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Measurement of radioactivity in the environment — Soil —

Part 5: Strontium 90 — Test method using proportional counting or liquid scintillation counting

Mesurage de la radioactivité dans l'environnement — Sol —

Partie 5: Strontium 90 — Méthode d'essai par comptage proportionnel et scintillation liquide

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 2, *Radiation protection*.

This second edition cancels and replaces the first edition (ISO 18589-5:2009), which has been technically revised.

The main change compared to the previous edition are as follows:

— The introduction has been reviewed accordingly to the generic introduction adopted for the standards published on the radioactivity measurement in the environment.

A list of all parts in the ISO 18589 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Everyone is exposed to natural radiation. The natural sources of radiation are cosmic rays and naturally occurring radioactive substances which exist in the earth and flora and fauna, including the human body. Human activities involving the use of radiation and radioactive substances add to the radiation exposure from this natural exposure. Some of those activities, such as the mining and use of ores containing naturally-occurring radioactive materials (NORM) and the production of energy by burning coal that contains such substances, simply enhance the exposure from natural radiation sources. Nuclear power plants and other nuclear installations use radioactive materials and produce radioactive effluent and waste during operation and decommissioning. The use of radioactive materials in industry, agriculture and research is expanding around the globe.

All these human activities give rise to radiation exposures that are only a small fraction of the global average level of natural exposure. The medical use of radiation is the largest and a growing man-made source of radiation exposure in developed countries. It includes diagnostic radiology, radiotherapy, nuclear medicine and interventional radiology.

Radiation exposure also occurs as a result of occupational activities. It is incurred by workers in industry, medicine and research using radiation or radioactive substances, as well as by passengers and crew during air travel. The average level of occupational exposures is generally below the global average level of natural radiation exposure (see Reference [1]).

As uses of radiation increase, so do the potential health risk and the public's concerns. Thus, all these exposures are regularly assessed in order to:

— improve the understanding of global levels and temporal trends of public and worker exposure;

— evaluate the components of exposure so as to provide a measure of their relative importance;

— identify emerging issues that may warrant more attention and study. While doses to workers are mostly directly measured, doses to the public are usually assessed by indirect methods using the results of radioactivity measurements of waste, effluent and/or environmental samples.

To ensure that the data obtained from radioactivity monitoring programs support their intended use, it is essential that the stakeholders (for example nuclear site operators, regulatory and local authorities) agree on appropriate methods and procedures for obtaining representative samples and for handling, storing, preparing and measuring the test samples. An assessment of the overall measurement uncertainty also needs to be carried out systematically. As reliable, comparable and 'fit for purpose' data are an essential requirement for any public health decision based on radioactivity measurements, international standards of tested and validated radionuclide test methods are an important tool for the production of such measurement results. The application of standards serves also to guarantee comparability of the test results over time and between different testing laboratories. Laboratories apply them to demonstrate their technical competences and to complete proficiency tests successfully during interlaboratory comparisons, two prerequisites for obtaining national accreditation.

Today, over a hundred International Standards are available to testing laboratories for measuring radionuclides in different matrices.

Generic standards help testing laboratories to manage the measurement process by setting out the general requirements and methods to calibrate equipment and validate techniques. These standards underpin specific standards which describe the test methods to be performed by staff, for example, for different types of sample. The specific standards cover test methods for:

— naturally-occurring radionuclides (including ^{40}K, ^{3}H, ^{14}C and those originating from the thorium and uranium decay series, in particular ^{226}Ra, ^{228}Ra, ^{234}U, ^{238}U and ^{210}Pb) which can be found in materials from natural sources or can be released from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or phosphate fertilizer production and use);

— human-made radionuclides, such as transuranium elements (americium, plutonium, neptunium, and curium), ^{3}H, ^{14}C, ^{90}Sr and gamma-ray emitting radionuclides found in waste, liquid and gaseous effluent, in environmental matrices (water, air, soil and biota), in food and in animal feed as a result of authorized releases into the environment, fallout from the explosion in the atmosphere of nuclear devices and fallout from accidents, such as those that occurred in Chernobyl and Fukushima.

The fraction of the background dose rate to man from environmental radiation, mainly gamma radiation, is very variable and depends on factors such as the radioactivity of the local rock and soil, the nature of building materials and the construction of buildings in which people live and work.

A reliable determination of the activity concentration of gamma-ray emitting radionuclides in various matrices is necessary to assess the potential human exposure, to verify compliance with radiation protection and environmental protection regulations or to provide guidance on reducing health risks. Gamma-ray emitting radionuclides are also used as tracers in biology, medicine, physics, chemistry, and engineering. Accurate measurement of the activities of the radionuclides is also needed for homeland security and in connection with the Non-Proliferation Treaty (NPT).

This document describes the requirements to quantify the activity of ^{90}Sr in soil samples after proper sampling, sample handling and test sample preparation in a testing laboratory or in situ.

This document is to be used in the context of a quality assurance management system (ISO/IEC 17025).

This document is published in several parts for use jointly or separately according to needs. These parts are complementary and are addressed to those responsible for determining the radioactivity present in soil, bedrocks and ore (NORM or TENORM). The first two parts are general in nature describe the setting up of programmes and sampling techniques, methods of general processing of samples in the laboratory (ISO 18589-1), the sampling strategy and the soil sampling technique, soil sample handling and preparation (ISO 18589-2). ISO 18589-3 to ISO 18589-5 deal with nuclide-specific test methods to quantify the activity concentration of gamma emitters radionuclides (ISO 18589-3 and ISO 20042), plutonium isotopes (ISO 18589-4) and ^{90}Sr (ISO 18589-5) of soil samples. ISO 18589-6 deals with non-specific measurements to quantify rapidly gross alpha or gross beta activities and ISO 18589-7 describes in situ measurement of gamma-emitting radionuclides.

The test methods described in ISO 18589-3 to ISO 18589-6 can also be used to measure the radionuclides in sludge, sediment, construction material and products following proper sampling procedure.

This document is one of a set of International Standards on measurement of radioactivity in the environment.

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: Strontium 90 — Test method using proportional counting or liquid scintillation counting

1 Scope

This document 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 counters (PC) or liquid scintillation counters (LSC). ^{90}Sr can be obtained from the test samples when the equilibrium between ^{90}Sr and ^{90}Y is reached or through direct ^{90}Y measurement. The selection of the measuring method depends on the origin of the contamination, the characteristics of the soil to be analysed, the required accuracy of measurement and the resources of the available laboratories.

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

In case of recent fallout immediately following a nuclear accident, the contribution of ^{89}Sr to the total amount of strontium activity will not be negligible. This standard provides the measurement method to determine the activity of ^{90}Sr in presence of ^{89}Sr.

The test methods described in this document can also be used to measure the radionuclides in sludge, sediment, construction material and products by following proper sampling procedure.

