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Second edition
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Water quality — Radium-226 —

Part 1:
Test method using liquid scintillation counting

Qualité de l'eau — Radium-226 —

Partie 1: Méthode d'essai par comptage des scintillations en milieu liquide

Reference number
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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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

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

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

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

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

This second edition cancels and replaces the first edition (ISO 13165-1:2013), which has been technically revised.

The main changes are as follows:

- the introduction has been updated;
- the list of symbols has been updated;
- the expression of results has been updated;
- the test report has been updated;
- the validation data has been updated;

A list of all parts in the ISO 13165 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

Radioactivity from several naturally-occurring and anthropogenic sources is present throughout the environment. Thus, water bodies (such as surface waters, ground waters, sea waters) can contain radionuclides of natural, human-made, or both origins.

— Natural 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, ^{210}Po and ^{210}Pb can be found in water for natural reasons (e.g. desorption from the soil and washoff by rain water) or can be released from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or phosphate fertilizers production and use).

— Anthropogenic radionuclides such as transuranium elements (americium, plutonium, neptunium, curium), ^{3}H, ^{14}C, ^{90}Sr and gamma emitting radionuclides can also be found in natural waters. Small quantities of these radionuclides are discharged from nuclear fuel cycle facilities into the environment as a result of authorized routine releases. Some of these radionuclides used for medical and industrial applications are also released into the environment after use. Anthropogenic radionuclides are also found in waters as a result of past fallout contaminations resulting from the explosion in the atmosphere of nuclear devices and accidents such as those that occurred in Chernobyl and Fukushima.

Radionuclide activity concentration in water bodies can vary according to local geological characteristics and climatic conditions and can be locally and temporally enhanced by releases from nuclear installations during planned, existing and emergency exposure situations.[1] Drinking water can thus contain radionuclides at activity concentrations which can present a risk to human health.

The radionuclides present in liquid effluents are usually controlled before being discharged into the environment[2] and water bodies. Drinking waters are monitored for their radioactivity content as recommended by the World Health Organization (WHO)[3] so that proper actions can be taken to ensure that there is no adverse health effect to the public. Following these international recommendations, national regulations usually specify radionuclide authorized concentration limits for liquid effluent discharged to the environment and radionuclide guidance levels for waterbodies and drinking waters for planned, existing and emergency exposure situations. Compliance with these limits can be assessed using measurement results with their associated uncertainties as specified by ISO/IEC Guide 98-3 and ISO 5667-20[4].

Depending on the exposure situation, there are different limits and guidance levels that would result in an action to reduce health risk. As an example, during planned or existing situation, the WHO guidelines for guidance level in drinking water is $1\ \text{Bq}\cdot\text{l}^{-1}$ for ^{226}Ra activity concentration.

NOTE 1 The guidance level (GL) is the activity concentration with an intake of $2\ \text{l}\cdot\text{d}^{-1}$ of drinking water for one year that results in an effective dose of $0{,}1\ \text{mSv}\cdot\text{a}^{-1}$ for members of the public. This is an effective dose that represents a very low level of risk and which is not expected to give rise to any detectable adverse health effects[7].

In the event of a nuclear emergency, the WHO Codex Guideline Levels[5] mentioned that the activity concentration can be greater.

NOTE 2 The Codex GLs apply to radionuclides contained in foods destined for human consumption and traded internationally, which have been contaminated following a nuclear or radiological emergency. These GLs apply to food after reconstitution or as prepared for consumption, i.e. not to dried or concentrated foods and are based on an intervention exemption level of 1 mSv in a year for members of the public (infant and adult)[5].

Thus, the test method can be adapted so that the characteristic limits, decision threshold, detection limit and uncertainties ensure that the radionuclide activity concentrations test results can be verified to be below the guidance levels required by a national authority for either planned/existing situations or for an emergency situation[6][7].

Usually, the test methods can be adjusted to measure the activity concentration of the radionuclide(s) in either wastewaters before storage or in liquid effluents before being discharged to the environment.

The test results will enable the plant/installation operator to verify that, before their discharge, wastewaters/liquid effluent radioactive activity concentrations do not exceed authorized limits.

The test method(s) described in this document may be used during planned, existing and emergency exposure situations as well as for wastewaters and liquid effluents with specific modifications that can increase the overall uncertainty, detection limit and threshold.

The test method(s) may be used for water samples after proper sampling, sample handling and test sample preparation (see the relevant part of the ISO 5667 series).

