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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, ^{3}H, ^{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), ^{3}H, ^{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.

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 ^{60}Co 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.

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.

Table 1 — Symbols and units

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 $\ln 2 \cdot t_{½}^{-1}$
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)
$t_{½}$	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:

$$N = \sum_{i=L}^{H} C_i \tag{1}$$

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:

$$B = \frac{n\left(C_L + C_H\right)}{2} \tag{2}$$

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:

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

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:

$$C_h = \frac{\sum_{i=L}^{H} i \cdot C_i'}{\sum_{i=L}^{H} C_i'} \tag{4}$$

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), $LaBr_3$(Ce), $CeBr_3$, 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.

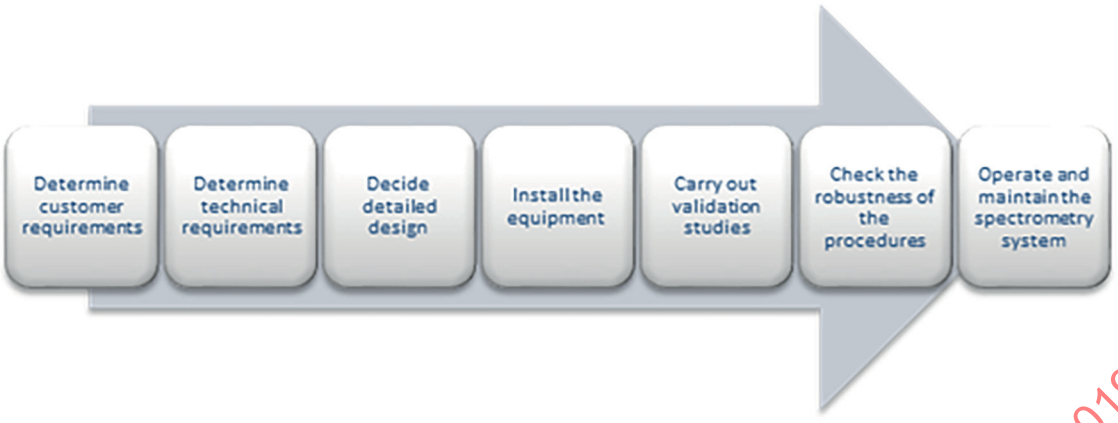


Figure 1 — Schematic diagram of validation process

6.2 Step 1: customer requirements

The specification for the spectrometry system shall be defined, including the energy range, characteristic limits, maximum activity and uncertainty required by the customer. In consultation with other members of staff as appropriate, the specification shall also take into account:

— compatibility with existing equipment;

— location (for example proximity to high activity radioactive sources, accelerators, reactors);

— environmental conditions;

— training requirements;

— documentation needed;

— sample throughput and turnaround times;

— activity of samples;

— measurement uncertainties required.

The user requirements may also be set by the customer or local, national or international requirements in terms of pass/fail criteria for the samples (for example testing if the activity content of a drinking water sample meets national regulations). The characteristic limits required should be estimated and the value for the detection limit shall be lower than the required limits.

6.3 Step 2: technical requirements

The technical requirements for the spectrometry system shall be defined to meet the requirements set out in Step 1 (see 6.2). These requirements shall include:

— characteristics of detector [for example detector type (see Annex D), detector geometry, detector window material, use of selected low-background materials];

— type of cooling system if required (liquid nitrogen, electro-cooling system or electro-mechanical cryocoolers);

— shielding and other methods to reduce background radiation;

— sample positioning system/auto-sample changer;

— environmental conditions;

— type of signal processing electronics (analogue or digital);

— type of preamplifier (resistive feedback or reset-type feedback);

— power supply requirements;

— software requirements for data collection, analysis and storage (duration);

— minimizing correction factors to meet requirements for measurement uncertainties (see Annexes B, C and F).

It is recommended that the following issues are taken into account in the technical requirements (see also References [3] and [24]):

a) The detector should normally be shielded from all sides (including the base), to reduce background signals originating from cosmic radiation, naturally occurring radionuclides from building materials or from other sources of ionising radiation such as an accelerator. It is usually sufficient to shield the detector with 10 cm thick, low-background lead walls. The walls may be lined with cadmium and copper sheets or other materials, to attenuate fluorescence X-rays from interactions of the gamma-rays with the lead, particularly if measurements below 100 keV are required.

b) Reduction of the radon inside the shield is desirable for measuring low-activity samples. This may be achieved by displacing the air inside the shield with nitrogen boiled off from the cooling Dewar.

c) Steps to reduce the background include using selected low-background lead, low-background materials for the detector itself, forced ventilation of the laboratory and air filtration. An anti-coincidence shield may also be used.

d) 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 Annex D and Reference [4].

e) For laboratory use, the spectrometer should be located in a facility with a stable ambient temperature. It should be noted that changes in ambient temperature can affect the amplifier gain, changing the energy calibration significantly. The power supply should be clean (free from spikes and dropouts) and stable, or provided with a power conditioner or uninterruptible power supply. Instrument grounds should be isolated and connected to local ground at one point only to avoid ground loops that may affect the detector resolution.

f) The specification should include the requirement for an automatic high voltage cut-off and alarm signal which is activated if the temperature of the crystal increases due to a malfunctioning of the cryostat or electrocooling system, or failure to maintain the correct level of liquid nitrogen in the dewar.

g) Due to the risk of anoxia in laboratories where liquid nitrogen is handled, sufficient air changes, secondary gas removal (such as a gas extraction hood) and/or an oxygen depletion alarm are recommended.

h) Depending on the required accuracy and the desired detection limit, it is generally necessary to use high-quality detectors whose energy resolution (FWHM) is less than 2 keV (for the ^{60}Co photopeak at 1,3 MeV) and with a peak-to-Compton ratio between 50 and 80 for the ^{60}Co photopeak.

i) The characteristic limits required should be taken into account in setting the technical specification (which determines the detector type and size, geometry and shielding needed). It may be necessary to refer to the scientific literature to estimate the characteristic limits or to previous experience with similar detectors.

j) Care shall be taken to ensure that the reference sources and test samples do not contaminate the detector, sample holders or shields. A protective film on the source holder may be useful.

k) Microphonics can result in an increase in the FWHM of the photopeaks; it may be necessary to place the detector on an anti-vibration mat.

6.4 Step 3: detailed design

Based on the above specification, identify the instruments and software available. The detailed design for the system shall be documented, listing the manufacturer and model numbers for the components chosen. A comparison with the initial specification and the justification for any deviations from the specification shall be made and documented.

6.5 Step 4: installation

The installation of the system shall be documented and retained, including:

— record of equipment type, manufacturer, location, serial number;

— parts check list;

— surface finish inspection;

— a record that utilities provided on site have the capacity for the equipment (for example electrical power supply);

— properties of the shielding (if applicable);

— a record of documents, including as-built drawings, maintenance program, manual and results from supplier's tests.

6.6 Step 5: validation studies

Tests shall be carried out and documented to demonstrate that the spectrometry system meets the specification and is fit for purpose. Appropriate statistical tests (such as the t-test) shall be used to demonstrate conformance with specification. The main characteristics that shall be tested include (see also References [3] and [24]):

— energy resolution (FWHM), which enables the detector to separate two neighbouring photopeaks. This test shall be carried out over the energy range of interest for measurements;

— detection efficiency, which specifies the number of photons detected in the photopeak relative to the number of photons emitted;

— the effects of variation in sample preparation, geometry and composition;

— the reproducibility of sample positioning;

— the effects of variation of ambient environmental conditions (e.g. temperature) on results (this may be achieved by monitoring results from quality control checks over a long period);

— characteristic limits;

— accuracy of the dead-time and pile-up correction technique chosen for the specified range of count rates (see B.4).

A maintenance and quality control program shall be defined, along with the operating procedure (further details of the quality control program are given in Annex A). Other tests can be carried out as recommended by the manufacturer, such as the peak-to-Compton ratio.

6.7 Step 6: robustness

Tests shall be conducted and documented to demonstrate that the spectrometry system meets the specification in routine use. This should include demonstrating consistent results for samples assayed by different members of staff under different conditions.

Authorisation for the spectrometry system to be placed into service shall be documented.

6.8 Step 7: operation and maintenance

Tests shall be implemented following the defined program and the results recorded to demonstrate that the spectrometry system continues to meet the specification. These tests may include:

— quality control tests of the energy calibration, the detection efficiency and the FWHM, covering the energy range of interest;

— measurement of the background spectrum;

— participation in proficiency test exercises;

— in-house cross check programme;

— detailed quality tests of the detection efficiency and comparison to previous measurements/ calibrations, which covers more photopeaks than in the frequent quality control tests.

