INTERNATIONAL STANDARD

ISO 18589-6

Second edition
2019-12

Measurement of radioactivity in the environment — Soil —

Part 6:

Gross alpha and gross beta activities — Test method using gas-flow proportional counting

Mesurage de la radioactivité dans l'environnement — Sol —

Partie 6: Mesurage des activités alpha globale et bêta globale — Méthode d'essai utilisant un compteur proportionnel à circulation gazeuse

Reference number
ISO 18589-6:2019(E)

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Published in Switzerland

Contents

Page

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 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 the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 18589-6:2009), which has been technically revised.

The main change compared to the previous edition is as follows:

- The introduction has been reviewed accordingly to the generic introduction adopted for the standards published on the radioactivity measurement in the environment.

A list of all parts in the ISO 18589 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Everyone is exposed to natural radiation. The natural sources of radiation are cosmic rays and naturally occurring radioactive substances which exist in the earth and flora and fauna, including the human body. Human activities involving the use of radiation and radioactive substances add to the radiation exposure from this natural exposure. Some of those activities, such as the mining and use of ores containing naturally-occurring radioactive materials (NORM) and the production of energy by burning coal that contains such substances, simply enhance the exposure from natural radiation sources. Nuclear power plants and other nuclear installations use radioactive materials and produce radioactive effluent and waste during operation and decommissioning. The use of radioactive materials in industry, agriculture and research is expanding around the globe.

All these human activities give rise to radiation exposures that are only a small fraction of the global average level of natural exposure. The medical use of radiation is the largest and a growing man-made source of radiation exposure in developed countries. It includes diagnostic radiology, radiotherapy, nuclear medicine and interventional radiology.

Radiation exposure also occurs as a result of occupational activities. It is incurred by workers in industry, medicine and research using radiation or radioactive substances, as well as by passengers and crew during air travel. The average level of occupational exposures is generally below the global average level of natural radiation exposure (see Reference [1]).

As uses of radiation increase, so do the potential health risk and the public's concerns. Thus, all these exposures are regularly assessed in order to:

- improve the understanding of global levels and temporal trends of public and worker exposure;
- evaluate the components of exposure so as to provide a measure of their relative importance;
- identify emerging issues that may warrant more attention and study. While doses to workers are mostly directly measured, doses to the public are usually assessed by indirect methods using the results of radioactivity measurements of waste, effluent and/or environmental samples.

To ensure that the data obtained from radioactivity monitoring programs support their intended use, it is essential that the stakeholders (for example nuclear site operators, regulatory and local authorities) agree on appropriate methods and procedures for obtaining representative samples and for handling, storing, preparing and measuring the test samples. An assessment of the overall measurement uncertainty also needs to be carried out systematically. As reliable, comparable and 'fit for purpose' data are an essential requirement for any public health decision based on radioactivity measurements, international standards of tested and validated radionuclide test methods are an important tool for the production of such measurement results. The application of standards serves also to guarantee comparability of the test results over time and between different testing laboratories. Laboratories apply them to demonstrate their technical competences and to complete proficiency tests successfully during interlaboratory comparisons, two prerequisites for obtaining national accreditation.

Today, over a hundred International Standards are available to testing laboratories for measuring radionuclides in different matrices.

Generic standards help testing laboratories to manage the measurement process by setting out the general requirements and methods to calibrate equipment and validate techniques. These standards underpin specific standards which describe the test methods to be performed by staff, for example, for different types of sample. The specific standards cover test methods for:

- naturally-occurring radionuclides (including ^{40}K, ^{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);

- 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 requirements to allow an estimation of gross radioactivity of alpha- and beta-emitters in soil 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).

