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BSI Standards Publication

Measurement of radioactivity in the environment — Soil

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

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National foreword

This British Standard is the UK implementation of ISO 18589-3:2015. It supersedes BS ISO 18589-3:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee NCE/2, Radiation protection and measurement.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Measurement of radioactivity in the environment — Soil —

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

Mesurage de la radioactivité dans l'environnement — Sol —

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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

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

The committee responsible for this document is ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 2, *Radiological protection*.

This second edition cancels and replaces the first edition (ISO 18589-3:2007), which has been technically revised.

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

— *Part 1: General guidelines and definitions*

— *Part 2: Guidance for the selection of the sampling strategy, sampling and pre-treatment of samples*

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

— *Part 4: Measurement of plutonium isotopes (plutonium 238 and plutonium 239 + 240) by alpha spectrometry*

— *Part 5: Measurement of strontium 90*

— *Part 6: Measurement of gross alpha and gross beta activities*

— *Part 7: In situ measurement of gamma-emitting radionuclides*

Introduction

This part of ISO 18589 is published in several parts to be used jointly or separately according to needs. ISO 18589-1 to ISO 18589-6, concerning the measurements of radioactivity in the soil, have been prepared simultaneously. These parts are complementary and are addressed to those responsible for determining the radioactivity present in soils. The first two parts are general in nature. ISO 18589-3 to ISO 18589-5 deal with radionuclide-specific measurements and ISO 18589-6 with non-specific measurements of gross alpha or gross beta activities. ISO 18589-7 deals with the measurement of gamma-emitting radionuclides using *in situ* spectrometry.

Additional parts can be added to ISO 18589 in the future if the standardization of the measurement of other radionuclides becomes necessary.

Measurement of radioactivity in the environment — Soil —

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

1 Scope

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

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

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

This part of ISO 18589 is addressed to people responsible for determining gamma-emitting radionuclides activity present in soils for the purpose of radiation protection. It is suitable for the surveillance of the environment and the inspection of a site and allows, in case of accidents, a quick evaluation of gamma activity of soil samples. This might concern soils from gardens, farmland, urban or industrial sites that can contain building materials rubble, as well as soil not affected by human activities.

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

2 Normative references

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

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

ISO 11074, *Soil quality — Vocabulary*

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

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

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

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

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

3 Terms, definitions and symbols

3.1 Terms and definitions

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

3.2 Symbols

m	mass of the test portion, in kilograms
A	activity of each radionuclide in the calibration source, at the calibration time, in becquerel
a, a_c	activity, in becquerel per kilogram, per unit of mass of each radionuclide, without and with corrections
t_g	sample spectrum counting time, in seconds
t_0	ambient background spectrum counting time, in seconds
t_s	calibration spectrum counting time, in seconds
$n_{\mathrm{N},E}$, $n_{\mathrm{N0},E}$, $n_{\mathrm{Ns},E}$	number of counts in the net area of the peak, at energy, E, in the sample spectrum, in the background spectrum and in the calibration spectrum, respectively
$n_{\mathrm{g},E}$, $n_{\mathrm{g0},E}$, $n_{\mathrm{gs},E}$	number of counts in the gross area of the peak, at energy, E, in the sample spectrum, in the background spectrum and in the calibration spectrum, respectively
$n_{\mathrm{b},E}$, $n_{\mathrm{b0},E}$, $n_{\mathrm{bs},E}$	number of counts in the background of the peak, at energy, E, in the sample spectrum, in the background spectrum and in the calibration spectrum, respectively
ε_E	efficiency of the detector at energy, E, with the actual measurement geometry
P_E	probability of the emission of gamma radiation with energy, E, for each radionuclide, per decay
$\mu_1(E)$, $\mu_2(E)$	linear attenuation coefficient at photon energy, E, of the sample and calibration source, respectively, per centimetre
$\mu_{\mathrm{m},i}(E)$	mass attenuation coefficient, in square centimetres per gram, at photon energy, E, of element i
h	height of the sample in the container, in centimetres
w_i	mass fraction of element i (no unit)
ρ	bulk density, in grams per cubic centimetre, of the sample
λ	decay constant of each radionuclide, per second
$u(a)$, $u(a_\mathrm{c})$	standard uncertainty, in becquerel per kilogram, associated with the measurement result, with and without corrections, respectively
U	expanded uncertainty, in becquerel per kilogram, calculated by $U = k \cdot u\,(a)$ with $k = 1, 2, \ldots$
a^*, a_c^*	decision threshold, in becquerel per kilogram, for each radionuclide, without and with corrections, respectively
$a^\#$, $a_\mathrm{c}^\#$	detection limit, in becquerel per kilogram, for each radionuclide, without and with corrections, respectively
$a^\triangleleft$, $a^\triangleright$	lower and upper limits of the confidence interval, for each radionuclide, in becquerel per kilogram

4 Principle

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

The nature and geometry of the detectors as well as the samples call for appropriate energy and efficiency calibrations.[1][2] Both coincidence and random summation effects need to be considered, particularly with container sitting directly on the detector and Marinelli type container, high activity levels or with well-type detectors used to measure small-mass samples (see 8.1.4).

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

5 Gamma-spectrometry equipment

Gamma-spectrometry equipment generally consists of

— a semiconductor detector with a cooling system (liquid nitrogen, cryogenic assembly, etc.),

— a shield, consisting of lead and/or other materials, against ambient radiation,

— appropriate electronics (high-voltage power supply; signal-amplification system; an analogue-to-digital converter),

— a multi-channel amplitude analyser, and

— a computer to display the measurement spectra and to process the data.

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

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

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

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

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

c) peak-to-Compton ratio.

Depending on the required accuracy and the desired detection limit, it is generally necessary to use high-quality detectors whose energy resolution is less than 2,2 keV (for the ^{60}Co peak at 1 332 keV) and with a peak/Compton ratio between 50 and 80 for ^{137}Cs (see IEC 61452).

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

The computer, in combination with the available hardware and software, shall be carefully selected.[5][6] It is recommended that the results of the computer analysis of the spectrum be visually checked regularly.

Comparison with a certified reference material is recommended to check the performance of the apparatus. Participation in proficiency and inter-laboratory tests and inter-comparison exercises can also help to verify the performance of the apparatus and the status of the analysis.[9][10]

6 Sample container

Measuring gamma radioactivity in soils requires sample containers that are suited to gamma spectrometry with the following recommended characteristics:

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

— be made of transparent material to see the level of content;

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

— be watertight and not react with the sample constituents;

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

— be unbreakable.

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

7 Procedure

7.1 Packaging of samples for measuring purposes

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

The procedure shall be carried out as follows.

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

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

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

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

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

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

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

When measuring Ra-226 through the short-lived decay products of Rn-222, the sealed container shall be stored long enough (30 d) to allow radioactive equilibrium to be reached between Ra-226 and Rn-222.

