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Measurement of radioactivity in the environment — Soil —

Part 3:
Measurement of gamma-emitting radionuclides

Mesurage de la radioactivité dans l'environnement — Sol —

Partie 3: Mesurages des radionucléides émetteurs gamma

Reference number
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Contents

Page

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 18589-3 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 2, *Radiation protection*.

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

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

Introduction

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

Additional parts may 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 3:
Measurement of gamma-emitting radionuclides

1 Scope

This part of ISO 18589 specifies the identification and the measurement of the activity in soils of a large number of gamma-emitting radionuclides using gamma spectrometry. This non-destructive method, applicable to large-volume samples (up to about 3 000 cm^3), covers the determination in a single measurement of all the γ-emitters present for which the photon energy is between 5 keV and 3 MeV.

This part of ISO 18589 can be applied by test laboratories performing routine radioactivity measurements as a majority of radionuclides is characterized by gamma-ray emission between 40 keV and 2 MeV.

This part of ISO 18589 is suitable for the surveillance of the environment and the inspection of a site and allows, in case of accidents, a quick evaluation of gamma activity.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 31-9, *Quantities and units — Part 9: Atomic and nuclear physics*

ISO 10703, *Water quality — Determination of the activity concentration of radionuclides — Method by high resolution gamma-ray spectrometry*

ISO 11074, *Soil quality — Vocabulary*

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

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

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

Guide to the expression of uncertainty in measurement (GUM), BIPM/IEC/IFCC/ISO/IUPAC/IUPAP/OIML

3 Terms, definitions and symbols

For the purposes of this document, the terms, definitions and symbols given in ISO 18589-1, ISO 11074, ISO 31-9 and ISO 10703 and the following symbols apply.

m	Mass of the test portion, in kilograms
A	Activity of each radionuclide in the calibration source, at the calibration time, in becquerel
a, a_c	Activity, in becquerel per kilogram, per unit of mass of each radionuclide, without and with corrections
t_g	Sample spectrum counting time, in seconds
t_0	Ambient background spectrum counting time, in seconds
t_s	Calibration spectrum counting time, in seconds
$n_{\mathrm{N},E}$, $n_{\mathrm{N0},E}$, $n_{\mathrm{Ns},E}$	Number of counts in the net area of the peak, at energy, E, in the sample spectrum, in the background spectrum and in the calibration spectrum, respectively
$n_{\mathrm{g},E}$, $n_{\mathrm{g0},E}$, $n_{\mathrm{gs},E}$	Number of counts in the gross area of the peak, at energy, E, in the sample spectrum, in the background spectrum and in the calibration spectrum, respectively
$n_{\mathrm{b},E}$, $n_{\mathrm{b0},E}$, $n_{\mathrm{bs},E}$	Number of counts in the background of the peak, at energy, E, in the sample spectrum, in the background spectrum and in the calibration spectrum, respectively
ε_E	Efficiency of the detector at energy, E, with the actual measurement geometry
P_E	Probability of the emission of gamma radiation with energy, E, for each radionuclide, per decay
$\mu_1(E)$, $\mu_2(E)$	Linear attenuation coefficient at photon energy, E, of the sample and calibration source, respectively, per centimetre
$\mu_{\mathrm{m},i}(E)$	Mass attenuation coefficient, in square centimetres per gram, at photon energy, E, of element i
h	Height of the sample in the container, in centimetres
w_i	Mass fraction of element i (no unit)
ρ	Bulk density, in grams per cubic centimetre, of the sample
λ	Decay constant of each radionuclide, per second
$u(a)$, $u(a_\mathrm{c})$	Standard uncertainty, in becquerel per kilogram, associated with the measurement result, with and without corrections, respectively
U	Expanded uncertainty, in becquerel per kilogram, calculated by $U = k \cdot u\left(a\right)$ with $k = 1, 2, \ldots$
a^*, a_c^*	Decision threshold, in becquerel per kilogram, for each radionuclide, without and with corrections, respectively
$a^\#$, $a_\mathrm{c}^\#$	Detection limit, in becquerel per kilogram, for each radionuclide, without and with corrections, respectively
$a^\triangleleft$, $a^\triangleright$	Lower and upper limits of the confidence interval, for each radionuclide, in becquerel per kilogram

4 Principle

The activity of gamma-emitting radionuclides present in the soil samples is determined using gamma spectrometry techniques based on the analysis of the energies and the peak areas of the full-energy peaks of the gamma lines. These techniques allow the identification and the quantification of the radionuclides [1], [2].

