
**Measurement of radioactivity in the
environment — Soil —**

**Part 1:
General guidelines and definitions**

Mesurage de la radioactivité dans l'environnement — Sol —

Partie 1: Lignes directrices générales et définitions



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Contents

Page

| | |
|--|----|
| Foreword | iv |
| Introduction | v |
| 1 Scope..... | 1 |
| 2 Normative references | 2 |
| 3 Terms and definitions | 2 |
| 3.1 General terms | 2 |
| 3.2 Terms relating to soils..... | 2 |
| 3.3 Terms relating to sampling | 3 |
| 4 Symbols | 5 |
| 5 Origins of the radioactivity in soils | 5 |
| 5.1 Natural radioactivity..... | 5 |
| 5.2 Other sources of radioactivity in soils..... | 6 |
| 6 Objectives of the study of soil radioactivity..... | 6 |
| 7 Principles and requirements of the study of soil radioactivity | 7 |
| 7.1 Planning process — Sampling strategy and plan | 8 |
| 7.2 Sampling process | 9 |
| 7.3 Laboratory process..... | 9 |
| 7.4 General procedural requirements | 11 |
| 7.5 Documentation | 11 |
| Bibliography | 12 |

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 18589-1 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*

The following parts are under preparation:

— *Part 2: Sampling strategy, sampling, and pre-treatment of samples*

— *Part 3: Measurements of gamma emitting radionuclides*

— *Part 4: Measurement of plutonium by alpha spectroscopy*

— *Part 5: Measurements of strontium 90*

— *Part 6: Measurements of gross alpha and gross beta activities*

Introduction

This document was prepared following discussions during meetings of WG 17 in Tokyo 2000-05-8/10, Recife 2001-09-17/19, Paris 2002-01-15/16, Paris 2002-03-26/27, Rinhals 2002-05-27/29, Paris 2002-10-14/15, Paris 2003-03-03/04, Paris 2003-06-16/17 and Paris 2003-12-08/09.

This part of ISO 18589 has been prepared simultaneously with five other parts concerning the measurements of radioactivity in the soil environment, and is complementary to the latter documents.

ISO 18589 Parts 1 to 6 are addressed to those responsible for determining the radioactivity present in soils. This International Standard is published in several parts to be used jointly or separately according to needs. Parts 1 and 2 are general in nature. Parts 3 to 5 deal with nuclide-specific measurements, Part 6 with non-specific measurements of gross alpha or gross beta activities.

Further parts may be added to this International Standard in the future if the standardization of measurements of other radionuclides becomes necessary.

Measurement of radioactivity in the environment — Soil —

Part 1: General guidelines and definitions

1 Scope

This part of ISO 18589 specifies the general requirements to carry out radionuclides tests on soil sample, including sampling.

This part of ISO 18589 is addressed to people responsible for determining the radioactivity present in soils for the purpose of radiation protection. This may concern soils from gardens and farmland, urban or industrial sites, as well as soil not affected by human activities.

This part of ISO 18589 is applicable to all laboratories regardless of the number of personnel or the extent of the scope of testing activities. When a laboratory does not undertake one or more of the activities covered by this part of ISO 18589, such as planning, sampling or testing, the requirements of those clauses do not apply.

This part of ISO 18589 is to be used in conjunction with other parts of ISO 18589 that outline the setting up of programmes and sampling techniques, methods of general processing of samples in the laboratory and also methods for measuring the radioactivity in soil. Its purpose is the following:

- define the main terms relating to soils, sampling, radioactivity and its measurement;
- describe the origins of the radioactivity in soils;
- define the main objectives of the study of radioactivity in soil samples;
- present the principles of studies of soil radioactivity;
- identify the analytical and procedural requirements when measuring radioactivity in soil.

