
**Measurement of radioactivity —
Gamma-ray emitting radionuclides —
Generic test method using gamma-ray
spectrometry**

*Mesurage de la radioactivité — Radionucléides émetteurs de
rayons gamma — Méthode d'essai générique par spectrométrie à
rayons 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 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*, SC 2, *Radiological protection*.

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, medicine 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 similar to 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,

- a) improve the understanding of global levels and temporal trends of public and worker exposure,
- b) evaluate the components of exposure so as to provide a measure of their relative importance, and
- c) identify emerging issues that may warrant more attention and study.

While doses to workers are mostly measured directly, doses to the public are usually assessed indirectly 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), and

- 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 describes the generic requirements to quantify the activity of gamma-ray-emitting radionuclides in samples after proper sampling, sample handling and test sample preparation in a testing laboratory or in situ.

This document is to be used in the context of a quality assurance management system (ISO/IEC 17025). It forms the basis for measurement tasks using gamma-ray spectrometry, such as those set out in ISO 18589-3, ISO 18589-7, ISO 10703, ISO 13164-2 and ISO 13165-3.

This document is one of a set of generic International Standards on measurement of radioactivity such as ISO 19361.

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Measurement of radioactivity — Gamma-ray emitting radionuclides — Generic test method using gamma-ray spectrometry

1 Scope

This document describes the methods for determining the activity in becquerel (Bq) of gamma-ray emitting radionuclides in test samples by gamma-ray spectrometry. The measurements are carried out in a testing laboratory following proper sample preparation. The test samples can be solid, liquid or gaseous. Applications include:

- routine surveillance of radioactivity released from nuclear installations or from sites discharging enhanced levels of naturally occurring radioactive materials;
- contributing to determining the evolution of radioactivity in the environment;
- investigating accident and incident situations, in order to plan remedial actions and monitor their effectiveness;
- assessment of potentially contaminated waste materials from nuclear decommissioning activities;
- surveillance of radioactive contamination in media such as soils, foodstuffs, potable water, groundwaters, seawater or sewage sludge;
- measurements for estimating the intake (inhalation, ingestion or injection) of activity of gamma-ray emitting radionuclides in the body.

It is assumed that the user of this document has been given information on the composition of the test sample or the site. In some cases, the radionuclides for analysis have also been specified if characteristic limits are needed. It is also assumed that the test sample has been homogenised and is representative of the material under test.

General guidance is included for preparing the samples for measurement. However, some types of sample are to be prepared following the requirements of specific standards referred to in this document. The generic recommendations can also be useful for the measurement of gamma-ray emitters in situ.

This document includes generic advice on equipment selection (see [Annex A](#)), detectors (more detailed information is included in [Annex D](#)), and commissioning of instrumentation and method validation. [Annex F](#) summarises the influence of different measurement parameters on results for a typical gamma-ray spectrometry system. Quality control and routine maintenance are also covered, but electrical testing of the detector and pulse processing electronics is excluded. It is assumed that any data collection and analysis software used has been written and tested in accordance with relevant software standards such as ISO/IEC/IEEE 12207.

Calibration using reference sources and/or numerical methods is covered, including verification of the results. It also covers the procedure to estimate the activity content of the sample (Bq) from the spectrum.

The principles set out in this document are applicable to measurements by gamma-ray spectrometry in testing laboratories and in situ. However, the detailed requirements for in situ measurement are given in ISO 18589-7 and are outside the scope of this document.

This document covers, but is not restricted to, gamma-ray emitters which emit photons in the energy range of 5 keV to 3 000 keV. However, most of the measurements fall into the range 40 keV to 2 000 keV. The activity (Bq) ranges from the low levels (sub-Bq) found in environmental samples to activities found in accident conditions and high level radioactive wastes.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 542, *Oilseeds — Sampling*

ISO 707, *Milk and milk products — Guidance on sampling*

ISO 5500, *Oilseed residues — Sampling*

ISO 5538, *Milk and milk products — Sampling — Inspection by attributes*

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste waters*

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

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

ISO 17604, *Microbiology of the food chain — Carcass sampling for microbiological analysis*

ISO 18400-101, *Soil quality — Sampling — Part 101: Framework for the preparation and application of a sampling plan*

ISO 18400-102, *Soil quality — Sampling — Part 102: Selection and application of sampling techniques*

ISO 18400-103, *Soil quality — Sampling — Part 103: Safety*

ISO 18400-104, *Soil quality — Sampling — Part 104: Strategies*

ISO 18400-107, *Soil quality — Sampling — Part 107: Recording and reporting*

ISO 18400-202, *Soil quality — Sampling — Part 202: Preliminary investigations*

ISO 18400-203, *Soil quality — Sampling — Part 203: Investigation of potentially contaminated sites*

ISO 18400-204, *Soil quality — Sampling — Part 204: Guidance on sampling of soil gas*

ISO 18400-205, *Soil quality — Sampling — Part 205: Guidance on the procedure for investigation of natural, near-natural and cultivated sites*

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 18589-7, *Measurement of radioactivity in the environment — Soil — Part 7: In situ measurement of gamma-emitting radionuclides*

ISO 24333, *Cereals and cereal products — Sampling*

ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

background continuum

events in the spectrum that form a smooth curve onto which the photopeaks are superimposed

Note 1 to entry: The continuum may arise from gamma-rays scattered inside the test sample or any surrounding materials, from cosmic radiation or from radionuclides in the surrounding materials.