The nature and geometry of the detectors as well as the samples call for appropriate energy and efficiency calibrations [3], [4], [5]. If well-type detectors are used to measure small-mass samples, it is necessary to take special care to consider coincidence and summation effects (see 8.1.4).

NOTE Sodium iodide detectors can be used for the measurement of radioactivity in soil only in certain cases. Therefore, this part of ISO 18589 deals exclusively with gamma spectrometry using semiconductor detectors.

5 Gamma-spectrometry equipment

Gamma-spectrometry equipment generally consists of

- a semiconductor detector with a cooling system (liquid nitrogen, cryogenic assembly, etc.),
- a shield, consisting of lead and/or other materials, against ambient radiation,
- appropriate electronics (high-voltage power supply; signal-amplification system; an analogue-to-digital converter),
- a multi-channel amplitude analyser,
- a personal computer to display the measurement spectra and to process the data.

The semiconductor detectors generally used are made of high-purity germanium crystals (HP Ge). The type and geometry of these detectors determine their field of application. For example, when detecting photons with an energy below 400 keV, the use of detectors with a thin crystal is recommended in order to limit interference from high-energy photons. However, it is better to use a large-volume, P-type coaxial detector to measure high-energy photons (above 200 keV) or an N-type coaxial detector to detect both low- and high-energy radiation.

At the level of natural radioactivity, it is advantageous for the measurement to use an ultra-low-level measuring instrument, i.e. a set-up arranged with a choice of materials for the detector and shielding that guarantees a very low background level. This includes very low-noise electronic preamplifiers and amplifiers. The shielding case should be large enough to allow sufficient distance from all walls and the detector set up in the centre of the case, when 1-l samples are inserted. This allows the use of a room with a very low specific activity of building materials and a very low radon concentration in the room air to be chosen. It is optimal to erect the measuring instruments in the middle of the room with the maximum distance available to the room walls. Forced ventilation of the measuring room can possibly contribute to stabilizing the background level. On the other hand, forced ventilation can then cause problems when the outside air drawn in contains excess radon as a result of a warming-up of the soil (in particular, when the soil thaws in spring). It is always good practice to fill the inner part of the shielding with nitrogen. For this, the gaseous nitrogen escaping from the Dewar vessel of the detector arrangement can be passed permanently into the shielding.

The main characteristics that allow the estimation of a detector performance are as follows:

a) energy resolution (total width at half maximum of the full-energy peak), which enables the detector to separate two neighbouring gamma peaks;

b) absolute efficiency, which specifies the percentage of photons detected in the full-energy peak relative to the number of photons emitted;

c) peak-to-Compton ratio.

Depending on the required accuracy and the desired detection limit, it is generally necessary to use high-quality detectors whose energy resolution is less than 2,2 keV (for the ^{60}Co peak at 1,332 keV) and with a peak/Compton ratio between 50 and 80 for ^{137}Cs.

Some natural radionuclides, e.g. ^{210}Pb and ^{238}U via ^{234}Th, can be measured only via gamma lines in the energy range of 100 keV. In this case, the use of an N-type detector is recommended. Low-energy, low-level detectors offered by manufacturers have been optimized for this purpose and can additionally be used in other areas of environmental monitoring, e. g. for measurements of ^{129}I and ^{241}Am in samples from the vicinity of nuclear facilities.

The computer, in combination with the available hardware and software, shall be carefully selected [6], [7]. It is recommended that the results of the computer analysis of the spectrum be visually checked regularly.

Comparison with a certified reference material is recommended to check the performance of the apparatus. Participation in proficiency and inter-laboratory tests and inter-comparison exercises can also help to verify the performance of the apparatus and the status of the analysis [10], [11].