This part of ISO 18589 is applicable if radionuclide measurements for the purpose of radiation protection are to be made in the following cases:

- initial characterization of radioactivity in the environment;
- routine surveillance of the impact of nuclear installations or of the evolution of the general territory;
- investigations of accident and incident situations;
- planning and surveillance of remedial action;
- decommissioning of installations or clearance of materials.

This part of ISO 18589 is not intended to cover scientific investigations of soil radioactivity and therefore does not apply to aspects of such measurements.

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 11074-1:1996, *Soil quality — Vocabulary — Part 1: Terms and definitions relating to the protection and pollution of the soil*

ISO 11074-2:1998, *Soil quality — Vocabulary — Part 2: Terms and definitions relating to sampling*

ISO 10381-1:2002, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 10381-2:2002, *Soil quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 10381-3:2001, *Soil quality — Sampling — Part 3: Guidance on safety*

ISO 11464, *Soil Quality — Pretreatment of samples for physico-chemical analyses*

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

GUM:1995, *Guide to the expression of uncertainty in measurement*, first edition BIPM/IEC/IFCC/ISO/IUPAC/IUPAP/OIML

3 Terms and definitions

For the purposes of all parts of ISO 18589, the terms and definitions given in ISO 11074 and the following apply.

3.1 General terms

3.1.1 routine surveillance

surveillance carried out periodically and designed to observe the potential changes of the soil's radioactive characteristics

3.1.2 analysis for characterization

set of observations that contribute, at a given time, to the characterization of the radioactive properties of a soil sample with a view to use them later as reference data

NOTE The test report may include other data characterizing the site studied.

3.1.3 vertical distribution of the radioactivity

determination of the radioactivity in the layers of soil sampled at different depths which describe the vertical profile of the distribution by a radionuclide or a group of radionuclides

3.2 Terms relating to soils

3.2.1 soil

upper layer of the Earth's crust composed of mineral particles, organic matter, water, air and living organisms

3.2.2 herbaceous cover

lower stratum of vegetation made up essentially of various herbaceous species found for example in meadows, lawns or fallow fields

3.2.3**soil horizon**

basic layer of soil, which is more or less parallel to the surface and is homogeneous in appearance for most morphological characteristics (colour, texture, structure, etc.)

NOTE The succession of soil horizons makes up a soil profile and allows, on the basis of certain analytical criteria, the morphogenetic nature of the soil to be defined.

3.3 Terms relating to sampling

The following definitions are adaptations of the definitions taken from ISO 11074 and ISO 10381.

3.3.1**sample**

portion of material selected from a larger quantity of material, collected and taken away for testing

3.3.2**sampling**

defined procedure whereby a part of the soil is taken for testing

NOTE 1 In certain cases, the sample might not be representative but is determined by availability.

NOTE 2 Sampling procedures shall describe all the processes necessary to provide the laboratory with the samples required to reach the objectives of the study of the soil radioactivity. This will include the selection, sampling plan, withdrawal and preparation of the samples from the soil.

3.3.3**sampling strategy**

set of technical principles that aim to resolve, depending on the objectives and site considered, the two main issues which are the sampling density and the spatial distribution of the sampling areas

NOTE The sampling strategy provides the set of technical options that will be required in the sampling plan.

3.3.4**sampling area**

area from which the different samples are collected

NOTE A site can be divided into several sampling areas.

3.3.5**sampling plan**

precise protocol that, depending on the application of the principles of the strategy adopted, defines the spatial and temporal dimensions of sampling, the frequency, the sample number, the quantities sampled, etc., and the human resources to be used for the sampling operation

3.3.6**random sampling**

collecting samples at random in space and time from the sampling area

3.3.7**systematic sampling**

collecting samples by some systematic method in space and time from the sampling area

3.3.8**random systematic sampling**

collecting samples at random from each sampling unit from a set of systematically defined sampling units

3.3.9

sampling unit

section of the sampling area whose limits can be physical or hypothetical

NOTE Sampling units are obtained by dividing the sampling area into grid box units according to the sampling pattern.

