures).

Origin		Old fallout				Fresh fallout			
Radionuclide contents		<sup>90</sup> Sr+ <sup>90</sup> Y				<sup>90</sup> Sr+ <sup>90</sup> Y <sup>89</sup> Sr			
	Element	Sr		► Y <sup>a</sup>		Sr			
Separation	Method	Chromatography <sup>b</sup>	Precipitation	Extraction	Precipitation	Chromatography <sup>b</sup>	Precipitation		
	Product	<sup>90</sup> Sr		<sup>90</sup> Y		<sup>90</sup> Sr+ <sup>89</sup> Sr			
Sel	Carrier or tracer <sup>c</sup>	<sup>85</sup> Sr or stat	<sup>85</sup> Sr or stable Sr		able Y	<sup>85</sup> Sr or stable Sr			
Measurement(s)	Equilibrium <sup>90</sup> Sr+ <sup>90</sup> Y	Yes —— 15 to 20 days (recommended)	No │ No	No		Yes 15 to 20 days (recommended)	No		
	Number	▼ One	•	One		Two or more			
	Emissions	<sup>90</sup> Sr <sup>90</sup> Y		<sup>90</sup> Sr <sup>90</sup> Y <sup>90</sup> Y <sup>89</sup> Sr					
	Equipment	PC or LSC (total)			or LSC Cerenkov)	PC or LSC (total)			
	Calibration of sources	iTeh <sup>90</sup> Sr+ <sup>90</sup> Y	STANI <sup>90</sup> Sr (Stoynd	DARD ards.it	PREV.	<sup>90</sup> Sr+ <sup>90</sup> Y <sup>89</sup> Sr	<sup>90</sup> Sr <sup>90</sup> Y <sup>89</sup> Sr		
<ul> <li>Y separation is performed following the <sup>90</sup>Sr – <sup>90</sup>Y equilibrium in the test sample. https://standards.iteh.ai/catalog/standards/sist/26dc82d1-299d-4c13-8619-</li> <li>Specific chromatography using crown-ether resin. 680086f6788d/iso-18589-5-2009</li> </ul>									
<sup>c</sup> Carrier or tracer measurements are done using gamma spectrometry for <sup>85</sup> Sr and gravimetry, atomic absorption (AAS) or mass spectrometry (MS) for Sr and Y.									

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

#### 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<sup>[6], [7], [8]</sup>. 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<sub>3</sub> precipitate and are presented in Annex B.

For the measurement procedure with <sup>90</sup>Sr and <sup>90</sup>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 <sup>89</sup>Sr, <sup>90</sup>Sr is measured by counting the beta emission of <sup>90</sup>Y or of <sup>90</sup>Y and <sup>90</sup>Sr in equilibrium.

When the presence of <sup>89</sup>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

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This technique is based on the extraction using an organic solvent of <sup>90</sup>Y at equilibrium with its radioactive parent <sup>90</sup>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.

<sup>90</sup>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 <sup>90</sup>Y should be measured by PC or by measuring the Cerenkov radiation from the <sup>90</sup>Y, by LSC.

The absence of other interfering  $\beta$ -emitters is verified during the decay of <sup>90</sup>Y by measuring the decrease in count rate of the <sup>90</sup>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 <sup>90</sup>Sr and <sup>90</sup>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 <sup>89</sup>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  $\beta$ -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,  ${}^{90}$ 

The blank source should be prepared following the method chosen starting with a test portion without <sup>90</sup>Sr (or directly with distilled water).

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#### 6.3.2 Source preparation for proportional counter

#### <u>ISO 18589-5:2009</u>

The proportional counter measures directly the  $\beta$ -emission from the source prepared from a thin-layer deposit to minimize the self-absorption effects. 680086f6788d/iso-18589-5-2009

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 (<sup>90</sup>Sr, <sup>89</sup>Sr, <sup>90</sup>Sr+<sup>90</sup>Y or <sup>90</sup>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 <sup>90</sup>Sr activity by its measurement with <sup>90</sup>Y in equilibrium, two cases arise.