Using samples sizes of 20 g and counting times of 1 000 min, detection limits of (0,1 to 0,5) $Bq \cdot kg^{-1}$ can be achievable for ^{90}Sr using conventional and commercially available proportional counter or liquid scintillation counter when the presence of ^{89}Sr can be neglected. If ^{89}Sr is present in the test sample, detection limits of (1 to 2) $Bq \cdot kg^{-1}$ can be obtained for both ^{90}Sr and ^{89}Sr using the same sample size, counting time and proportional counter or liquid scintillation counter as in the previous situation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 11074, *Soil quality — Vocabulary*

ISO 11929 (all parts), *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application*

ISO 19361, *Measurement of radioactivity — Determination of beta emitters activities — Test method using liquid scintillation counting*

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

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*

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

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11074, ISO 18589-1, ISO 11929 (all parts) and ISO 80000-10 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.2 Symbols

m	mass of the test portion, in unit of mass
a_i	activity per unit of mass, of radionuclide i, in becquerel per unit of mass
$A_{s,i}$	reference measurement standard activity of radionuclide i, at the calibration time, in becquerel
A_i	sample source activity of radionuclide i, at time $t = 0$, in becquerel
t_g	sample counting time, in seconds
t_0	background counting time, in seconds
t_s	reference measurement standard counting time
r_g	gross count rate, in per second
r_0	background count rate, in per second
r_s	reference measurement standard count rate, in per second
$R_{c,i}$	chemical yield of the extraction of radionuclide i
ε_i	counting efficiency of radionuclide i
λ_i	decay constant of radionuclide i
t_d, t_f	start and stop time respectively of the measurement, referred to $t = 0$, in seconds
r_{gj}	gross count rate, for measurement j, in per second
r_{0j}	background count rate, for measurement j, in per second
r_j	net count rate, for measurement j, in per second
t_j	start time of the measurement j, referred to $t = 0$
$u(X)$	standard uncertainty associated with X
U	expanded uncertainty, calculated by $U = k \cdot u(X)$ with $k = 1, 2, \ldots$

a_i^* decision threshold of radionuclide *i*, in becquerel per unit of mass

$a_i^\#$ detection limit of radionuclide *i*, in becquerel per unit of mass

$a_i^\triangleleft$, $a_i^\triangleright$ lower and upper limits of the confidence interval, of radionuclide *i*, in becquerel per unit of mass

4 Principle

4.1 General

From a detection perspective, it can be consider that ^{90}Sr, ^{90}Y and ^{89}Sr are pure beta emitter radionuclides. Their beta emission energies and half-lives are given in Table 1[2][3].

Table 1 — Beta emission average 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 y	2,67 d	50,6 d

^{89}Sr and ^{90}Sr can be directly measured. ^{90}Sr can be estimated through the measurement of its daughter product ^{90}Y. All the measurements are based on a chemical separation step followed by beta counting of the element using a PC or using a LSC (See Table 2).

In a previous step, strontium will be desorbed from the soil test portion by acid treatment and will be in solution in the leachate fraction. The tracer or carrier is added at the start of this step of the procedure, and time allowed, usually up to one day, to obtain equilibrium before starting the strontium desorption.

A strontium desorption method for soil samples is specified in Clause 6.

4.2 Chemical separation

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

The separation step should maximise the extraction of the pure element. The method chosen shall be selective with a high radiochemical separation yield. As thorium, lead, and bismuth radioisotopes are present in soil at high activity levels they shall be removed from the sample because they may interfere with ^{90}Sr or ^{90}Y or ^{89}Sr emissions during the detection step. Other matrix constituents which can interfere, such as alkaline earth elements, calcium for strontium, or transuranic and lanthanide elements for yttrium, shall 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 like atomic absorption spectroscopy (AAS) or atomic emission spectroscopy (ICP-AES) or 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, its original concentration shall be known in the test sample before the addition of the carrier to avoid the overestimation of the radiochemical separation yield.

There are three usual techniques for the chemical separation: precipitation liquid-liquid extraction and chromatography extraction using selective crown-ether resin[4][5][6]. Clauses 7, 8 and 9 give a procedure for each of these techniques.

4.3 Detection

4.3.1 General

The use of a LSC is recommended, depending on the compatibility between the LSC solvent and the sample, because it can provide energy spectra and discriminate interference from unwanted radionuclides. LSC can also be used, with water as the solvent, to count Cerenkov photons. However, LSC is subject to optical and chemical quenching. A PC, on the other hand, does not distinguish between emissions from different beta emitters, but can exclude alpha contamination and is not subject to the Cerenkov effect or sample quenching. When a 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.

4.3.2 Source preparation for liquid scintillation counter

The test portion is mixed with the scintillation cocktail in a counting vial to obtain a homogeneous medium (scintillation source). Beta particles emitted from the test portion transfer their energy to the scintillation cocktail molecules, causing them to become excited. Upon returning to the ground state, photons are emitted, which can then be detected by photoelectron multiplier tubes (phototubes). To properly carry out measurements by using a LSC, recommendations contained in ISO 19361 shall be taken into account.

The Sr or Y precipitate is dissolved and mixed with the scintillation cocktail. The solution volume depends on the equipment (vial size) and the specific scintillation cocktail used.

The reference measurement standard 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. Methods that allow the calibration with different radionuclide are also available.

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

4.3.3 Source preparation for proportional counter

The proportional counter measures directly the beta 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 on a stainless steel dish by direct evaporation.

The filter or dish size diameter should be determined by the counter requirements, i.e. the detector diameter and source holder dimensions.

The reference measurement standard 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.

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

4.3.4 Background determination

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

Table 2 — Determination procedures for strontium depending on its origin

Origin.		Old Fallout				Fresh Fallout	
Radionucl. Contents		$^{90}Sr+^{90}Y$				$^{90}Sr+^{90}Y$ ^{89}Sr	
Separation	Element	Sr		Y [a]		Sr	
	Method	Chromatography [b]	Precipitation	Extraction	Precipitation	Chromatography [c]	Precipitation
	Product	^{90}Sr		^{90}Y		$^{90}Sr+^{89}Sr$	
	Carrier or Tracer [d]	^{85}Sr or Stable Sr		Stable Y		^{85}Sr or Stable Sr	
Measurement(s)	Equilibrium $^{90}Sr+^{90}Y$	Yes 15-20 days (recommended)	No	No		Yes 15-20 days (recommended)	No
	Number	One		One		Two or more	
	Emissions	^{90}Sr ^{90}Y		^{90}Y		^{90}Sr ^{90}Y ^{89}Sr	
	Equipment	PC or LSC (total)		PC or LSC (total or Cerenkov)		PC or LSC (total)	
	Cal. 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 will be performed following the ^{90}Sr –^{90}Y equilibrium in the test sample.

[b] Specific chromatography using crown ether resin.

[c] Specific chromatography using crown ether resin.

[d] A 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 Clauses 7, 8 and 9.

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

6 Procedure of strontium desorption

6.1 Principles

The Sr contents in the test portion of soil can be extracted by an acid leach cycle (nitric acid, hydrochloric acid, acid mixture).

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

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

6.2 Technical resources

6.2.1 Equipment

The equipment to be used is as follows:

a) analytical balance of an accuracy of 0,1 mg;

b) hot plate;

c) centrifuge and tubes;

d) furnace.

6.2.2 Chemical reagents

The reagents required are as follows:

a) Sr^{2+} carrier, ^{85}Sr tracer or Y metal carrier;

b) nitric acid, solution, $c(HNO_3)$ = 8 mol/l;

c) hydrogen peroxide, $w(H_2O_2)$ = 30 %;

d) sodium nitrite ($NaNO_2$).