3.3.10

sampling pattern

system of sampling locations based on the results of statistical procedures

NOTE This leads to a set of predetermined sampling points designed to monitor one or more specified sites. The sampling area is divided into several sampling units or basic grid box units, which are usually square or rectangular (but circular or linear grid boxes are not excluded depending upon the characteristics of the pollution source).

3.3.11

increment

portion of material collected in a single operation using a sampling device

NOTE Increments can be grouped to form a composite sample.

3.3.12

sub-sample

sample in which the material of interest is randomly distributed in parts of equal or unequal size

3.3.13

single sample

representative quantity of the material, presumed to be homogeneous, taken from a sampling unit, kept and treated separately from the other samples

3.3.14

composite sample

two or more increments mixed together in appropriate proportions, either discretely or continuously (blended composite sample), from which the average value representative of a desired characteristic may be obtained

3.3.15

sorted sample

single sample or composite sample taken from the same sampling unit, obtained after the elimination of coarse elements that are larger than 2 cm and before drying

3.3.16

laboratory sample

sorted sample intended for laboratory inspection or testing

NOTE 1 When the laboratory sample is further prepared (reduced) by subdividing, mixing, grinding or combinations of these operations, the result is the test sample. When no preparation is required, the initial laboratory sample is considered as the test sample. Depending on the number of analyses to be performed, test portions are isolated from the test sample for analysis.

NOTE 2 The laboratory sample is the final sample from the point of view of the sample collection step, but it is the initial sample from the point of view of the test step.

3.3.17

test sample

sample treated in accordance with ISO 18589-2, prepared for testing

NOTE The test sample is prepared from the laboratory sample. It is a fine dry homogeneous soil in a powder state.

3.3.18

test portion

part of the test sample prepared for specific testing

4 Symbols

Table 1 — Definitions and symbols

| Quantity | Common notation | Unit | Definition |
|-------------------------|-----------------|--|--|
| Activity | A | becquerel Bq | number of decays per second of a radionuclide |
| Activity concentration | A_m | becquerel per kilogram Bq·kg ⁻¹ | radionuclide activity per unit dry mass of material |
| Activity per unit area | A_s | becquerel per square metre Bq·m ⁻² | radionuclide activity per unit area used to characterize the activity at the soil surface, at a depth or integrated activity over a soil column |
| Gross α activity | $A'(\alpha)$ | Becquerel Bq | number of α decays per second of a mixture of radionuclides determined by non-nuclide-specific measurement techniques whose efficiency is calibrated using a specific radionuclide such as ²³⁹ Pu, ²⁴¹ Am, ... |
| Gross β activity | $A'(\beta)$ | Becquerel Bq | number of β decays per second of a mixture of radionuclides determined by non-nuclide-specific measurement techniques whose efficiency is calibrated using a specific radionuclide such as ³⁶ Cl, ⁴⁰ K, ⁹⁰ Sr+ ⁹⁰ Y, ... |

5 Origins of the radioactivity in soils

5.1 Natural radioactivity

Soils are naturally radioactive, primarily because of their mineral content. The main natural radionuclides are potassium 40 (⁴⁰K) and the radioactive nuclides of the uranium 238 (²³⁸U) and thorium 232 (²³²Th) decay series. The natural radioactivity may vary considerably from one type of soil to another. Table 2 gives the order of magnitude of the activity concentrations of these elements in soils of some large regions of the world [3].