The detection efficiency shall also be measured following a repair of the measurement set-up, a long warm up, or a major software or hardware change that could have an impact. This includes also any changes to the shielding or the sample properties (e.g. the sample container, geometry, physical or chemical form) that could impact the detection efficiency. If any significant changes are observed the spectrometer shall be recalibrated.

The spectrometer shall also be recalibrated at a defined frequency, the interval between calibrations taking into account any drifts observed in the quality control checks, decay of the check sources, the application (including local regulations) and the requirement to ensure staff skills are maintained.

It is considered best practice to perform a detector efficiency calibration once every 1 year to 5 years, and to compare the detector efficiency curves to those from previous calibrations. If there are any differences greater than 5 %, the cause should be investigated and correction action taken.

If numerical methods are used to calibrate the detector, the manufacturer's recommendations for recalibration shall be followed.

It is recommended that the quality control tests are conducted at least weekly. More frequent tests are recommended if samples containing low activities are to be measured, as there may be no photopeaks in the spectrum to use as internal checks.

7 Nuclear decay data

7.1 Recommended nuclear decay data

Nuclear decay data (half-lives, gamma-ray energy and emission probabilities) are available from the scientific literature, databases and other publications. There are often differences between the values depending on the original data used and the evaluation method.

To ensure consistent results from gamma-ray spectrometry measurements, nuclear decay data used for instrument calibration or for estimating the activity content of samples, should therefore be taken from the Decay Data Evaluation Project (http://www.lnhb.fr/nuclear-data/). If no evaluation of the

nuclear decay data of the radionuclide of interest is available in this database, the following databases should be consulted:

— Joint Evaluated Fission and Fusion (JEFF) Library (http://www.oecd-nea.org/dbdata/);

— National Nuclear Data Center database (https://www.nndc.bnl.gov/);

— Evaluated Nuclear Data File (ENDF) (https://www-nds.iaea.org/exfor/endf.htm).

7.2 Selection of gamma-ray photopeaks for inclusion in spectrum analysis libraries

Most gamma-ray spectrometry analysis packages require the user to select in advance the radionuclides for analysis, the gamma-ray emissions and those to be used in estimating the activity of the radionuclides present. This information is recorded in a spectrum analysis library and the reasons for the selection should be documented. The following criteria should be used to select gamma-ray photopeaks for inclusion in the spectrum analysis library:

— intense photopeak with low uncertainty on emission probability;

— well separated (several FWHM) from other photopeaks in the decay of the radionuclide, photopeaks from other radionuclides present in the sample or present in the background spectrum, and artefacts in the spectrum (such as backscatter peaks, escape peaks and Compton edge);

— in an energy region where the calibration of the instrument is well known (typically above 300 keV);

— low correction for true summing or accurately estimated correction.

Use of the 511 keV photopeak from positron annihilation should be avoided for the following reasons: annihilation may take place outside of the sample so the geometry of the sample is ill-defined; positron annihilation-in-flight shall be corrected for; the photopeak is broadened due to the Doppler effect so the net photopeak area may not be determined correctly by the software package; high energy photons produced in other decays or by other radionuclides may result in pair production in the sample or surrounding materials, resulting in additional counts in the 511 keV photopeak.

The use of X-rays should also be avoided in estimating sample activities. Due to the very short lifetime of the excited atomic states, Lorentzian broadening of the photopeaks can be significant resulting in low- and high-energy tails on photopeaks; as a result, photopeak analysis software can underestimate the photopeak area if these tails are omitted from the model used. As the X-rays are element rather than radionuclide specific, X-rays may originate from more than one radionuclide in the sample.

Additional photopeaks not meeting the above criteria should be included in the spectrum analysis library to ensure that interferences from other radionuclides are subtracted from the radionuclide of interest. However, only photopeaks meeting the above criteria should be used in the calculation of the radionuclide activity. It is recommended that only one photopeak is used to calculate the radionuclide activity in order to simplify the calculation of the measurement uncertainty.

7.3 Decay chains

The algorithm used by the analysis software to determine the activities of radionuclides present in the sample shall be reviewed to assess how the activity of radionuclides in decay chains is calculated. If no correction is made for decay chains, the activities of the radionuclides affected may have to be calculated manually (see Reference [5]). Alternatively, if radionuclides in a decay chain are in equilibrium with a long lived parent radionuclide, the half-life of the daughter radionuclides may be set equal to that of the parent radionuclide.

Errors may result in an analysis if this effect is not considered, as the analysis software may reject the presence of radionuclides with very short half-lives or calculate their activity based on the short half-lives.

8 Detector energy and efficiency calibration

8.1 Energy calibration

For reliable radionuclide identification, the energy calibration should be accurate to better than 0,2 keV over the energy range (see A.6). The energy calibration of the spectrometer shall therefore be established using one or more sources containing radionuclides that emit gamma-rays that cover the energy range of interest. This calibration establishes the relationship between the channel number of the photopeak centroid and the known energy of the photons. This task is normally carried out using commercially-available software to determine the photopeak centroid (channel) and to match this to the energy (keV). The software also determines the full width half maximum of the full energy photopeaks. This information is used by the photopeak search algorithm and for the calculation of characteristic limits.

The count rate in the detector shall be such that the photopeak shape is not distorted due to pulse pile-up; a dead time less than 10 % normally suffices. For a Germanium spectrometer, the photopeak position should be determined with an accuracy of better than 0,2 keV.

NOTE A photopeak area of approximately 50 000 counts normally meets this requirement for a Germanium spectrometer.

A calibration curve (energy vs channel) should be calculated by least-squares fitting, so that photopeak energies may be interpolated. A linear or quadratic function is normally sufficient but higher order polynomials may be required. A similar process should be used to determine the full width half maximum as a function of energy.

Care shall be taken when extrapolating the energy calibration curve. Photopeaks in the background spectrum or X-rays should be used to check the accuracy of the extrapolated curve, and the extrapolation should be plotted to check for large deviations from a linear function due to quadratic or higher order components.

8.2 Efficiency calibration

The following approaches may be considered for determining the detection efficiency:

— direct comparison with a reference source of the same radionuclide in the same matrix and geometry. This approach is appropriate if the aim of the measurement is to determine the activity of a single radionuclide or if the photon energy is in an energy region that is difficult to calibrate (typically 20 keV to 100 keV). It may also be required if true coincidence summing corrections are significant (for example, when measuring a gamma-ray emitting radionuclide with a complex decay scheme using a high efficiency detector such as a well detector);

— measurement of the full energy photopeak detection efficiency as a function of energy;

— calculation of the full energy photopeak detection efficiency as a function of energy by Monte Carlo simulation or other modelling technique. Such numerical models are sensitive to input parameters such as the detector dimensions, and therefore shall be checked using at least one reference source containing radionuclides that emit gamma-rays covering the energy range of interest. If a discrepancy is found between efficiency calculated using the model and the reference source, the discrepancy shall be investigated and corrections applied to the numerical model.

The same algorithm for analysis of the spectrum shall be used for both calibration and sample measurement.

The detection efficiency is affected by the following factors:

— the detector;

— the geometry of the sample with respect to the detector (solid angle);

— the density of the sample and the sample container characteristics;

— the sample mass and chemical composition;

— the heterogeneity of the sample matrix with respect to activity, density and chemical composition.

When one of these factors is changed, the detection efficiency shall be re-evaluated for the new conditions.

If no numerical model is used, the test sample measurement shall be performed with comparable measuring conditions as used for calibrating the gamma-ray spectrometry system. In particular, the measurement geometry, the position of the reference source in relation to the detector and the test sample and reference source matrices should be identical. Corrections should be applied to account for differences in the matrix and other factors that can affect the detection efficiency, as appropriate. If the efficiency calibration method uses a numerical model, the parameters used to describe the sample shall match the item being measured.

In most practical measurements, the instrument dead time for the calibration measurements is higher than for the sample measurements. The activity of the efficiency calibration source shall be such that count rate does not distort the photopeak shape and that an accurate dead-time correction is applied by the analysis software; a dead time of less than 5 % normally suffices. Higher dead times may be used but evidence of the accuracy of the dead-time correction shall be required.

The photopeaks in the spectrum shall contain sufficient counts such that the uncertainty in the photopeak area is small in comparison with other components of uncertainty. A photopeak area of 10 000 counts is normally sufficient. The photopeak shall be discarded from the efficiency calibration data set if visual inspection shows that it is not Gaussian in shape.

The detection efficiency for each photopeak in the calibration spectrum shall be calculated as follows:

$$\varepsilon_E = \frac{n_{N_{s,E}}}{A \cdot P_E \cdot t_s} \tag{5}$$

NOTE This formula assumes negligible decay of the calibration source during the acquisition time.