6.3 Procedure

The procedure described below is based on a cycle of acid leaching and dry evaporation:

a) weigh between 0,1 g and 50 g of the test sample prepared in compliance with ISO 18589-1 and ISO 18589-2. The mass of the test portion shall depend on the assumed activity of the sample, the desired detection limit and the method chosen. It is advisable to ash the sample (i.e. 500 °C for 2 h) to destroy organics before the HNO_3 leaching;

b) gradually add 10 ml per gram of sample of 8 mol/l HNO_3;

c) add the tracer or carrier and stir to homogenize;

d) digest on a hot plate for at least 8 h with sporadic additions of H_2O_2;

e) transfer the sample to a centrifuge tube and centrifuge it. The supernatant is stored;

It is possible once cooled to filter using a fibreglass filter instead of centrifuge.

f) rinse the beaker with 8 mol/l HNO_3, add to the same tube, stir and centrifuge. Pour the supernatant together with that stored in step e). Repeat the process until the supernatant is clear;

g) evaporate the supernatant to dryness;

h) add 50 ml of 8 mol/l HNO_3;

i) add 0,6 g of $NaNO_2$ and heat to remove the nitrous fumes and leave to cool.

Sample is ready to undergo a radiochemical separation.

The dissolution can also be carried out using a microwave mineralization apparatus. Operating conditions for each system should be adjusted to the type of apparatus used and the nature of the soil to be mineralised.

7 Chemical separation procedure by precipitation

7.1 Principles

The precipitation technique is suited to separate all mineral elements, including strontium, in soil 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. In this document, the most usual procedures lead to a $SrCO_3$ precipitate are presented. See References [4][5][6].

The Sr 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 $SrCO_3$, can be measured by proportional counting or by liquid scintillation counting after being dissolved. Another possibility is when the yttrium has reached equilibrium to separate it from the strontium by precipitation in the form of oxalate with a view to measurement by PC or LSC.

For the measurement procedure 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 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 an yttrium precipitate, usually under the form of an oxalate precipitate.

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, this technique giving direct measurement of strontium at two or more different times shall be chosen.

The mass of the test portion shall take into account the presumed activity of the sample and the desired detection limit. The procedure presented below is given for solid samples from 1 g to 50 g.

7.2 Technical resources

7.2.1 Equipment

The equipment to be used is as follows:

a) standard laboratory equipment;

b) analytical balance of an accuracy of 0,1 mg;

c) oven;

d) furnace;

e) hot plate with temperature control and magnetic stirring;

f) pH meter;

g) centrifuge and tubes;

h) atomic absorption spectrosmeter (AAS) or atomic emission spectroscope (ICP-AES or ICP-MS) or mass spectrometer (ICP-MS) or gamma spectrometer;

i) proportional or liquid scintillator counter;

j) filtration apparatus for fibreglass or cellulose filters with diameters 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);

k) cellulose and fibreglass filter;

l) Büchner funnel;

m) infrared lamps;

n) desiccator;

o) stainless steel test dish with a diameter compatible with the geometry of the counter or polyethylene vials;

p) plastic flasks.

7.2.2 Chemical reagents

The reagents to be used are as follows:

a) demineralised water;

b) ammonia concentrated, $w(NH_4OH) = 25\ \%$;

c) nitric acid, concentrated, $w(HNO_3) = 65\ \%$;

d) nitric acid, solution, $c(HNO_3) = 8$ mol/l;

e) nitric acid, solution, $c(HNO_3) = 2{,}5$ mol/l;

f) sodium or potassium chromate, solution, $c(Na_2CrO_4$ or $K_2CrO_4) = 1$ mol/l;

g) sodium carbonate (Na_2CO_3);

h) Fe^{3+} carrier solution (20 mg/ml), $w(FeCl_3) = 30\%$;

i) barium acetate solution ($Ba(C_2H_3O_2)_2$);

j) hydrochloric acid concentrated, $w(HCl) = 37\ \%$;

k) oxalic acid, saturated solution ($H_2C_2O_4 \cdot 2H_2O = 140$ g/l);

l) scintillation cocktail, compatible with nitric acid;

m) yttrium carrier solution (20 mg/ml in 0,1 mol/l HNO_3) (only in case of Y separation);

n) methanol or ethanol.

7.3 Procedure

7.3.1 Separation of alkaline metals and calcium

This phase consists of the following steps:

a) evaporate the nitric solution obtained after the test portion preparation until a volume between 50 ml to 100 ml is obtained or until the salts appear;

b) add 200 ml of concentrated HNO_3; evaporate until 100 ml or salt appears. Cover with a watch glass and leave to cool;

c) add 50 ml of concentrated HNO_3;

d) stir for 30 min;

e) filter the $Sr(NO_3)_2$ obtained using the filter pump and fibreglass filter;

f) leave the precipitate to dry. Clean with concentrated HNO_3. Throw away the filtrate;

g) dissolve the precipitate in hot demineralised water. Clean the filter to obtain 50 ml of solution.

7.3.2 Separation of barium, radium and lead

This phase consists of the following steps:

a) adjust the pH to 4 to 6 with concentrated ammonia;

b) add 4 ml of barium acetate and heat;

c) add 1 ml of 1 mol/l sodium or potassium chromate and stir;

d) leave to cool, filter using a filter pump and fibreglass filter or Büchner funnel;

e) clean the filter with demineralised water. Throw away the filter with the precipitate.

7.3.3 Separation of fission products and yttrium

This phase consists of the following steps:

a) transfer the filtrate to a 250 ml glass beaker, add 1 ml of Fe^{3+} carrier solution;

b) adjust the pH to 9 using ammonia;

c) Heat for 10 min, avoid boiling;

d) leave to cool and filter using a fibreglass filter;

e) clean the filter with 10 ml of demineralised water. Throw away the filter with the precipitate;

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

7.3.4 Strontium purification

7.3.4.1 General

This phase consists of the following steps:

a) adjust the pH to 8 using ammonia, add 20 ml of sodium carbonate in saturated solution. Stir for 30 min;

b) filter the $SrCO_3$ obtained using the filter pump and a fibreglass filter. Rinse the beaker and the precipitate with 0,1 mol/l sodium carbonate solution and after with demineralised water;

c) throw away the filtrate, leave the precipitate to dry with the vacuum pump from the filter system turning on for at least 10 min;

d) turn off the vacuum pump and add slowly through the precipitated 100 ml of concentrated HNO_3;

e) turn on the vacuum pump and leave the precipitate to dry;

f) dissolve the precipitate with water and transfer the solution into a glass beaker. The use of hot water is available;

g) adjust the pH to 8 to 9 using ammonia. Heat for a few minutes, avoid boiling;

h) add 10 ml of sodium carbonate in saturated solution. Stir for 30 min;

i) filter the $SrCO_3$ obtained using the filter pump and a pre-weighed fibreglass or cellulose filter. Rinse the beaker and the precipitate with 1 % ammonia dissolution and after with demineralised water or methanol.

7.3.4.2 Source preparation to be measured by PC

a) put the filter with the $SrCO_3$ precipitate directly into a pre-weighed test dish, this is the source to be measured;

b) leave the source to dry in an oven until a constant mass, leave to cool in a dryer system;

c) after weighing put the source in the drying system until measured.