7.2 Laboratory background level

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

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

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

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

7.3 Calibration

7.3.1 Energy calibration

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

Further information is given in IEC 61452, ISO 10703, and References [7] and [8].

7.3.2 Efficiency calibration

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

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

The sample measurement shall be performed with the same measuring conditions as used for calibrating the gamma spectrometry system. In particular, the settings of the electronics (gain and high voltage), the measurement geometry, the position of the source in relation to the detector and the sample and standard matrices shall be identical.

For this purpose, a calibration source should have the same physical and chemical properties as the sample. It might, for instance, be produced by spiking an appropriate sample of soil.

With these conditions, the efficiency at energy E shall be calculated as given in Formula (1):

$$\varepsilon_E = \frac{n_{\mathrm{Ns},E} / t_\mathrm{s}}{A \cdot P_E} \quad (1)$$

For a single peak at an energy E, the count, $n_{\mathrm{Ns},E}$, in the net-peak area of a γ-spectrum is calculated as given in Formula (2):

$$n_{\mathrm{Ns},E} = n_{\mathrm{gs},E} - n_{\mathrm{bs},E} \quad (2)$$

When the physical and chemical nature of the sample (chemical composition, bulk density) is different from the conditions of the efficiency calibration, a correction for the self-absorption of gamma radiation should be applied.

Further information is given in IEC 61452, ISO 10703, and References [7] and [8].

7.4 Measurements of and corrections for natural radionuclides

If activities of natural radionuclides in the soil are being measured, the areas of full-energy peaks used for evaluating their activities shall be corrected for the background contribution of those same radionuclides inside the detector shielding, taking into account potential differences of the duration of the sample and background measurements.

Special advice to take into account during the measurement of natural radionuclides in soil and information on spectroscopic interferences is given in Annex B.

The gamma ray of the radionuclides in the background and/or of natural radionuclides inside the sample can also interfere with measurements of artificial radionuclides and can require appropriate corrections.

8 Expression of results

8.1 Calculation of the activity per unit of mass

8.1.1 General

The activity per unit of mass, a of each radionuclide present in the sample is obtained from the net count, $n_{\mathrm{N},E}$, from the peak of an individual γ-line without interference using Formula (3):

$$a = \frac{n_{\mathrm{N},E} / t_\mathrm{g}}{P_E \cdot \varepsilon_E \cdot m \cdot f_E} \quad (3)$$

where

f_E is the correction factor considering all necessary corrections according to Formula (4).

$$f_E = f_\mathrm{d} - f_{\mathrm{att},E} - f_{\mathrm{cl},E} - f_{\mathrm{s},E} \tag{4}$$

where

f_d is the factor to correct for decay for a reference date;

$f_{\mathrm{att},E}$ is the factor to correct for self-absorption;

$f_{\mathrm{cl},E}$ is the factor to correct for coincidence losses;

$f_{\mathrm{s},E}$ is the factor to correct summing-up effects by coincidences.

For an undisturbed peak with energy, E, the count, $n_{\mathrm{N},E}$, in the net-peak area of a γ-spectrum is calculated by Formula (5):

$$n_{\mathrm{N},E} = n_{\mathrm{g},E} - n_{\mathrm{b},E} \tag{5}$$

Thus, Formula (3) can be expressed as given in Formula (6):

$$a = \frac{n_{\mathrm{N},E}/t_\mathrm{g}}{P_E \cdot \varepsilon_E \cdot m \cdot f_E} = \frac{n_{\mathrm{g},E} - n_{\mathrm{b},E}}{P_E \cdot \varepsilon_E \cdot m \cdot f_E \cdot t_\mathrm{g}} = (n_{\mathrm{g},E} - n_{\mathrm{b},E}) \cdot w / t_\mathrm{g} \text{ with } w = \frac{1}{P_E \cdot \varepsilon_E \cdot m \cdot f_E} \tag{6}$$

if the net-peak area, $n_{\mathrm{N},E}$, is obtained by unfolding of a multiplet Formula (6) is valid; but, special care is needed in calculating the uncertainties according to 8.2.

If a peak is disturbed by an interfering γ-line of another radionuclide and cannot be resolved by unfolding methods due to the limited resolution of the detector, and if the contribution of the interfering radionuclide can be estimated from another γ-line of the interfering radionuclide, the procedure described in 8.6 should be applied.

For nuclides characterized by more than one line, the activity can be computed using several peaks taking into account the known branching ratios described by their decay scheme and the efficiency curve.

8.1.2 Decay corrections

Depending on the half-life of the radionuclide being measured, the activity per unit of mass shall be corrected by f_d. To take into account the radioactive decay during the counting time and during the time between the reference instant ($t = 0$) and the measuring instant ($t = t_i$), f_d shall be calculated by Formula (7):

$$f_\mathrm{d}^{-1} = e^{\lambda \cdot t_i} \cdot \left[\frac{\lambda \cdot t_\mathrm{g}}{1 - e^{-\lambda \cdot t_\mathrm{g}}} \right] \tag{7}$$

8.1.3 Self-absorption correction

Measurement of radioactivity in soils by gamma spectrometry can involve sample whose matrix is different from that of the calibrated source. In this case, a correction factor should be applied to the result obtained. The lower the radiation energy, the larger the correction factor.

Different techniques can be used to determine this correction factor:

- measurement of the attenuation coefficient of gamma radiation in the sample material at a given energy;
- mathematical calculation that takes into account the chemical composition and bulk density of the sample.

For cylindrical sample containers at the level of the detector, the value of the attenuation correction factor, $f_{\text{att},E}$, can be estimated using Formula (8):

$$f_{\text{att},E} = \frac{\mu_2(E)\cdot\left(1-e^{-\mu_1(E)\cdot X}\right)}{\mu_1(E)\cdot\left(1-e^{-\mu_2(E)\cdot X}\right)} \tag{8}$$

where

X is the average test sample thickness in the container, expressed in centimetres.

The linear attenuation coefficient, $\mu(E)$, depends on the photon energy, bulk density, chemical composition of the sample and expresses the exponential decrease of the flux density of gamma rays with distance. It can be calculated using Formula (9):

$$\mu(E) = \left[\sum_i w_i \mu_{\text{m},i}(E)\right]\rho \tag{9}$$

As an approximation and for soils of the same nature, the linear attenuation coefficient, $\mu(E)$, can be obtained directly by multiplying the mass attenuation coefficient by the density.

8.1.4 Summation effects or coincidence losses corrections

For radionuclides with cascade transitions, counting losses due to coincidence summing are to be expected, especially at high counting efficiencies.

These corrections are important for point as well as thin source samples measured very close to the detector surface; they are specific for each radionuclide, detector, measuring geometry and sample-to-detector distance.

Most of the theoretical methods for such calculations are related to the use of transport theory and Monte-Carlo techniques (Geant, EGSnrc, MCNP, Penelope, etc.; see References [13] to [16]); given the difficulties associated with modelling detectors, some experimental procedures can be applied for each specific situation.