The detection efficiency as a function of energy should be calculated and, if required, a function fitted to enable detection efficiency to be interpolated. The detection efficiency is a rapidly varying function of energy and software packages offer a choice of different functions. The selection of the fit function shall be based on an evaluation of the goodness of fit parameter (chi-squared) and visual inspection of the function to confirm that the fitted function does not deviate from a smooth curve due to using too many variable parameters. Extrapolation of the efficiency calibration function to energies below or above the range of the calibration points shall not be used unless supported by other evidence such as modelling.

8.3 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 photopeak shape is not distorted and pulse pile-up avoided.

The source(s) shall emit gamma-rays spread across the energy range of interest. The number of photopeaks to use depends on the order of polynomial needed for the energy vs. channel calibration curve; normally 5 to 10 photopeaks 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 photopeaks may be used.

8.4 Reference source(s) for efficiency calibration

8.4.1 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).

8.4.2 Reference sources for laboratory systems

Reference sources for laboratory-based spectrometry systems shall match, as closely as possible, the geometry, density and matrix 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).

8.4.3 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.

9 Sample container

Sample containers shall be used taking into account the following factors:

— reproducibility of measurements (Annex F contains information on the impact of sample geometry and positioning on reproducibility);

— the effect of sample geometry and self-absorption;

— optimisation of detection efficiency (for example the use of Marinelli beakers to maximize detection efficiency for low activity samples or the use of jigs to position high activity samples at a fixed distance from the detector to reduce dead-time effects);

— the risk of evaporation of solutions and the loss of volatile elements such as iodine or radioactive gases such as radon;

— “radiopurity” (little or no radioactive contamination) of the container materials;

— the risk of chemical reactions with the test sample or reference material;

— ease of filling (a wide-necked opening is recommended);

— transparency, with a clear fill line to ensure consistent fill height;

— robustness if dropped accidently (any contamination of the detector or the surrounding materials is extremely difficult to remove);

— absorption of low-energy gamma-rays and X-rays in the container material (use of thin material to maximize detection efficiency, or a thicker material to absorb low energy gamma-rays and X-rays to reduce corrections for true summing or dead time).

If containers are re-used, they shall be cleaned thoroughly to ensure sample integrity and to avoid the risk of cross-contamination. It may also be useful to place containers in clean, thin plastic bags to reduce the risk of contaminating work surfaces or the spectrometer.

10 Procedure

10.1 Sample measuring procedure

10.1.1 Sampling

Samples shall be collected in accordance with the relevant standard (See Table 2).

The following information shall be recorded:

— a unique identifier for the sample;

— location;

— reference to the sampling procedure followed;

— sampling date and time;

— sample description (physical, chemical, biological form, mass and volume, as appropriate);

— name of the operator.

Special handling of samples may be required to avoid degradation if there is risk of a long delay (several days) between sampling and measurement. If samples are perishable, it is recommended that they are transported and stored in the absence of light and at a temperature between 1 °C and 5 °C before processing and measurement.

10.1.2 Sample preparation

The techniques for sample preparation shall be documented. All the information gathered during sampling as described in 10.1.1 shall be handed over to the personnel responsible for sample preparation, measurements and analysis. Additional information may be provided to the analyst on the sampling procedure (date, location, etc.), in order to inform the spectrum analysis process.

Samples shall be prepared following the requirements of the standards listed in Table 2; if no relevant standard is available samples shall be prepared for measurement in accordance with the customer’s requirements, the radionuclides analysed, the risk of loss of radionuclides of interest during preparation, the sample container chosen and the desired measurement sensitivity. Alternative sample preparation procedures can be used but shall be validated in compliance with the requirements of ISO/IEC 17025.

Consideration shall be given to ensuring homogeneity, optimising the sample geometry, minimising the density correction, minimising the loss of activity (for example, of volatile radionuclides) and

the shelf life required. Objects in the sample which could cause heterogeneity shall be removed and measured separately if necessary to determine if the object is not representative of the sample and contains significantly higher or lower activity. The effect of inhomogeneity shall be assessed and taken into account in the uncertainty budget. If there is a requirement, samples may be fractionated (e.g. according to particle size, solid or colloidal phase). The preparation technique may also depend on the sample size available.

Solid samples may be measured as supplied, dried or ashed. Oven-drying and freeze-drying, calcination, and fractionation and comminution can be used.

Liquid samples may be measured as supplied, concentrated or diluted. Concentration by evaporation, percolation using ion exchange resin or concentration by precipitation using selective precipitating agents can be used.

Gaseous samples may be measured as supplied in gas-tight containers under atmosphere pressure or robust, vacuum-tight and pressure-tight containers with special couplings for filling and measuring under pressure.

The sample condition (fresh, dry, or ashed) shall be recorded so the analyst can determine the appropriate calibration to use and correction factors for self-absorption to apply.

NOTE Similarly, the concentration level (fresh/dry weight ratio, liquid concentration) and radionuclide activity levels should be specified to identify situations deviating from routine laboratory measurements.

If a concentration technique is used such as evaporation or percolation, the chemical yield of process for the relevant elements shall be determined including its uncertainty and variability.

Table 2 — Standards for sampling and sample preparation of different types of sample

Type of Sample	Standard references
Water	
Potable water, seawater, rainwater and ground water	ISO 10703 ISO 5667-1
Waste water, sewage sludge	ISO 5667-10
Food	
Milk and milk products	ISO 707 ISO 5538
Meat	ISO 17604
Cereals and cereal products	ISO 24333
Vegetation	
Oil seeds	ISO 542
Oil seeds residue	ISO 5500
Soil, rock, bedrock, building materials	
Soil, rock, bedrock, building materials	ISO 18589-2
Soil from gardens, farmland, urban or industrial sites that can contain building materials rubble, as well as soil not affected by human activities	ISO 18400-101 ISO 18400-102 ISO 18400-104 ISO 18400-107 ISO 18400-202 ISO 18400-203 ISO 18400-205

Table 2 *(continued)*

Type of Sample	Standard references
Soil gas	ISO 18400-204
In situ measurement	
Surface contamination and contamination of soil (environment) and of walls of a building (decommissioning)	ISO 18589-7

10.1.3 Loading the sample container

The sample container shall be loaded as follows (see also ISO 18589-3):

— fill the container to the same height as that used for the calibration source. The filling height and flatness shall be checked by eye and material added until the reference line is reached. A correction factor may be required if there is insufficient material to fill the container up to the desired height (Annex F may be consulted to estimate the order of magnitude of the effect). A mechanical filling device such as a compactor, tapping device or vibrating table can be used to ensure consistent filling by flattening-out the filled volume;

— record the sample mass and/or volume;

— seal the container;

— clean the outside of the container to remove potential contamination during the filling process. The container may be sealed in a clean plastic bag to reduce the risk of contaminating the detector and the sample holder.

When measuring gaseous decay products the sealed container shall be stored long enough to allow radioactive equilibrium to be reached.

10.1.4 Recording the sample spectrum

The test sample shall be placed in the counting chamber for measurement in an accurately reproducible position, at the same position as the calibration source. The spectrum shall be recorded, along with the start time and date of the measurement. The dead time of the instrument shall be within the range of dead times covered in the validation process; if the dead time is higher than this value, a positioning jig can be constructed to increase the source to detector distance but a new detection efficiency calibration shall be carried out for this position.

The spectrum shall be acquired until the required characteristic limit is reached or there are sufficient counts in the net photopeak area(s) to meet the required uncertainty, taking into account the requirement to subtract the background spectrum. This may take many hours.