This document has been developed to answer the need of test laboratories carrying out these measurements, that are sometimes required by national authorities, as they can be required to obtain a specific accreditation for radionuclide measurement in drinking water samples.

This document is one of a set of International Standards on test methods dealing with the measurement of the activity concentration of radionuclides in water samples.

Water quality — Radium-226 —

Part 1:
Test method using liquid scintillation counting

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of any other restrictions.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies the determination of radium-226 (^{226}Ra) activity concentration in non-saline water samples by extraction of its daughter radon-222 (^{222}Rn) and its measurement using liquid scintillation analysis.

The test method described in this document, using currently available scintillation counters, has a detection limit of approximately 50 mBq·l^{-1}. This method is not applicable to the measurement of other radium isotopes.

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 3696, *Water for analytical laboratory use — Specification and test methods*

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

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

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

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

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 80000-10, ISO/IEC Guide 98-3 apply.

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

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

4 Symbols and units

For the purposes of this document, the symbols given in Table 1, ISO 80000-10 and ISO/IEC Guide 98-3 apply.

Table 1 — Symbols

Symbol	Description	Unit
a	Massic activity of the sample at the measuring time	Bq·g^{-1}
a_S	Massic activity of the ^{226}Ra standard solution at the measuring time	Bq·g^{-1}
a^*	Decision threshold for the massic alpha-activity	Bq·g^{-1}
$a^\#$	Detection limit for the massic alpha-activity	Bq·g^{-1}
$a^\triangleleft$, $a^\triangleright$	Lower and upper limits of the probabilistically symmetric coverage interval	Bq·g^{-1}
$a^<$, $a^>$	Lower and upper limits of the shortest coverage interval	Bq·g^{-1}
c	Concentration	mol· l^{-1}
c_A	Activity concentration	Bq·l^{-1}
k	Coverage factor	—
m	Mass of the test sample	g
m_1	Mass of initial sample subject to heating or possibly concentration	g
m_2	Mass of heated or concentrated sample	g
m_3	Mass of heated or concentrated sample transferred in the vial	g
m_S	Mass of ^{226}Ra standard solution used for the preparation of the calibration sample	g
p	Probability (for instance $p = 1 - \alpha$, $1- \beta$ or $1- \gamma /2$)	—
q	Probability	—
r_0	Blank sample count rate in the alpha-window	s^{-1}
r_g	Sample gross count rate in the alpha-window	s^{-1}
r_S	Count rate of the calibration sample in the alpha-window	s^{-1}
t_0	Blank sample counting time	s
t_g	Sample counting time	s
t_S	Calibration sample counting time	s
$u(a)$	Standard uncertainty associated with the measurement result	Bq·l^{-1}
$\tilde{u}(\tilde{a})$	Standard uncertainty of a as a function of its true value	—
u_rel	Relative standard uncertainty	—
u_c	Combined uncertainty	—
U	Expanded uncertainty, calculated using $U = ku(a)$, with $k = 1, 2, \ldots$	Bq·l^{-1}
w	Factor equal to $1/\varepsilon\, m$	—
ε	Alpha-efficiency, relative	—
ρ	Density	g·l^{-1}
Φ	Distribution function of the standardized normal distribution	—
ω	Auxiliary quantity	—
γ	Coverage interval probability	—

5 Principle

The massic activity of ^{226}Ra is indirectly determined by isolating its progeny ^{222}Rn by liquid scintillation counting (LSC). ^{222}Rn is in secular equilibrium with its parent, ^{226}Ra, after 30 d (99,56 %). ^{222}Rn is extracted from the aqueous solution using a scintillation cocktail, within the scintillation vial, that is immiscible in water (see References [8], [9], [10] and [11]).

The aqueous sample is acidified, heated and, if possible, concentrated by slow evaporation in order to desorb ^{222}Rn and to achieve a better detection limit. The concentrated aqueous sample is transferred into a radon-tight scintillation vial and a water-immiscible scintillation cocktail is added.

After 30 d, the sample is measured by liquid scintillation counting (LSC) applying alpha and beta discrimination; only alpha-emission of ^{222}Rn and that of its short-lived progenies (^{218}Po and ^{214}Po) are considered, as this counting condition ensures a better detection limit.

6 Reagents and equipment

6.1 Reagents

All reagents shall be of recognized analytical grade and, except for 6.1.4, shall not contain any detectable alpha- and beta-activity.