Table 2 — Activity concentrations of natural radionuclides in soils [3]

| Region/Country | Activity concentration Bq·kg ⁻¹ | | | | | |
|----------------------------------|---|--------------|------------------|----------|-------------------|----------|
| | ⁴⁰ K | | ²³⁸ U | | ²³² Th | |
| | Mean | Range | Mean | Range | Mean | Range |
| North America (USA) | 370 | 100 to 700 | 35 | 4 to 140 | 35 | 4 to 130 |
| South America (Argentina) | 650 | 540 to 750 | — | — | — | — |
| East Asia (China R.P.) | 440 | 9 to 1 800 | 33 | 2 to 690 | 41 | 1 to 360 |
| West Asia (Armenia) | 360 | 310 to 420 | 46 | 20 to 78 | 30 | 29 to 60 |
| North Europe (Lithuania) | 600 | 350 to 850 | 16 | 3 to 30 | 25 | 9 to 46 |
| West Europe (Ireland) | 350 | 40 to 800 | 37 | 8 to 120 | 26 | 3 to 60 |
| East Europe (Russian Federation) | 520 | 100 to 1 400 | 19 | 0 to 67 | 30 | 2 to 79 |
| South Europe (Greece) | 360 | 12 to 1 570 | 25 | 1 to 240 | 21 | 1 to 190 |

5.2 Other sources of radioactivity in soils

The sources of radioactivity in soils other than those of natural origin ^[3] are mainly due to the following:

- fallout from past atmospheric explosions of nuclear devices and following nuclear accidents;
- routine authorized low-level radioactive effluent discharges or accidental release into the environment from nuclear installations, mining and mineral extraction industries, industries working with mineral materials enriched in naturally radioactive elements (e.g. fertilizer factories or manufacturers of rare earth), and various economic sectors in which naturally or artificially radioactive elements are used;
- extensive use of fertilizers rich in phosphates for agricultural purposes.

Generally, and excluding limited areas of any high levels of contamination, the levels of artificial radioactivity in soils are between one and several orders of magnitude less than those of natural radioactivity. Therefore, before undertaking very low level activity measurements of soils samples, the main and secondary objectives of the study should be determined as accurately as possible to define the sampling strategy and the measurement protocols.

6 Objectives of the study of soil radioactivity

The main objective of the measurement of soil radioactivity is to assess the impact of release or remobilization of radioactive materials on the environment and on the population through its direct and indirect exposures (inhalation and ingestion pathways). Any potential protection procedures shall rely on knowledge of the activity concentrations in the soil and of their horizontal and vertical distributions, the particle sizes associated with the radionuclides as well as their chemical speciation inside the soil. Radioactivity measurements can be performed in a variety of different situations. This part of ISO 18589 shall apply to the following cases:

a) Characterization of radioactivity in the environment

Characterization of environmental radioactivity may be performed at a particular site where a nuclear installation is planned in order to establish background levels before any operation starts on the site. Comparison of the data obtained with those collected subsequently, under identical sampling conditions, during routine surveillance, serves to quantify the environmental impact of the installation [see 6 b)].

Characterization of environmental radioactivity may also be performed at a variety of locations in a territory. It provides the distribution patterns of environmental radioactivity with the goal of identifying areas of potentially enhanced exposures. This investigation gives the baseline values to estimate the exposure resulting from the natural radioactivity or enhanced radioactivity due to human activity.

b) Routine surveillance of the impact of nuclear installations or of the evolution of the general territory

Routine surveillance covers the systematic and periodic investigation of a specific site, such as the area around a nuclear installation. Sites where investigations may be necessary include installations of the nuclear fuel cycle (uranium mining and milling, isotope enrichment facilities, fuel element fabrication, power plants, reprocessing plants, storage facilities, final nuclear waste repositories) as well as radiochemical laboratories and nuclear medicine installations. Surveillance is performed to help quantify the impact of authorized radioactive effluents discharged from nuclear installations into the environment. The periodic checks will help ensure that the installation remains in compliance with legal requirements.