10.2 Analysis of the spectrum

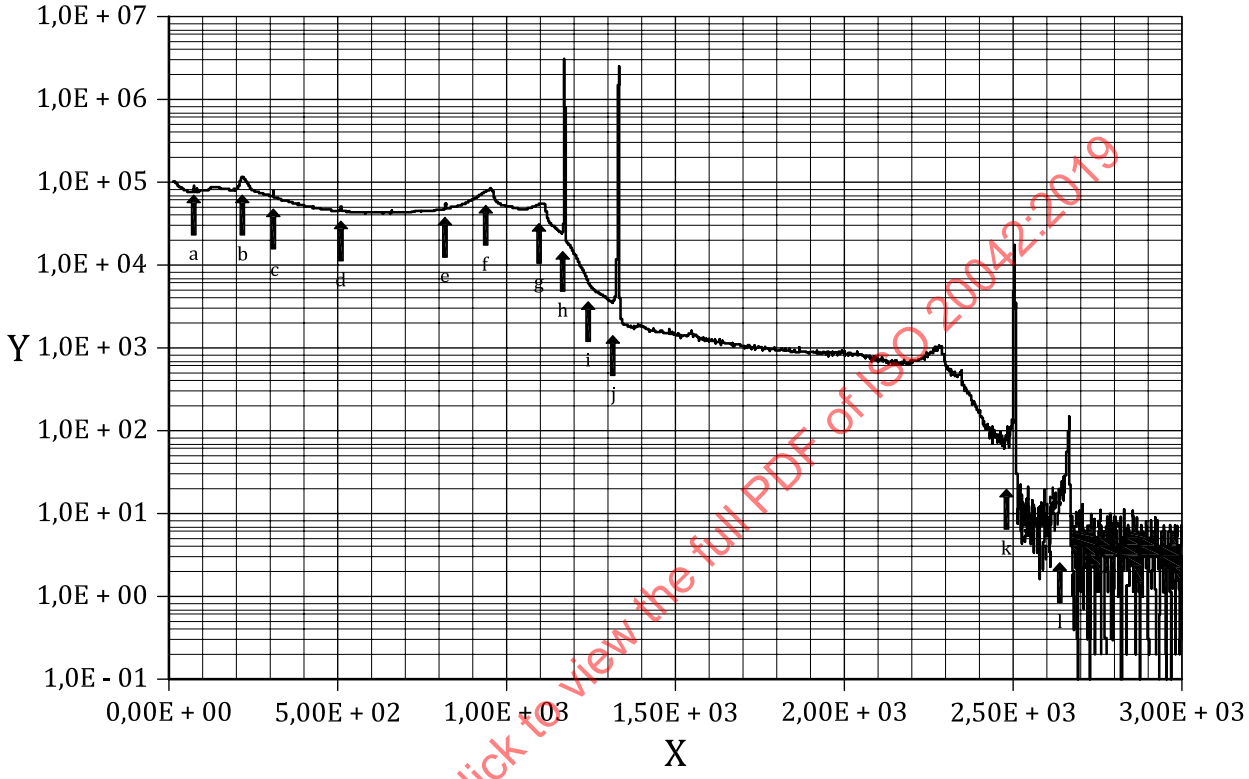
10.2.1 Procedure for laboratory-based measuring systems

The energy and the net photopeak areas of the photopeaks in the spectrum shall be calculated to determine the sample activity or sample activity concentration using the same spectrum analysis software as that used to calibrate the instrument. Deconvolution of overlapping photopeaks may require manual adjustment of analysis parameters to ensure a good fit to the data.

Photopeaks shall be used to identify the radionuclides in the sample. The identification should take into account the accuracy and stability of the energy calibration, the accuracy of the determination of the energy of the photopeaks in the sample spectrum and any additional information available on which radionuclides may be present in the sample. Possible radionuclides can be included in the nuclear data library used for the analysis.

Depending on the application and local regulations, it is recommended that all spectra are inspected visually to ensure that all the photopeaks in the spectra have been identified and overlapping photopeaks de-convoluted correctly.

Artefacts in the spectrum may be observed and should be identified, such as sum photopeaks, fluorescent X-rays from shielding materials, escape photopeaks and Compton edges (see Figure 2). Detailed information can be found in Reference [4] and the scientific literature.



Key

X	energy, in keV	f	Compton edge from 1 172 keV photopeak.
Y	counts	g	Compton edge from 1 333 keV photopeak.
a	Lead X-rays from shield.	h	1 172 keV photopeak.
b	Backscatter peak.	i	Summing.
c	Double escape peak.	j	1 333 keV photopeak.
d	Positron annihilation (511 keV).	k	Sum peak (1 172 + 1 333 keV).
e	Single escape peak.	l	Sum peak (2 × 1 333 keV).

Figure 2 — Typical spectrum from a high resolution gamma-ray spectrometer

Figure 2 shows the number of counts versus energy (keV) from a ^{60}Co source. The Gaussian-shaped photopeaks are shown (footnotes h and j), along with the background continuum and examples of spectrum artefacts. Further information on the origin of the artefacts is given in Reference [5].

10.2.2 Background corrections

Areas of photopeaks used for evaluating activities of radionuclides shall be corrected for the background contribution, taking into account any difference in the duration of the sample and background measurements.

The detector, the sample container or the detector surroundings may be contaminated or may contain naturally-occurring radionuclides (see also ISO 18589-3). This can give rise to photopeaks in the spectrum not originating from the sample. For this reason, a background spectrum shall be recorded at

frequent defined intervals for subtraction from the sample spectrum and to identify any contamination of the detector so that it may be cleaned, if required.

Additional advice to take into account during the measurement of natural radionuclides in soil and information on spectroscopic interferences is given in ISO 18589-3.

Large samples may shield the detector from the ambient background radiation. The background spectrum should be obtained using a blank sample, in the same geometry as the samples to be measured.

The background acquisition time shall be greater than or equal to the acquisition time used for sample measurements.

The background spectrum may be analysed to identify the origin of the photopeaks in the spectrum (for example, naturally occurring radioactive impurities in the detector materials) in order to check for contamination of the detector, shield, sample containers or sample holders.

Other influences on background spectrum can include airborne radioactivity such as radon or radioactive sources stored in the vicinity of the detector.

11 Expression of results

11.1 Calculation of activity and activity per kg (or m^3) of sample

The activity, a, of each radionuclide in the sample is given by Formulae (5), (6) and (7):

$$a = n_{N,E} \cdot f_d \cdot \left(P_E \cdot \varepsilon_E \cdot t\right)^{-1} \tag{6}$$

where

$$f_d = e^{\lambda t_i} \cdot \left[\frac{\lambda \cdot t}{1 - e^{-\lambda \cdot t}}\right] \tag{7}$$

corrects for radioactive decay during the counting time t, and t_i is the time between the reference time for the results and the start of the count time.

NOTE 1 t_i is positive if the reference time is before the measurement time.

NOTE 2 This calculation can usually be performed by the analysis software package.

The result shall be stated as required by the customer: for example, as activity per mass (a/m Bq·kg^{-1}) or per volume (a/V Bq·m^{-3}). Prefixes to units may be used, for example, ml in place of m^3.

Corrections shall be applied to the results for the following effects:

— differences between the test sample and the reference source used for instrument calibration;

— dead time and pile up;

— true coincidence summing.

Recommendations on applying corrections for these effects are summarised in Annex B.

Recommendations on estimating the measurement uncertainties are given in Annex C. Advice is also included on implementing the uncertainty budget in commercial software packages.

11.2 Determination of the characteristic limits

Characteristic limits shall be estimated following ISO 11929 unless an alternative method is specified by the customer, local, national or international regulations. The formulae in References [18], [19] or [20] can be used.

NOTE References [18] and [20] also cover the case where there is an interfering photopeak in the spectrum.

An example for gamma-ray spectrometric investigation of a sample with ^{137}Cs is given in Annex E.

12 Test report

The test report shall conform to ISO/IEC 17025 requirements and shall also contain the following information:

— a statement of conformity with this document;

— the decision threshold or detection limits for any radionuclides specified by the customer or depending on the application (this may be indicated on the report as 'not greater than' or '<' or '≤' depending on customer requirements);

— the method used to estimate the detection limits or a statement of conformity to ISO 11929;

NOTE In some measurements, the activity calculated by the software may be below the decision threshold or detection limit defined in ISO 11929. Depending on customer requirements, the result may be presented as follows:

— if the activity is below the decision threshold a^*, the result of the measurement can be expressed as $\leq a^*$;

— if the activity is below the detection limit $a^\#$, the result of the measurement can be expressed as $\leq a^\#$;

— the levels of significance or coverage factors used for expressing the decision threshold or detection limits;

— any factors or assumptions that may affect the results reported (for example homogeneity or composition of the samples).

Annex A
(informative)

Quality assurance and quality control program

A.1 General

Quality control operations shall meet the requirements of ISO/IEC 17025. The influencing variables affecting each measurement method are discussed in the relevant ISO/IEC standards.

A.2 Influencing variables

Care shall be taken in order to limit as much as possible the influence of parameters that may bias the measurement and lead to wrong results. Failure to take sufficient precautions requires correction factors to be applied to the measurement result. The influencing variables affecting this measurement method are discussed in Annex B.

A.3 Instrument verification

Major instrument parameters (for example detection efficiency and background) shall be checked periodically as part of a quality assurance program established by the laboratory and in accordance with the manufacturer's instructions.

A.4 Method verification

A verification of method accuracy shall be run periodically. This may be accomplished by measurement of reference sources.

Laboratories are also recommended to participate in national or international proficiency test exercises. In these exercises, participants receive samples of gamma-ray-emitting radionuclides and are asked to identify and quantify the radionuclides present (and to state the estimated uncertainties). The laboratory's results shall be compared to the published data. Any discrepancies with the stated activity content shall be investigated and corrective actions shall be taken.

For in situ measurements, verification exercises are organized by national or international organisations. All participants perform and analyse measurements on a selected area with a nominal known activity per unit of surface area and/or mass (i.e. by evaluating samples or known sources). The results of the participating teams are compared to the nominal values and to the mean values calculated by the other teams.