6.1.1 Laboratory water, distilled or deionized, conforming with ISO 3696, grade 3.

Deionized water can contain detectable amounts of ^{222}Rn and short-lived progeny. It is therefore strongly recommended that water be boiled under vigorous stirring and allowed to stand for 1 d before use. Otherwise, purge it with nitrogen for about 1 h for 2 l.

6.1.2 Nitric acid, $c(\text{HNO}_3) = 15{,}8\ \text{mol}\cdot \text{l}^{-1}$, $\rho = 1{,}42\ \text{g ml}^{-1}$, mass fraction $w(\text{HNO}_3) = 70\ \%$.

6.1.3 Scintillation cocktail, commercially available scintillation cocktails, water immiscible and suitable for alpha and beta discrimination (e.g. diisopropylnaphthalene-based cocktails).

6.1.4 ^{226}Ra standard solution

^{226}Ra standard solutions shall be provided with calibration certificates containing at least the activity concentration, measurement uncertainty and/or statement of compliance with an identified metrological specification.

6.2 Equipment

6.2.1 Balance.

6.2.2 Hotplate with magnetic stirrer and stirring bar.

6.2.3 pH-meter.

6.2.4 Wide-mouth high density polyethylene (HDPE) sample bottles, volumes between 100 ml and 500 ml.

6.2.5 Liquid scintillation counter, with alpha and beta discrimination option, with thermostated counting chamber and preferably an ultra-low level counter to achieve better detection limits.

6.2.6 Polyethylene scintillation vials, polytetrafluoroethylene (PTFE) coated, 20 ml.

PTFE-coated polyethylene vials are the best choice, since they prevent both the diffusion of the cocktail into the wall of the vial and the absorption of radon from the environment. Glass vials exhibit a considerably higher background and generally degrade the achievable alpha and beta discrimination.

7 Sampling

It is the responsibility of the laboratory to ensure the suitability of this test method for the water samples tested.

Collect the sample in accordance with ISO 5667-1. Store the water sample (from 0,1 l to 1 l) in a bottle (6.2.4) according to ISO 5667-3.

If necessary, carry out filtration immediately on collection and before acidification.

When pre-concentration is desired, acidify the sample to pH 1 to pH 3 with HNO_3 (6.1.2).

The acidification of the water sample minimizes the loss of radioactive material from solution by plating on the wall of the sample container. If filtration of the sample is required, the acidification is performed afterwards, otherwise radioactive material already adsorbed on the particulate material can be desorbed.

If the sample is not acidified, the sample preparation should start as soon as possible and always less than one month after the sampling date (see ISO 5667-3).

8 Instrument set-up and calibration

8.1 Preparation of calibration sources

Transfer an accurately known mass, m_S, of the ^{226}Ra standard solution (6.1.4) into a scintillation vial (6.2.6). Let the massic activity of the ^{226}Ra standard solution at the measuring time be a_s. Dilute with water (6.1.1) to the previously chosen volume, for example, 10 ml. Add the scintillation cocktail (6.1.3), for example, 10 ml.

Store the sample for at least 30 d to reach secular equilibrium.

Ensure that the diluted standard solutions are between pH 0 and pH 2.

Store samples so as to ensure optimum preservation. Storage in the dark is recommended. Select a single generally applicable temperature in order not to affect distribution coefficients. This temperature shall be consistent with the characteristics of the scintillation cocktail (6.1.3, see manufacturer's instructions). Generally, if possible, storage in the scintillation chamber at around 15 °C is suitable.

8.2 Optimization of counting conditions

Set the alpha-counting window so that the energies of all the three alpha-emitters present in the cocktail phase: ^{222}Rn (5,49 MeV); ^{218}Po (6,00 MeV); and ^{214}Po (7,69 MeV); are covered. The observed energy range should also include ^{212}Po (8,78 MeV) as discussed in Clause 12.

Count the ^{226}Ra calibration sample in alpha and beta-discrimination mode (see manufacturer instructions) for an appropriate period, under different discriminator settings.

The best discriminator setting (working point) is chosen by visual inspection of the spectra in order to obtain an alpha-spectrum free of beta counts (see Annex A).

NOTE Since no water is present in the scintillation cocktail phase, the quenching is low and constant, while the alpha and beta discrimination is quite sharp.