Routine surveillance covers also the systematic and periodic investigations of an entire territory. It is based on pre-selected sampling areas spread over the territory in order to identify local or widespread changes of environmental radioactivity, e.g. in the EU dense and sparse networks ^[4].

c) Investigations of accident and incident situations

Measurements of the radioactivity in soil following accidents and incidents allow quantification of the spatial distribution of the contamination by the radionuclides potentially released. These measurements provide a basis for decisions about protection measures for the population and the future use of the soil.

d) Planning and surveillance of remedial action

Planning and surveillance of remedial operations at particular sites or areas, which could have been contaminated due to past activities, require radioactivity measurements. Such measurements serve to characterize the contamination including the quantification of radioactive inventories and radioactivity levels. They are used to assess the transfer of contamination through different pathways, to select appropriate remedial measures and to control their effectiveness.

e) Decommissioning of installations or clearance of materials

Activity measurements of materials, low-level contaminated soil or rubble, to be released for re-use, recycling or disposal as non-radioactive waste arising from soil remedial operation and decommissioning of nuclear facilities, in order to demonstrate compliance with established criteria for unrestricted clearance.

This part of ISO 18589 gives advice on adequate sampling-planning and sampling processes, sample preparation methods and techniques, and procedures for laboratory measurements of radioactivity in soils. It was elaborated following a review of published procedures described in various national standards and other relevant documents internationally available. See References [5] to [13].

It defines general procedures of gamma spectrometry measurement, and of nuclide-specific analysis of alpha- and beta-emitting radionuclides. For completeness, measurements of gross alpha or gross beta radioactivity are also described. These methods are still used for purposes such as a rapid screening of soil samples in order to select those with higher contamination levels that need specific radionuclide measurement in the laboratory^[14]. Finally, it defines the general analytical and procedural requirements for measurements of radioactivity in soil.

7 Principles and requirements of the study of soil radioactivity

Three steps can be identified:

- a) planning process:** depending upon the objective, a sampling strategy can be defined that will lead to the definition of a sampling plan;
- b) sampling process:** the resulting sampling operation in the field will lead to sorted samples that will be packed and transported to the laboratory (Figure 1);
- c) laboratory process:** the preparation of test samples for laboratory measurement.

The laboratory in charge of sampling shall have a documented sampling plan with sampling procedures. The sampling plan shall be available at the location where the sampling is undertaken (see ISO/IEC 17025).