A.5 Quality assurance plan

A quality assurance plan shall be established, describing the actions to assure that measurement results reported meet the customer's specification, in accordance with ISO/IEC 17025.

The quality assurance plan shall include:

— periodical quality checks;

— verification/test measurements;

— staff training and assessment;

— documentation of results from quality control checks.

The frequency of the checks shall be based on results from the validation studies and historical records.

A.6 Energy calibration

The energy calibration shall be checked periodically by counting reference source(s) emitting gamma-rays whose energies are precisely known and cover the energy range of interest. The energy calibration shall be repeated if any of the measured energies lie outside the desired range (typically 0,2 keV).

A.7 Efficiency calibration

The reproducibility of the full energy photopeak efficiencies shall be checked periodically using a reference source with a long half-life, emitting gamma-rays that cover the energy range of interest. The same counting geometry shall be used for these checks with a reproducible source-detector distance (preferably greater than 10 cm). At least 10 000 counts should be acquired in each photopeak. Shewart control charts (see Reference [15]) should be used to identify deviations in photopeak counting rates. Any deviations shall be investigated and corrective action taken if necessary.

A.8 Full width half maximum

The resolution (FWHM) at both high and low energies shall also be measured and recorded during the periodic detection efficiency test. Shewart control charts (see Reference [15]) should be used to identify deviations in the measured FWHM and deviations shall be investigated and corrective action taken if necessary.

Any upward long-term trend in the FWHM shall also be investigated and corrective action taken if necessary, as it can indicate degradation of components in the preamplifier or other faults.

A.9 Background

The background shall be checked periodically in detail by comparing the latest background spectrum with previous spectra. The count rates in the complete spectra and the photopeak areas should be compared and the photopeak energies should be compared, to confirm that the detector or its surroundings have not been contaminated. Any significant variation in the background shall be investigated and corrective action taken if necessary.

NOTE Background spectra can vary with time due to variations in the concentration of naturally occurring radioactive materials in air.

A.10 Staff training and assessment

A training plan and periodic assessment shall be established for personnel who plan and carry out measurements, perform data analysis and prepare reports.

It is recommended that staff carrying out in situ measurements follow specialist courses on the measurement techniques and participate in measurement campaigns organized by experts, educational establishments, equipment manufacturers or software producers.

A.11 Documentation

Records of the results of all quality control checks and dates shall be retained. Any discrepancies and corrective actions taken shall also be recorded.

Annex B
(informative)

Corrections to the analysis process

B.1 General

The following factors, if not corrected, may lead to biases in estimates of the activities of radionuclides in the test sample:

— differences between the geometry of the test sample and the reference source;

— differences between the composition and/or density of the test sample and the reference source;

— true coincidence summing;

— pulse pile-up effect.

The magnitude of each of these biases shall be estimated and compared to the measurement uncertainty required to meet the customer requirement. If necessary, a correction shall be applied to the measured activities and a component included in the uncertainty budget for the uncertainty of the correction. If no correction is made, a component shall be included in the uncertainty budget based on the estimate of the magnitude of the effect. Examples of the magnitude of some of these effects are given in Annex F.

Tables B.1 to B.4 summarize methods for minimizing the effect, estimating the magnitude of the effect and estimating correction factors more accurately. Other methods may be used but shall be recorded and documented.

Other correction factors may be required depending on the details of the measurement technique used (for example if the spectrometer is equipped with an anti-coincidence shield). These factors shall also be identified, the magnitude estimated and corrections applied.

B.2 Sample geometry

Table B.1 — Sample geometry correction

Reason for the correction	The sample geometry (usually the fill height in the container) may not match the geometry used for calibrating the instrument, resulting in a change in the detection efficiency which varies as a function of energy. NOTE In most arrangements, the correction is smaller than could be expected as the counts in the detector are dominated by the part of the sample closest to the detector.
Minimising the correction	The correction may be minimised by ensuring that the same geometry is used for the samples and the reference sources. For liquid samples, the volume may be controlled by dispensing a known volume using a calibrated flask or cylinder rather than using markings on the sample container. The geometry of solid sources may be controlled by using packing tools to ensure that the sample container is filled to a controlled height.
Estimating the magnitude	The magnitude of the effect may be estimated by preparing samples of known activity at the lowest and highest values of the parameter of interest (e.g. fill height) and comparing the estimates of activity. Alternatively, Monte Carlo or semi-empirical models may be used.
Estimating the correction factor	The correction factor may be estimated by preparing a set of sources of known activity, varying the geometry (for example the fill height). Monte Carlo or semi-empirical models may also be used.

B.3 Composition and density correction (internal absorption)

Table B.2 — Composition and density correction

Reason for the correction	Photons emitted in the decay of the radionuclides in the sample can be attenuated by the photoelectric effect, Compton scattering and pair production as they pass through the sample matrix before reaching the detector. The attenuation depends on the elemental composition and density of the sample matrix which may differ from that of the reference source used to calibrate the instrument. This correction varies rapidly with energy; it is most important for low energy photons and samples containing high atomic number elements and may be very significant.
Minimising the correction	Corrections may be minimised by selecting sample containers and fill height to minimise the distance that photons pass through the sample matrix. The composition and density of the reference material should be selected to match those of the sample.
Estimating the magnitude	If the density and approximate elemental composition of the sample is known, the magnitude of this effect may be determined by first estimating the maximum pathlength of photons through the sample material. The attenuation factor (C_{att}) can then be calculated as a function of energy from tabulated attenuation coefficients such as XCOM (see Reference [8]): $$C_{\text{att}} = \exp\left[-\mu(E)\cdot h\right] \quad \text{(B.1)}$$ where h is the maximum pathlength; $\mu(E)$ is the linear attenuation coefficient of the sample material for photon energy E. Alternatively, for a cylindrical source of height x placed on the axis of symmetry of the detector, the self-attenuation correction c_{self} is given approximately by (see Reference [2]): $$C_{\text{self}} = \frac{1-\exp\left[-\mu(E)\cdot x\right]}{\mu(E)\cdot x} \quad \text{(B.2)}$$ The calculation should be repeated for the reference source using the same value for the pathlength. The magnitude of the correction factor is given by the ratio of the two attenuation factors. The correction factor may also be determined experimentally by measuring the attenuation of gamma-rays from one or more point sources through the sample, relative to the attenuation through the reference source. The point source(s) shall emit gamma-rays not present in the sample or source.
Estimating the correction factor	The correction factor may be estimated using a software model of the detector and sample geometry. Monte Carlo simulation or semi-empirical models may be used (see Reference [2] and references therein). The uncertainty in the correction may be estimated by a sensitivity analysis of the correction to variations in the sample density and composition.

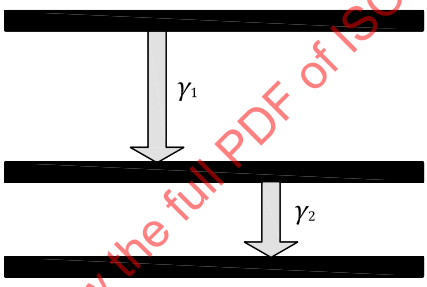
B.4 Pulse pile-up and dead time

Table B.3 — Pulse pile-up and dead-time correction

Reason for the correction	The pulse processing electronics and analysis software normally apply a correction for the dead time introduced by the electronics. However, the analogue pulse may extend beyond this time period; if a second pulse arrives on the tail of the pulse, the pulse height may be distorted and a pulse may be lost from the photopeak in the spectrum. The effect may be seen in the spectrum as increased photopeak widths (FWHM) and low-energy tails. The effect shall not be neglected for low-activity measurements as it may be significant for the reference source used for calibration. The effect depends on the instrument and electronics used. An additional correction may be needed if the dead time changes significantly during the measurement time (for example when measuring high-activity short half-life samples). Specialist dead-time correction systems are needed in this case (see Reference [23]).
Minimising the correction	Low count rates (less than a few thousand pulses per second, equivalent to dead times of a few percent) should be used when possible, by choosing appropriate sample sizes and geometry. Short shaping times may be selected on the main amplifier to reduce pulse lengths, However, this may have the effect of increasing the FHWM of photopeaks. Care shall be taken to ensure that the pole-zero adjustment has been made as an incorrect setting may affect the magnitude of this effect.
Estimating the magnitude	For dead times less than 10 %, the correction for pile-up is typically less than 1 %, though the magnitude of the correction depends on the spectrometer type and settings. If higher dead times are used, a correction should be applied as described below.
Estimating the correction factor	a) Repeated measurements of a short half-life radionuclide such as ^{99m}Tc. The observed count rate for each measurement should be decay corrected to a common reference time and plotted versus dead time. The correction may be estimated assuming that the count rate is unaffected by pile-up at low dead times (less than a few percent). b) Two source method: position a single radionuclide source (e.g. ^{137}Cs) at a fixed distance from the spectrometer. Carry out repeated measurements, varying the dead time by moving a second source (e.g. ^{60}Co) closer to the spectrometer in stages. Plot the count rate in the peak from the first source versus dead time (as for method 1). c) Pulser method: simulated detector pulses may be passed through the pulse-processing electronics and losses from the peak in the spectrum used to determine the correction factor (see Reference [9]).