8.3 Detection efficiency

Let the counting rate be r_S for the counts of the ^{226}Ra calibration sample in the alpha-window, as measured with the previously defined best discriminator setting.

Determine the relative alpha-efficiency given in Formula (1):

$$\varepsilon = \frac{r_S - r_0}{a_S\, m_S} \tag{1}$$

Acceptance limits for efficiency should be defined.

NOTE The alpha-efficiency includes both counting and extraction efficiency. Usual values (in percent) are in the range 200 % to 300 % (^{222}Rn, ^{218}Po and ^{214}Po alpha-emissions).

It is advisable to check the linearity of the method. Assess the efficiency using calibration samples whose activities cover the whole working range.

A more accurate estimate of efficiency can be obtained by preparing and measuring a sufficient number of calibration samples.

Verify efficiencies at a periodicity established by the laboratory and whenever there are changes in materials (e.g. scintillation cocktail) or when maintenance operations are performed on the scintillation counter (6.2.5). A verification or a recalibration is necessary when the quality control requirements of the instrument (see Clause 10) are not met.

8.4 Blank sample preparation and measurement

Acidify a laboratory water sample to between pH 0 and pH 2. Transfer the chosen quantity, for example, 10 ml, into the scintillation vial (6.2.6). Add the scintillation cocktail (6.1.3), for example, 10 ml, and mix thoroughly.

Store the blank sample for 30 d and then count it using the chosen optimum counting conditions. Let the measured counting rate in the alpha-window be r_0. If a preconcentration procedure is normally employed, prepare blank samples by the same method.

Acceptance limits for blank samples should also be defined on the basis of the sensitivity desired. The use of control charts (see ISO 7870-2[12]) is advisable for this purpose.

It is recommended that blank samples be counted for the same period of time as the test portions.

Perform blank measurements at a periodicity established by the laboratory (e.g. monthly) and whenever changes in materials (e.g. scintillation cocktail batch) or when maintenance operations are made on the scintillation counter (6.2.5). Verification or a recalibration is necessary when instrument quality control requirements (see Clause 10) are not met.

9 Procedure

9.1 Direct counting

Transfer a weighed (6.2.1) aliquot (m_1) of the initial water sample (approximately 50 g) into a beaker. If the sample has not yet been acidified, adjust to pH 0 and pH 2 using nitric acid (6.1.2) and verify with a pH-meter (6.2.3).

Heat to approximately 80 °C under stirring for 30 min in a covered beaker, to allow degassing of dissolved ^{222}Rn. Allow the sample to cool and reweigh it to account for loss due to evaporation (m_2).

9.2 Thermal preconcentration

Thermal preconcentration can be used when soft waters are examined (e.g. dry residue <500 $\text{mg}\cdot\text{l}^{-1}$, as in most drinking waters) in order to decrease the detection limit of the method. Hard waters can give rise to salt precipitations which are difficult to dissolve completely. If unknown, only a rough evaluation of the dry residue is needed. Any commonly used technique can be adopted.

Transfer a weighed (6.2.1) aliquot (m_1) of the initial water sample (about 200 g) into a beaker. If the sample has not yet been acidified, adjust to between pH 1 and pH 3 with nitric acid (6.1.2) and verify with a pH-meter (6.2.3).

Slowly evaporate the sample on a hotplate (6.2.2) down to about 20 g. Allow the sample to cool to room temperature and weigh the concentrated sample (m_2). Adjust the concentrated sample to between pH 0 and pH 2.

No precipitation should be observable, otherwise direct counting (see 9.1) or smaller preconcentration factors shall be applied.

If unknown, only a rough evaluation of the dry residue is needed. Any commonly used technique can be adopted.

NOTE The volume of acid required is small (normally about 0,1 ml of concentrated HNO_3 in 200 g sample when preconcentration is adopted) and its mass can be neglected.

9.3 Sample preparation

Transfer a weighed (6.2.1) amount (m_3) of the heated or concentrated sample (e.g. 10 ml) into a scintillation vial (6.2.6). Add the scintillation cocktail (6.1.3), for example, 10 ml, clean the outside vial surface with ethanol and store for 30 d (see 8.1, paragraph 2). Calculate the exact mass, m, of test sample analysed as indicated in Formula (2):

$$m = \frac{m_1\, m_3}{m_2} \tag{2}$$

9.4 Sample measurement

Count the sample after at least 30 d from its preparation using the chosen optimum counting conditions. Let the count rate in the alpha-window be r_g.