Some of these experimental procedures use data from specialized literature, but given the wide range of detector possibilities and measuring conditions, direct measurement as given in a) to c) below can be made.

a) Prepare a source containing the multi-line photon-emitting radionuclide whose correction factor at energy, E, shall be calculated along with another radionuclide emitting at a similar energy, E', which is mono-energetic or has negligible summing corrections. The geometry shall be the same as that used for the sample source.

b) Make a measurement with this source at a large distance from the detector. Calculate the relationship between the net peak counts at energies E and E'.

c) Make a measurement with the sample in the normal measuring position. The relationship between the net peak counts at energies E and E' is similar to that calculated above and the theoretical net peak counts, $n_{\text{N},E}^{\text{T}}$, at energy E can be estimated.

The relationship between the theoretical net peak counts, $n_{\text{N},E}^{\text{T}}$, and the measured net peak counts, $n_{\text{N},E}$, is the summing correction factor for energy E of the multi-line photon emitting radionuclide that shall be applied to the analysis of the calibration and source sample spectrum.

Further information is given in References [2] and [8].

8.2 Standard uncertainty

According to ISO/IEC Guide 98-1:2009,[18] the standard uncertainty of a is calculated by Formula (10):

$$u(a)=\sqrt{\left(w/t_{\mathrm{g}}\right)^{2}\cdot\left[u^{2}\left(n_{\mathrm{g},E}\right)+u^{2}\left(n_{\mathrm{b},E}\right)\right]+a^{2}\cdot u_{\mathrm{rel}}^{2}(w)} \tag{10}$$

where the uncertainty of the counting time is neglected.

The relative standard uncertainty of w is calculated by Formula (11):

$$u_{\mathrm{rel}}^{2}(w)=u_{\mathrm{rel}}^{2}(P_E)+u_{\mathrm{rel}}^{2}(m)+u_{\mathrm{rel}}^{2}(\varepsilon_E)+u_{\mathrm{rel}}^{2}(f_E) \tag{11}$$

Taking Formula (1) into account, the relative standard uncertainty of ε_E is calculated by Formula (12):

$$u_{\mathrm{rel}}^{2}(\varepsilon_E)=u_{\mathrm{rel}}^{2}(n_{\mathrm{Ns},E})+u_{\mathrm{rel}}^{2}(A)+u_{\mathrm{rel}}^{2}(P_E)=+u_{\mathrm{rel}}^{2}(n_{\mathrm{gs},E}-n_{\mathrm{bs},E})+u_{\mathrm{rel}}^{2}(A)+u_{\mathrm{rel}}^{2}(P_E) \tag{12}$$

where $u_{\mathrm{rel}}(A)$ includes all the uncertainties related to the calibration source: standard certificate, preparation of the calibration source.

For the calculation of the characteristic limits (see ISO 11929), it is necessary to know $\tilde{u}(\tilde{a})$, i.e. the standard uncertainty of a as a function of its true value. For a true value $\tilde{a}$, from $n_{\mathrm{g},E}=\tilde{a}\cdot t_{\mathrm{g}}/w+n_{\mathrm{b},E}$ and with $u^{2}(n_{\mathrm{g}})=n_{\mathrm{g}}$, one obtains Formula (13):

$$\tilde{u}(\tilde{a})=\sqrt{\left(w/t_{\mathrm{g}}\right)^{2}\cdot\left[\left(t_{\mathrm{g}}/w\right)\cdot\tilde{a}+n_{\mathrm{b},E}+u^{2}(n_{\mathrm{b},E})\right]+\tilde{a}^{2}\cdot u_{\mathrm{rel}}^{2}(w)} \tag{13}$$

The uncertainties $u(n_{\mathrm{N}})$, $u(n_{\mathrm{g}})$, and $u(n_{\mathrm{b}})$ shall be calculated in accordance with ISO/IEC Guide 98-1:2009,[18] taking into account that the individual counts, n_i, in channel i of a multi-channel spectrum are the result of a Poisson process and hence $u^2(n_i)$ = n_i holds. The values of n_{N}, n_{g}, and n_{b} and their associated standard uncertainties $u(n_{\mathrm{N}})$, $u(n_{\mathrm{g}})$, and $u(n_{\mathrm{b}})$ can be calculated with a computer program. Since there are various methods of subtracting the background below a peak in order to derive the number of counts in the net peak area, no generally applicable formula can be given. An example of the simple case of linear background subtraction is given in Annex A.

If the net-peak area $n_{\mathrm{N},E}$, is obtained by a software using unfolding techniques, the software should yield $n_{\mathrm{N},E}$ and its associated standard uncertainty. When it provides both $n_{\mathrm{b},E}$ with its associated standard uncertainty, the uncertainties can be calculated according to Formulae (10) to (13). If the code gives directly a decision threshold and a detection limit for the activity a, these characteristic limits should be calculated according to ISO 11929, Annex C, in particular C.5. This procedure of ISO 11929 based on Reference [17] is not intended for users but rather for code developers.

8.3 Decision threshold

The decision threshold, a^*, is obtained from Formula (13) for $\tilde{a}=0$ (see ISO 11929). This yields Formula (14):

$$a^{*}=k_{1-\alpha}\cdot\tilde{u}(0)=k_{1-\alpha}\cdot\left(w/t_{\mathrm{g}}\right)\sqrt{n_{\mathrm{b},E}+u^{2}\left(n_{\mathrm{b},E}\right)} \tag{14}$$

α = 0,05 and $k_{1-\alpha}$ = 1,65 are often chosen by default.

8.4 Detection limit

The detection limit, $a^{\#}$, is calculated by Formula (15) (see ISO 11929):

$$\begin{aligned} a^{\#} &= a^{*} + k_{1-\beta} \cdot \tilde{u}(a^{*}) \\ &= a^{*} + k_{1-\beta} \cdot \sqrt{w^{2}\left[\left(a^{\#}/w + n_{\mathrm{b},E}/t_{\mathrm{g}}\right)/t_{\mathrm{g}} + u^{2}(n_{\mathrm{b},E})/t_{\mathrm{g}}^{2}\right] + a^{\#2} \cdot u_{\mathrm{rel}}^{2}(w)} \end{aligned} \tag{15}$$

$\beta = 0{,}05$ and $k_{1-\beta} = 1{,}65$ are often chosen by default.

The detection limit can be calculated by solving Formula (15) for $a^{\#}$ or, more simply, by iteration, starting with the approximation $a^{\#} = 2 \cdot a^{*}$.

