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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 document 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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

For an explanation of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 2, *Radiological protection*.

This third edition cancels and replaces the second edition (ISO 18589-3:2015), which has been technically revised.

The main change is:

- a correction to **Formula (4)**.

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.

This corrected version of ISO 18589-3:2023 incorporates the following corrections:

- In Annex B, **Formula (B.8) and Formula (B.10) have been corrected.**

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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 [4]-[11]).

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 samples. The specific standards cover test methods for:

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Measurement of radioactivity in the environment — Soil —

Part 3: Test method of gamma-emitting radionuclides using gamma-ray spectrometry

1 Scope

This document 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 l), 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.

Generic test method and fundamentals using gamma-ray spectrometry are described in ISO 20042.

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

The method can be implemented using a germanium or other type of detector with a resolution better than 5 keV.

This document addresses methods and practices for determining gamma-emitting radionuclides activity present in soil, including rock from bedrock and ore, construction materials and products, pottery, etc. This includes such soils and material containing naturally occurring radioactive material (NORM) or those from technological processes involving Technologically Enhanced Naturally Occurring Radioactive Materials (TENORM) (e.g. the mining and processing of mineral sands or phosphate fertilizer production and use) as well as of sludge and sediment. This determination of gamma-emitting radionuclides activity is typically performed for the purpose of radiation protection. It 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 of soil samples. This might concern soils from gardens, farmland, urban or industrial sites that can contain building materials rubble, as well as soil not affected by human activities.

When the radioactivity characterization of the unsieved material above 200 μm or 250 μm , made of petrographic nature or of anthropogenic origin such as building materials rubble, is required, this material can be crushed in order to obtain a homogeneous sample for testing as described in ISO 18589-2.

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.

~~<std>ISO 10703, Water quality — Gamma-ray emitting radionuclides — Test method using high resolution gamma ray spectrometry</std>~~

~~<std>ISO 11074, Soil quality — Vocabulary</std>~~

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<std>ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories</std>

<std>ISO 18589-1, Measurement of radioactivity in the environment — Soil — Part 1: General guidelines and definitions</std>

<std>ISO 10703, Water quality — Gamma-ray emitting radionuclides — Test method using 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 20042, Measurement of radioactivity — Gamma-ray emitting radionuclides — Generic test method using gamma-ray spectrometry</std>

<std>ISO 80000-10, Quantities and units — Part 10: Atomic and nuclear physics</std>

<std>ISO/IEC Guide 98-1, Uncertainty of measurement — Part 1: Introduction to the expression of uncertainty in measurement</std>

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

ISO/IEC Guide 98-1, Uncertainty of measurement — Part 1: Introduction to the expression of uncertainty in measurement

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 10703, ISO 11074, ISO 18589-1, ISO 20042 and ISO 80000-10 apply.

3.2 Symbols

For the purposes of this document, the symbols given in ISO 10703, ISO 11074, ISO 18589-1, ISO 20042 and ISO 80000-10 apply.

Symbol	Meaning	Unit
A	Activity of each radionuclide in calibration source, at the calibration time	Bq
a, a_c	Activity per unit of mass of each radionuclide, without and with corrections	Bq·kg ⁻¹
$\epsilon_E, \epsilon_{Ea}$	Efficiency of the detector at energy, E , with the actual measurement geometry	
f_a	Correction factor considering all necessary corrections	
$f_{att}(E), f_{att}(E)_a$	Correction factor for self-attenuation at photon energy E	
$F_{att}(E), F_{att}(E)_{sample}$	Attenuation factor at photon energy E respectively for the sample and the standard	