This document is published in several parts for use jointly or separately according to needs. These parts are complementary and are addressed to those responsible for determining the radioactivity present in soil, bedrocks and ore (NORM or TENORM). The first two parts are general in nature describe the setting up of programmes and sampling techniques, methods of general processing of samples in the laboratory (ISO 18589-1), the sampling strategy and the soil sampling technique, soil sample handling and preparation (ISO 18589-2). ISO 18589-3 to ISO 18589-5 deal with nuclide-specific test methods to quantify the activity concentration of gamma emitters radionuclides (ISO 18589-3 and ISO 20042), plutonium isotopes (ISO 18589-4) and ^{90}Sr (ISO 18589-5) of soil samples. ISO 18589-6 deals with non-specific measurements to quantify rapidly gross alpha or gross beta activities and ISO 18589-7 describes in situ measurement of gamma-emitting radionuclides.

The test methods described in ISO 18589-3 to ISO 18589-6 can also be used to measure the radionuclides in sludge, sediment, construction material and products following proper sampling procedure.

This document is one of a set of International Standards on measurement of radioactivity in the environment.

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 6:
Gross alpha and gross beta activities — Test method using gas-flow proportional counting

1 Scope

This document provides a method that allows an estimation of gross radioactivity of alpha- and beta-emitters present in soil samples. It applies, essentially, to systematic inspections based on comparative measurements or to preliminary site studies to guide the testing staff both in the choice of soil samples for measurement as a priority and in the specific analysis methods for implementation.

The gross α or β radioactivity is generally different from the sum of the effective radioactivities of the radionuclides present since, by convention, the same alpha counting efficiency is assigned for all the alpha emissions and the same beta counting efficiency is assigned for all the beta emissions.

Soil includes rock from bedrock and ore as well as construction materials and products, potery, etc. using naturally occurring radioactive materials (NORM) or those from technological processes involving Technologically Enhanced Naturally Occurring Radioactive Materials (TENORM), e.g. the mining and processing of mineral sands or phosphate fertilizer production and use.

The test methods described in this document can also be used to assess gross radioactivity of alpha- and beta-emitters in sludge, sediment, construction material and products following proper sampling procedure[2][3][4][5][7][8].

For simplification, the term “soil” used in this document covers the set of elements mentioned above.

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 11074, *Soil quality — Vocabulary*

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

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

ISO 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 18589-4, *Measurement of radioactivity in the environment — Soil — Part 4: Plutonium 238 and plutonium 239 + 240 — Test method using alpha spectrometry*

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

3 Terms and definitions

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

4 Symbols

m	mass of the test portion, expressed in kilograms;
a	activity per unit of mass, expressed in becquerel per kilogram;
A_α, A_β	activity of the standard in the α and β reference measurement standards, expressed in becquerel;
t_g	sample counting time, expressed in seconds;
t_0	background counting time, expressed in seconds;
$t_{s\alpha}$, $t_{s\beta}$	α and β reference measurement standards counting time, expressed in seconds;
$r_{g\alpha}$, $r_{g\beta}$	gross count rate per second, from the α and β windows, respectively
$r_{0\alpha}$, $r_{0\beta}$	background count rate per second, from the α and β windows, respectively;
$r_{s\alpha}$, $r_{s\beta}$	calibration count rate per second, from the α and β windows, respectively;
ε_α, ε_β	counting efficiency for α and β, respectively;
$r_{s\alpha\rightarrow\beta}$	count rate in the β window when the α reference measurement standard is measured;
χ	alpha-beta cross-talk, percentage of α count going into β window from the reference measurement standard;
$u(a)$	standard uncertainty associated with the measurement result;
U	expanded uncertainty, calculated by $U = k \cdot u(a)$ with k = 1, 2,..., expressed in becquerel per kilogram;
a^*	decision threshold, expressed in becquerel per kilogram;
$a^\#$	detection limit, expressed in becquerel per kilogram;
$a^\triangleleft$, $a^\triangleright$	lower and upper limits of the confidence interval, expressed in becquerel per kilogram.

5 Principle

Gross α and β radioactivity are determined by using gas-flow proportional counting or solid scintillation counting on a thin layer of fine soil on a planchette[1][9][10]. Gross α and β determinations are not absolute determinations of the radioactivity of the sample, but relative determinations referring to a specific α- or β-emitter that constitutes the reference measurement standard. These types of determinations are also known as the alpha index and beta index.