By setting $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution to Formula (15) is given by Formula (16):

$$a^{\#} = \frac{2 \cdot a^{*} + \left(k^{2} \cdot w\right)/t_{\mathrm{g}}}{1 - k^{2} \cdot u_{\mathrm{rel}}^{2}(w)} \tag{16}$$

8.5 Confidence limits

The lower, $a^{\triangleleft}$, and upper, $a^{\triangleright}$, limits of the confidence interval are calculated using Formulae (17) and (18), respectively (see ISO 11929):

$$a^{\triangleleft} = a - k_{\mathrm{p}} \cdot u(a) \quad \text{where } p = \omega \cdot (1 - \gamma/2) \tag{17}$$

$$a^{\triangleright} = a + k_{\mathrm{q}} \cdot u(a) \quad \text{where } q = 1 - \omega \cdot \gamma/2 \tag{18}$$

where $\omega = \Phi\left[y/u(y)\right]$, Φ being the distribution function of the standardized normal distribution.

If $a \geq 4 \cdot u(a)$, ω can be set equal to 1 and Formula (19) applies:

$$a^{\triangleleft,\triangleright} = a \pm k_{1-\gamma/2} \cdot u(a) \tag{19}$$

$\gamma = 0{,}05$ and $k_{1-\gamma/2} = 1{,}96$ are often chosen by default.

8.6 Corrections for contributions from other radionuclides and background

8.6.1 General

In gamma spectrometry, it is frequently necessary to correct for two types of contributions.

a) The gamma line of the radionuclide being determined contains contributions from gamma radiation of another radionuclide in the sample. The contributing radionuclide has another gamma line from which the contribution to the line in question can be estimated taking into account the emission probabilities of the gamma lines.

b) The gamma line of the radionuclide being determined occurs also in the background of the spectrometer. By measuring a background spectrum without a sample for a counting time, t_0, this contribution can be corrected, taking into account the different counting times for the two spectra.

For both cases, the activity per unit of mass can be calculated using a model given in Formula (20):

$$a_\mathrm{c} = \left(n_{\mathrm{N},E} / t_\mathrm{g} - x \cdot n_{\mathrm{N0},E} / t_0\right) \cdot w \tag{20}$$

where

x is a factor that is a function of the type of correction.

For the both types of contributions, this model gives the necessary correction.

8.6.2 Contribution from other radionuclides

The gamma line being corrected at the energy, E_1, has the net peak area, n_{N,E_1}. The contribution of the radionuclide is calculated using the ratio of the contributing radionuclide for gamma energy, E_1. Formula (20) gives the necessary correction with $x = P_{E_1} \cdot \varepsilon_1 / P_{E_2} \cdot \varepsilon_2$ and $t_0 = t_g$. This yields Formula (21):

$$a_\mathrm{c} = \left(n_{\mathrm{N},E_1} - x \cdot n_{\mathrm{N},E_2}\right) \cdot w / t_\mathrm{g} \tag{21}$$

Neglecting the standard uncertainty of x, the standard uncertainty of a_c is calculated from Formula (22):

$$u^2(a_\mathrm{c}) = \left(w / t_\mathrm{g}\right)^2 \left\{n_{\mathrm{g},E_1} + u^2\left(n_{\mathrm{b},E_1}\right) + x^2\left[n_{\mathrm{g},E_2} + u^2(n_{\mathrm{b},E_2})\right]\right\} + a_\mathrm{c}^2 \cdot u_\mathrm{rel}^2(w) \tag{22}$$

and with a true value $\tilde{a}_\mathrm{c}$ of a_c, Formula (23) can be derived:

$$\tilde{u}^2(\tilde{a}_\mathrm{c}) = \left(w / t_\mathrm{g}\right)^2 \left\{\tilde{a}_\mathrm{c} t_\mathrm{g} / w + n_{\mathrm{b},E_1} + u^2\left(n_{\mathrm{b},E_1}\right) + x\left(n_{\mathrm{g},E_2} - n_{\mathrm{b},E_2}\right) + x^2\left[n_{\mathrm{g},E_2} + u^2\left(n_{\mathrm{b},E_2}\right)\right]\right\} + \tilde{a}_\mathrm{c}^2 \cdot u_\mathrm{rel}^2(w) \tag{23}$$

Then, the decision threshold, a_c^*, is given by Formula (24):

$$a_\mathrm{c}^* = k_{1-\alpha} \cdot \left(w / t_\mathrm{g}\right) \cdot \sqrt{n_{\mathrm{b},E_1} + u^2(n_{\mathrm{b},E_1}) + x(n_{\mathrm{g},E_2} - n_{\mathrm{b},E_2}) + x^2\left[n_{\mathrm{g},E_2} + u^2(n_{\mathrm{b},E_2}\right]} \tag{24}$$

and the detection limit, $a_\mathrm{c}^\#$, by Formula (25)

$$a_\mathrm{c}^\# = a_\mathrm{c}^* + k_{1-\beta} \sqrt{(w / t_\mathrm{g})^2 \left\{a_\mathrm{c}^\# t_\mathrm{g} / w + n_{\mathrm{b},E_1} + u^2(n_{\mathrm{b},E_1}) + x\left(n_{\mathrm{g},E_2} - n_{\mathrm{b},E_2}\right) + x^2\left[n_{\mathrm{g},E_2} + u^2(n_{\mathrm{b},E_2})\right]\right\} + a_\mathrm{c}^{\#2} u_\mathrm{rel}^2(w)} \tag{25}$$

The detection limit can be calculated by solving Formula (25) for $a_\mathrm{c}^\#$ or, more simply, by an iteration starting with the approximation $a_\mathrm{c}^\# = 2 \cdot a_c^*$.

By setting $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution to Formula (25) is given by Formula (26):

$$a_\mathrm{c}^\# = \frac{2 \cdot a_c^* + \left(k^2 \cdot w\right) / t_\mathrm{g}}{1 - k^2 \cdot u_\mathrm{rel}^2(w)} \tag{26}$$

8.6.3 Contribution from background

In this case, Formula (20) is used for the correction by setting $x = 1$ and $u(x) = 0$. $n_{\mathrm{N}0,E}$ is the net peak area of the gamma line in the background spectrum and t_0 is the counting time of the background spectrum. This yields Formula (27):

$$a_\mathrm{c} = \left(n_{\mathrm{N},E} / t_\mathrm{g} - n_{\mathrm{N}0,E} / t_0 \right) \cdot w \tag{27}$$

The standard uncertainty of a_c is calculated by Formula (28):

$$u^2(a_\mathrm{c}) = w^2 \left(n_{\mathrm{g},E} / t_\mathrm{g}^2 + n_{\mathrm{g}0,E} / t_0^2 + u^2\left(n_{\mathrm{b},E}\right) / t_\mathrm{g}^2 + u^2\left(n_{\mathrm{b}0,E}\right) / t_0^2 \right) + a_\mathrm{c}^2 \cdot u_\mathrm{rel}^2(w) \tag{28}$$

and with a true value $\tilde{a}_c$ of a_c, Formula (29) is obtained*:*

$$\tilde{u}^2(\tilde{a}_\mathrm{c}) = w^2 \left\{ \tilde{a}_\mathrm{c} / t_\mathrm{g} w + \left[n_{\mathrm{b},E} + u^2\left(n_{\mathrm{b},E}\right) \right] / t_\mathrm{g}^2 + \left[n_{\mathrm{g}0,E} + u^2\left(n_{\mathrm{b}0,E}\right) \right] / t_0^2 + \left(n_{\mathrm{g}0,E} - n_{\mathrm{b}0,E} \right) / t_0 t_\mathrm{g} \right\} + \tilde{a}_\mathrm{c}^2 u_\mathrm{rel}^2(w) \tag{29}$$