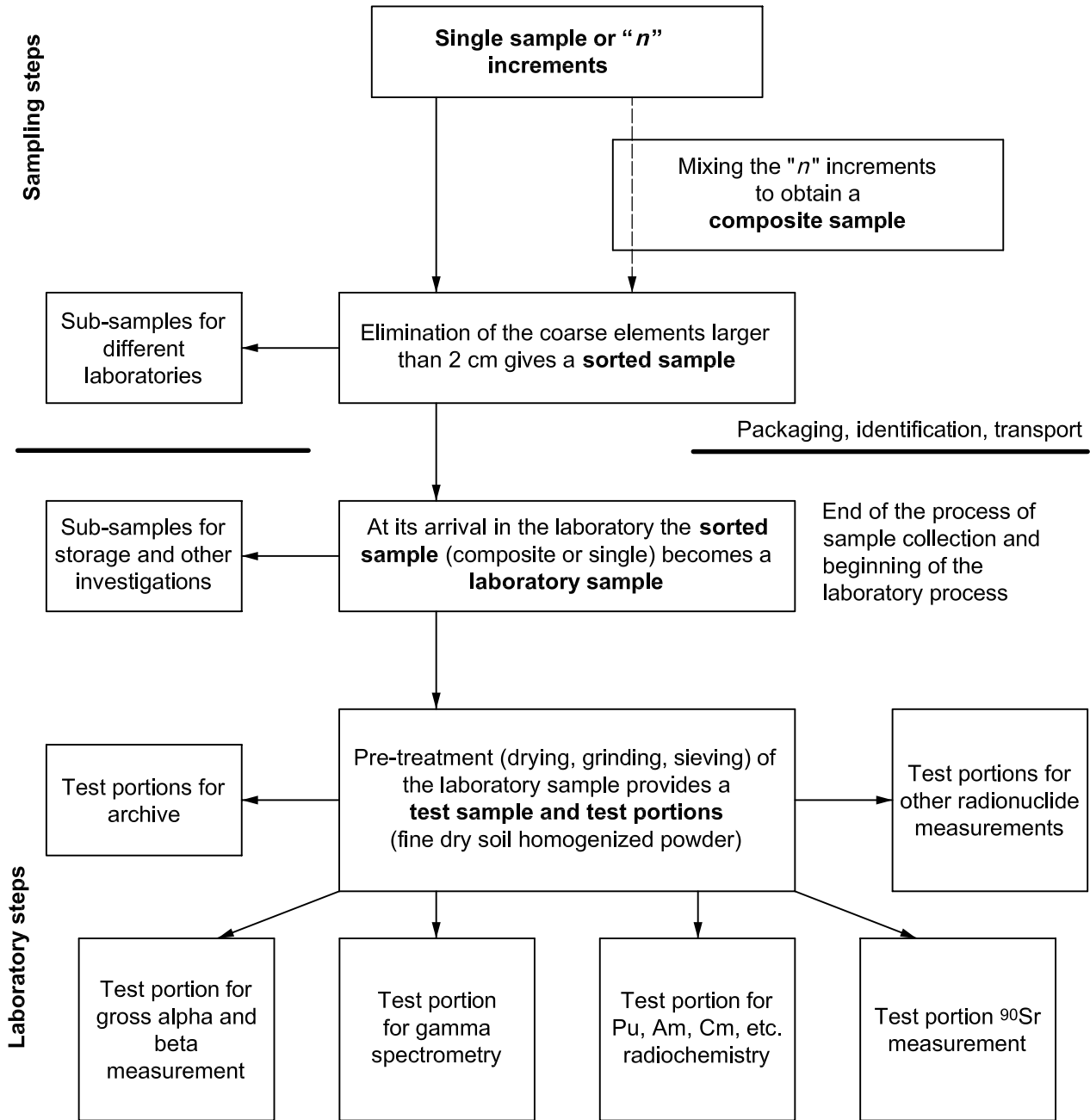


Figure 1 — Diagram of the evolution of the sample characteristics from the sampling site to the test laboratory

7.1 Planning process — Sampling strategy and plan

The sampling process will be performed following different approaches or sampling strategies depending on the objective pursued. Whatever this objective might be, the sampling strategy shall be carefully selected as it determines a large number of decisions and can generate important and costly activities.

Measurements of the radioactivity of a soil sample can only be correctly interpreted if the sampling is representative of the soil to be characterized [15], [16]. The sampling strategy shall ensure that the radioactivity of samples is representative of the distribution of radionuclides in the soil of the area under investigation. Nevertheless, in certain cases, the sample might not be representative but is determined by availability.

The definition of the sampling strategy should follow, as far as possible, the following stages:

- a) analysis of records to enable an historic study of the previous use of the sampling site;
- b) site reconnaissance (in some cases, analytic investigation techniques, using portable radioactivity detectors, may be used to identify the areas to be studied in details);
- c) identification of preferential migration pathways and/or accumulation areas;
- d) site reconnaissance with respect to the sampling to be undertaken.

The implementation of this strategy, which also includes the definition of the data quality objectives according to the parameters to be analysed, gives rise to the sampling plan.

NOTE For the past several years, attempts have taken place to streamline and increase the efficiency of field data collection programmes, by encouraging project managers to develop data quality objectives (DQOs) prior to initiation of the sampling ^[17].

The sampling plan shall define the operations to be carried out as defined in 3.3.5 and any other complementary operations such as the following:

- the precise location of the sampling unit and the type of equipment needed, depending upon the type of sample being collected (surface layer or layers with depth);
- the grouping of the increments to be taken, the homogenization to be carried out and the reduction of the sample to be made in order to obtain the mass necessary for the test samples (all test portions) needed for all the laboratory measurements;
- the packaging of the sample for its transport to the laboratory to avoid any loss or contamination from external sources or, conversely, to avoid all contamination of the premises or operators;
- the identification of the samples, together with a sampling data sheet stating the relevant details describing the sampling process.