B.5 True coincidence summing correction

Table B.4 — True coincidence summing correction

Reason for the correction	Some radionuclides decay emitting gamma-rays in cascade; in most such transitions, the gamma-rays and associated X-rays are emitted effectively simultaneously and may be detected as a single event. As an example for the measurement of a point source, the observed photopeak area in a spectrum for γ_1 in the simple decay scheme shown in Figure B.1 is given by: $$n_{N,E} = A \cdot P_E \cdot t \cdot \varepsilon_E \cdot \left(1 - \varepsilon_{E_2}^{T}\right) \quad \text{(B.3)}$$ where $\varepsilon_{E_2}^{T}$ is the total detection efficiency for γ_2 and is the probability that γ_2 is detected in the photopeak or, importantly, at lower energies in the Compton continuum.  **Figure B.1** The correction factor $1 - \varepsilon_{E_2}^{T}$ is due to events when γ_1 is detected in the photopeak but γ_2 is also detected resulting in a pulse with a higher energy than the photopeak, losing counts from the photopeak. The total detection efficiency may be much higher than the photopeak detection efficiency, so the correction factor can be significant (10 % to 20 % in some cases (see Reference [10]). The correction is independent of count rate but depends on the decay scheme of the radionuclide and the detection efficiency. The correction is complex to estimate for radionuclides with multiple decay paths. In addition, the formula above is only valid for point sources: for extended samples (e.g. Marinelli beakers), the correction shall be determined for each voxel of the sample and then integrated.
Minimising the correction	The correction may be minimized by using a large sample to detector distance (typically greater than 15 cm) and a detector with a small relative detection efficiency. The correction may be eliminated by calibrating the spectrometer using a reference source of the radionuclide of interest and estimating the sample activity by a simple ratio to the certificated value.
Estimating the magnitude	The magnitude of the effect depends on the sample geometry and matrix, the detector itself and the radionuclides used for calibration and required for measurement. The scientific literature may be consulted and an estimate taken from a similar set-up.
Estimating the correction factor	For the reasons stated above, estimating the correction factor is a complex task. Modelling software may be used to apply an appropriate correction [see Reference [2] and references therein]; References [16] and [17] describe an alternative empirical approach. The uncertainty (for a coverage factor of $k = 1$) may be taken to be 1/3 of the correction.

Annex C
(informative)

Uncertainty budget

C.1 General

For some applications of gamma-ray spectrometry, the uncertainty of the activity in the test samples is small compared to uncertainties in sampling or it may have little impact on the decisions taken on the basis of the measurements (for example, if the technique is used to determine the activity of aqueous discharges from a nuclear site and the activity is a small fraction of the regulatory limit). Even in such cases, this document requires that an estimate of the uncertainty of measurement is conducted, so that it can be demonstrated that the results are fit for purpose and for possible future re-analysis of the data.

The uncertainty in the measurement results shall be estimated and documented in accordance with the requirements of ISO/IEC Guide 98-3:2008. In accordance with ISO/IEC 17025, the major components of uncertainty shall be estimated; for high accuracy measurements (for example for nuclear decay studies or for nuclear forensics), the uncertainty budget shall be estimated in detail.

A complete analysis of the uncertainties is very complex (see Reference [2]), as correlations shall be taken into account in the detection efficiency calibration and the measurement; the measurement uncertainty also cannot be better than the uncertainty in primary standards of radioactivity. For these reasons, measurement uncertainties are unlikely to be less than 5 % (k = 2) for routine assays. If the uncertainty budget is of the order of 1 % then it is likely that correlations have not been addressed correctly in the calculation.

C.2 Uncertainty components

Table C.1 summarizes likely components of the uncertainty budget. Table C.1 assumes that the overall uncertainty in the measurement is no better than 5 % and components are quasi-independent; for more accurate measurements, correlations shall be considered but this is outside the scope of this document.

Table C.1 — Type A components

Component	Possible estimation method	Distribution	Notes
Photopeak area estimate	From software analysis packages	Gaussian	Detailed calculations are given in Reference [2] including issues with overlapping photopeaks. It should also be noted that photopeak area calculations can be subject to large errors if artefacts in the background continuum interfere with the photopeak and background shape.
Sample positioning	From repeated measurements, removing and replacing the sample		Accurate sample positioning jigs should be used to minimise this component.
Sample fill height	From repeated sample preparations		This component can be minimised by packing material to a reproducible height in the container. Modelling codes may be useful for estimating the magnitude.
Variations in sample container construction	From repeated sample preparations		Due to variations in the thickness of the base of the container and shape affecting the sample to detector distance.
Sample homogeneity	From repeated sample preparations		Difficult to estimate and potentially significant. Particular care is needed if measuring radon or other gaseous daughter radionuclides, as the gas may be distributed throughout the container rather than the sample material (see Reference [2]).

Table C.2 — Type B components

Component	Possible estimation method	Distribution	Notes
Detector efficiency (determined experimentally)	Detector efficiency calibration software package	Gaussian	Varies rapidly as a function of energy. Correlations between data points may have to be considered.
Detector efficiency (determined through Monte Carlo modelling or by semi-empirical methods)	Comparison with traceable reference materials supported by consultation of reviews in the scientific literature.		Subject to large uncertainties and dependent on the details of the detector parameters used in the model. Discrepancies of up to 30 % have been observed in the literature (see Reference [11]).
Dead-time correction	From linearity tests		Typically 10 % of the correction may be assumed.
True coincidence summing correction	See Annex B		Typically 10 % to 30 % of the correction may be assumed.
Self-absorption	See Annex B		
Emission probability	From nuclear data tables	Gaussian	
Decay correction	From nuclear data tables	Gaussian	The standard uncertainty is negligible for most radionuclides (see Reference [2]).
Deconvolution of overlapping photopeaks	From photopeak fitting algorithms		
Background subtraction	From photopeak analysis software		

C.3 Implementation of uncertainty budget

The method used by the software analysis package shall be reviewed, including the algorithm used to combine the uncertainties, and compared to the uncertainty budget devised using the steps described above. Parameters in the software shall be set (with the reasons documented) so that the overall measurement uncertainties match as closely as practicable to the uncertainty budget estimated. If the activity of a radionuclide is estimated by taking a weighted mean of results from more than one gamma-ray emission, care shall be taken to ensure that the uncertainty budget remains realistic.

If a more accurate estimation of the uncertainty is required, it may be necessary to develop in-house software or spreadsheets rather than using commercial analysis software.

Annex D
(informative)

Detector types

The type of detector to use depends on the application. High efficiency detectors are more sensitive but may also be subject to high dead times, high correction factors for true coincidence summing and high ambient background. Detectors with beryllium windows require careful handling to avoid breaking the fragile window. Low energy response may not generate additional quantitative information as low energy gamma-rays and X-rays may be subject to large correction factors for self-absorption in the sample. The shape of the crystal is also important; a thin but wide detector is more sensitive to radioactive material placed on the axis of symmetry of the crystal than to active material at the side – reducing the background from materials to the side but also reducing the usefulness of Marinelli beakers.

Tables D.1 and D.2 summarise the main characteristics of semi-conductor and scintillation detectors. Figure D.1 shows example spectra from different detector types.

Table D.1 — Typical characteristics of semi-conductor detectors

Type of crystal	End cap material	Window material	Resolution (FWHM) at 662 keV %	Range of energy	Relative detection efficiency[8][9] %	Notes
Germanium p-Type coaxial	Al, Cu, CF[a]	same as end cap	0,2	50 keV to 10 MeV	20 to 175	2
Germanium p-Type broad energy	Al, Cu, CF	Carbon composite, Be	0,2	3 keV to 3 MeV 3 keV to 10 MeV	7 to 65 20 to 175	3, 4, 5
Germanium p-Type planar	Al	Be	—	3 keV to 1 MeV		
Germanium n-Type coaxial+	Al, Cu, CF	Carbon composite Be	0,2	3 keV to 10 MeV	10 to 150	2, 3, 4, 6
Germanium n-Type planar	Al	Carbon composite, Be	0,2	3 keV to 3 MeV	20 to 50	3, 4, 5
Germanium Well type coaxial	Al, Cu	Al	0,2	10 keV to 10 MeV	40 to 150	
CdZnTe	Al	Al	2,0	30 keV to 3 MeV	—	
Silicon Lithium-Drifted Planar	Al	Be	Resolution at 5,9 keV: 3,5	1 keV to 30 keV	—	

[a] Carbon fibre.