6 Chemical reagents and equipment

6.1 Degreasing solvent.

6.2 **Fixer**, for example cellulose nitrate (Parlodion®[1]), up to 10 g/l in acetone.

6.3 **Planchette**, stainless steel, with a diameter adapted to the measuring apparatus.

6.4 **Analytical balance**, accurate to 0,1 mg.

6.5 **Gas-flow proportional counter** or **solid-state scintillation counter** (such as ZnS), designed to discriminate between the alpha and beta radioactivity.

7 Procedure

7.1 Preparation of sources

The preparation of sources involves the following stages.

a) Clean the planchette (6.3) using a degreasing solvent (6.1).

b) Evenly deposit a known mass of the test sample, *m*, prepared in accordance with ISO 18589-2 in order to obtain the thinnest possible layer with a surface deposit below 20 mg/cm^2.

c) The mass of the sample shall fall between the maximum and the minimum values of the calibration curve.

d) Place the planchette on a horizontal surface.

e) It is recommended to cover the deposit using the fixer (for example 10 ml of Parlodion® dissolved in a 130 mm test dish) and leave it to evaporate at ambient temperature (take the same quantity of fixer as used to prepare the standard sources). The purpose of this operation is to permanently fix a thin film on the surface of the sample on the planchette.

f) Identify/label the planchette.

The source is ready for the determination of the gross α and gross β radioactivity indices using previously calibrated measuring equipment.

The planchette should be suitably stored before measuring, to avoid any risk of contamination.

IMPORTANT —

— **The self-absorption phenomena that depend on the matrix constituent as well as the thickness of the deposit tend to underestimate the gross radioactivity, especially the α radioactivity.**

— **It is difficult to prepare thin sources with a uniform thickness, which shall be constant from one preparation to another.**

7.2 Calibration

7.2.1 Principle

The counting efficiency and associated uncertainty are determined using soil reference measurement standards[11][12][13] prepared by

a) metrological bodies using the reference soil supplied;

b) the laboratory using soil of the same nature as the soil being analysed, whose homogeneity has been thoroughly evaluated. These soils become reference soils after spiking, separately, with calibrated α

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[1] Parlodion® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

and β solutions with sufficient activity to allow the activity of the natural or artificial radionuclides present in the soil to represent less than 2 % of the total activity in the reference soils.

Among the standard solutions available for artificial α-emitting radionuclides, ^{241}Am and ^{239}Pu are commonly selected. When ^{239}Pu is used, it is necessary to take into account the presence of the ^{241}Pu impurity. Among the standard solutions available for artificial β-emitting radionuclides, ^{90}Sr+^{90}Y is commonly selected. Finally, among the natural standard solutions, U_{nat} and ^{40}K are commonly selected.

An example of the method of the preparation of sources calibrated with plutonium to measure the gross α-radioactivity is given in Annex A. A similar procedure may be implemented by spiking.

The reference measurement standards shall be stored in a desiccator due to the hydrophilic nature of the soil.

It is advisable to choose the α and β windows so that the beta-alpha cross-talk is close to zero. Then it is sufficient to consider only the alpha-beta cross-talk correction factor. If this cannot be done, it is necessary to consider both the alpha-beta and the beta-alpha cross-talk correction factors.

7.2.2 Procedure

7.2.2.1 β calibration

The procedure for calibrating the counters is as follows.

a) Deposit a thin layer of the reference soil following the procedure given in 7.1.

b) Select the width of the α and β windows.

c) Select $t_{s\beta}$ to collect at least 10^4 counts in the β window.

d) Determine the count rate of the reference measurement standard in its window.

e) Verify that the count rate in the α window is zero or compatible with the equipment background count rate.