The counting time depends on the sample count rate and also on precision and detection limit required.

Avoid delays considerably longer than 30 d since chemical degradation of the scintillation cocktail can occur.

10 Quality control

Measurement methods shall be selected and associated procedures performed by suitably skilled staff under a quality assurance programme with quality control.

Maintain confidence in the measurement results by regular use of certified reference materials and participation in interlaboratory comparisons and proficiency testing in accordance with ISO/IEC 17025.

Laboratory procedures shall ensure that laboratory and equipment contamination as well as cross-sample contamination is avoided.

11 Expression of results

11.1 Calculation of massic activity

Calculate the ^{226}Ra massic activity, a, of the test water sample using Formula (3):

$$a = \frac{r_g - r_0}{\varepsilon\, m} = \left(r_g - r_0\right) w \qquad w = \frac{1}{\varepsilon\, m} \tag{3}$$

To express the result as an activity concentration, c_A, in becquerels per litre, multiply the initial result expressed in becquerels per gram by the density, ρ, in gram per litre, of the water sample, i.e. $c_A = \rho a$.

11.2 Standard uncertainty

According to ISO/IEC Guide 98-3, the standard uncertainty of a is calculated by Formula (4):

$$u(a) = \sqrt{w^2\left[u^2\left(r_g\right) + u^2\left(r_0\right)\right] + a^2\, u_{rel}^2(w)} = \sqrt{w^2\left(r_g / t_g + r_0 / t_0\right) + a^2\, u_{rel}^2(w)} \tag{4}$$

where the uncertainty of the counting time is neglected.

The relative standard uncertainty of w is calculated by Formula (5):

$$u_{rel}^2(w) = u_{rel}^2(\varepsilon) + u_{rel}^2(m) \tag{5}$$

The relative standard uncertainty of ε is calculated by Formula (6):

$$u_{rel}^2(\varepsilon) = u_{rel}^2\left(r_S - r_0\right) + u_{rel}^2\left(a_S\right) + u_{rel}^2\left(m_S\right) = \frac{\left(r_S / t_S + r_0 / t_0\right)}{\left(r_S - r_0\right)^2} + u_{rel}^2\left(a_S\right) + u_{rel}^2\left(m_S\right) \tag{6}$$

If replicate efficiency determinations are available, average efficiency and its uncertainty should be accordingly calculated.

The relative standard uncertainty of m is calculated using Formula (7):

$$u_{rel}^2(m) = u_{rel}^2\left(m_1\right) + u_{rel}^2\left(m_2\right) + u_{rel}^2\left(m_3\right) \tag{7}$$

Mass uncertainties $u_{rel}(m_1)$, $u_{rel}(m_2)$ and $u_{rel}(m_3)$ should be estimated based on laboratory experience and can be greater than balance uncertainty since they also take into account also the occurrence of phenomena like sample evaporation.

For the calculation of the characteristic limits according to ISO 11929-1,[13] $\tilde{u}(\tilde{a})$, i.e the standard uncertainty of a as a function of its true value, is required from Formula (8):

$$\tilde{u}(\tilde{a}) = \sqrt{w^2\left[\frac{\left(\tilde{a}/w + r_0\right)}{t_g} + \frac{r_0}{t_0}\right] + \tilde{a}^2\, u_{rel}^2(w)} \tag{8}$$

11.3 Decision threshold

The decision threshold, a^*, is obtained from Formula (8) for $\tilde{a}=0$. The yields of which are given in Formula (9):

$$a^* = k_{1-\alpha}\,\tilde{u}(0) = k_{1-\alpha}\,w\sqrt{\frac{r_0}{t_g}+\frac{r_0}{t_0}} \tag{9}$$

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

11.4 Detection limit

The detection limit, $a^\#$, is calculated using Formula (10):

$$a^\# = a^* + k_{1-\beta}\,\tilde{u}\left(a^\#\right) = a^* + k_{1-\beta}\sqrt{w^2\left[\frac{\left(a^\#/w+r_0\right)}{t_g}+\frac{r_0}{t_0}\right]+a^{\#2}u_{\mathrm{rel}}^2(w)} \tag{10}$$

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

The detection limit can be calculated by solving Formula (10) for $a^\#$ or, more simply, by iteration with a starting approximation $a^\# = 2a^*$.

When taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k'$, the solution of Formula (10) is given by Formula (11):

$$a^\# = \frac{2a^* + \left(k'^2\,w\right)/t_g}{1-k'^2\,u_{\mathrm{rel}}^2(w)} \tag{11}$$

11.5 Limits of the coverage intervals

11.5.1 Limits of the probabilistically symmetric coverage interval

In accordance with ISO 11929-1[13], the lower, $a^\triangleleft$, and upper, $a^\triangleright$, limits of the coverage interval are calculated using Formulae (12) and (13):

$$a^\triangleleft = a - k_p\,u(a);\quad p = \omega(1-\gamma/2) \tag{12}$$

$$a^\triangleright = a + k_q\,u(a);\quad q = 1-\omega\gamma/2 \tag{13}$$

where

$$\omega = \Phi\left[\frac{y}{u(y)}\right]$$

in which Φ is the distribution function of the standardized normal distribution.

$1-\gamma$ is the probability for the coverage interval of the measurand;

$\omega = 1$ may be set if $a \geq 4u(a)$ and Formula (14) applies:

$$a^{\triangleleft,\triangleright} = a \pm k_{1-\gamma/2}\,u(a) \tag{14}$$

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

11.5.2 Shortest coverage interval

As described in detail in ISO 11929-1[13], the lower limit of the shortest coverage interval, $a^{<}$, and the upper limit of the shortest coverage interval, $a^{>}$, are calculated from a primary measurement result, *a*, of the measurand and the standard uncertainty, *u*(*a*), associated with *a*, either by Formula (15):

$$a^{<}, a^{>} = a \pm k_p \,; p = (1 + \omega \cdot (1 - \gamma))/2 \tag{15}$$

or if $a^{<} < 0$ were the result, by Formula (16):

$$a^{<} = 0; a^{>} = a \pm k_q \cdot u(a); q = 1 - \omega \cdot \gamma \tag{16}$$

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

The relations $0 \le a^{<} < a^{>}$ apply and the approximation of Formula (14) is valid.

11.6 Calculations using the activity concentration

The activity concentration can be calculated by multiplying the massic activity by the density, *ρ*, in gram per litre, as given in Formula (17) and Formula (18):

$$c_A = \frac{r_g - r_0}{\varepsilon\, m} \rho = (r_g - r_0) w$$

$$w = \frac{\rho}{\varepsilon \; m} \tag{17}$$

$$u_{\mathrm{rel}}^2(w) = u_{\mathrm{rel}}^2(\varepsilon) + u_{\mathrm{rel}}^2(m) + u_{\mathrm{rel}}^2(\rho) \tag{18}$$

The uncertainty, the characteristic limits and the limits of the coverage interval can be calculated using the previous expression [see Formulae (4), (9), (10), (12) and (13)].

12 Interference control

In ground waters, concentrations of ^{222}Rn can be some orders of magnitude higher than those of ^{226}Ra. Insufficient removal of ^{222}Rn can lead to an overestimation of ^{226}Ra activity.

If a relevant amount of ^{224}Ra is present, ^{220}Rn can be absorbed in the scintillation cocktail phase and interfere with ^{226}Ra assessment. Interference due to ^{220}Rn and its progeny is indicated by the detection of the ^{212}Po alpha-peak (8,78 MeV), whose energy is higher than any other alpha-particle involved. When ^{212}Po is present, the peak is clearly visible on the right side of the alpha-spectrum. When the ^{212}Po peak is detected, this method does not produce a reliable estimate of ^{226}Ra and should not be used.

13 Test report

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

a) reference to this document (ISO 13165-1:2022);

b) identification of the sample;

c) units in which the results are expressed;

d) the test result:

  1) when the massic activity, a is compared with the decision threshold (see ISO 11929-1);

   — if the result is less than the decision threshold, the result of the measurement is expressed as $\leq a^{*}$;

   — if the result is greater than the decision threshold, the result of the measurement is expressed as $a \pm u_c(a)$ or $a \pm U$ with the associated k value,

  2) when the massic activity, a is compared with the detection limit;

   — if the result is less than the detection limit, the result of the measurement is expressed as $\leq a^{\#}$;

   — if the result is greater than the detection limit, the result of the measurement is expressed as $a \pm u_c(a)$ or $a \pm U$ with the associated k value;

e) the method used (if the standard includes several);

f) the result(s), including a reference to the clause which explains how the results were calculated;

g) any deviations from the procedure;

h) any unusual features observed;

i) the date of the test.