7.3.4.3 Source preparation to be measured by LSC

a) dissolve the $SrCO_3$ precipitate in a maximum volume of 35 ml of 2,5 mol/l nitric acid. Put the solution in a pre-weighed flask and note the mass;

b) transfer 14 ml of this dissolution to a 20 ml pre-weighed polyethylene vial, note the mass;

c) add 6 ml of scintillation cocktail and stir until dissolved; this is the source to be measured;

d) keep at (15 ± 2) °C until measurement.

7.3.5 Yttrium extraction

7.3.5.1 General

a) dissolve the $SrCO_3$ precipitate in a maximum volume of 35 ml of 2,5 mol/l nitric acid. Put the solution in a pre-weighed plastic flask and note the mass;

b) add the yttrium carrier;

c) store for two weeks in order to reach more than 95 % equilibrium of ^{90}Y;

d) pour the solution at equilibrium into a centrifuge tube. Adjust the pH to 8 using ammonia. Heat in the water bath to 90 °C. Cold centrifuge to separate the precipitate;

e) collect the supernatant in a beaker. It contains the Sr carrier or tracer, which will be used to determine the extraction yield of the strontium;

f) note the date and time of the separation of the strontium;

g) dissolve the yttrium precipitate in 5 drops of hydrochloric acid and 30 ml of demineralised water;

h) add whilst stirring 5 ml of oxalic acid in saturated solution and adjust the pH to 2 to 2,5 using ammonia. 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. Rinse the centrifuge tube with demineralised water.

7.3.5.2 Source preparation to be measured by PC

a) put the filter with the yttrium oxalate precipitate directly into a pre-weighed test dish, this is the source to be measured;

b) leave the source to dry in an oven until constant mass, leave to cool in a dryer system;

c) after weighing put the source in the dryer system until measured.

7.3.5.3 Source preparation to be measured by LSC

a) dissolve the yttrium oxalate precipitate in a maximum volume of 35 ml of 2,5 mol/l nitric acid. Put the solution in a pre-weighed plastic flask and note the mass;

b) transfer 14 ml of this dissolution to a 20 ml pre-weighed polyethylene vial and note the mass;

c) add 6 ml of scintillation cocktail and stir until dissolution; this is the source to be measured;

d) keep to (15 ± 2) °C until measured.

7.3.6 Determination of the chemical yields

a) The chemical yield of the yttrium ($R_{c,Y}$) is calculated by the ratio of the mass of the collected oxalate precipitate over the mass of the equivalent yttrium oxalate added as a carrier in the middle of the procedure, see Formula (1):

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

where

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

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

b) The chemical yield of the strontium ($R_{c,Sr}$) is calculated from strontium carrier or tracer by one of the following procedures, see Formula (2) or Formula (3):

1) chemical yield calculated as the ratio of the mass of the collected carbonate precipitate over the mass of the equivalent strontium carbonate added as a carrier at the start of the procedure:

$$R_{c,Sr} = \frac{m_{c,p}}{m_{c,Sr}} \quad (2)$$

where

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

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

2) chemical yield calculated as the ratio of the activity of the collected carbonate precipitate, measured by γ spectrometry, over the known activity of the ^{85}Sr added as a tracer at the start of the procedure.

$$R_{c,Sr} = \frac{A_{Sr85,m}}{A_{Sr85,t}} \quad (3)$$

where

$A_{Sr85,m}$ is the activity of ^{85}Sr measured by gamma spectrometry;

$A_{Sr85,t}$ is the known activity of ^{85}Sr added at the start of the procedure.

8 Chemical separation procedure by liquid-liquid extraction

8.1 Principle

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 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 may 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 β emitting radionuclide contents. One of the most general methods is presented here. In it yttrium is extracted from the sample solution using an organic solvent of HDEHP (di-(2-ethylhexyl)phosphoric acid), with a pH of 1,4. After washing the organic phase in 1 mol/l HCl and re-extracting the yttrium from the same phase using 9 mol/l HCl, the solution is again purified using a TOM solution (trioctyl methyl ammonium chloride in toluene). Finally, the yttrium is precipitated in oxalate form, and calcinated in an oven at 900 °C, before being measured by β counting using a proportional counter or a liquid scintillation counter.

The absence of other interfering β 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.

The carrier used is inactive yttrium in metal powder form. It is added to the sample during the mineralisation 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 5 g to 10 g solid samples.

8.2 Technical resources

8.2.1 Equipment

The equipment to be used is as follows:

a) scales to an accuracy of 0,1 mg;

b) muffle furnace with programmable temperature control;

c) hot plate with temperature controls;

d) refrigerated centrifuge;

e) pH meter;

f) filtration apparatus for ashless filter paper;

g) gas flow proportional counter or liquid scintillation counter;

h) 0,45 µm membrane filter or ashless filter paper with an average porosity of 0,45 µm;

i) 250 ml separating funnel;

j) stainless steel test dish with edges with a diameter compatible with the geometry of the counter or polyethylene vials;

k) 250 ml centrifuge pot;

l) silica crucible.

8.2.2 Chemical reagents

The reagents used are as follows:

a) demineralised water;

b) hydrochloric acid, solution, c(HCl) = 9 mol/l;

c) hydrochloric acid, solution, c(HCl) = 1 mol/l;

d) heptane;

e) 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);

f) toluene;

g) TOM solution (trioctyl methyl ammonium chloride): 333 ml of TOM for 666 ml of toluene;

h) Ammonia, concentrated, $w(NH_4OH)$ = 28 % minimum (mass fraction);

i) oxalic acid, saturated solution ($H_2C_2O_4 \cdot 2H_2O$ = 140 g/l);

j) yttrium in metallic powder form.

8.3 Procedure

8.3.1 General

The sample is dissolved according to the method described in Clause 6. The tracer (5 mg to 10 mg of metal yttrium per gram of test material) is added to the sample during this mineralization phase. In principle, this leads to a nitric solution for which the organic separation procedure described below applies.

8.3.2 Chemical separation of yttrium

The chemical separation phase of yttrium consists of the following steps:

a) evaporate until almost dry the nitric solution obtained following the dissolution of the sample;

b) add 100 ml of 1 mol/l HCl. Adjust the pH to 1,4 with a diluted ammonia solution;

c) pour the sample into a 250 ml separating funnel and add 50 ml of HDEHP solution;

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

e) wash the organic phase five times in 20 ml of 1 mol/l HCl: stir for 1 min, leave to settle for 2 min and throw the aqueous phase away each time;

f) extract the ^{90}Y from the organic phase five times using 10 ml of 9 mol/l HCl: stir for 1 min and leave to settle for 2 min each time. Recover the aqueous phases;

g) transfer the aqueous phases into another 250 ml separating flask;

h) add 50 ml of TOM solution, stir vigorously for several minutes and leave to settle for 15 min;

i) collect the aqueous phase in a 250 ml beaker and add 50 ml of demineralised water. If this phase is cloudy, begin the purification again with 50 ml of TOM solution;

j) add 60 ml to 80 ml of concentrated ammonia solution and boil for 2 min to achieve flocculation of the yttrium hydroxide. Leave to settle and cool for approximately 20 min;

k) transfer into the centrifuge pots and centrifuge for 10 min at 3 000 $r \cdot min^{-1}$;

l) remove the supernatant and recover the residue by adding 5 ml of 2 mol/l HNO_3;

m) transfer into a 100 ml beaker and add 10 ml of saturated oxalic acid solution. Make up to a 50 ml solution with water;

n) bring to boiling point and stop as soon as bubbles appear. Leave to settle for approximately 30 min;

o) filter using a 0,45 µm membrane to collect the yttrium oxalate precipitate.