f) Calculate the counting efficiency of the counter, dividing the recorded count rate by the activity of the reference measurement standard as given by Formula (1):

$$\varepsilon_\beta = \left(r_{s\beta} - r_{0\beta}\right)/A_\beta \quad (1)$$

7.2.2.2 α calibration

The procedure for calibrating the counters is as follows.

a) Deposit a thin layer of the reference soil following the procedure given in 7.1.

b) Select the width of the α and β windows.

c) Select the $t_{s\alpha}$ to collect at least 10^4 counts in the α window.

d) Determine the count rate of the reference measurement standard in its window.

e) Calculate the alpha-beta cross-talk correction factor, χ, as given by Formula (2):

$$\chi = r_{s\alpha\rightarrow\beta}/r_{s\alpha} \quad (2)$$

where $r_{s\alpha\rightarrow\beta}$ is the count rate in the β window when the α reference measurement standard is measured.

f) Calculate the counting efficiency of the counter by dividing the recorded count rate by the activity of the reference measurement standard as given by Formula (3):

$$\varepsilon_\alpha = \left(r_{s\alpha} - r_{0\alpha} \right) / A_\alpha \tag{3}$$

7.3 Calibration curves

In general, it is difficult to produce a sample source identical to the reference measurement standard. To avoid self-absorption differences between them, it is convenient to construct a calibration curve of sample mass versus counting efficiency, ε (m). The self-absorption effect is more important in α counting.

7.4 Background determination

Measure the background using a planchette prepared in a manner similar to that for the sample source. Let the measured count rates be $r_{0\alpha}$ and $r_{0\beta}$.

7.5 Measurement

The α and β activities per unit mass are estimated by counting the sample source for an appropriate time.

The same equipment conditions should be used for measurements of the sample, the background and the reference measurement standard.

The counting time depends on the sample and background count rates and the required detection limit and decision threshold.

$r_{g\alpha}$ and $r_{g\beta}$ are corrected for the background contribution and $r_{g\beta}$ for the alpha-beta cross-talk contribution.

8 Expression of results

8.1 Activities per unit mass

8.1.1 Calculation of alpha activity per unit of mass

The gross α activity per unit of mass, a_α, is calculated as given by Formula (4):

$$a_\alpha = \frac{r_{g\alpha} - r_{0\alpha}}{m \cdot \varepsilon_\alpha (m)} = \left(r_{g\alpha} - r_{0\alpha} \right) \cdot w \tag{4}$$

where $w = \dfrac{1}{m \cdot \varepsilon_\alpha (m)}$.

8.1.2 Calculation of beta activity per unit of mass

The gross β activity per unit of mass, a_β, is calculated as given by Formula (5):

$$a_\beta = \frac{r_{g\beta} - r_{0\beta} - \chi \left(r_{g\alpha} - r_{0\alpha} \right)}{m \cdot \varepsilon_\beta (m)} = \left[r_{g\beta} - r_{0\beta} - \chi \left(r_{g\alpha} - r_{0\alpha} \right) \right] \cdot w \tag{5}$$

where $w = \dfrac{1}{m \cdot \varepsilon_\alpha (m)}$.

If the alpha-beta cross-talk can be neglected, $\chi = 0$ and Formula (5) becomes similar to Formula (4).

8.2 Standard uncertainty

8.2.1 Standard uncertainty of the alpha activity per unit of mass

According to ISO/IEC Guide 98-3, the standard uncertainty of a_α is calculated as given by Formula (6):

$$u(a_\alpha) = \sqrt{w^2 \cdot \left[u^2(r_{g\alpha}) + u^2(r_{0\alpha})\right] + {a_\alpha}^2 \cdot u_{\mathrm{rel}}^2(w)} = \sqrt{w^2 \cdot (r_{g\alpha}/t_g + r_{0\alpha}/t_0) + a_\alpha^2 \cdot u_{\mathrm{rel}}^2(w)} \tag{6}$$

where the uncertainty of the counting time is neglected.