Table D.2 — Typical Characteristics of scintillation detectors

Type of crystal	End cap material	Window material	Resolution at 662 keV %	Energy range	Relative detection efficiency[8][9] %	Notes
NaI(Tl)	Al, Cu	Same as end cap	5 to 7	30 keV to 3 MeV	100 (by definition for a 3” × 3” detector)	
$LaBr_3(Ce)$	Al	Same as end cap	3,5	30 keV to 3 MeV	140	10
$CeBr_3$	Al	Same as end cap	4	30 keV to 3 MeV	140	

NOTE 1 The terms “broad energy” and “planar” are used differently by different suppliers.

NOTE 2 Very large detectors can have disadvantages, for example, high count rates can result in the need for large corrections for dead time and consequently higher measurement uncertainties.

NOTE 3 Window materials made of Carbon composite are more robust than Be windows. Great care should be taken to avoid breaking the fragile Be windows.

NOTE 4 Corrections for self-absorption in the sample matrix may significantly increase measurement uncertainties at low energies, limiting the usefulness of low energy detectors.

NOTE 5 For some of the crystal types listed here, the detection efficiency can depend strongly on the direction of incidence of the photons. This factor should be taken into account when selecting sample containers. However, one advantage is that the detectors can be relatively insensitive to photons from background radiation.

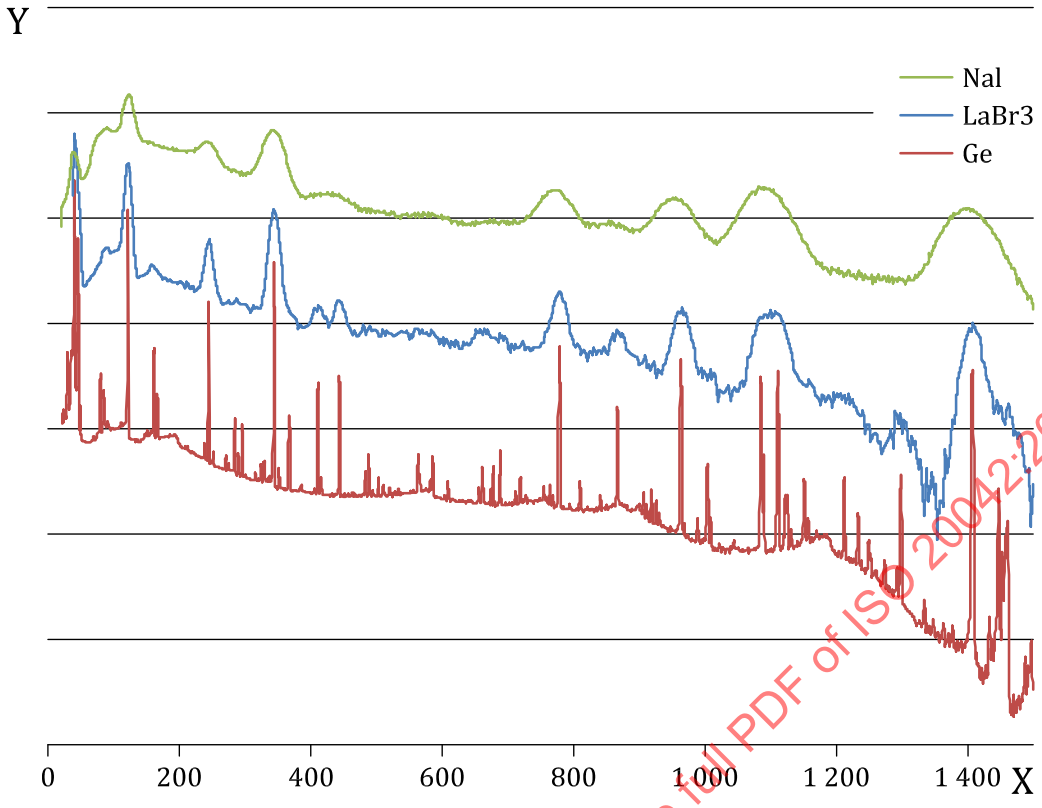
NOTE 6 Crystals with a relative detection efficiency greater than 100 % are also available.

NOTE 7 Different materials are available, including Al.

NOTE 8 For germanium spectrometers, by convention, the relative detection efficiency is defined to be the photopeak detection efficiency at 1,3 MeV for a point source placed on the cylindrical axis of symmetry of the detector at 25 cm from the detector relative to the detection efficiency of a 3” × 3” NaI(Tl) detector using the same measurement geometry.

NOTE 9 The relative detection efficiency depends strongly on the size of crystal.

NOTE 10 $LaBr_3$(Ce) detectors contain ^{138}La and ^{227}Ac, both of which are radioactive and result in higher observed background count rates and hence lower detection sensitivity.



Key

X energy, in keV

Y counts, in log scale

Figure D.1 — Typical gamma spectra from NaI, $LaBr_3$ and Ge spectrometers, showing the difference in energy resolution between the different types of detector

Annex E
(informative)

Example: Calculation of ^{137}Cs activity content and characteristic limits in an aqueous sample

E.1 Principle

This example illustrates a typical application of high resolution gamma-ray spectrometry: the determination of the ^{137}Cs content and the characteristic limits. The 662 keV photopeak from the decay of ^{137m}Ba is used as the signature for ^{137}Cs. To simplify the example, it is assumed that the spectrometer has been calibrated using a multi-radionuclide standardized solution with the same composition and geometry as the sample. Also, for simplicity, it is assumed that no corrections are needed for true summing, dead time, decay and sample composition/positioning.

E.2 Measurement

The sample was placed on the spectrometer in the same position as the reference source (a jig was used to ensure a reproducible position) and a spectrum was acquired for 60 000 s.

E.3 Calculation of activity content

The activity of ^{137}Cs in the sample is given by the following measurement formula:

$$a_m = \left(\frac{n_{N,E}}{t} \right) \cdot w = \frac{(n_{b,s} - z_{0,s})}{t} \cdot w \tag{E.1}$$

with

$$w = \frac{1}{\varepsilon} \cdot \frac{1}{P_E} \cdot \frac{1}{m} \cdot f_1 \cdot f_2 \cdot f_3 \cdot f_4 \cdot f_5 \tag{E.2}$$

where

a_m is the activity per kilogram of sample (Bq·kg^{-1}) at the reference time;

$n_{N,E}$ is the area (counts) in photopeak in spectrum from sample;

t is the counting time (s) of sample (live time);

$n_{b,s}$ is the gross counts in photopeak region in spectrum;

$z_{0,s}$ is the counts in background continuum in photopeak region of the sample spectrum;

f_1 is the attenuation correction factor (assumed 1,0);

f_2 is the true-summing correction factor (assumed 1,0);

f_3 is the dead-time correction factor (assumed 1,0);

f_4 is the decay correction factor (assumed 1,0);

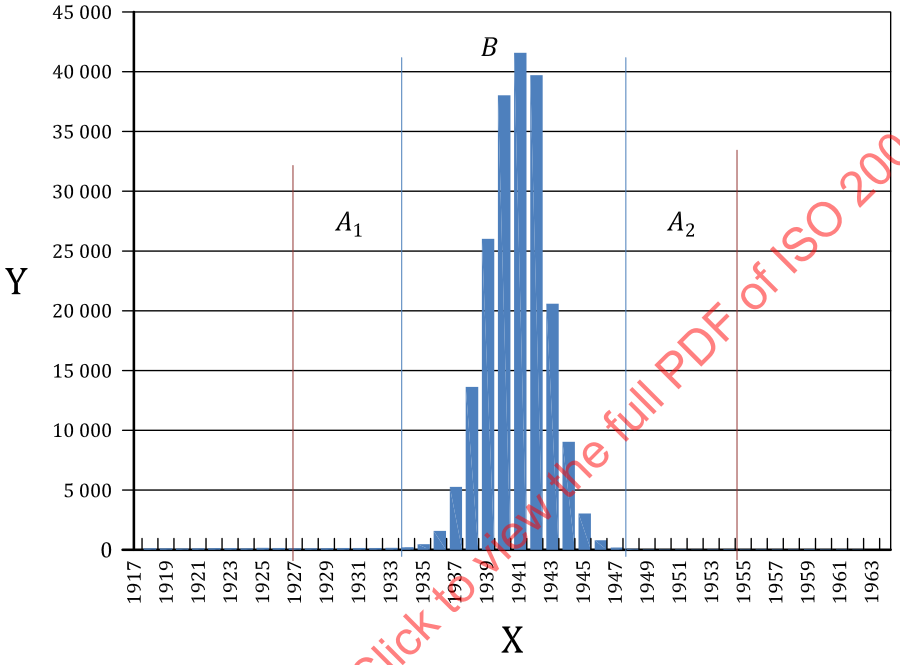
f_5 is the sample versus reference source positioning/height correction (assumed 1,0);

m is the sample mass (kg);

ρ_E is the gamma-ray-emission probability;

ε_E is the detection efficiency, taken from the calibration curve.