Independent of the strategy chosen and of the sampling plan, the sampling and sample-preparation processes shall be performed in accordance with ISO 18589-2.

7.2 Sampling process

The collection of samples in the field shall match the sampling plan. It shall lead to the production of single samples or increments (composite sample).

The elimination of coarse elements during the sampling step, when feasible, shall produce the sorted sample. When necessary, sub-samples of the sorted sample shall be made in order to share the sample among different laboratories.

The preparation of sorted samples shall be produced by reduction of single or composite samples. A sorted sample should be representative of the average value of one or more given soil characteristics.

The identification, labelling, packaging, transporting procedures of the sorted samples to the laboratory shall guarantee the conservation of their characteristics.

7.3 Laboratory process

7.3.1 Preparation of samples

After arrival in the laboratory, the sorted samples are considered as laboratory samples for storage and further pre-treatment before their analysis.

In all cases, prior to the measurement of the radioactive characteristics, the laboratory sample shall be subject to pre-treatment in the laboratory, except if otherwise requested by the users of the measurement results. This

pre-treatment, according to in ISO 18589-2, shall be used to obtain a test sample whose physical-chemical characteristics (after drying, crushing, sieving and homogenization) are constant over time for all radioactive analyses to which the sample might be submitted, thus rendering the results easier to interpret (see ISO 11464).

Following this first pre-treatment step, certain radionuclide measurements require mineralization by ashing, leaching or dissolution of the test sample.

7.3.2 Radioactivity measurements

7.3.2.1 Nuclide-specific measurements

Generally, nuclide-specific measurements shall be preferred to those that cannot identify individual radionuclides, because any estimate of the exposure resulting from the radioactivity in the soil has to consider the individual radionuclides present. Analyses should be linked to the monitoring objectives.

NOTE There can be a regulatory requirement for gross alpha or/and gross beta monitoring and if these regulatory criteria are met, further radionuclide analytical characterization may not be needed.

The nuclide-specific measurement methods presently available include the following:

- gamma spectrometry for radionuclides whose gamma-decay energy range lies between 20 keV and 2 000 keV. This technique allows detection limits of the order of $\text{Bq}\cdot\text{kg}^{-1}$ for most radionuclides. For nuclide-specific measurement techniques, gamma spectrometry is the first method to be applied, since it allows the simultaneous determination of a wide range of natural and man-made gamma-emitting radionuclides without requiring any chemical sample preparation. In ISO 18589-3, evaluated methods are given for measuring gamma-emitting radionuclides;
- alpha spectrometry for alpha-emitting radionuclides and more particularly plutonium, americium and curium radio-isotopes. The determination of alpha-emitters first requires the dissolving of the soil test sample, then the sequential selective separation of the radionuclides from the solution. Finally, thin, solid sources of the extracted radionuclides are prepared for alpha spectrometry. In ISO 18589-4, several evaluated methods are given for measuring alpha-emitting radionuclides;
- beta measurements for the determination of pure beta-emitting nuclides after selective physical or chemical separation from a sample. In ISO 18589-5, several evaluated methods are given for measuring strontium 90;
- mass spectrometry of long-lived radionuclides is becoming increasingly important for the analysis of long-lived radionuclides such as ^{10}Be , ^{14}C , ^{26}Al , ^{36}Cl , ^{40}Ca , ^{53}Mn , ^{60}Fe , ^{59}Ni , ^{129}I , ^{135}Cs as well as Am, U, and Pu isotopes in environmental samples. Such measurements also require the chemical separation of the nuclide under investigation from the matrix and the preparation of a sample for measurement by mass spectrometry. At present, a number of mass spectrometric methods such as ICP-MS, AMS, and RIMS are scientifically and technically well established and respective chemical separation schemes exist. These are, however, not considered in this part of ISO 18589.