The relative standard uncertainty of w is calculated as given by Formula (7):

$$u_{\mathrm{rel}}^2(w) = u_{\mathrm{rel}}^2\left[\varepsilon_\alpha(m)\right] + u_{\mathrm{rel}}^2(m) \tag{7}$$

and the relative standard uncertainty of ε_α is calculated as given by Formula (8):

$$\begin{aligned} u_{\mathrm{rel}}^2\left[\varepsilon_\alpha(m)\right] &= u_{\mathrm{rel}}^2(r_{s\alpha} - r_{0\alpha}) + u_{\mathrm{rel}}^2(A_\alpha) \\ &= (r_{s\alpha}/t_{s\alpha} + r_{0\alpha}/t_0) \big/ (r_{s\alpha} - r_{0\alpha})^2 + u_{\mathrm{rel}}^2(A_\alpha) \end{aligned} \tag{8}$$

$u_{\mathrm{rel}}^2(A_\alpha)$ includes all the uncertainties related to the reference measurement standard, that is, in the standard solution and the preparation of the reference measurement standard.

The calculation of the characteristic limits [in accordance with ISO 11929 (all parts)] requires the calculation of $\tilde{u}(\tilde{a}_\alpha)$, i.e. the standard uncertainty of a_α as a function of its true value, calculated as given by Formula (9):

$$\tilde{u}(\tilde{a}_\alpha) = \sqrt{w^2 \cdot \left[(\tilde{a}_\alpha/w + r_{0\alpha})/t_g + r_{0\alpha}/t_0\right] + \tilde{a}_\alpha^2 \cdot u_{\mathrm{rel}}^2(w)} \tag{9}$$

8.2.2 Standard uncertainty of the beta activity per unit of mass

According to ISO/IEC Guide 98-3, the standard uncertainty of a_β is calculated as given by Formulae (10) and (11):

$$u(a_\beta) = \sqrt{w^2 \left[(r_{g\beta}/t_g + r_{0\beta}/t_0) + T(\chi)\right] + a_\beta^2 \cdot u_{\mathrm{rel}}^2(w)} \tag{10}$$

where

$$T(\chi) = (r_{g\alpha} - r_{0\alpha})^2 u^2(\chi) + \chi^2 (r_{g\alpha}/t_g + r_{0\alpha}/t_0) \tag{11}$$

where the uncertainty of the counting time is neglected.

The relative standard uncertainty of w is calculated as given by Formula (12):

$$u_{\mathrm{rel}}^2(w)=u_{\mathrm{rel}}^2\left[\varepsilon_\beta(m)\right]+u_{\mathrm{rel}}^2(m) \tag{12}$$

The relative standard uncertainty of ε_β is calculated as given by Formula (13):

$$u_{\mathrm{rel}}^2\left[\varepsilon_\beta(m)\right]=u_{\mathrm{rel}}^2\left(r_{\mathrm{s}\beta}-r_{0\beta}\right)+u_{\mathrm{rel}}^2\left(A_\beta\right)=\left(r_{\mathrm{s}\beta}/t_{\mathrm{s}\beta}+r_{0\beta}/t_0\right)\Big/\left(r_{\mathrm{s}\beta}-r_{0\beta}\right)^2+u_{\mathrm{rel}}^2\left(A_\beta\right) \tag{13}$$

The relative standard uncertainty of χ is calculated as given by Formula (14):

$$u(\chi)=\sqrt{\chi(\chi+1)/(r_{\mathrm{s}\alpha}\cdot t_{\mathrm{s}\alpha})} \tag{14}$$

For the calculation of the characteristic limits [in accordance with ISO 11929 (all parts)], one needs $\tilde{u}\left(\tilde{a}_\beta\right)$, i.e. the standard uncertainty of a_β as a function of its true value, calculated as given by Formula (15):

$$\tilde{u}\left(\tilde{a}_\beta\right)=\sqrt{w^2\left\{\left[\tilde{a}_\beta/w+\chi\left(r_{\mathrm{g}\alpha}-r_{0\alpha}\right)+r_{0\beta}\right]/t_{\mathrm{g}}+r_{0\beta}/t_0+T(\chi)\right\}+\tilde{a}_\beta^2\cdot u_{\mathrm{rel}}^2(w)} \tag{15}$$

If the alpha-beta cross-talk can be neglected, χ = 0, and Formulae (10) and (15) become similar to Formulae (6) and (9).