8.3.3 Source preparation to be measured by PC

a) transfer the filter into a silica crucible, cover and place in the oven at 900 °C for 5 min. Leave to cool;

b) recover the calcination residue (yttrium oxide) by adding 5 ml of demineralised water and spread out using a pipette point on a pre-weighed stainless steel test-dish;

c) weigh the test dish containing the yttrium oxide deposit. Note the mass of the Y_2O_3.

8.3.4 Source preparation to be measured by LSC

a) dissolve the yttrium oxalate precipitate in a maximum volume of 35 ml of 2,5 mol/l nitric acid. Put the solution in a pre-weighed plastic flask and note the mass;

b) transfer 14 ml of this dissolution to a 20 ml pre-weighed polyethylene vial and note the mass;

c) add 6 ml of scintillation cocktail and stir until homogenous dissolution; this is the source to be measured;

d) keep at (15 ± 2) °C until measured.

8.3.5 Determination of the chemical yields

The chemical yield is calculated from:

a) For PC measurement the chemical yield of the yttrium ($R_{c,Y}$) is calculated by the ratio of the mass of the measured yttrium oxide precipitate over the mass of the metallic yttrium added as carrier, corrected with a factor of 1,27 as given in Formula (4).

$$R_{c,Y} = \frac{m_{ox,p}}{m_{m,Y} \cdot 1,27} \tag{4}$$

where

$m_{ox,p}$ is the mass of the oxide precipitate collected;

$m_{m,Y}$ is the mass of the metallic yttrium carrier added.

1,27 is the ratio of molecular weight used to normalise the metallic yttrium mass to the yttrium oxide mass.

b) For LSC measurements the chemical yield of the yttrium ($R_{c,Y}$) is calculated by the ratio of the mass of the collected oxalate precipitate over the mass of the equivalent yttrium oxalate added as a carrier see Formula (5):

$$R_{c,Y} = \frac{m_{o,p}}{m_{o,Y}} \tag{5}$$

where

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

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

9 Chemical separation procedure by chromatography (crown ether resin)

9.1 Principles

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. See References [7][8][9].

The alkaline earth elements (strontium, calcium, barium) are precipitated in the form of phosphates in a basic medium. The barium, if present in large quantities, is removed in nitrate form. The strontium is selectively set on a specific "crown ether" column in an HNO_3 medium (between 3 mol/l and 8 mol/l) then eluted using a 0,05 mol/l HNO_3 solution. The final source to be measured is a strontium precipitate. The fixation yield is determined from the inactive strontium added initially as a carrier and the strontium measured subsequently by atomic absorption.

For the measurement procedure 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 can be measured after the final separation from strontium. In this latter case, the chemical recovery 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 precipitate.

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, this technique giving direct measurement of strontium at two or more different times shall be chosen.

9.2 Technical resources

9.2.1 Equipment

The equipment to be used is as follows:

a) scales to an accuracy of 0,1 mg;

b) centrifuge;

c) ventilated oven or hot plate with temperature control;

d) atomic absorption spectrophotometer, ICP/AES or ICP/MS;

e) pH meter;

f) evaporation system;

g) β counting apparatus equipped with a gas flow proportional counter or a liquid scintillation counter;

h) stainless steel test dish if proportional counting is chosen; if not, a flask adapted to the liquid scintillation counter is used;

i) 50 ml centrifuge pot.

9.2.2 Chemical reagents

The products used are as follows:

a) a support, coated with a crown ether specific to the extraction of strontium;

b) inactive strontium salt;

c) water, complying with grade 3 as defined in ISO 3696, or better;

d) reference strontium solution for spectrophotometric determination;

e) phosphoric acid, solution, $c(H_3PO_4)$ = 9 mol/l;

f) 0,5 mol/l aluminium nitrate ($Al(NO_3)_3$)solution in a nitric medium between 3 mol/l and 8 mol/l;

g) nitric acid, solution, $c(HNO_3)$ between 3 mol/l and 8 mol/l;

h) nitric acid, solution, $c(HNO_3)$ = 0,05 mol/l;

i) concentrated ammonia (NH_4OH): 28 % minimum;

j) scintillating cocktail if counting by liquid scintillation is chosen, compatible with nitric acid.

9.3 Procedure

9.3.1 General

The sample is dissolved. The carrier (10 mg of inactive strontium) is added to the sample during the mineralization phase. After dissolution, the nitric solution obtained is made up to 100 ml with distilled water.

9.3.2 Chemical separation of the strontium

The steps of the chemical isolation procedure of ^{90}Sr are as follows:

a) add 5 ml of 9 mol/l H_3PO_4;

b) create a basic medium by adding 28 % NH_4OH until a pH of between 9,5 and 10 is achieved;

c) collect the precipitate by centrifuging (several times if necessary);

d) heat the precipitate in an oven (70 °C) or on a hot plate in the same container until almost dry;

e) dissolve the salts in at least 10 ml of a solution containing 0,5 mol/l $Al(NO_3)_3$ and between 3 mol/l and 8 mol/l HNO_3 (more if necessary. Dissolution shall be complete);

f) place a column containing 2,8 g of a support coated with crown ether on a holder;

g) let the water in the column drip into a beaker;

h) prepare the resin by adding 20 ml of 3 mol/l to 8 mol/l HNO_3;

i) filter the sample through the resin column in 2,5 ml fractions;

j) rinse the container which contained the solution with 3 ml of between 3 mol/l and 8 mol/l HNO_3. Pour the solution through the column;

k) rinse the column at least three times with 3 ml of between 3 mol/(l and 8 mol/l HNO_3;

l) note the date and time after rinsing;

m) dry the drops on the sides of the column container;

n) place a small container under the column and elute the strontium with at least 10 ml (V_{eluted}) of 0,05 mol/l HNO_3 solution;

o) take an aliquot fraction of the solution to determine the quantity of strontium by atomic absorption or ICP/AES or ICP/MS in order to evaluate the chemical yield;

p) use an aspirator to evaporate the volume remaining on a stainless test dish if measuring activity by proportional counting or prepare the source for measurement by liquid scintillation counting.

9.3.3 Determination of chemical yield

The chemical yield of the strontium is determined by the following Formula (6):

$$R_{c,Sr} = \frac{m_{Sr}}{m_{Sr,T}} \tag{6}$$

where

m_{Sr} is the mass of the strontium in the eluate measured by AA, ICP/AES or ICP/MS;

$m_{Sr,T}$ is the mass of strontium added initially.

10 Measurement

10.1 General

The same equipment conditions should be used for measurements of the sample, the background and the reference measurement standard.

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

10.2 Liquid scintillation counter

LSC measures directly the photons, produced following the excitement of the scintillation cocktail by the beta particles. The advantage of this method is the possibility of discriminating beta particles energies by setting so called energy "windows" that allows one to check the presence of other beta contaminants in the source. When this type of equipment is chosen for radiostrontium determinations, recommendations contained in ISO 19361 should be taken into account.