Complementary information can be provided such as:

j) the uncertainty can also be expressed as the limits of the probabilistically symmetric coverage interval $a^{\triangleleft}, a^{\triangleright}$ and/or the limits of the shortest coverage interval $a^{<}, a^{>}$;

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

l) decision threshold and the detection limit;

m) if the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose;

n) mention of any relevant information likely to affect the results.

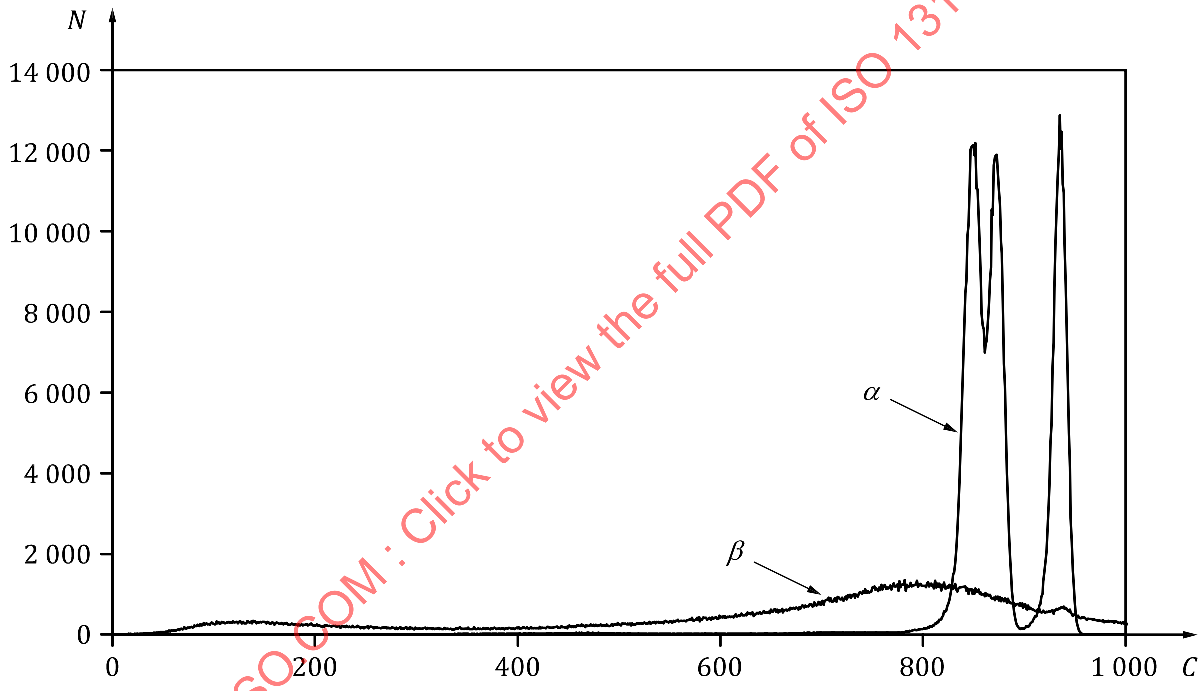
NOTE Occasionally, it is requested by the customer or regulator to compare the primary measurement result, a, with the detection limit, $a^{\#}$, in order to decide whether the physical effect is recognized or not. Such stipulations are not in accordance with ISO 11929-1. They have the consequence that it is decided too frequently that the physical effect is absent when in fact it is not absent.

Annex A
(informative)

Set-up parameters and validation data[13]

A.1 Instrument set-up and calibration

Measurements are performed by applying alpha and beta discrimination with the discriminator setting chosen by visual inspection of spectra (see 8.2). Alpha and beta spectra appear in Figure A.1 (counter: PerkinElmer Quantulus 1220; scintillation cocktail: PerkinElmer Ultima Gold F; vial: Zinsser Polyvials SLD).



Key

N	net counts	*C*	channel numbers

Figure A.1 — LSC spectrum

This annex considers an alpha-counting window between channels 750 and 950.

Three replicate sources containing 15 Bq of ^{226}Ra are prepared (see 8.1) and measured. The alpha-efficiency (extraction plus counting) is determined (see Table A.1).

For preparation of blank samples, see 8.4. Blank samples are prepared and measured (60 000 s) every month over one year. Results are reported in Table A.1. The uncertainty is calculated as a standard deviation of the whole set of measurements.