The net photopeak areas were calculated by a variation of the summation method. The spectrum and channel contents from the sample are shown below, in the region of the 662 keV photopeak (see Figure E.1).



Key

X channel number

Y number of counts

Figure E.1 — ^{137}Cs photopeak in a gamma-ray spectrum of an aqueous test sample

The region of interest of the photopeak was divided into three sub-regions as follows:

— A_1 is the low energy background continuum area;

— B is the photopeak area;

— A_2 is the high energy background continuum area.

Table E.1 — Measured counts in the region of the ^{137}Cs photopeak in the test sample placed on the detector, t_m = 60 000 s

Channel	Region A_1 counts	Channel	Peak B counts	Channel	Region A_2 counts
		1 934	208	1 948	92
		1 935	472	1 949	37
		1 936	1 594	1 950	44
		1 937	5 269	1 951	40
		1 938	13 633	1 952	33
		1 939	26 019	1 953	36
		1 940	38 034	1 954	47
1 927	133	1 941	41 584		
1 928	138	1 942	39 706		
1 929	144	1 943	20 584		
1 930	160	1 944	9 040		
1 931	158	1 945	3 046		
1 932	165	1 946	798		
1 933	173	1 947	196		

In this example, the number of counts in the photopeak was calculated by subtracting the mean background continuum count under the photopeak.

Table E.2 — Counts in defined regions of spectrum

Region	Number of channels	Counts in region	Background count (per channel)	Background under photopeak	Photopeak area (counts)
A_1	7	1 071	153		
B	14	200 183	Mean: 110	1 540	198 643
A_2	7	329	39,3		

Assuming that the detection efficiency is 0,5 %, taking the emission probability for the 662 keV gamma-ray photopeak to be 0,849 9 (see Reference [30]) and using a sample mass of 1,0 kg the activity in the sample is:

$$a_m = \frac{n_{N,E}}{\varepsilon_E \cdot t \cdot p_E \cdot m} = \frac{198\,643}{60\,000 \cdot 0{,}005 \cdot 0{,}8499 \cdot 1{,}0} \cdot \frac{1}{\mathrm{s \cdot kg}} = 779{,}1\ \mathrm{Bq \cdot kg^{-1}} \quad \text{(E.3)}$$

The main components of uncertainty are:

a) Photopeak area calculation.

b) Detection efficiency. The uncertainty in the detection efficiency is calculated by the software package used and depends on the curve fitting software and the uncertainty in the reference sources (typically 1 % to 2 %), plus uncertainties that may be introduced when dispensing the reference sources. For the purpose of this example, the relative uncertainty is assumed to be 5 % using a coverage factor of k = 1.

c) Gamma-ray emission probability.

This may be taken from published decay data; the relative uncertainty is taken to be 0,24 % in this example using a coverage factor of k = 1.

d) Additional components of the uncertainty from the correction factors (although assumed to be unity) should normally be included but are assumed negligible for this example.

The number of net counts in the photopeak area is given by:

$$n_{\mathrm{NP},s} = n_{\mathrm{b},s} - c_{0,s} \cdot n_{0,s} = n_{\mathrm{b},s} - z_{0,s} \tag{E.4}$$

with

$$n_{\mathrm{b},s} = \sum_{B} n_i \;,\; n_{0,s} = \sum_{A_1} n_{s,i} + \sum_{A_2} n_{s,i} \;\text{ and }\; c_{0,s} = \frac{b}{2l} \tag{E.5}$$

$c_{0,s} = c_{0,r} = \dfrac{b}{2l}$ should hold

where

b is the number of channels in the photopeak area B;

l is the number of channels in each of the areas A_1 and A_2.

The uncertainty ($k = 1$) in the photopeak area is given by:

$$u^2(n_{\mathrm{NP},s}) = n_{\mathrm{b},s} + c_{0,s}^2 \cdot n_{0,s} \tag{E.6}$$

$$a = (n_{NP,s} / t \cdot w = w \cdot (n_{b,s} - z_{0,s}) / t \tag{E.7}$$

The uncertainty can be estimated using the approach described in the GUM and is given by:

$$u^2(a) = w^2 \cdot \left(n_{b,s} + c_{0,s}^2 \cdot n_{0,s} \right) / t^2 + a^2 \cdot u_{\mathrm{rel}}^2(w) \tag{E.8}$$

E.4 Characteristic limits

For low-activity measurements, it may be necessary to state the characteristic limits [the decision threshold and the detection limit (see ISO 11929)] of the measurement, to indicate the limitations of the measurement. The characteristic limits are used to decide whether activity is present or not, and to estimate the sensitivity of the measurement. The characteristic limits depend on the measurement equipment, the background, the measurement parameters and the sample itself.

E.5 Decision threshold

The decision threshold is the value of the activity a^* above which it is decided that activity is detected in the sample (with a given probability α for a wrong decision that there is an activity if in reality there is none).

For $\tilde{a}$ one expects $n_{b,s} = \dfrac{\tilde{a}}{w} \cdot t + z_{0,s}$ with $u^2(n_{\mathrm{b},s}) = n_{\mathrm{b},s}$ (E.9)

$$\tilde{u}^2(\tilde{a}) = w^2 \cdot \left(\frac{\tilde{a}}{w} \cdot t + c_{0,s} \cdot n_{0,s} + c_{0,s}^2 \cdot n_{0,s} \right) / t^2 + \tilde{a}^2 \cdot u_{\mathrm{rel}}^2(w) \tag{E.10}$$

$$a^* = k_{1-\alpha} \cdot w \cdot \sqrt{c_{0,s} \cdot n_{0,s} + c_{0,s}^2 \cdot n_{0,s}} \tag{E.11}$$

E.6 Detection limit

The detection limit $a^{\#}$ is the smallest true value of the activity $\tilde{a}$ that can be detected with a defined probability β for a wrong decision that the activity is not detected while in reality it is present.

$$a^{\#} = a^{*} + k_{1-\beta} \cdot \sqrt{w^2 \cdot \left(\frac{a^{\#}}{w} \cdot t + c_{0,s} \cdot n_{0,s} + c_{0,s}^2 \cdot n_{0,s} \right) / t^2 + a^{\#2} \cdot u_{\mathrm{rel}}^2 (w)} \tag{E.12}$$

which can be solved by iteration with a starting value of $a^{\#} = 2a^{*}$.

If $\alpha = \beta$ and hence $k_{1-\alpha} = k_{1-\beta} = k$ an exact solution for the detection limit is given by:

$$a^{\#} = \frac{2 \cdot a^{*} + (k^2 \cdot w)/t}{1 - k^2 \cdot u_{\mathrm{rel}}^2 (w)} \tag{E.13}$$

Annex F
(informative)

Example: Simulating correction factors for sample positioning, geometry, matrix, density and true summing

F.1 Introduction

This annex shows the impact on measurement results of variations in sample positioning and geometry for a typical gamma-ray spectrometry system, estimated using a Monte Carlo modelling software package. The results apply only to the detector and sample dimensions used in this example and may be used to indicate the order of magnitude of the effects mentioned below. However, any correction factors needed to implement this document shall be calculated for the particular detector and samples being used.

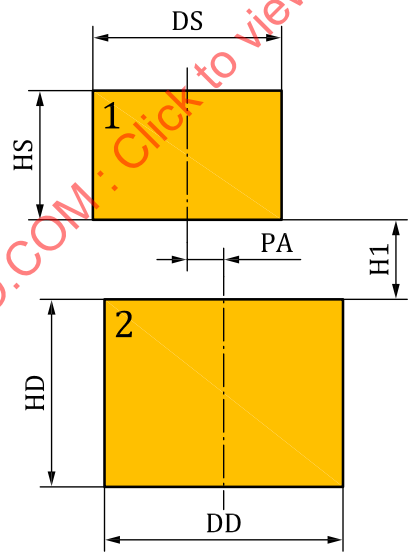
The annex covers the effects of

— the variation of the dimensions of the source,

— the variation of the composition of the source (density and matrix), and

— the variation of the position of the source with respect to the detector.

The parameters describing the system geometry and default values are defined in Table F.1.



Key

1 source:
— Ma (Matrice): material of a certain density (RO)

2 detector

Figure F.1 — Parameters describing the system geometry and default values