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

— where 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;

— or, 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.

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.

10.3 Gas flow proportional counter

A gas flow proportional counter is a system that measures directly the alpha and beta radiations, without energy discrimination, from the source usually prepared as a thin layer deposit.

When a strontium test source is measured, the use of double window (α and β) in this type of counter allows the presence of alpha emitting contaminants in the source. If other short half-life beta emitters are present they can be detected by performing successive measurements of the source at given times.

Other systems, incorporating a plastic scintillation detector, Geiger-Müller detector or a silicon-charged particle detector [passivated implanted planar silicon (PIPS)], can also be used.

When using a gas-flow proportional counter, it is advisable to choose the beta window with minimal beta-alpha cross-talk. If some alpha contamination is present, data should be corrected for the alpha-beta cross-talk. If equipment other than gas-flow proportional counters is used, then cross-talk may be insignificant and ignored.

If a windowless gas-flow proportional counter is used, carry out regular checks for possible contamination of the counting system by counting blank samples.

10.4 Calculation of counting efficiency

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 reference measurement standard;

— calculate the counting efficiency of the counter by dividing the count rate measured with the activity of the calibration source, at measuring time, as given in Formula (7):

$$\varepsilon_i = (r_s - r_0)/A_{s,i} \tag{7}$$

— when a PC is used, reference measurement standards of various mass per area should be measured so that actual efficiency can be used in evaluation depending on Sr recovery.

11 Expression of results

11.1 General

When ^{90}Sr is determined through or by ^{90}Y it is considered that the measurement should be carried out when both are in radioactive equilibrium. In other case, appropriate formulae should be developed following ISO 11929 (all parts).

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

11.2.1 Calculation of the activity per unit of mass

The activity per unit 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 in and is in equilibrium with the parent nuclide ^{90}Sr. This occurs 15 to 20 days after $t = 0$; where $t = 0$ is the point in time when 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 the ^{89}Sr contained in the test sample shall be neglected.

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 plus ^{90}Y ($a_{^{90}\mathrm{Sr+Y}}$) shall be calculated as given in Formula (8):

$$a_{^{90}\mathrm{Sr+Y}} = \frac{r_g - r_0}{m \cdot R_{\mathrm{c,Sr}} \cdot \varepsilon_{^{90}\mathrm{Sr+Y}}} \tag{8}$$

and, the activity per unit of mass of ^{90}Sr, shall be calculated as given in Formula (9):

$$a_{^{90}\mathrm{Sr}} = \frac{a_{^{90}\mathrm{Sr+Y}}}{2} = (r_g - r_0) \cdot w_{^{90}\mathrm{Sr}} \text{ with } w_{^{90}\mathrm{Sr}} = \frac{1}{2 \cdot m \cdot R_{\mathrm{c,Sr}} \cdot \varepsilon_{^{90}\mathrm{Sr+Y}}} \tag{9}$$

11.2.2 Standard uncertainty

According to the ISO/IEC Guide 98-3, the standard uncertainty of $a_{^{90}\mathrm{Sr}}$ is calculated by Formula (10):

$$u(a_{^{90}\mathrm{Sr}}) = \sqrt{w_{^{90}\mathrm{Sr}}^2 \cdot (r_\mathrm{g}/t_\mathrm{g} + r_0/t_0) + a_{^{90}\mathrm{Sr}}^2 \cdot u_\mathrm{rel}^2(w_{^{90}\mathrm{Sr}})} \tag{10}$$

Where the uncertainties of the sample and background counting times are neglected and the relative standard uncertainty of w is calculated by Formula (11):

$$u_\mathrm{rel}^2(w_{^{90}\mathrm{Sr}}) = u_\mathrm{rel}^2(R_{\mathrm{c,Sr}}) + u_\mathrm{rel}^2(m) + u_\mathrm{rel}^2(\varepsilon_{^{90}\mathrm{Sr+Y}}) \tag{11}$$

the relative standard uncertainty of $\varepsilon_{\mathrm{Sr+Y}}$ is calculated by Formula (12):

$$u_\mathrm{rel}^2(\varepsilon_{^{90}\mathrm{Sr+Y}}) = (r_\mathrm{s}/t_\mathrm{s} + r_0/t_0)/(r_\mathrm{s} - r_0)^2 + u_\mathrm{rel}^2(A_{\mathrm{s},^{90}\mathrm{Sr+Y}}) \tag{12}$$

$u_\mathrm{rel}(A_{\mathrm{s},^{90}\mathrm{Sr+Y}})$ includes all the uncertainties related to the reference measurement standard, that is, those of the standard solution and the preparation of the reference measurement standard. $u_\mathrm{rel}(R_{\mathrm{c,Sr}})$ is the uncertainty related to the chemical yield, and depends on its method of evaluation.

11.2.3 Decision threshold

In accordance with ISO 11929 (all parts), the decision threshold, $a^*_{^{90}\mathrm{Sr}}$, is obtained through $\tilde{u}(\tilde{a}_{^{90}\mathrm{Sr}})$, i.e. the standard uncertainty of $a_{^{90}\mathrm{Sr}}$ as a function of its true value, this yields Formula (13):

$$a^*_{^{90}\mathrm{Sr}} = k_{1-\alpha} \cdot \tilde{u}(0) = k_{1-\alpha} \cdot w_{^{90}\mathrm{Sr}} \cdot \sqrt{r_0/t_\mathrm{g} + r_0/t_0} \tag{13}$$

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

11.2.4 Detection limit

In accordance with ISO 11929 (all parts), the detection limit, $a^\#_{^{90}\mathrm{Sr}}$, is calculated by Formula (14):

$$\begin{aligned} a^\#_{^{90}\mathrm{Sr}} &= a^*_{^{90}\mathrm{Sr}} + k_{1-\beta} \cdot \tilde{u}(a^\#_{^{90}\mathrm{Sr}}) \\ &= a^*_{^{90}\mathrm{Sr}} + k_{1-\beta} \cdot \sqrt{w_{^{90}\mathrm{Sr}}^2 \cdot [(a^\#_{^{90}\mathrm{Sr}}/w_{^{90}\mathrm{Sr}} + r_0)/t_\mathrm{g} + r_0/t_0] + a^{\#2}_{^{90}\mathrm{Sr}} \cdot u_\mathrm{rel}^2(w_{^{90}\mathrm{Sr}})} \end{aligned} \tag{14}$$

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

The detection limit can be calculated by solving Formula (14) for $a^\#_{^{90}\mathrm{Sr}}$ or, more simply, by iteration with a starting approximation $a^\#_{^{90}\mathrm{Sr}} = 2 \cdot a^*_{^{90}\mathrm{Sr}}$.