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Symbol	Meaning	Unit
x	Unit thickness	cm
X	Thickness of the sample crossed by a photon flux; it also represents the filling height of the sample in the measurement containers	cm
w_i	Mass fraction of element i (no unit)	
a^*, a_c^*, a_c^*	Decision threshold, without and with corrections	Bq·kg ⁻¹
$a^{\#}, a_c^{\#}, a_c^{\#}$	Detection limit, without and with corrections	Bq·kg ⁻¹
$a^{\leftarrow}, a^{\rightarrow}, a_c^{\leftarrow}, a_c^{\rightarrow}$	Lower and upper limits of the probabilistically symmetric coverage interval	Bq·kg ⁻¹
$a^{\leftarrow}, a^{\rightarrow}, a_c^{\leftarrow}, a_c^{\rightarrow}$	Lower and upper limits of the shortest coverage interval	Bq·kg ⁻¹
k_p	Quantile of the standardized normal distribution for the probability p (for instance $p = 1 - \alpha, 1 - \beta$ or $1 - \gamma/2$)	
k_q	Quantile of the standardized normal distribution for the probability q (for instance $q = 1 - \omega - \gamma/2$ or $q = 1 - \omega \cdot \gamma/2$)	
ϕ, ϕ_x	Distribution function of the standardized normal distribution	
ω, ω_x	Auxiliary quantity	

4 Principle

Gamma-rays produce electron-hole pairs when interacting with matter. When a voltage is applied across a semiconductor detector, these electron hole-pairs are, after proper amplification, detected as current pulses. The pulse height is related to the energy absorbed from the gamma-photon or photons in the resolving time of the detector and electronics. By discriminating between the height of the pulses, a gamma-ray pulse height spectrum is obtained. After analysis of the spectrum, the various peaks are assigned to the radionuclides which emitted the corresponding gamma rays using an established detector energy calibration curve.

The activity of gamma-emitting radionuclides present in the soil samples is calculated using the established energy-dependent detector efficiency curve. These techniques allow the identification and the quantification of the radionuclides [2][3].

The nature and geometry of the detectors as well as the samples call for appropriate energy and efficiency calibrations [2][3]. True coincidence summing effects need to be considered, in particular when analysing samples with high activity levels or in applications with high detections efficiencies (e.g. when using Marinelli type containers or well-type detectors) or when the sample container is placed directly on the detector (see 10.1.5).10.1.5).

Fundamentals to gamma-spectrometry, definition and terms and generic description of gamma-spectrometry equipment are summarized in ISO 20042.

NOTE This part deals exclusively with gamma spectrometry using semiconductor detectors.

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5 Reference sources

5.1 Source(s) for energy calibration

The energy calibration of the spectrometer shall be established using one or more sources containing radionuclides that emit gamma-rays that cover the energy range of interest. Sources can be of any form but the dead time of the spectrometer for the measurements shall be such that the full energy peak shape is not distorted and pulse pile-up avoided.

The number of peaks (full energy peaks) required depends on the order of polynomial needed for the energy vs. channel calibration curve; normally 5 to 10 peaks should be sufficient. Sources containing long-lived radionuclides (for example ¹⁵²Eu, ²⁴¹Am, ⁶⁰Co or ¹³⁷Cs) are recommended for this purpose. For periodical checks of the energy calibration, a smaller number of energy peaks may be used.

5.2 Reference source(s) for efficiency calibration

5.2.1 General

The general method to calibrate the spectrometer is to establish the detection efficiency as a function of energy for a defined geometry and energy range. One or more reference sources containing single or multiple radionuclides may be used for this purpose. The activity or emission rates of the radionuclide(s) in the reference source(s) shall be traceable to national or international standards.

The energies of the emitted gamma-rays shall be distributed over the entire energy range of interest, in such a way that the energy-dependent efficiency of the spectrometer for the specific geometry can be determined in a sufficiently accurate way. For most purposes, the accuracy is sufficient for an energy range of 60 keV to 1 836 keV if a multi-radionuclide source is used containing all or most of the following radionuclides: ²⁴¹Am, ¹⁰⁹Cd, ⁵⁷Co, ¹³⁹Ce, ²⁰³Hg, ⁵¹Cr, ¹¹³Sn, ⁸⁵Sr, ¹³⁷Cs, ⁵⁴Mn, ⁵⁹Fe, ⁶⁰Co, ⁶⁵Zn or ⁸⁸Y.