Then, the decision threshold, a_c^*, is given by Formula (30):

$$a_\mathrm{c}^* = k_{1-\alpha} \cdot w \cdot \sqrt{\left[n_{\mathrm{b},E} + u^2(n_{\mathrm{b},E}) \right] / t_\mathrm{g}^2 + \left[n_{\mathrm{g}0,E} + u^2\left(n_{\mathrm{b}0,E}\right) \right] / t_0^2 + \left(n_{\mathrm{g}0,E} - n_{\mathrm{b}0,E} \right) / t_0 t_\mathrm{g}} \tag{30}$$

and the detection limit $a_\mathrm{c}^\#$ by Formula (31):

$$a_\mathrm{c}^\# = a_\mathrm{c}^* + k_{1-\beta} \sqrt{w^2 \left\{ a_\mathrm{c}^\# / t_\mathrm{g} w + \left[n_{\mathrm{b},E} + u^2\left(n_{\mathrm{b},E}\right) \right] / t_\mathrm{g}^2 + \left[n_{\mathrm{g}0,E} + u^2\left(n_{\mathrm{b}0,E}\right) \right] / t_0^2 + \left(n_{\mathrm{g}0,E} - n_{\mathrm{b}0,E} \right) / t_0 t_\mathrm{g} \right\} + a_\mathrm{c}^{\#2} u_\mathrm{rel}^2(w)} \tag{31}$$

The detection limit can be calculated by solving Formula (25) for $a_\mathrm{c}^\#$ or, more simply, by iteration with a starting approximation $a_\mathrm{c}^\# = 2 \cdot a_c^*$.

When taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of Formula (31) is given by Formula (32):

$$a_\mathrm{c}^\# = \frac{2 \cdot a_\mathrm{c}^* + \left(k^2 \cdot w \right) / t_\mathrm{g}}{1 - k^2 \cdot u_\mathrm{rel}^2(w)} \tag{32}$$

The limits of the confidence interval are calculated according to Formulae (17) and (18).

9 Test report

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

a) reference to this part of ISO 18589;

b) identification of the sample;

c) units in which the results are expressed;

d) test result, $a \pm u$ or $a \pm U$, with the associated k value.

Complementary information can also be reported with items a to d such as:

— probabilities α, β and $(1 - \gamma)$;

— decision threshold and the detection limit;

- depending on the customer request, there are different ways to present the result:
    - when the activity per unit of mass, a, is compared with the decision threshold (see ISO 11929), the result of the measurement should be expressed as $\leq a^*$ when the result is below the decision threshold;
    - when the activity per unit of mass, a, is compared with the detection limit, the result of the measurement can be expressed as $\leq a^\#$ when the result is below the detection limit. If the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose;
- mention of any relevant information likely to affect the results.

Annex A
(informative)

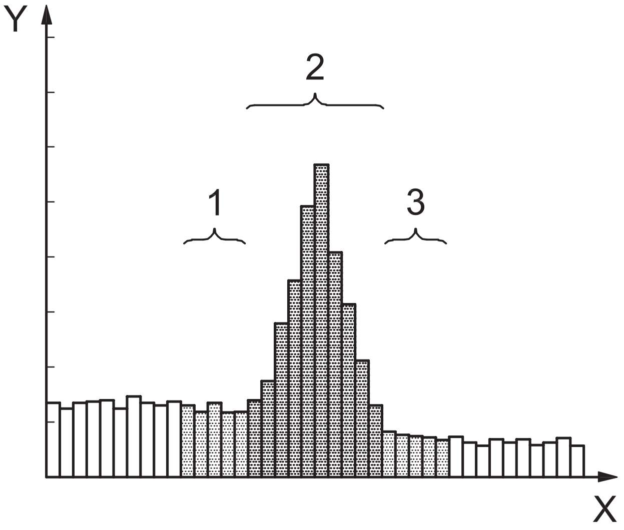
Calculation of the activity per unit mass from a gamma spectrum using a linear background subtraction

Frequently, the net peak area is calculated by subtraction of a linear background. In this case, three channel areas are defined in the spectrum: an area, *P*, symmetrical around the peak maximum covering *p* channels and two areas, *B*1 and *B*2, each covering *b* channels on either side of *P*; see Figure A.1. With the full width at half peak height, *h*, a length of the peak area $p \approx 2{,}5h$ and $b \approx p/2$ are frequently chosen. Then, n_N can be calculated according to Formula (5), as given in Formula (A.1):

$$n_\mathrm{g} = \sum_{i \in P} n_i \,,\; n_{B1} = \sum_{i \in B1} n_i \,,\; n_{B2} = \sum_{i \in B2} n_i \,, \textit{ and } \quad n_\mathrm{b} = \frac{p}{2b} \cdot (n_{B1} + n_{B2}) \tag{A.1}$$

The standard uncertainties are given in Formula (A.2):

$$u(n_\mathrm{g}) = \sqrt{n_\mathrm{g}} \;,\; u(n_\mathrm{b}) = \frac{p}{2b} \cdot \sqrt{n_{B1} + n_{B2}} \;, \text{ and } u(n_\mathrm{N}) = \sqrt{n_\mathrm{g} + \left(\frac{p}{2b}\right)^2 \cdot (n_{B1} + n_{B2})} \tag{A.2}$$



Key

X channel number, *i*
Y counts, n_i
1 area *B*1, with length *b*
2 area *P*, with length *p*
3 area *B*2, with length *b*

Figure A.1 — Scheme of linear background subtraction in gamma spectrometry

The standard uncertainty for the specific activity according to Formula (10) is given by Formula (A.3):

$$u^2(a) = w^2 \cdot \left[n_\mathrm{g} + \left(\frac{p}{2b}\right)^2 \cdot (n_{B1} + n_{B2}) \right] / t_\mathrm{g}^2 + a^2 \cdot u_\mathrm{ref}^2(w) \tag{A.3}$$