3.2

blank sample

sample of a similar material to the test sample but containing radioactive impurities negligible in comparison with the test sample

3.3

calcination

thermal treatment of the powder in order to remove volatile impurities or to change the density or specific surface area of the powder

[SOURCE: ISO 13779-6:2015, 3.4]

Note 1 to entry: Calcination is commonly used for samples such as soil.

3.4

comminution

operation of reducing particle size by crushing, grinding or pulverisation

3.5

dead time

time during spectrum acquisition (real time) during which pulses are not recorded or processed

Note 1 to entry: Dead time is given by real time minus live time.

Note 2 to entry: The time is given in seconds.

3.6

decision threshold

value of the estimator of the measurand, which when exceeded by the result of an actual measurement using a given measurement procedure of a measurand quantifying a physical effect, one decides that the physical effect is present

[SOURCE: ISO 11929:2010, 3.6]

3.7

detection efficiency

probability that a gamma-ray emitted at a particular energy (keV) in the decay of a radionuclide in a test sample is detected in the photopeak corresponding to that energy

3.8

detection limit

smallest true value of the measurand which ensures a specified probability of being detectable by the measurement procedure

[SOURCE: ISO 11929:2010, 3.7]

3.9

fractionation

separation of a product into several fractions by an appropriate technique such as distillation or crystallization

[SOURCE: ISO 1998-4:1998, 4.20.300]

3.10

full width half maximum

FWHM

width of a gamma-ray photopeak at half the maximum of the photopeak distribution

Note 1 to entry: The width is given in kiloelectronvolts.

3.11

in situ

use of a portable gamma-ray spectrometer for the direct measurement (e.g. in the environment and buildings) for determination of activity such as per unit of surface area or per mass unit of gamma-emitting radionuclides present in or deposited on the soil surface or content of large items such as waste drums

3.12

live time

time during which pulses are processed during an acquisition (real) time

Note 1 to entry: The time is given in seconds.

3.13

net photopeak area

area (number of counts) observed in the photopeak

3.14

pathlength

distance a photon travels through matter

3.15

peak-to-Compton ratio

ratio of the number of counts in the biggest channel of the 1 332,5 keV ⁶⁰Co peak to the average number of counts in the channels representing the range from 1 040 through 1 096 keV

[SOURCE: 325-1996-IEEE Standard Test Procedures for Germanium Gamma-Ray Detectors]

3.16

percolation

separation technique to enrich selective ions of one element (e.g. by ion exchange or precipitation)

3.17

photopeak

peak observed above the background continuum in a gamma-ray spectrum due to events that deposit the full energy of the photon in the detector material, usually approximately Gaussian in shape

3.18

radionuclide

radioactive nuclide

[SOURCE: IEV 881-02-36]

3.19

real time

time taken to acquire a spectrum

Note 1 to entry: The time is given in seconds.

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3.20**reference source**

source containing one or more radionuclides in solid, liquid or gaseous form, sealed in a suitable container, of known activity (Bq, Bq·g⁻¹ or Bq·ml⁻¹), prepared such that the activity is traceable to national or international primary standards of radioactivity

3.21**region of interest**

part of the spectrum that brackets a photopeak

3.22**spectrometry system**

complete assembly of the sensor and associated pulse-processing electronics that converts the gamma-rays detected by the sensor into a pulse-height spectrum

3.23**test sample**

artefact (for example sample of soil in a plastic container) for measurement of the content of gamma-ray emitting radionuclides

3.24**true coincidence summing**

simultaneous detection of two or more gamma-rays in the spectrometry system, due to the emission of a cascade of gamma-rays in the decay of a single nucleus in the test sample

4 Symbols and units

For the purpose of this document, the following symbols apply.