For determining the activity of radionuclides emitting gamma-ray or X-rays in the energy region less than 60 keV, the spectrometry system can be calibrated using a reference source containing the radionuclides of interest.

It may be necessary to take into account true coincidence summing corrections for the calibration radionuclides (for example ⁶⁰Co and ⁸⁸Y).

5.2.2 Reference sources for laboratory gamma spectrometry systems

Reference sources for laboratory gamma spectrometry systems shall match, as closely as possible, the geometry, density and matrix composition of the samples to be measured. Reference sources may be prepared from standardized solutions or purchased as sealed sources. Only standardized solutions or reference sources that are traceable to national or international primary standards of radioactivity shall be used.

If no reference materials are available to match the samples, correction factors shall be calculated, documented and be applied to results from the measurements to take into account differences in detection efficiency due to geometry, density and matrix effects.

NOTE Reference material used for calibration should be prepared according to ISO 17034.

If a reference source is prepared by dilution from a standardized solution, the supplier's recommendation of the chemical form of the diluent shall be followed. It is also recommended that the dispensing process includes checks for possible losses of active material and on the accuracy of dispensing (for example gravimetric, volumetric and radiometric techniques should be used and cross-checked).

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For this purpose, a calibration source should have the same physical and chemical properties as the sample. It might, for instance, be produced by spiking an appropriate sample of soil. In this case, it is essential to ensure the homogeneity of the spiking soil.

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5.2.3 Reference sources used with numerical methods

Reference sources for gamma-ray spectrometry systems based on numerical models shall be used following the manufacturer's recommendations (see 9.3.2). The activity or the emission rates of the reference sources shall be traceable to national or international standards.

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6 Gamma spectrometry equipment

6.1 General description

The operation of the measurement system is as follows: in semi-conductor detectors, freed charge (the positive and negative charge carriers, holes and electrons) is generated by the interaction of ionising radiation with the detector material (through the photoelectric effect, the Compton effect or pair production). A high-voltage supply applies a bias voltage to the detector crystal resulting in an electric field. The freed charge is accelerated by the electric field towards the detector electrodes. The collected charge is converted into an output voltage pulse by a preamplifier and the output pulse is shaped and amplified by the main amplifier.

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Two types of electronic systems can be used to process the signal from the detector preamplifier; an analogue amplifier combined with digital analogue converter (ADC), or a digital signal processor (DSP) system. Both systems convert the pulse amplitude and the pulse-height histogram (spectrum) is stored using a multichannel analyser (MCA). The height of the pulse is proportional to the amount of freed charge and hence to the energy of the ionising radiation striking the detector.

The spectrum stored by the MCA shows a set of peaks (full energy peaks) superimposed on a background continuum from scattered radiation. The full energy peaks are approximately Gaussian in shape. The channel number of the peak centroid depends on the energy of the photon detected. The net full energy peak area is proportional to the number of photons of that energy that have interacted with the detector during the counting period (corrected for dead time). The net full energy peak area is normally determined in the analysis software package by one of two different techniques – summation or fitting.

For laboratory use, the spectrometer should be located in a facility with stable temperature following the manufacturer recommendations. It should be noted that changes in temperature can affect the amplifier gain, changing the energy calibration substantially.

The apparatus shall consist of the following necessary parts from 6.2 to 6.8.

6.2 Detector types

The three main geometries of germanium or other type of detectors available are planar, coaxial and well-type. Each has specific advantages depending on the circumstances. Coaxial detectors are generally used with large volume samples, whereas the well-type detectors are most efficient for small volume samples. Planar detectors can be useful for detecting photons with energies below 200 keV as they can have better energy resolution than coaxial detectors at these energies. More detailed information on the detectors is given in ISO 20042:2019, Table D.1.

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Microphonics phenomena can result in an increase in the Full Width at Half Maximum (FWHM) of the full energy peak. It may be necessary to place the detector on an anti-vibration mat.

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