8.3 Decision threshold

8.3.1 Decision threshold of the alpha activity per unit of mass

The decision threshold, a_α^* [in accordance with ISO 11929 (all parts)], is obtained from Formula (9) for $\tilde{a}_\alpha=0$, which yields to Formula (16):

$$a_\alpha^*=k_{1-\alpha}\cdot\tilde{u}(0)=k_{1-\alpha}\cdot w\cdot\sqrt{r_{0\alpha}/t_{\mathrm{g}}+r_{0\alpha}/t_0} \tag{16}$$

A significance level of α = 0,05 with $k_{1-\alpha}=1{,}65$ is often chosen by default.

8.3.2 Decision threshold of the beta activity per unit of mass

The decision threshold, a_β^* [in accordance with ISO 11929 (all parts)], is obtained from Formula (15) for $\tilde{a}_\beta=0$, which yields to Formula (17):

$$a_\beta^*=k_{1-\alpha}\cdot\tilde{u}(0)=k_{1-\alpha}\cdot w\cdot\sqrt{\left[\chi\left(r_{\mathrm{g}\alpha}-r_{0\alpha}\right)+r_{0\beta}\right]/t_{\mathrm{g}}+r_{0\beta}/t_0+T(\chi)} \tag{17}$$

A significance level of α = 0,05 with $k_{1-\alpha}=1{,}65$ is often chosen by default.

If the alpha-beta cross-talk can be neglected, χ = 0, and Formula (17) becomes similar to Formula (16).

8.4 Detection limit

8.4.1 Detection limit of the alpha activity per unit of mass

The detection limit, $a_\alpha^\#$ [in accordance with ISO 11929 (all parts)], is calculated as given by Formula (18):

$$\begin{aligned} a_\alpha^\# &= a_\alpha^* + k_{1-\beta} \cdot \tilde{u}\left(a_\alpha^\#\right) \\ &= a_\alpha^* + k_{1-\beta} \cdot \sqrt{w^2 \left[\left(a_\alpha^\# / w + r_{0\alpha}\right) / t_\mathrm{g} + r_{0\alpha} / t_0\right] + \left(a_\alpha^\#\right)^2 \cdot u_\mathrm{rel}^2(w)} \end{aligned} \tag{18}$$

A significance level of $\beta = 0{,}05$ with $k_{1-\beta} = 1{,}65$ is often chosen by default.

The detection limit can be calculated by solving Formula (18) for $a_\alpha^\#$ or, more simply, by iteration with a starting approximation of $a_\alpha^\# = 2 \cdot a_\alpha^*$.

By taking an equal significance level of $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$, and the solution of Formula (18) is given by Formula (19):

$$a_\alpha^\# = \frac{2 \cdot a_\alpha^* + \left(k^2 \cdot w\right) / t_\mathrm{g}}{1 - k^2 \cdot u_\mathrm{rel}^2(w)} \tag{19}$$

8.4.2 Detection limit of the beta activity per unit of mass

The detection limit, $a_\beta^\#$ [in accordance with ISO 11929 (all parts)], is calculated as given by Formula (20):

$$\begin{aligned} a_\beta^\# &= a_\beta^* + k_{1-\beta} \cdot \tilde{u}\left(a_\beta^\#\right) \\ &= a_\beta^* + k_{1-\beta} \cdot \sqrt{w^2 \left\{\left[a_\beta^\# / w + \chi\left(r_{\mathrm{g}\alpha} - r_{0\alpha}\right) + r_{0\beta}\right] / t_\mathrm{g} + r_{0\beta} / t_0 + T(\chi)\right\} + \left(a_\beta^\#\right)^2 \cdot u_\mathrm{rel}^2(w)} \end{aligned} \tag{20}$$

A significance level of $\beta = 0{,}05$ with $k_{1-\beta} = 1{,}65$ is often chosen by default.