In this case, $\tilde{u}^2(\tilde{a})$ is calculated by Formula (A.4):

$$\tilde{u}^2(\tilde{a}) = (w / t_\mathrm{g})^2 \cdot \left\{ \tilde{a} \cdot t_\mathrm{g} / w + \left[(p/2b) + (p/2b)^2 \right] \cdot (n_{B1} + n_{B2}) \right\} + \tilde{a}^2 \cdot u_\mathrm{rel}^2(w) \tag{A.4}$$

where

$$u_\mathrm{rel}(w) = \frac{u(w)}{w} \tag{A.5}$$

The decision threshold is calculated as given in Formula (A.6):

$$a^* = k_{1-\alpha} \cdot \tilde{u}(0) = k_{1-\alpha} \cdot (w / t_\mathrm{g}) \cdot \sqrt{\frac{p}{2b} \cdot (n_{B1} + n_{B2}) + \left(\frac{p}{2b}\right)^2 \cdot (n_{B1} + n_{B2})} \tag{A.6}$$

and the detection limit, as given in Formula (A.7):

$$\begin{aligned} a^\# &= a^* + k_{1-\beta} \cdot \tilde{u}(a^\#) \\ &= a^* + k_{1-\beta} \cdot \sqrt{(w / t_\mathrm{g})^2 \cdot \left[a^\# \cdot (t_\mathrm{g} / w) + (p/2b) \cdot (n_{B1} + n_{B2}) + (p/2b)^2 \cdot (n_{B1} + n_{B2}) \right] + a^{\#2} \cdot u_\mathrm{rel}^2(w)} \end{aligned}$$

(A.7)

When $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of Formula (A.4) is given by Formula (A.8):

$$a^\# = \frac{2 \cdot a^* + \left(k^2 \cdot w\right) / t_\mathrm{g}}{1\text{-}\mathrm{k}^2 \cdot u_\mathrm{rel}^2(w)} \tag{A.8}$$

The limits of the confidence interval are calculated according to Formulae (17) and (18).

Annex B
(informative)

Analysis of natural radionuclides in soil samples using gamma spectrometry

B.1 General

Among the natural radionuclides, those besides K-40, belonging to a natural decay chain that can be measured using gamma spectrometry include U-238, Ra-226; Pb-210 of the uranium/radium decay chain; U-235 and Th-227 of the uranium/actinium decay chain; as well as Th-232, Ra-228 and Th-228 of the thorium decay chain; see Figure B.1.

Some radionuclides of the natural decay chains (e.g. U-238, Ra-228, Th-228) cannot be determined directly by gamma spectrometry but only by measuring their daughter radionuclides. In these cases, it is necessary to ensure that there is equilibrium between the parent radionuclide and the daughter radionuclides being measured. Radioactive equilibrium can be disrupted within the media being examined due to the different chemical or biochemical behaviour of the respective elements. For example, the radioactive equilibrium can be shifted strongly due to the different transfer behaviour of parent radionuclide and daughter radionuclides in the soil-vegetation-animal-milk chain.

In these cases, it is recommended to keep the samples for a sufficiently long period before measuring them. On the other hand, interference of the radioactive equilibrium in the sample for measurement can result from escaping radon. In the case of measuring the short-lived decay products of Rn-222, the sample material shall additionally be put into a gas-tight glass container such that the dead volume in the glass container between the sample and the lid is as low as possible, and the sample left until radioactive equilibrium has been achieved. Since, as a rule, the severity of the interference with the equilibrium is not known, to be on the safe side, one should assume, when estimating the waiting period that initially there are practically no daughter radionuclides. In the case of a long-lived parent radionuclide and a short-lived decay product, this means that the waiting period should be at least six half-lives of the decay product.

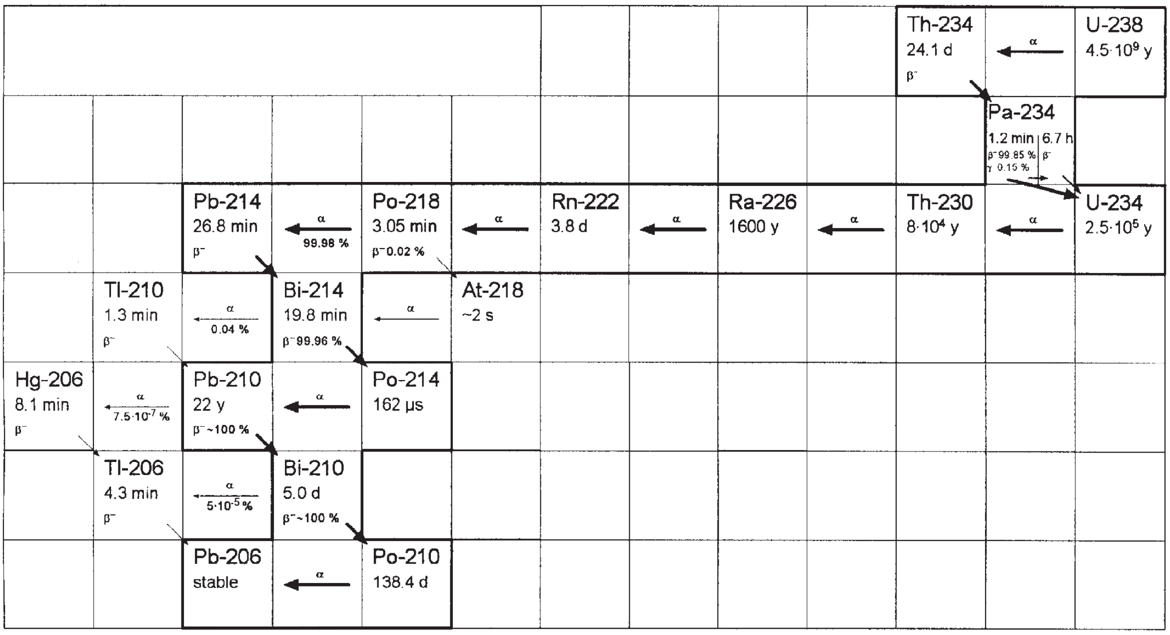
Measured activity values for the radionuclides of the natural decay chains that are not in radioactive equilibrium with the respective longer-lived parent radionuclide shall be counted back to a reference date; in most cases this should be the sampling date. Both the radioactive decay of the respective radionuclide and its decay products from its parent radionuclide shall be taken into account. Examples of such radionuclide pairs are Th-232/Ra-228, Ra-228/Th-228, Ra-226/Pb-210.

Another problem for the gamma spectrometric determination of natural radionuclides is the fact that some radionuclides show gamma lines that are identical or so near each other that it is not possible to resolve them by hardware or software means. In these cases, it is necessary to make corrections using other gamma lines; see 8.6.1 a).