NOTE Nuclide-specific measurements are dealt with in ISO 18589-3, ISO 18589-4 and ISO 18589-5. The list of the parts is given in the Foreword.

7.3.2.2 Measurements of gross alpha and beta activity

This involves measuring the global activity of a sample (ISO 18589-6) to provide information about the gross activity of all alpha- or beta-emitting radionuclides present without however enabling detailed identification of the radionuclides:

- gross alpha activity concentration or per unit area;
- gross beta activity concentration or per unit area.

The activity concentrations, commonly measured in the soils, are in the order of several hundred $\text{Bq}\cdot\text{kg}^{-1}$ for gross alpha radioactivity and almost a thousand $\text{Bq}\cdot\text{kg}^{-1}$ for gross beta-emitting radioactivity. These activities

are essentially due to potassium 40 and alpha and beta emitters of the uranium and thorium radioactive decay chains.

A result of this type of measurement (carried out on a fine layer of the test sample) using a proportional counter type equipment can be quickly obtained. If previous results on soils taken from the same source and treated in identical conditions are available, a simple comparison allows the analyst to report any abnormal abundance of the radioactivity and allows the choice of the specific analysis methods to be implemented.

7.4 General procedural requirements

All steps and procedures carried out to establish the radioactivity of soil samples shall be completely traceable as specified in ISO/IEC 17025. This implies a complete documentation of the sampling strategy and plan chosen, the sampling operations performed, the chain of custody of the samples, the analytical protocol and all steps undertaken during analysis. Though the measurement procedures described in ISO 18589 have been evaluated in many instances, they must be performed under a quality assurance programme and control. They include the use of certified reference materials, the participation in interlaboratory comparisons and proficiency testing. Laboratory procedures shall ensure that laboratory and equipment contamination as well as cross sample contamination is avoided. When chemical separations are performed, the complete analytical blank shall be documented together with the results obtained.

For any measurement result, the standard uncertainty associated with it shall be determined in accordance with GUM:1995, taking into account all known sources of uncertainty.

For the determination of the decision threshold, the detection limit and the limits of the confidence interval, the probabilities of the errors of the first and second kind as well as the confidence level shall be specified and documented. The characteristic limits have to be determined in accordance with ISO 11929-7. The following steps to provide complete documentation are required.

- For the qualification of the analytical method, the detection limit must be compared with legal or other requirements and the observed detection limit of the method must be below the value specified in those requirements to qualify the method ^[18]. If the result is lower than the detection limit, the result of the measurement shall be reported as “below the detection limit”. For each measurement, the detection limit shall be specified in the test report.
- The actual result of a measurement shall be compared with the decision threshold obtained. If the result is below the decision threshold, a true value of the measurand equal to zero cannot be excluded. In this case, the result of the measurement shall be reported as “below the decision threshold”.
- If the confidence interval is asymmetric around the measurement result, the best estimate of the true value of the measurand in accordance with ISO 11929-7 shall be determined and reported.

7.5 Documentation

The test report shall refer to the sampling report, as well as the analytical procedure and the quality assurance measures (ISO/IEC 17025). It shall specify any relevant information likely to have affected the result and shall contain the following:

- the result of the measurement with its standard uncertainty;
- the detection limit;
- the limits of the confidence interval.

Records shall be kept detailing the test conditions under which the results were obtained in order to confirm the measurement results. Each such record shall be approved, dated, and signed by an authorized person to attest the correctness of the results.

Records of all relevant information on the measuring equipment needed for the confirmation process of a result shall be kept. These records shall demonstrate that each item of the measuring equipment satisfies the metrological requirements specified within the confirmation process for the equipment. Calibration certificates or verification reports and other relevant information shall be available.

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