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Table 1 — Symbols and units
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A	Activity (Bq) of each radionuclide in the calibration source at the time of calibration (t_c).
a, a_m	Activity (Bq) of radionuclide in the sample, activity per unit mass (Bq·kg ⁻¹), in the sample
a^*	Decision threshold (Bq)
$a^\#$	Detection limit (Bq)
\tilde{a}	True value of the activity (Bq)
ε_E	Detection efficiency at energy, E
f_d	Factor to correct for the radioactive decay during the counting time, t and t_i
P_E	Probability of the emission by a radionuclide of a gamma-ray with energy, E , per decay
λ	Decay constant of a radionuclide (s ⁻¹). The decay constant equals $\ln 2 \cdot t_{1/2}^{-1}$
m	Sample mass (kg)
$n_{N,E}$	Number of counts in the net area of the photopeak at energy, E , in the sample spectrum
$n_{Ns,E}$	Number of counts in the net area of the photopeak at energy, E , in calibration spectrum
u	Standard uncertainty associated with the measurement result (Bq)
U	Expanded uncertainty calculated by $U = k \cdot u$ where k is the coverage factor (Bq)
t	Sample spectrum counting time (live time) (s)
t_i	Time between the reference time for the results and the start of the count time (s)
t_s	Calibration spectrum counting time (live time) (s)
$t_{1/2}$	Half-life of a radionuclide (s)
V	Sample volume (m ³)

5 Principle

5.1 General

The activity of gamma-ray emitting radionuclides in test samples is commonly determined using high resolution gamma-ray spectrometry techniques based on the analysis of the energies and the areas of the photopeaks. These techniques allow the identification and the quantification of the radionuclides and are normally performed by the analysis software.

NOTE Lower-resolution detectors, such as sodium iodide or other scintillation materials, can be used for the measurement of radioactivity in test samples in certain cases (see ISO 19581). For example, low-resolution detectors are useful for rapid screening of samples of foodstuffs in the case of a nuclear incident but high-resolution spectrometry is essential for samples that can contain complex mixtures of radionuclides, such as environmental samples.

The nature and geometry of the detectors as well as the test samples call for appropriate energy and efficiency calibrations. For 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. The pulse amplitude is converted to a digital value by an analog-to-digital converter (ADC) 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 sensitive volume of the detector. Digital data acquisition systems are also available that carry out the same function as the analogue electronics.

The spectrum stored by the MCA shows a set of peaks (photopeaks) superimposed on a background continuum from scattered radiation; Reference [21] contains examples of gamma-ray spectra. The photopeaks are approximately Gaussian in shape. The channel number of the photopeak centroid depends on the energy of the photon detected. The net photopeak 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 photopeak area is normally determined in the analysis software package by one of two different techniques – summation or fitting.

5.2 Summing method

The number of counts in the photopeak is calculated by summing the total number of counts in a region of interest around the photopeak and subtracting counts in the background continuum. The total number of counts is given by:

$$N = \sum_{i=L}^H C_i \quad (1)$$

where

N is the total number of counts from channel L (lowest) to channel H (highest) in the region of interest;

C_i is the number of counts in channel number i .

Assuming the background continuum under the photopeak is linear, the background in the same region of interest is given by:

$$B = \frac{n(C_L + C_H)}{2} \quad (2)$$

where

B is the number of counts in the background from channel L to channel H ;

n is the number of channels in the region of interest ($n = H - L + 1$).

The net photopeak area is given by $N - B$ and the standard uncertainty in the photopeak area (assuming a Poisson distribution for the contents of each channel) is given by:

$$u = \left(\sum_{i=L}^H C_i + \frac{n^2 (C_L + C_H)}{4} \right)^{1/2} \quad (3)$$

Different software packages use different methods to determine the upper and lower bounds of the region of interest and the shape of the background function. The region of interest shall be selected carefully, particularly when the photopeak is near to discontinuities in the spectrum, near another photopeak or located on a high background continuum (see Reference [2]).

The photopeak position is generally determined from the net counts in each channel:

$$C_h = \frac{\sum_{i=L}^H i \cdot C'_i}{\sum_{i=L}^H C'_i} \quad (4)$$

where

C_h is the photopeak position (channel);

C'_i is the net count in channel i .

5.3 Fitting method

In this method, the net photopeak area is determined by non-linear least squares fitting of an analytical function to the counts in the region of interest. The analytical function for an individual photopeak is normally Gaussian, but some approaches include one or more exponential tails to approximate the photopeak shape more closely. The net photopeak area and photopeak position are determined from the values of the fitted parameters. Further details on the uncertainty in the photopeak area using this approach are given in Reference [2].

The fitting method shall be used to determine the net areas of overlapping photopeaks in a spectrum.

The radionuclides in the test sample may be identified from the energies of the photopeaks present; the activity (Bq) in the test sample may also be determined from the count rate observed in the photopeak, corrected for factors such as detection efficiency, gamma-ray-emission probability and decay. Care shall also be taken to apply corrections for effects not covered by many commercial spectrum analysis software packages, such as true coincidence summing.

NOTE This description applies to semi-conductor detectors including CdZnTe but similar principles can also be applied to other detectors [NaI(Tl), LaBr₃(Ce), CeBr₃, etc.].

6 Validating measurements by gamma-ray spectrometry

6.1 General

This subclause describes the steps to be followed from setting out the customer requirements and selecting the equipment through to operation and maintenance, as also required by ISO/IEC 17025. Documented evidence shall be available to demonstrate that the measurement procedures meet customer requirements. The validation process is summarized in [Figure 1](#).