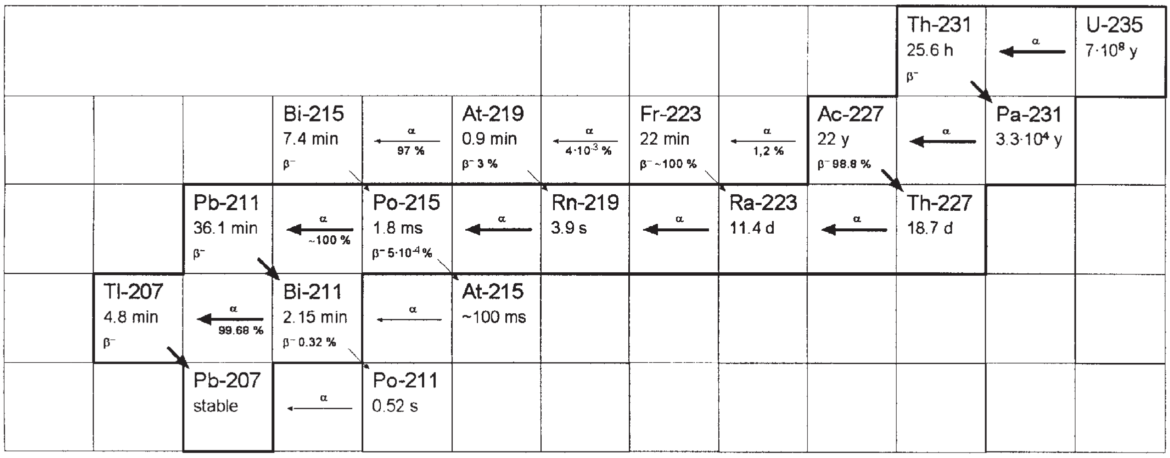
If this procedure cannot be carried out, a correction can be performed only by measuring radionuclides with other methods (e.g. alpha spectrometry or an emanation measurement). It is necessary to use these more sensitive methods when required detection limits cannot be achieved through gamma spectrometry (e.g. due to insufficient sample quantities).

B.2 to B.10 give additional explanations regarding the gamma spectrometric determination of common natural radionuclides in soil. For some radionuclides, the requirement of correcting self-absorption and summation losses are mentioned. Table B.1 lists the photon energies, E_γ, and the emission probabilities, p_E, of selected radionuclides.

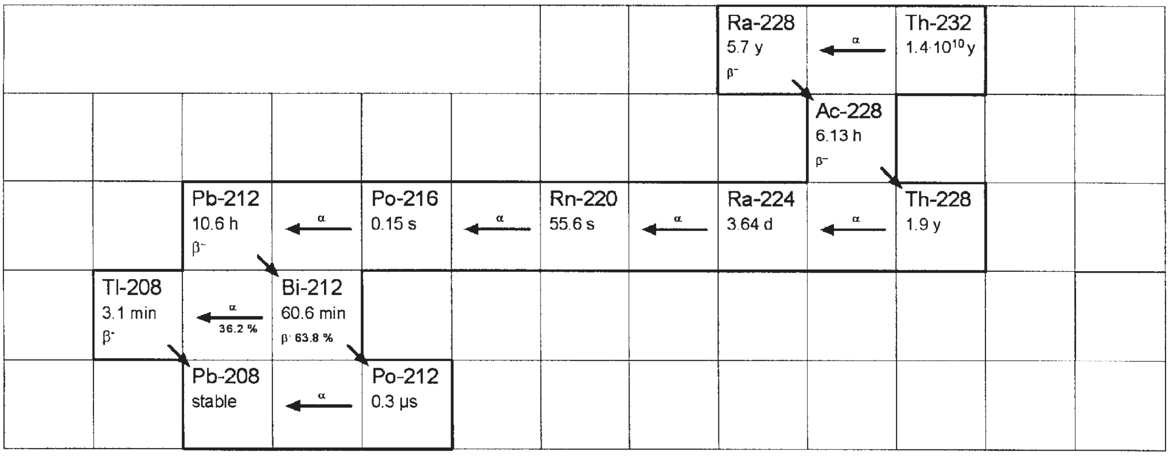
When the decay chains are in equilibrium, the listed emission probabilities refer to the decay of the parent nuclide. For example, in the determination of the Th-228 content through Tl-208, it is not necessary to take into account that the branching ratio of the Bi-212 into Tl-208 is only 36,2 %; see Figure B.1 c). This is already considered when stating the emission probability in Table B.1.



a) Uranium/radium ($A = 4n + 2$)



b) Uranium/actinium ($A = 4n + 3$)



c) Thorium ($A = 4n$)

Figure B.1 — Natural decay chain

B.2 Determination of uranium-238

U-238 with its half-life of 4,468 · 10^9 years is the parent radionuclide in the uranium/radium decay chain. U-238 cannot be measured by gamma spectrometry but only through its decay radionuclides, Th-234 and Pa-234m. Very often, the 63,28 keV-line of Th-234 is used. At this low energy, however, matrix variations between calibration and test sample can have considerable influence on the result due to different self-absorption and therefore it is necessary that they be taken into account. The 63,28 keV line is additionally interfered with by the 63,81 keV line of the Th-232 with an emission probability of 0,263 %. The use of the double line energies 92,37 keV and 92,79 keV of Th-234 with an emission probability of 4,81 % (2,42 % + 2,39 %) is not recommended to quantify U-238.

The lines of Pa-234m at 1001,03 keV and 766,37 keV are more suitable due to their higher energies, although their emission probabilities are very low and summing coincidences have to be considered. When evaluating the 1 001,03 keV line, it is necessary to observe that the emission probability is 0,839 %. The lines suitable for evaluation have been compiled In Table B.1.

Additional information for the determination of U-238 is the nearly constant natural U-238/U-235 activity ratio of 21,7 in samples of natural origin.

B.3 Determination of radium-226

Ra-226 is a long-lived radionuclide in the uranium/radium decay chain with a half-life of 1 600 years. For the gamma spectrometric determination of Ra-226 in soil samples, there are, in particular, two possibilities. Either Ra-226 is evaluated at its 186,1 keV line or the short-lived decay radionuclides Pb-214 and Bi-214 of Rn-222 are measured after the radioactive equilibrium between Ra-226, Rn-222, Pb-214 and Bi-214 has been established.

If the line at 186,1 keV is used, it is necessary to take into account the contribution of the overlapping line at 185,72 keV of U-235. The contribution of this overlap can be corrected according to 8.6 a) if the U-235 activity concentration is known. The activity concentration of uranium in a sample can also be derived using the results from other uranium determinations, e.g. fluorimetry, phosphorimetry, X-ray fluorescence measurements, mass spectrometry. If radioactive equilibrium can be assumed in the uranium/radium chain, the activity concentration of U-238 equals that of Ra-226, and in case of natural uranium, the activity concentration of U-235 can be calculated since the specific activity of U-235 is approximately 1/22 of the specific activity of U-238.

If radioactive equilibrium or the natural composition of uranium cannot be assumed, the contribution of U-235 can be corrected only if its activity concentration is determined through other lines of U-235.

