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Measurement of radioactivity in the environment — Soil —

Part 3:

Test method of gamma-emitting radionuclides using gamma-ray spectrometry

Mesurage de la radioactivité dans l'environnement — Sol —

Partie 3: Méthode d'essai des radionucléides émetteurs gamma par spectrométrie gamma

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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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: [Foreword — Supplementary information](#).

The committee responsible for this document is 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 changes are

- a correction to [Formula \(4\)](#)
- etc

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 , ^3H , ^{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), ^3H , ^{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 is to be used in the context of a quality assurance management system (ISO/IEC 17025).

ISO 18589 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, ISO 18589-4 and 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.

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 people responsible for determining gamma-emitting radionuclides activity present in soil, including rock from bedrock and ore, construction materials and products, pottery, etc. using 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, 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, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 20042:2019, *Measurement of radioactivity — Gamma-ray emitting radionuclides — Generic test method using gamma-ray spectrometry*

ISO 10703, *Water quality — Gamma-ray emitting radionuclides — Test method using high resolution gamma-ray spectrometry*

ISO 11074, *Soil quality — Vocabulary*

ISO 11929-1, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 1: Elementary applications*

ISO 11929-2, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 2: Advanced applications*

ISO 11929-3, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 3: Applications to unfolding methods*

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*

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

IEC 61452, *Nuclear instrumentation — Measurement of gamma-ray emission rates of radionuclides — Calibration and use of germanium spectrometer*

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

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

m	mass of the test portion	kg
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
t_g	Test sample spectrum counting time	s
t_0	Background spectrum counting time	s
t_i	Time between the reference time and the start of the measuring time	s
t_S	Calibration spectrum counting time	s
$n_{N,E}, n_{N0,E}, n_{Ns,E}$	Number of counts in the net area of the peak, at energy E , in the test sample spectrum, in the background spectrum and in the calibration spectrum, respectively	
$n_{g,E}, n_{g0,E}, n_{gs,E}$	Number of counts in the gross area of the peak, at energy E , in the test sample spectrum, in the background spectrum and in the calibration spectrum, respectively	
$n_{b,E}, n_{b0,E}, n_{bs,E}$	Number of counts in the background of the peak, at energy E , in the test 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	
f_E	Correction factor considering all necessary corrections	
$f_{att}(E)$	Correction factor for self-attenuation at photon energy E	
$F_{att}(E)$	Attenuation factor at photon energy E	
f_d	Correction factor for decay for a reference date	
$f_{cl,E}$	Correction factor for coincidence losses (summing-out)	

$f_{su,E}$	Correction factor for summing-in effects by coincidences	
$f_{dt pu,E}$	Correction factor for dead time and pile up	
P_E	Probability of the emission of a gamma-ray with energy E of each radionuclide, per decay	
$\mu(E)$	linear attenuation coefficient at photon energy E	cm^{-1}
$\mu_m(E)$	mass attenuation coefficient, at photon energy, E	$\text{cm}^2 \cdot \text{g}^{-1}$
h	height of the sample in the container	cm
w_i	mass fraction of element i (no unit)	
ρ	bulk density, in grams per cubic centimetre, of the sample	$\text{g} \cdot \text{cm}^{-3}$
λ	Decay constant of each radionuclide	s^{-1}
$u(a), u(a_c)$	Standard uncertainty associated with the measurement result (without and with corrections)	$\text{Bq} \cdot \text{kg}^{-1}$
U	Expanded uncertainty calculated with $k = 2$.	$\text{Bq} \cdot \text{kg}^{-1}$
a^*, a_c^*	Decision threshold, without and with corrections	$\text{Bq} \cdot \text{kg}^{-1}$
$a^\#, a_c^\#$	Detection limit, without and with corrections	$\text{Bq} \cdot \text{kg}^{-1}$
$a^<, a^>$	Lower and upper limits of the probabilistically symmetric coverage interval	$\text{Bq} \cdot \text{kg}^{-1}$
$a^{<}, a^{>}$	Lower and upper limits of the shortest coverage interval	$\text{Bq} \cdot \text{kg}^{-1}$

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 response 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]}. Both coincidence and random summation effects need to be considered, particularly with container sitting directly on the detector and Marinelli type container, high activity levels or with well-type detectors used to measure small-mass samples (see 8.1.4).

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

NOTE ISO 18589 deals exclusively with gamma spectrometry using semiconductor detectors.

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 ^{152}Eu , ^{241}Am , ^{60}Co or ^{137}Cs) 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 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: ^{241}Am , ^{109}Cd , ^{57}Co , ^{139}Ce , ^{203}Hg , ^{51}Cr , ^{113}Sn , ^{85}Sr , ^{137}Cs , ^{54}Mn , ^{59}Fe , ^{60}Co , ^{65}Zn or ^{88}Y .

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 ^{60}Co and ^{88}Y).

5.2.2 Reference sources for laboratory systems

Reference sources for laboratory-based 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 standardised solutions or purchased as sealed sources. Only standardised 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.

If a reference source is prepared by dilution from a standardised solution, the supplier's recommendation on 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).

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.

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. The activity or the emission rates of the reference sources shall be traceable to national or international standards.

6 Gamma spectrometry equipment

6.1 General description

The operation of the measurement system is as follows: in semi-conductor detectors, freed charge 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.

Two types of electronic systems can be used to process the signal from the detector preamplifier; an analog amplifier combined with a digital analog converter (ADC), or a digital DSP (Digital Signal Processor) system. Both systems convert the pulse amplitude and the pulse-height histogram (spectrum) is stored using a multichannel analyzer (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 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.

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.

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 114 332 keV) and with a peak/Compton ratio between 50 and 80 for ^{137}Cs (see IEC 61452).

Some natural radionuclides, e.g. ^{210}Pb and ^{238}U through ^{234}Th , can be measured only through 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.

6.3 High voltage power supply

When in operation, the power supply should be free from spikes and dropouts, or provided with a power conditioner and/or uninterruptible power supply. Instrument earth connections should be isolated and connected to local ground at one point only to avoid ground loops that may affect the detector resolution.