When taking $\alpha = \beta$ then $k_{1-a} = k_{1-\beta} = k$ and the solution of Formula (9) is given by Formula (15):

$$a^\#_{^{90}\mathrm{Sr}} = \frac{2 \cdot a^*_{^{90}\mathrm{Sr}} + \left(k^2 \cdot w_{^{90}\mathrm{Sr}}\right)/t_\mathrm{g}}{1 - k^2 \cdot u_\mathrm{rel}^2\left(w_{^{90}\mathrm{Sr}}\right)} \tag{15}$$

11.3 Determination of ^{90}Sr by the ^{90}Y

11.3.1 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 gives 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 per unit of mass of ^{90}Sr (a_{Sr}) is calculated at time $t = 0$, using Formula (16):

$$a_{Sr} = a_Y = (r_g - r_0) \cdot w_Y \text{ with } w_Y = \frac{\lambda_Y \cdot t_g}{(e^{-\lambda_Y t_d} - e^{-\lambda_Y t_f})} \cdot \frac{1}{\varepsilon_Y \cdot m \cdot R_c} \quad (16)$$

Being $R_c = R_{c,Sr} \cdot R_{c,Y}$

This equation allows to correct the activity of the decay of ^{90}Y during the counting time ($t_g = t_f - t_d$).

11.3.2 Standard uncertainty

According to *GUM*, the standard uncertainty of $a_{^{90}Sr}$ is calculated by Formula (17):

$$u(a_{^{90}Sr}) = \sqrt{w_Y{}^2 \cdot (r_g / t_g + r_0 / t_0) + a^2_{^{90}Sr} \cdot u^2_{rel}(w_Y)} \quad (17)$$

Where the uncertainties of the sample and background counting times are neglected and the relative standard uncertainty of w is calculated by Formula (18):

$$u^2_{rel}(w_Y) = u^2_{rel}(R_c) + u^2_{rel}(m) + u^2_{rel}(\varepsilon_Y) \quad (18)$$

the relative standard uncertainty of ε_Y is calculated by Formula (19):

$$u^2_{rel}(\varepsilon_Y) = (r_s / t_s + r_0 / t_0) / (r_s - r_0)^2 + u^2_{rel}(A_{s,Y}) \quad (19)$$

$u_{rel}(A_{s,Y})$ includes all the uncertainties related to the reference measurement standard, that is, those of the standard solution and the preparation of the reference measurement standard. $u_{rel}(R_c)$ is the uncertainty related to the chemical yield. It can be calculated by Formula (20):

$$u^2_{rel}(R_c) = u^2_{rel}(R_{c,Sr}) + u^2_{rel}(R_{c,Y}) \quad (20)$$

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 should be depend on their method of evaluation.

11.3.3 Decision threshold

In accordance with ISO 11929 (all parts), the decision threshold, $a^*_{^{90}Sr}$, is obtained through $\tilde{u}(\tilde{a}_{^{90}Sr})$, i.e. the standard uncertainty of $a_{^{90}Sr}$ as a function of its true value, this yields Formula (21):

$$a^*_{^{90}Sr} = k_{1-\alpha} \cdot \tilde{u}(0) = k_{1-\alpha} \cdot w_Y \cdot \sqrt{r_0 / t_g + r_0 / t_0} \quad (21)$$

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

11.3.4 Detection limit

In accordance with ISO 11929 (all parts), the detection limit, $a^{\#}_{^{90}\mathrm{Sr}}$, is calculated by Formula (22):

$$\begin{aligned} a^{\#}_{^{90}\mathrm{Sr}} &= a^{*}_{^{90}\mathrm{Sr}} + k_{1-\beta}\cdot\tilde{u}\left(a^{\#}_{^{90}\mathrm{Sr}}\right) \\ &= a^{*}_{^{90}\mathrm{Sr}} + k_{1-\beta}\cdot\sqrt{w_{\mathrm{Y}}^{2}\cdot\left[\left(a^{\#}_{^{90}\mathrm{Sr}}/w_{\mathrm{Y}}+r_{0}\right)/t_{\mathrm{g}}+r_{0}/t_{0}\right]+a^{\#2}_{^{90}\mathrm{Sr}}\cdot u^{2}_{\mathrm{rel}}\left(w_{\mathrm{Y}}\right)} \end{aligned} \tag{22}$$

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

The detection limit can be calculated by solving Formula (22) for $a^{\#}_{^{90}\mathrm{Sr}}$ or, more simply, by iteration with a starting approximation $a^{\#}_{^{90}\mathrm{Sr}} = 2\cdot a^{*}_{^{90}\mathrm{Sr}}$.

When taking $\alpha = \beta$ then $k_{1-a} = k_{1-\beta} = k$ and the solution of Formula (22) is given by Formula (23):

$$a^{\#}_{^{90}\mathrm{Sr}} = \frac{2\cdot a^{*}_{^{90}\mathrm{Sr}} + \left(k^{2}\cdot w_{\mathrm{Y}}\right)/t_{\mathrm{g}}}{1-k^{2}\cdot u^{2}_{\mathrm{rel}}\left(w_{\mathrm{Y}}\right)} \tag{23}$$

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

11.4.1 Calculation of the activity per unit of mass

This method is based in the realisation 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 portion. It is suggested that the same counting time, t_{g}, is used for both measurements. The net counts rates, r_j, of these measurements can be calculated from the gross count rates, r_{gj} and the background count rates, r_{0j}, as:

$$r_j = r_{gj} - r_{0j} \tag{24}$$

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 Formula (25) below, considering that the ^{90}Sr and ^{89}Sr activities are constant during the counting time, and the appropriate decay constants.

$$\begin{aligned} r_1 &= 2\cdot A_{^{90}\mathrm{Sr}}\cdot\varepsilon_{^{90}\mathrm{Sr}+Y} + \varepsilon_{^{89}\mathrm{Sr}}A_{^{89}\mathrm{Sr}}e^{-\lambda_{^{89}\mathrm{Sr}}t_1} \\ r_2 &= 2\cdot A_{^{90}\mathrm{Sr}}\cdot\varepsilon_{^{90}\mathrm{Sr}+Y} + \varepsilon_{^{89}\mathrm{Sr}}A_{^{89}\mathrm{Sr}}e^{-\lambda_{^{89}\mathrm{Sr}}t_2} \end{aligned} \tag{25}$$

The activity per unit of mass (a_i) of the radionuclide *i* is calculated using Formula (26):

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

and

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

with $w_{90} = \dfrac{1}{m\cdot R_{\mathrm{c,Sr}}\cdot 2\cdot\varepsilon_{^{90}\mathrm{Sr+Y}}\cdot(1-c)}$ and $c = e^{-\lambda_{^{89}\mathrm{Sr}}(t_2-t_1)}$

$$a_{89_{Sr}} = w_{89} \cdot (r_2 - r_1) \tag{28}$$

with $w_{89} = \dfrac{e^{+\lambda_{89_{Sr}} t_1}}{m \cdot R_{c,Sr} \cdot \varepsilon_{89_{Sr}} \cdot (c-1)}$ and $c = e^{-\lambda_{89_{Sr}}(t_2 - t_1)}$

11.4.2 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 by Formula (29):

$$u(a_{90_{Sr}}) = \sqrt{w_{90}{}^2 \cdot [u^2(r_2) + c^2 \cdot u^2(r_1)] + a^2_{90_{Sr}} \cdot u^2_{rel}(w_{90})}$$

$$u(a_{89_{Sr}}) = \sqrt{w_{89}{}^2 \cdot [u^2(r_1) + u^2(r_2)] + a^2_{89_{Sr}} \cdot u^2_{rel}(w_{89})} \tag{29}$$