For the determination of activities of the short-lived decay products, gas-tight containers are required since, otherwise, the values determined for Pb-214 and Bi-214 can be too low due to the loss of Rn-222. The period between filling the sample into a gas-tight vessel and starting the measurement is determined by the half-life of Rn-222 (3,8 d) and should be at least 23 d. The application of special types of activated charcoal can be useful to prevent the radon losses. It is necessary to correct the summation if all detectable gamma-lines of Pb-214 and Bi-214 are used for the determination of Ra-226. If the gamma line of Pb-214 at 351,93 keV is used, the summation effects become negligible.

B.4 Determination of lead-210

Pb-210 is a radionuclide of the uranium/radium decay chain with a half-life of 22,3 years and a gamma energy line at 46,54 keV. This energy gamma line is usually measured by gamma spectrometry using an N-type coaxial high-purity germanium detector. Different material compositions of calibration sources and test samples play an important role because of different self-absorption effects. It is necessary to consider the material composition appropriate for the correction of these effects (see 8.1.3).

B.5 Determination of uranium-235

U-235 is the parent radionuclide of the uranium/actinium decay chain with a half-life of $7{,}037 \cdot 10^8$ years. U-235 can be measured through the lines at 143,8 keV, 163,3 keV, 185,7 keV, and 205,3 keV. The line with the highest emission probability at 185,7 keV is not suitable if the Ra-226 activity is not known. The line with the second highest emission probability at 143,8 keV overlaps the 144,2 keV line of Ra-223. The two other lines have lower emission probabilities, which are not sufficient for measuring samples with a low activity concentration of U-235. Alternative measurement techniques, such as alpha-spectrometry or mass-spectrometry, can also be used.

B.6 Determination of actinium-227

Ac-227 is a radionuclide of the uranium/actinium decay chain with a half-life of 21,8 years. Ac-227 can be determined by gamma spectrometry through its short-lived decay radionuclides Th-227, Ra-223 and Rn-219. Generally the 235,97 keV line of Th-227 is preferred, since the other lines either have low emission probabilities or overlap with lines of other radionuclides.

B.7 Determination of thorium-232

Th-232 with a half-life of $1{,}41 \cdot 10^{10}$ years is the parent radionuclide of the thorium decay chain. Th-232 has a line at 63,81 keV with a very low emission probability of 0,263 % which overlaps a line of Th-234 at 63,28 keV with a higher emission probability of 4,1 %, so that Th-232 cannot be determined directly by gamma spectrometry in soil samples. Determination through its decay radionuclides Ac-228, Pb-212 and Tl-208 can be performed only if one assumes that these radionuclides are in radioactive equilibrium with each other and with Th-232.

This assumption is not always true in soil samples because of the presence of Ra-228 with a half-life of 5,7 years in the decay chain between Th-232 and Ac-228 (half-life of 6,3 h), which can cause a radioactive disequilibrium different from that of thorium due to its solubility.

B.8 Determination of radium-228

Ra-228 is a radionuclide of the thorium decay chain with a half-life of 5,75 years. Ra-228 is determined through measuring its decay radionuclide Ac-228, which is available in radioactive equilibrium with Ra-228 due to its short half-life of 6,15 h. Depending on the chosen measurement geometry, considerable counting losses can be expected as a result of summation effects, which require correction.

If the derived activity concentrations for Ra-224, Pb-212, and Tl-208 do not differ from Ac-228, they can also be used for the evaluation of the Ra-228 activity concentration.

B.9 Determination of thorium-228

Th-228 is a radionuclide of the thorium decay chain with a half-life of 1,91 years. Th-228 can be determined through measuring its short-lived decay radionuclides Ra-224, Pb-212, and Tl-208 if there is a radioactive equilibrium. Equilibrium is established within about 22 d between Th-228 and its short-lived daughters.

Pb-212 lines are subject to interference and should not be used to determine Th-228. Better choices are the 727,4 keV line from Bi-212 and the 583 keV line from Tl-208, which are not normally subject to interference.

When measuring Ra-224 at 240,99 keV, it is necessary to consider the contribution of the line of Pb-214 at an energy of 242,00 keV. Due to the half-life of 3,66 d for Ra-224, radioactive equilibrium with Th-228 can be assumed in the soil sample so that the measured results for Ra-224 also apply to Th-228.

A condition for the measurement of Pb-212 and Tl-208 is that Rn-220 remains in the test sample. This is generally given due to its low half-life of 54 s. Preferably, the 238,63 keV line of Pb-212 is used for evaluation. However, as a result of the summation effect, it is necessary to take counting losses into account.

B.10 Determination of potassium-40

K-40 is included in the isotope mixture of natural potassium (K-39, K-40, K-41) with an isotopic abundance of 0,011 9 % and decays with a half-life of $1,29 \times 10^9$ years to Ca-40 (β-decay) or Ar-40 (electron capture), respectively.

K-40 is determined by gamma spectrometry using its 1 460,83 keV line.

Table B.1 — Selected gamma lines (above 25 keV) for the determination of natural radionuclides and their interferences[11][12]

Radionuclide determined	Radionuclide measured	Energy E_γ keV	Emission probability P_E %	Interfering radionuclide (E_γ, P_E)
U-238	Th-234	63,30	3,75	Th-232 (63,811 keV; 0,259 %)
		92,38	2,18	Th-Kα1 (93,35 keV; 5,6 %)
		92,80	2,15	—
U-238	Pa-234m	766,361	0,323	—
		1 001,026	0,847	
Ra-226	Ra-226	186,211	3,555	U-235 (185,720 keV; 57,0 %)
Ra-226	Pb-214	295,224	18,414	Bi-211 (351,03 keV; 13,00 %)
		351,932	35,60	—
Ra-226	Bi-214	609,312	44,49	—
		1 120,287	14,91	—
		1 764,494	15,31	—
Pb-210	Pb-210	46,539	4,252	—
U-235	U-235	143,767	10,94	Ra-223 (144,27 keV; 3,36 %)
		163,356	5,08	—
		185,720	57,0	Ra-226 (186,211 keV; 3,555 %)
		205,316	5,02	
Ac-227	Th-227	235,97	12,3	—
Th-232	Ac-228	see Th-228		—
	Th-228			—
	Tl-208			—
Ra-228	Ac-228	209,248	3,97	—
		338,320	11,40	Ra-223 (338,282 keV; 2,85 %)
		911,196	26,20	—
		968,96	15,90	—
Th-228	Ra-224	240,986	4,12	Pb-214 (241,997 keV; 7,268 %)
Th-228	Pb-212	238,632	43,6	—
		300,089	3,18	Th-227 (300,00 keV; 2,70 %)
				Pa-231 (300,07 keV; 2,47 %)
Th-228	Tl-208	277,37	6,6	Ac-228 (278,80 keV; 0,204 %)
		583,187	85,0	Ac-228 (583,391 keV; 0,120 %)
		860,53	12,4	—
		2 614,511	99,755	—
K-40	K-40	1 460,822	10,55	Ac-228 (1 459,131 keV; 0,87 %)

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