The detection limit can be calculated by solving Formula (20) for $a_\beta^\#$ or, more simply, by iteration with a starting approximation of $a_\beta^\# = 2 \cdot a_\beta^*$.

If the alpha-beta cross-talk can be neglected, $\chi = 0$, and Formula (20) becomes similar to Formula (18).

By taking an equal significance level of $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$, and the solution of Formula (20) is given by Formula (21):

$$a_\beta^\# = \frac{2 \cdot a_\beta^* + \left(k^2 \cdot w\right) / t_\mathrm{g}}{1 - k^2 \cdot u_\mathrm{rel}^2(w)} \tag{21}$$

8.5 Confidence limits

The lower confidence limit, $a^\triangleleft$, and the upper confidence limit, $a^\triangleright$, are calculated using Formulae (22) and (23):

$$a^\triangleleft = a - k_p \cdot u(a); \quad p = \omega \cdot (1 - \gamma/2) \tag{22}$$

$$a^\triangleright = a + k_q \cdot u(a); \quad q = 1 - \omega \cdot \gamma/2 \tag{23}$$

where $\omega = \Phi\left[y/u(y)\right]$

where Φ is the distribution function of the standardized normal distribution.

ω may be set equal to 1 if $a \geq 4 \cdot u(a)$, yielding Formula (24):

$$a^{\triangleleft,\triangleright} = a \pm k_{1-\gamma/2} \cdot u(a) \tag{24}$$

A significance level of $\gamma = 0{,}05$ with $k_{1-\gamma/2} = 1{,}96$ is often chosen by default.

9 Test report

The test report shall be in accordance with the ISO/IEC 17025 requirements and shall contain the following information:

a) reference to this document, i.e. ISO 18589-6:2019;

b) identification of the sample;

c) units in which the results are expressed;

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

Complementary information can be provided, such as the following:

- probabilities α, β and $(1 - \gamma)$;
- the 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 [in accordance with ISO 11929 (all parts)], 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;
- any relevant information likely to affect the results.

Annex A
(informative)

Preparation of reference measurement standards with plutonium 239

A.1 Principle

A known quantity of ^{239}Pu is added to a soil sample in an aqueous medium. Any loss in activity during the operation shall be taken into account.

A.2 Preparation of the spiked sample

The preparation of the spiked sample consists of the following stages.

a) The soil shall be prepared, following the procedure described in ISO 18589-2.

b) Transfer an aliquot of this soil (approximately 5 g) into a beaker, B1, whose weight is known.

c) Dry in an oven at (105 ± 10) °C for 1 h, leave to cool in a dessicator and determine the mass of the soil present by calculating the difference in mass, m_1.

d) Add 10 ml of water and stir vigorously for 1 h until a uniform solution is obtained.

e) Add by weighing a known quantity of standard ^{239}Pu, A_{th}, and stir for approximately 1 h.

f) Heat the solution whilst stirring until the sample has solidified.

g) Desiccate in the oven at (105 ± 10) °C to a constant mass.

h) Leave it to cool, then pass the spiked sample through a 200 µm sieve using a spatula. This is the reference soil.

A.3 Determination of the activity of the spiked sample

To determine the activity of the reference soil, the following operations shall be carried out.

a) Determine the mass of the reference soil, m_2.

b) Determine the activity of the reference soil, A_{exp}, following the procedure in ISO 18589-4.

A.4 Determination of the residual activity

The residual activity corresponds to the loss of activity during the preparation of the spiked sample. To determine this activity, the following operations can be carried out.

a) Place the sieve and agitator used in beaker B1.

b) Rinse with a nitric acid solution, $c(HNO_3)$ = 4 mol/l, and retain the rinsing solution.

c) Determine the residual activity, A_{res}, of ^{239}Pu by measuring the α-activity of the rinsing solution.