6 Sample container

Measuring gamma radioactivity in soils requires sample containers that are suited to gamma spectrometry. These containers should have the following characteristics:

— be made of materials with low absorption of gamma radiation;

— have volumes adapted to the shape of the detector for maximum efficiency;

— be watertight and not react with the sample constituents;

— have a wide-necked, airtight opening to facilitate filling;

— be unbreakable.

In order to verify easily that the content of the container conforms to the standard counting geometry, a transparent container with a mark to check the filling can be selected.

7 Procedure

7.1 Packaging of samples for measuring purposes

The soil samples packaged for gamma spectrometry measurements are usually dried, crushed, and homogenized in accordance with ISO 18589-2.

The procedure shall be carried out as follows.

a) Choose the container that is best suited to the volume of the sample so as to measure as much material as possible. To decrease self-absorption effects, the height of the contents should be minimized.

b) Fill the container to the level of the volume mark. It is recommended to use a mechanical filling device (for example, a vibrating table) to pack the sample to avoid any future losses in volume.

c) Note the sample mass. This information is useful when using the measurements to express the result as specific activity and when carrying out self-absorption corrections.

d) Visually check the upper level of the sample and make sure that it is horizontal before measuring. Where applicable, add more material to the sample until the mark has been reached and adjust the noted sample mass accordingly.

e) Hermetically seal the container if volatile or natural radionuclides are being measured.

f) Clean the outside of the container to remove potential contamination due to the filling process.

If measurements are required quickly, the processing method described in ISO 18589-2 can be ignored. This shall be mentioned in the test report and the results cannot be expressed in becquerels per kilogram of dry soil.

When measuring Rn-222 via decay products of Ra-226, the sealed container shall be stored long enough to allow radioactive equilibrium to be reached.

7.2 Laboratory background level

As some radionuclides found in the soil (see Annex B) are the same as in building materials, the detector and sample shall be adequately shielded against natural background radiation. Frequently, it is sufficient to shield the detector in a 10 cm thick, low-background lead case wall. Reduction of radon inside the shield is desirable. Further information is given in references [1], [2].

The natural radionuclides and their decay products occur widely and with large concentration ranges in floors, walls, ceilings, the air of the measuring rooms and in the materials of which detectors and shielding are made.

There are isotopes of the decay chain of the rare gas radon, whose emanation from the materials surrounding the measuring instruments depends on various physical parameters. Thus, large fluctuations in the concentration of radon and of the decay products can occur in room air and in the air of the detector shielding. This is a particular problem in basements of old buildings with defective floors.

The background of the measuring instruments shall be kept as low as possible and, in particular, as stable as possible by appropriate measures. This includes vacuuming the shielding and removing the dust by filtration. Frequent measurements of the background level permit the verification of its stability. This is necessary because the peaks of the background spectrum shall be subtracted from those of a sample spectrum.

7.3 Calibration

7.3.1 Energy calibration

Energy calibration is carried out using sources of a radionuclide with different emission lines (for example ^{152}Eu) or sources containing a mixture of several radionuclides. This calibration allows the establishment of the relationship between the channel numbers of the analyser and the known energy of the photons [12], [13], [14]. Generally, this task is carried out with appropriate software, which uses the standard spectra to automatically convert the channel scale of the multi-channel analyzer into a photon energy scale and to record the useful information necessary for future analyses. By using the energy calibration spectra, the full-width at half the maximum of the full-energy peaks can be determined as a function of the gamma energy. This information is usually required by the spectrometry analysis software.

Further information is given in ISO 10703 and References [8] and [9].

7.3.2 Efficiency calibration

Efficiency calibration is carried out either via *ab initio* calculations of the detector efficiency using transport theory and Monte Carlo techniques (not covered in this part of ISO 18589) or by using a radionuclide source having different emission lines or a mixed-radionuclide source. This calibration allows the establishment of the detection efficiency of the detector as a function of the energy of the radiation.

When using a radionuclide source with different emission lines for calibration, summation effects or coincidence losses should be taken into account.