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

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

$$u^2(r_j) = r_{gj} / t_g + r_{0j} / t_0 \tag{30}$$

the relative standard uncertainties of w_{90} and w_{89} are calculated by Formula (31):

$$u^2_{rel}(w_{90}) = u^2_{rel}(R_{c,Sr}) + u^2_{rel}(m) + u^2_{rel}(\varepsilon_{90_{Sr+Y}})$$

$$u^2_{rel}(w_{89}) = u^2_{rel}(R_{c,Sr}) + u^2_{rel}(m) + u^2_{rel}(\varepsilon_{89_{Sr}}) \tag{31}$$

The relative standard uncertainty of ε_i is calculated by Formula (32):

$$u^2_{rel}(\varepsilon_i) = u^2_{rel}(r_s - r_0) + u^2_{rel}(A_{s,i}) = (r_s / t_s + r_0 / t_0) / (r_s - r_0)^2 + u^2_{rel}(A_{s,i}) \tag{32}$$

$u_{rel}(A_{s,i})$ includes all the uncertainties related to the reference measurement standard, that is, those of the standard solution and the preparation of the reference measurement standard. $u_{rel}(R_{c,Sr})$ is the uncertainty related to the chemical yield, and depends on its method of evaluation.

11.4.3 Decision threshold

In accordance with ISO 11929 (all parts), the decision thresholds, a_i^*, is obtained through $\tilde{u}(\tilde{a}_i)$, i.e. the standard uncertainty of a_i as a function of its true value, this yields Formula (33):

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

$$a^*_{89_{Sr}} = k_{1-\alpha} \cdot \tilde{u}(0) = k_{1-\alpha} \cdot w_{89} \cdot \sqrt{(r_{01} + r_{02})\left(\frac{1}{t_0} + \frac{1}{t_g}\right) + \frac{2}{t_g (1-c)} \frac{a_{90_{Sr}}}{w_{90}}} \tag{33}$$

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

11.4.4 Detection limit

In accordance with ISO 11929 (all parts), the detection limits, $a_i^{\#}$, are calculated by Formula (34):

$$\begin{aligned} a^{\#}_{90\,\mathrm{Sr}} &= a^{*}_{90\,\mathrm{Sr}} + k_{1-\beta}\cdot\tilde{u}(a^{\#}_{90\,\mathrm{Sr}}) \\ &= a^{*}_{90\,\mathrm{Sr}} + k_{1-\beta}\cdot\sqrt{w_{\mathrm{Y}}^2\cdot[(a^{\#}_{90\,\mathrm{Sr}}/w_{\mathrm{Y}}+r_0)/t_{\mathrm{g}}+r_0/t_0]+a^{\#2}_{90\,\mathrm{Sr}}\cdot u^2_{\mathrm{rel}}(w_{\mathrm{Y}})} \end{aligned} \tag{34}$$

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

The detection limit can be calculated by solving Formula (34) for $a_i^{\#}$ or, more simply, by iteration with a starting approximation $a_i^{\#} = 2\cdot a_i^{*}$.

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

$$a^{\#}_{90\,\mathrm{Sr}} = \frac{2\cdot a^{*}_{90\,\mathrm{Sr}} + k^2\cdot w_{90}\cdot(1+c^2)/t_{\mathrm{g}}\cdot(1-c)}{1-k^2\cdot u^2_{\mathrm{rel}}(w_{90})}$$

$$a^{\#}_{89\,\mathrm{Sr}} = \frac{2\cdot a^{*}_{89\,\mathrm{Sr}} + k^2\cdot w_{89}(1+c)/(c-1)\cdot t_{\mathrm{g}}}{1-k^2\cdot u^2_{\mathrm{rel}}(w_{89})} \tag{35}$$

11.5 Confidence limits

Confidence limits can be calculated in accordance with ISO 11929 (all parts). The values of lower limit, $a_i^{\triangleleft}$, and upper limit, $a_i^{\triangleright}$, are calculated by Formula (36) and Formula (37):

$$a_i^{\triangleleft} = a_i - k_p\cdot u(a_i);\ p = \omega\cdot(1-\gamma/2) \tag{36}$$

$$a_i^{\triangleright} = a_i + k_q\cdot u(a_i);\ q = 1-\omega\cdot\gamma/2 \tag{37}$$

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

$\omega = 1$ may be set if $a_i \geq 4\cdot u(a_i)$. In this case Formula (38):

$$a_i^{\triangleleft,\triangleright} = a_i \pm k_{1-\gamma/2}\cdot u(a_i) \tag{38}$$

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

12 Test report

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

a) a reference to this document, i.e. ISO 18589-5:2019;

b) identification of the sample;

c) units in which the results are expressed;

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

Complementary information can be provided such as:

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

— decision threshold and the detection limit;

— depending on the customer request there are different ways to present the result:

  — when the activity per unit of mass a_i is compared with the decision threshold, in accordance with ISO 11929 (all parts), the result of the measurement should be expressed as $\leq a_i^*$ 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 $\leq a_i^\#$ 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;

— mention of any relevant information likely to affect the results.

Annex A
(informative)

Examples of evaluation models

A.1 General considerations

For both equipment, PC and LSC, and for the three examples described in this annex, 20 g of dry soil sample are taken and counting times for blank and test samples of 60 000 s are taken with the aim to be able to compare the obtained results. Standard uncertainties of times involved in the calculus process as well as those of decay constants are neglected.

$\alpha, \beta = 0{,}05$ with $k_{1-\alpha} = 1{,}65$ and $k_{1-\beta} = 1{,}65$ are chosen by default.

Values provided for input quantities correspond to the typical measurements by proportional counter and by liquid scintillation counter.

A.2 Determination of ^{90}Sr in equilibrium with ^{90}Y

A.2.1 Input quantities and values

Table A.1 — Input quantities and values

Quantity	Symbol	Xi (PC)	Xi (LSC)	Unit
Mass of dry soil	m	20	20	g
Background counting time	t_0	60 000	60 000	s
Sample counting time	t_g	60 000	60 000	s
Reference measurement standard counting time	t_s	3 600	3 600	s
Gross count rate	r_g	0,15	0,49	s^{-1}
Background count rate	r_0	0,01	0,16	s^{-1}
Reference measurement standard count rate	r_s	3,33	8,01	s^{-1}
Chemical yield of Strontium	$R_{c,Sr}$	0,78	0,78	
Counting efficiency of ^{90}Sr+Y	$\varepsilon_{^{90}Sr+Y}$	0,41	0,96	
Relative uncertainty of ^{90}Sr+Y reference measurement standard (square)	$u^2_{rel}(A_{s,^{90}Sr+Y})$	2,50E-03	2,50E-03	
Relative uncertainty of mass (square)	$u^2_{rel}(m)$	1,00E-04	1,00E-04	
Relative uncertainty of yield (square)	$u^2_{rel}(R_{c,Sr})$	2,50E-03	2,50E-03	

Using values from Table A.1 and formulae from 11.2, the following values are obtained:

A.2.2 Activity concentration

With $w_{90} = \dfrac{1}{2 \cdot m \cdot R_{c,Sr} \cdot \varepsilon_{^{90}Sr+Y}}$ taken a value of: