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Water quality — Radon-222 —

Part 3: Test method using emanometry

Qualité de l'eau — Radon 222 —

Partie 3: Méthode d'essai par émanométrie

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Foreword

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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, www.iso.org/directives.

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The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

ISO 13164 consists of the following parts, under the general title *Water quality — Radon-222*:

— *Part 1: General principles*

— *Part 2: Test method using gamma-ray spectrometry*

— *Part 3: Test method using emanometry*

The following part is under preparation:

— *Part 4: Test method using two-phase liquid scintillation counting*

Introduction

Radioactivity from several naturally occurring and human-made sources is present throughout the environment. Thus, water bodies (surface waters, groundwaters, sea waters) can contain radionuclides of natural and human-made origin.

- Natural radionuclides, including potassium-40, and those of the thorium and uranium decay series, in particular radium-226, radium-228, uranium-234, uranium-238, lead-210, can be found in water for natural reasons (e.g. desorption from the soil and wash-off by rain water) or releases 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, curium), tritium, carbon-14, strontium-90 and gamma-emitting radionuclides can also be found in natural waters as they can be authorized to be routinely released into the environment in small quantities in the effluent discharged from nuclear fuel cycle facilities and following their used in unsealed form in medicine or industry. They are also found in water due to the past fallout of the explosion in the atmosphere of nuclear devices and the accidents at Chernobyl and Fukushima.

Drinking-water can thus contain radionuclides at activity concentration which could present a risk to human health. In order to assess the quality of drinking-water (including mineral waters and spring waters) with respect to its radionuclide content and to provide guidance on reducing health risks by taking measures to decrease radionuclide activity concentrations, water resources (groundwater, river, lake, sea, etc.) and drinking water are monitor for their radioactivity content as recommended by the World Health Organization (WHO).

Standard test methods for radon-222 activity concentrations in water samples are needed by test laboratories carrying out such measurements in fulfillment of national authority requirements. Laboratories may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

The radon activity concentration in surface water is very low, usually below 1 Bq l^{-1}. In groundwater, the activity concentration varies from 1 Bq l^{-1} up to 50 Bq l^{-1} in sedimentary rock aquifers, from 10 Bq l^{-1} up to 300 Bq l^{-1} in wells, and from 100 Bq l^{-1} up to 1 000 Bq l^{-1} in crystalline rocks. The highest activity concentrations are normally measured in rocks with high concentration of uranium (Reference [15]).

High variations in the activity concentrations of radon in aquifers have been observed. Even in a region with relatively uniform rock types, some well water may exhibit radon activity concentration greatly higher than the average value for the same region. Significant seasonal variations have also been recorded (see Annex A).

Water may dissolve chemical substances as it passes from the soil surface to an aquifer or spring waters. The water may pass through or remain for some time in rock, some formations of which may contain a high concentration of natural radionuclides. Under favourable geochemical conditions, the water may selectively dissolve some of these natural radionuclides.

Guidance on radon in drinking-water supplies provided by WHO in 2008 suggests that controls should be implemented if the radon concentration of drinking-water for public water supplies exceeds 100 Bq l^{-1}. It also recommended that any new, especially public, drinking-water supply using groundwater should be tested prior to being used for general consumption and that if the radon concentration exceeds 100 Bq l^{-1}, treatment of the water source should be undertaken to reduce the radon levels to well below that level (Reference [16]).

This International Standard is one of a series dealing with the measurement of the activity concentration of radionuclides in water samples.

The origin of radon-222 and its short-lived decay products in water and other measurement methods are described generally in ISO 13164-1.

Water quality — Radon-222 —

Part 3:
Test method using emanometry

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 ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This part of ISO 13164 specifies a test method for the determination of radon-222 activity concentration in a sample of water following its transfer from the aqueous phase to the air phase by degassing and its detection. It gives recommendations for rapid measurements performed within less than 1 h.

The radon-222 activity concentrations, which can be measured by this test method utilizing currently available instruments, range from 0,1 Bq l^{-1} to several hundred thousand becquerels per litre for a 100 ml test sample.

This test method is used successfully with drinking water samples. The laboratory is responsible for ensuring the validity of this test method for water samples of untested matrices.

This test method can be applied on field sites or in the laboratory.

Annexes A and B give indications on the necessary counting conditions to meet the required sensitivity for drinking water monitoring.

2 Normative references

The following referenced documents are indispensable for the application 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 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 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 13164-1, *Water quality — Radon-222 — Part 1: General principles*

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*

IEC 61577-1, *Radiation protection instrumentation — Radon and radon decay product measuring instruments — Part 1: General principles*

IEC 61577-2, *Radiation protection instrumentation — Radon and radon decay product measuring instruments — Part 2: Specific requirements for radon measuring instruments*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 80000-10 and ISO 13164-1 apply.

3.2 Symbols

For the purposes of this document, the symbols defined in ISO 80000-10. ISO 13164-1, and the following apply.

c	measured radon activity concentration in the air of the measuring system after degassing, in becquerels per cubic metre
c_0	radon activity concentration in the air of the measuring system before degassing, in becquerels per cubic metre
c_A	activity concentration of radon in water, in becquerels per litre
c_A^*	decision threshold, in becquerels per litre
$c_A^\#$	detection limit, in becquerels per litre
$c_A^{\triangleleft}$, $c_A^{\triangleright}$	lower and upper limits of the confidence interval, in becquerels per litre
f_c	conversion factor from cubic metre to litre: 0,001
f_d	correction factor for the decay of radon during time interval t, dimensionless
k_p, k_q	quantiles of the standardized normal distribution for the probabilities, p and q, respectively
L	Ostwald coefficient
$T_{\mathrm{H_2O}}$	water temperature, in Celsius
t	time interval between the sampling and the measurement, in seconds
U	expanded uncertainty calculated by $U = ku(c_A)$ with $k = 2$
$u(c_A)$	standard uncertainty associated with the measurement result
$V_{\mathrm{H_2O}}$	volume of test sample, in litres
V_a	volume of air in the measurement system, in cubic metres
α, β	probability of the error of the first and second kind, respectively
γ	probability for the confidence interval of the activity concentration
λ	decay constant of radon-222, in reciprocal second
Φ	distribution function of the standardized normal distribution

4 Principle

The determination of radon-222 activity concentration in water by degassing into the air phase is based on the:

- collection of a representative sample of the water at time *t* in a suitable container;
- transfer of radon dissolved in the water to the air phase by degassing;
- detection of the alpha-radiation emitted by the radon or its solid decay products present in the air.

The radon activity concentration in the water is determined from the activity concentration in the air phase, taking account of the Ostwald coefficient (see ISO 13164-1).

5 Sampling

5.1 General requirement

The sample shall be representative of the environment to be analysed at a given time.

5.2 Sampling requirement

The sampling shall be carried out in compliance with the conditions and techniques specified in ISO 5667-1, ISO 5667-3, and ISO 13164-1. The temperature of the water shall be measured and recorded during the sampling process.

Fill the container completely and fit the cap in such a way as to avoid the presence of air above the sample.

The container shall be filled in such a way as to avoid degassing the radon in the water sample. The sampling techniques to be used vary according to the actual situation.

When the analytical laboratory is not in charge of sampling, the laboratory shall supply the container for the measurement and specify the sampling procedure to the person carrying out the sampling operation.

It is recommended that several discrete samples be taken in case of problems arising in relation to the sampling conditions or transportation of the samples.

5.3 Sample volume

Experience shows that a sample volume of at least 1 l is needed for the sample to be representative of the environment to be analysed.

At least 1 l samples are recommended, but for the effective determination smaller test portions are used.

5.4 Container characteristics

The choice and preparation of a suitable container are important (see ISO 5667-3).

The container and cap used to contain the sample shall comply with the following requirements.

- They shall be made from inert materials, impermeable to radon, non-hydrophobic, and conductive (in order not to adsorb radon and its decay products from the surrounding atmosphere).
- They shall be shock-proof.

The volume of the container should be compatible with the water volume required by the degassing technique used.

6 Transportation and storage

During transportation and storage, the sample shall be maintained at a temperature below that of the original water (but above 0 °C) until it is ready for analysis. The container shall be protected and tightly sealed. The container shall be packed in an appropriate manner in order to prevent any leakage.

The period of transportation and storage prior to analysis shall be as short as possible given the half-life of radon-222, the expected activity concentration, and the detection limit of the measurement method to be used.

On arrival at the laboratory, the sample shall be maintained at a temperature below that of the original water (but above 0 °C), if it cannot be analysed immediately. The sample shall be analysed as soon as possible.

Experience indicates that it is essential that the time between sampling and analysis not exceed 48 h.

7 Transfer of radon by degassing

7.1 Purpose

This technique is used to transfer the radon dissolved in the water into the air so that it can be detected and measured in its gaseous state.

7.2 Principle

As the Ostwald coefficient of radon in water is fairly low, the dissolved radon degasses naturally into the air with relatively slow kinetics (over a few hours) (see ISO 13164-1).

In order to accelerate the degassing process, several means may be used:

- shaking the sample;
- sparging radon-free air through the water sample using a fine air bubble to increase the air exchange surface;
- decreasing the pressure in the air phase.

In order to improve the detection limit of the measurement method, it is necessary for the radon activity concentration in the air used for the degassing process to be as low as possible and to be measured before degassing the radon from the water.

8 Detection

8.1 Objective

The purpose of the detector is to quantify the alpha-radiation emitted by the radon and/or its solid decay products that is directly related to the activity concentration of the radon in the air phase.

8.2 Principle

A number of detection techniques can be used (see ISO 11665-1[1]).

8.3 Silver-activated zinc sulfide ZnS(Ag) scintillation

Some electrons in scintillating media, such as ZnS(Ag), have the particular feature of emitting photons by returning to their ground state when they are excited by an alpha-particle. These emitted photons can be detected using a photomultiplier.

This is the principle adopted for scintillation cells (such as Lucas cells) used for radon spot measurement (see References [5]–[7] and ISO 11665-6[3]).

8.4 Air ionization

When it travels through the air, each alpha-particle creates several tens of thousands of ion pairs which, under some experimental conditions, produce an ionization current. Although very low, this current can be measured using an ionization chamber that gives the activity concentration of radon and its decay products. When the sampling is performed through a filtering medium, only radon diffuses into the ionization chamber and the signal is proportional to the radon activity concentration (see References [8]–[10] and ISO 11665-5[2]).

8.5 Semiconductor (alpha-detection)

A semiconductor detector, e.g. made of silicon, converts the energy from an incident alpha-particle into electric charges. These are converted into pulses with an amplitude proportional to the energy of the alpha-particles emitted by the radon and its short-lived decay products (see References [11]–[13]).

NOTE This detection principle is occasionally associated with electrostatic precipitation of the alpha-emitter isotopes.

9 Quality assurance and quality control programme

9.1 General

Quality control operations shall meet the requirements of ISO/IEC 17025.

9.2 Influence quantities

Various quantities can lead to measurement bias that could induce non-representative results. In the specific case of the emanometric method, influence quantities can affect the following stages in the measurement process: sampling; transportation and storage of the sample; transfer of radon from the aqueous phase to another; and the measurement of the radon activity concentration.

During the sampling, consider particularly the:

- water temperature;
- turbulence in the water;
- volume of air in the container.

During the transfer of the radon from the water to the air by degassing, the influence of the water temperature shall be taken into account.

During measurement, consider particularly the:

- detector storage conditions prior to beginning the measurement;
- stability of the characteristics of the detection system (contamination of the detection surface, saturation, etc.);
- possible presence of other alpha-emitters (radon isotopes) in the detection volume.

When the delay between the sampling and the analysis is too long, it is possible that the presence of dissolved radium in the water needs to be taken into account as an influence quantity.

When the presence of ^{226}Ra is suspected, take a second measurement of the same sample after a period equal to 10 half-lives of ^{222}Rn (38 days). If the radon activity concentration is insignificant relative to the initial measurement result, the contribution of ^{226}Ra is considered to be negligible. If this is not the case, determine the activity concentration of ^{226}Ra present in the water sample.

9.3 Instrument verification

Major instrument parameters (efficiency, background) shall be periodically checked within a quality assurance programme established by the laboratory and following the manufacturer's instructions.

9.4 Method verification

Periodically verify the accuracy of the method by:

- participating in intercomparison exercises;
- analysing reference materials.

Method repeatability shall also be checked, e.g. by replicate measurements.

The acceptance limits of the tests mentioned in the preceding shall be defined.

9.5 Demonstration of analyst capability

If an analyst has not used this procedure before, a precision and bias test shall be performed by running a duplicate measurement of a reference or spiked material. Acceptance limits shall be defined by the laboratory.

A similar test shall be performed by analysts routinely using this procedure with a periodicity defined by the laboratory. Acceptance limits shall be defined.

10 Expression of results

10.1 Activity concentration

The activity concentration of radon in the water, c_A, expressed at the date and time of sampling can be obtained using Formula (1):

$$c_A = (c - c_0)\left(L + \frac{V_a}{V_{H_2O}}\right) f_c f_d = (c - c_0)\omega \tag{1}$$

where

$$\omega = \left(L + \frac{V_a}{V_{H_2O}}\right) f_c f_d \tag{2}$$

$$f_d = \exp(\lambda t) \tag{3}$$

The Ostwald coefficient may be expressed by Formula (4) (Reference [14]):

$$L = 0{,}105 + 0{,}403\exp\left(-0{,}050\,2\,T_{H_2O}\right) \tag{4}$$

10.2 Standard uncertainty of the activity concentration

According to ISO/IEC Guide 98-3,[4] the standard uncertainty of c_A is calculated as given in Formula (5):

$$u(c_A)=\sqrt{\omega^2\left[u^2(c)+u^2(c_0)\right]+c_A^2u_{\mathrm{rel}}^2(\omega)} \qquad (5)$$

where

$$u_{\mathrm{rel}}^2(\omega)=\left[\frac{u^2(V_{\mathrm{a}})}{V_{\mathrm{H_2O}}^2}+\frac{V_{\mathrm{a}}^2u^2(V_{\mathrm{H_2O}})}{V_{\mathrm{H_2O}}^4}\right]\Big/\omega^2 \qquad (6)$$

where the standard uncertainties of the Ostwald coefficient, L, f_{c}, and f_{d} are neglected.

10.3 Decision threshold and detection limit

Calculate the characteristic limits associated with the activity concentration in accordance with ISO 11929. An example of the calculations of uncertainties and characteristic limits is detailed in Annexes A and B for two specific measurement methods.

10.4 Confidence limits

The lower, $c_A^{\triangleleft}$, and upper, $c_A^{\triangleright}$, confidence limits are calculated using Formulae (7) and (8) (see ISO 11929):

$$c_A^{\triangleleft}=c_A-k_pu(c_A) \quad \text{with} \quad p=\omega\left(1-\frac{\gamma}{2}\right) \qquad (7)$$

$$c_A^{\triangleright}=c_A+k_qu(c_A) \quad \text{with} \quad q=1-\frac{\omega\gamma}{2} \qquad (8)$$

where

$\omega = \Phi[y/u(y)]$ in which Φ is the distribution function of the standardized normal distribution;

ω can be assumed to be 1 if $c_A \geq 4u(c_A)$.

In this case:

$$c_A^{\triangleleft\triangleright}=c_A\pm k_{1-\gamma/2}u(c_A) \qquad (9)$$

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

11 Calibration

Calibrations shall be carried out under conditions specified in IEC 61577-1 and IEC 61577-2.

12 Test report

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

a) the test method used, together with a reference to this part of ISO 13164 (ISO 13164-3:2013);

b) measurement method;

c) identification of the sample;

d) measuring date and time;

e) units in which the results are expressed;

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

Complementary information can be provided such as the following:

g) sampling date and time;

h) sampling location;

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

j) the decision threshold and the detection limit — depending on the needs of the customer, there are different ways to present the result:

- when the activity concentration is compared with the decision threshold (see ISO 11929), the result of the measurement shall be expressed as $\leq c_A^*$ when the result is below the decision threshold,
- when the activity concentration is compared with the detection limit, the result of the measurement can be expressed as $\leq c_A^\#$ 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;

k) activity concentration of ^{226}Ra if its presence is detected in the water sample;

l) mention of any relevant information likely to have affected the results.

The results are expressed in a similar format to that shown in ISO 13164-1 (see Annex B).

Annex A
(informative)

Examples of measurement methods using scintillation cells

A.1 General

This annex deals only with the scintillation cells methods among the various methods able to meet the requirements of this part of ISO 13164. The two methods described differ in the degassing technique used and the volume of test sample. These methods are suitable for use either in the laboratory or on field sites.

For the purpose of this annex, the following symbols and those given in Clause 3 apply.

F_c	calibration factor per alpha for a counting carried out with a radioactive equilibrium between the radon and its short-lived decay products, in pulses per second per becquerel
f_d	correction factor for the decay of radon in the volume detection, dimensionless
f_p	correction factor for atmospheric pressure, dimensionless
N_0	number of background counts
$\bar{N}_0$	average number of background counts
N_s	number of gross counts
$\bar{N}_s$	average number of gross counts
n	number of counting of each sample
$n_\alpha(t)$	number of alpha-emitters present in the cell per becquerel of radon after a waiting time between filling and counting the cell (n_α is approximately 3 at a waiting time of 3 h for 1 Bq of radon)
t_c	counting duration (common to N_s, N_0), in seconds
V_{sc}	cell volume, in cubic metres
p_r	pressure measured in the cell after sampling, in hectopascals
p_v	pressure measured in the cell once under vacuum, in hectopascals
Δt	elapsed time between the end of the sampling, $t = 0$, and the cell counting, in seconds
λ	decay constant of radon-222, in reciprocal seconds

A.2 Method A

A.2.1 General

This test method covers the measurement of radon in water in activity concentrations above 10 Bq l^{-1} using a small test sample volume. The degassing technique used is the decrease of the air phase pressure.

A.2.2 Equipment

Usual laboratory equipment and in particular the following.

A.2.2.1 Borosilicate glass sample container fitted with a **self-healing rubber stopper**.

A.2.2.2 Hypodermic syringe to take the test sample.

A.2.2.3 Holder for filter medium for taking the air sample in the detection volume.

A.2.2.4 Scintillation cell fitted with a cap containing a hydrophilic cotton plug to enclose the detection volume.

A scintillation cell is a hermetically sealed glass flask with defined geometry and volume. The internal surface of the cell, apart from the bottom, is covered in silver-activated zinc sulfide [ZnS(Ag)].

A.2.2.5 Vacuum-creating device for the cell.

A.2.2.6 Pressure-measuring device for the cell.

A.2.2.7 Counting chain equipped with a **photomultiplier**.

A.2.2.8 Thermometer to measure the temperature of the water to be analysed.

A.2.3 Principle

A test portion of the water to be analysed (1 ml to 2 ml) is introduced into the scintillation cell (A.2.2.4) by injecting the water with a syringe (A.2.2.2) into the hydrophilic cotton plug placed in the cap. Because of the partial vacuum created in the cell prior the injection, radon is extracted from the water. Radon-free filtered air is then introduced into the scintillation cell to allow the pressure to return to normal (see Figure A.1).

The alpha-particles produced by the decay the radon and its short-lived decay products transfer their energy as they pass through the scintillation medium. As they return to their ground state, the excited electrons in the scintillation medium emit photons from the ZnS coating that can be detected by a photomultiplier (A.2.2.7). The photomultiplier converts the photons into electrical pulses that are then counted. The pulse count is directly proportional to the radon activity concentration in the air inside the cell.

A.2.4 Sampling

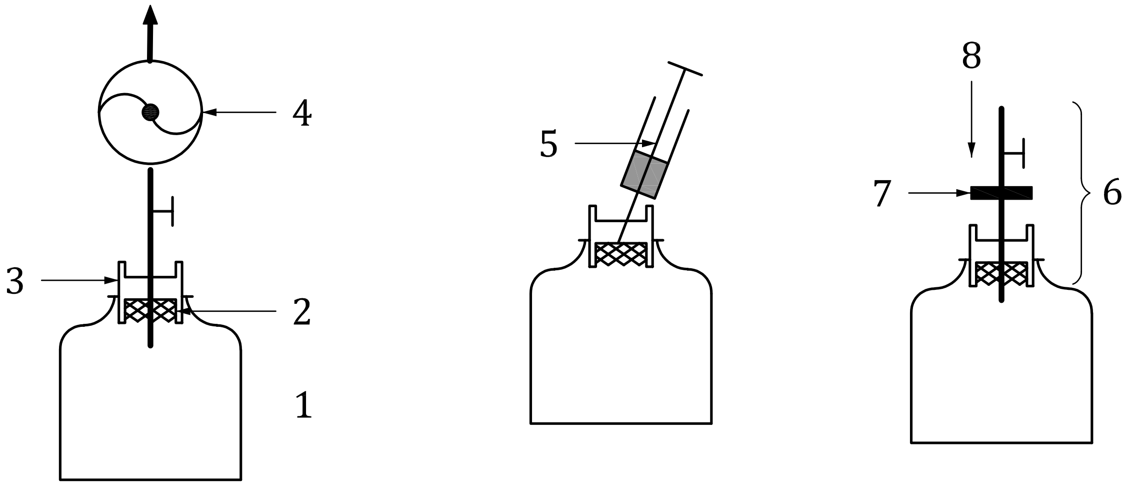
Sampling shall be performed in accordance with the requirements specified in Clause 5.

The test portion consists of 1 ml or 2 ml of water, taken through the self-healing rubber stopper using a hypodermic syringe.

A.2.5 Transfer of the radon from the water phase to the air phase

A partial vacuum at least 10 kPa (100 mbar) below ambient is created inside the scintillation cell using a vacuum pump. The test water sample is then injected into the cotton plug inside the cap of the scintillation cell. Because of the partial vacuum in the cell, radon is extracted from the water. The pressure in the cell returns to normal by allowing the introduction of clean radon-free filtered air. The radon equilibrium, governed by the Ostwald coefficient, is achieved between radon dissolved in water and radon in the air phase.

It is essential that the partial vacuum not be too high in order to avoid vaporizing the water inside the cell and damaging the zinc sulfide coating.



a) b) c)

Key

1	scintillation cell	5	syringe containing the test sample
2	hydrophilic cotton plug	6	air sampling device
3	cap	7	air filter
4	vacuum pump	8	air circulation

Figure A.1 — Principle of measuring radon in water by degassing and detection using a scintillation cell

A.2.6 Detection and counting

Before using the scintillation cell, its background count is checked by recording the count from a pre-calibrated photomultiplier placed in a lightproof enclosure for a suitable period of time.

For an optimum count, 3 h should elapse after injecting the water into the cotton. The accuracy sought dictates the counting duration and the number of counting for the sample.

A.2.7 Measurement procedure

The measurement procedure is as follows:

a) before using the cells, determination of the background of each scintillation cell by counting the photons emitted before sampling for a suitable duration with a pre-calibrated photomultiplier in a lightproof enclosure;

b) creation of a vacuum of 10 kPa (100 mbar) in the scintillation cell;

c) measurement of the residual pressure in the cells;

d) choice and location of the sampling place;

e) taking of one or more water samples;

f) taking a test portion using a syringe;

g) injection of the water into the hydrophilic cotton plug;

h) introduction of clean filtered air (free of radon) into the scintillation cell so that pressure returns to normal;

i) measurement of the pressure after filling the cells and maintenance of the pressure at atmospheric pressure;

j) recording of the location, date, and time of the sampling;

k) establishment of a radioactive equilibrium between the ^{222}Rn and its short-lived decay products (^{214}Po, ^{218}Po) in the cell by waiting, for an optimum counting, 3 h after the sampling;

l) counting, by means of a pre-calibrated photomultiplier placed in a lightproof enclosure, of the number of photons emitted by the scintillation medium when excited by alpha-particles produced by the decay of radon and its short-lived decay products present in the cells;

m) determination of the activity concentration by calculation.

It is assumed that the sample counting time and the background counting time are the same.

A.3 Method B

A.3.1 General

This test method covers the measurement of radon in water in activity concentrations above 0,1 Bq l^{-1}. The degassing technique used is air bubbling. The sensitivity of the emanometric method is enhanced by degassing radon from a large sample of water to a small volume of air.

A.3.2 Equipment

Usual laboratory equipment and in particular the following.

A.3.2.1 Borosilicate glass sample container fitted with a **self-healing rubber stopper**.

A.3.2.2 Degassing glass container.

A.3.2.3 Air filter to protect the scintillation cell against droplets of water.

A.3.2.4 Air pump.

A.3.2.5 Air stream-dividing device to create several tiny streams of air bubbles (such as a porous membrane).

A.3.2.6 Scintillation cell enclosing the detection volume, consisting of a hermetically sealed glass flask with defined geometry and volume. The internal surface of the cell, apart from the bottom, is covered in silver-activated zinc sulfide [ZnS(Ag)].

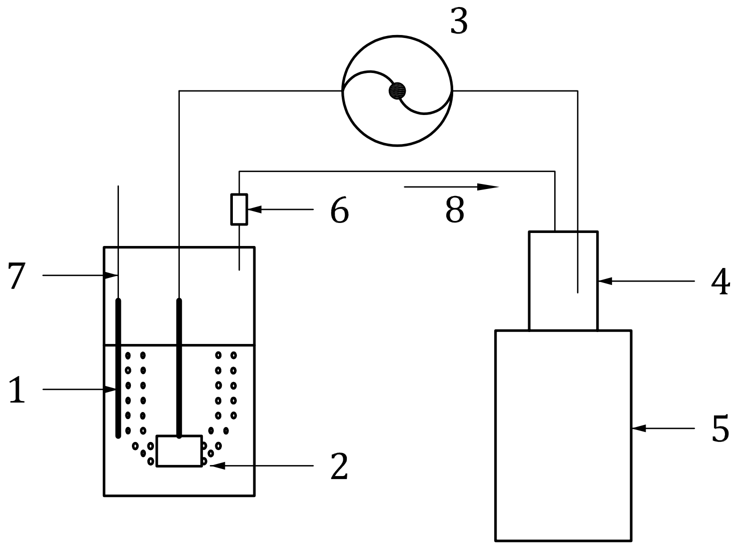
A.3.2.7 Counting chain equipped with a **photomultiplier**.

A.3.2.8 Temperature sensor for the water to be analysed.

A.3.3 Principle

The measurement is based on flushing water sample with air in closed loop with a scintillation cell (see Figure A.2).

A test portion consisting of a known volume (0,75 l) of the water to be analysed is introduced into the degassing water container. A pump is used to circulate the air in the system, bubbling it through the degassing container to extract the radon dissolved in the water. When equilibrium is achieved between radon dissolved in water and radon flushed into the air, a scintillation cell of suitable volume measures the activity concentration of the radon in the air within the system. The radon activity concentration in the test portion is determined from the activity concentration in the air within the system.



Key

1	degassing water container	5	counting chain
2	degassing system	6	air filter
3	pump	7	temperature sensor
4	scintillation cell	8	air circulation

Figure A.2 — Closed loop system for measuring radon in water by degassing and detection using a scintillation cell

A.3.4 Sampling

Sampling shall be performed in accordance with the requirements specified in Clause 5.

The test portion consists of 0,75 l of water.

A.3.5 Transfer of the radon from the water phase to the air phase

The test portion is introduced into the degassing container. The air present in the system is circulated by a pump during a flushing time of 5 min so that it bubbles through the water in the cell and degasses the radon dissolved in the water. The air flow rate is fixed at 0,4 l min^{-1}. The air volume of the entire system (scintillation cell, air pump and connections) shall be smaller than the test sample volume and is equal to 0,485 l.

A.3.6 Detection and counting

Before using the scintillation cell, its background count is checked by recording the count from a pre-calibrated photomultiplier for a suitable period of time.

When degassing of radon is achieved; measurement of the radon activity concentration in the air is performed for a suitable period of time.

It is recommended that both sample counting time and background counting time be the same. They are fixed at 10 min.

A.3.7 Measurement procedure

The measurement procedure is as follows:

a) before using the cells, determine the background of each scintillation cell by counting the photons emitted before sampling for a period of 10 min with a pre-calibrated photomultiplier in a lightproof enclosure;

b) choose and identify the location of the sampling place;

c) take one or more water samples;

d) record the location, date, and time of the sampling;

e) take of a test portion of 0,75 l;

f) inject the water into the degassing container;

g) close of the measuring system;

h) measure the water temperature;

i) start the pump in order to circulate the air in the measuring system;

j) stop the pump after running for 5 min;

k) measure the radon activity concentration in the air of the measuring system for a period of 10 min;

l) determine the activity concentration by calculation.

A.4 Expression of results

A.4.1 Activity concentration

The radon activity concentration, c_A, is obtained from Formula (1). This yields Formula (A.1):

$$c_A = \left(c - c_0\right)\omega = \left(\bar{N}_\mathrm{s} - \bar{N}_0\right)\omega_\mathrm{sc}\omega \qquad \text{(A.1)}$$

According to ISO 11665-6,[3] c and c_0 are given by Formulae (A.2) and (A.3), respectively:

$$c = \frac{\left(\bar{N}_s\right) f_p}{t_\mathrm{c} F_\mathrm{c} n_\alpha \left(t\right) V_\mathrm{sc}} = \left(\bar{N}_s\right)\omega_\mathrm{sc} \qquad \text{(A.2)}$$

$$c_0 = \frac{\left(\bar{N}_0\right) f_p}{t_\mathrm{c} F_\mathrm{c} n_\alpha \left(t\right) V_\mathrm{sc}} = \left(\bar{N}_0\right)\omega_\mathrm{sc} \qquad \text{(A.3)}$$

where

$$\bar{N}_\mathrm{s} = \sum_{j=1}^{n} N_{\mathrm{s}\,j/n} \quad \text{and} \quad \bar{N}_0 = \sum_{j=1}^{n} N_{0\,j/n}$$

$$\omega_\mathrm{sc} = \frac{f_\mathrm{p}}{t_\mathrm{c} F_\mathrm{c} n_\alpha \left(t\right) V_\mathrm{sc}} \qquad \text{(A.4)}$$

in which

$$f_\mathrm{p} = \frac{p_\mathrm{r}}{p_\mathrm{r} - p_\mathrm{v}} \qquad \text{(A.5)}$$

$$\omega=\left(L+\frac{V_a}{V_{H_2O}}\right)f_c f_d=\left(L+\frac{V_{sc}-V_{H_2O}}{V_{H_2O}}\right)f_c f_d$$

A.4.2 Standard uncertainty of activity concentration

The standard uncertainty of c_A is obtained from Formula (5). This yields Formula (A.6):

$$u(c_A)=\sqrt{\left(\bar{N}_s+\bar{N}_0\right)\frac{\omega^2\omega_{sc}^2}{n}+c_A^2\left[u_{rel}^2(\omega)+u_{rel}^2(\omega_{sc})\right]} \tag{A.6}$$

The relative standard uncertainty of ω is calculated using Formula (6).

The relative standard uncertainty of ω_{sc} is calculated using Formula (A.7).

$$u_{rel}^2(\omega_{sc})=u_{rel}^2(F_c)+u_{rel}^2(V_{sc}) \tag{A.7}$$

where the standard uncertainties of the counting time, the decay constant, the number of alpha-emitters, and the pressure are neglected.

The calculation of the characteristic limits according to ISO 11929 requires the calculation of $\tilde{u}(\tilde{c}_A)$, i.e. the standard uncertainty of c_A as a function of its true value, as given in Formula (A.8):

$$\tilde{u}(\tilde{c}_A)=\sqrt{\frac{\omega^2\omega_{sc}^2}{n}\times 2\times N_0+\tilde{c}_A\frac{\omega\omega_{sc}}{n}+(\tilde{c}_A)^2\left[u_{rel}^2(\omega)+u_{rel}^2(\omega_{sc})\right]} \tag{A.8}$$

A.4.3 Decision threshold

The decision threshold, c_A^*, is obtained from Formula (A.8) for $\tilde{c}_A=0$ (see ISO 11929).

This yields Formula (A.9):

$$c_A^*=k_{1-\alpha}\tilde{u}(0)=k_{1-\alpha}\omega\omega_{sc}\sqrt{2\times\frac{\bar{N}_0}{n}} \tag{A.9}$$

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

A.4.4 Detection limit

The detection limit, $c_A^\#$, is calculated as given in Formula (A.10) (see ISO 11929):

$$c_A^\#=c_A^*+k_{1-\beta}\tilde{u}\left(c_A^\#\right)=c_A^*+k_{1-\beta}\sqrt{\frac{\omega^2\omega_{sc}^2}{n}\times 2\times\bar{N}_0+\frac{\omega\omega_{sc}}{n}c_A^\#+c_A^{\#2}\left[u_{rel}^2(\omega)+u_{rel}^2(\omega_{sc})\right]} \tag{A.10}$$

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

The detection limit can be calculated by solving Formula (A.10) for $c_A^\#$ or, more simply, by iteration with a starting approximation $c_A^\#=2c_A^*$ in terms of the right side of Formula (A.10).

With $k_{1-\alpha}=k_{1-\beta}=k$, $c_A^\#$ is given by:

$$c_A^\#=\frac{2c_A^*+k^2(\omega\omega_{sc}/n)}{1-k^2\left[u_{rel}^2(\omega)+u_{rel}^2(\omega_{sc})\right]} \tag{A.11}$$

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

A.4.5 Example

The measurement of the radon activity concentration in a spring water at a sampling temperature of $T_{H_2O} = 20\,°C$ is performed using method A.

The test sample of volume $V_{H_2O} = 0{,}001 \pm 2 \times 10^{-5}$ is injected immediately after sampling.

The counting of the cell is carried out 3 h after sampling and for a period of 3 min. It gives for the cell with an average number of background counts $\bar{N}_0 = 3$ pulses:

$$N_{s_1} = \frac{280+268}{2} = 274 \text{ pulses}$$

$$f_p = \frac{1030}{1030-35} = 1{,}035$$

F_c = (0,6 ± 0,06) pulse s^{-1} Bq^{-1} per alpha

V_{sc} = ($125 \times 10^{-6} \pm 2{,}2 \times 10^{-6}$) m^3

NOTE Uncertainties of the calibration factor and volume of the scintillation cells have been estimated by the manufacturer from measurements carried out from a sample of 100 cells.

t_c = 180 s (the standard uncertainty of this variable is considered negligible).

n_α is approximately 3 for a waiting time of 3 h (the standard uncertainty of this variable is considered negligible).

$\lambda = 2{,}1 \times 10^{-6}\ s^{-1}$ (the standard uncertainty of this variable is considered negligible).

L = 0,253 at $T_{H_2O} = 20\,°C$.

The radon activity concentration, in becquerels per litre, in the water is:

$$c_A = \frac{(274-3)}{3 \times 60 \times 3 \times 0{,}6 \times 125 \times 10^{-6}} \left(0{,}253 + \frac{0{,}125-0{,}001}{0{,}001}\right) \frac{1}{1000} = 838$$

NOTE The radon decay between the water sampling and the counting is considered to be negligible.

The standard uncertainty of c_A, in becquerels per litre, is calculated using Formula (A.6):

$u(c_A) = 52$

Lastly, the result expression, in becquerels per litre, is:

$u(c_A) = 838 \pm 52$

The decision threshold, c_A^*, in becquerels per litre, is obtained from Formula (A.9)

$$c_A^* = k_{1-\alpha}\tilde{u}(0) = 12$$

The detection limit, $c_A^\#$, in becquerels per litre, is calculated by Formula (A.11)

$$c_A^\# = 33$$

Annex B
(informative)

Example of a measurement method using an ionization chamber

B.1 General

This annex deals only with a method using an ionization chamber from among the various methods meeting the requirements of this part of ISO 13164. It is suitable for use either in the laboratory or in the field.

For the purposes of this annex, the following symbols and those given in Clause 3 apply.

$\bar{E}$	mean energy, in joules, released by the radiation in the air of the chamber for a temperature T, in kelvin, and pressure, p, in hectopascals
e	electron charge, in coulomb ($e = 1{,}602 \times 10^{-19}$ C)
F_c	calibration factor, in becquerels per cubic metre per ampere
f_p	correction factor for atmospheric pressure, dimensionless
f_T	correction factor for temperature, dimensionless
I	ionization current due to radon, in amperes
I_0	background ionization current, in amperes
p_e	atmospheric pressure when calibrating the chamber, in hectopascals
p_m	atmospheric pressure when operating the chamber, in hectopascals
T_e	temperature of the air when calibrating the chamber, in kelvin
T_m	temperature of the air when operating the chamber, in kelvin
V_ch	volume of air present in the chamber, in cubic metres
ω_a	mean energy expended for the production of an ion pair in the air of the chamber, in joules

B.2 Equipment

Usual laboratory equipment and in particular the following.

B.2.1 Borosilicate glass container for the water sample.

B.2.2 Hypodermic syringe to inject the test portion.

B.2.3 Degassing system for radon dissolved in water, consisting of a degassing cell, a buffer cell, and free tubes, e.g. of flexible silicone.1)).

B.2.4 Pump.

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1)) Tygon 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.

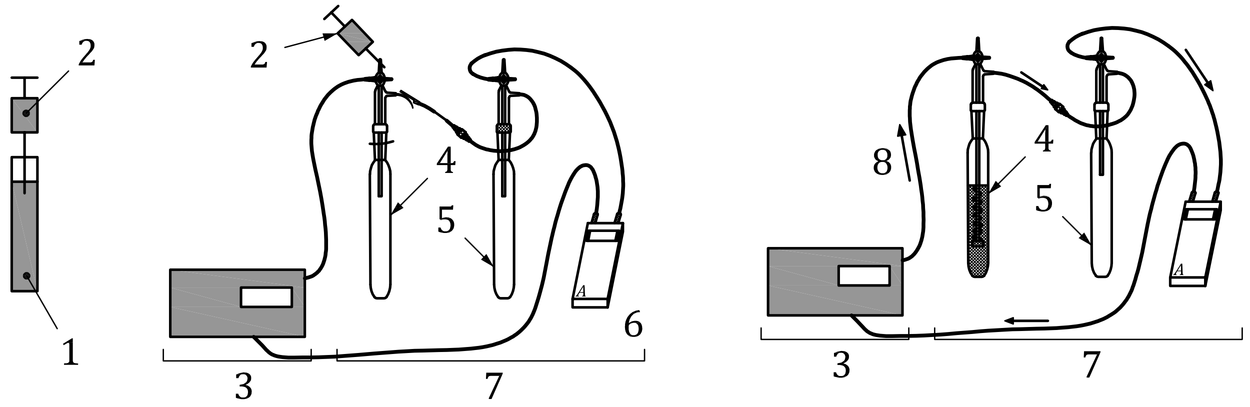
B.2.5 Ionization chamber.

B.2.6 Thermometer to measure the temperature of the water to be analysed at the time of making the measurement.

B.3 Principle

This technique uses a closed circuit consisting of a system to induce the degassing of the radon contained in the water and an ionization chamber to measure continuously the quantity of radon present in the air in the system. The radon activity concentration in the test sample is determined from the activity concentration in the air within the system.

A test portion consisting of a known volume (0,1 l) of the water to be analysed is introduced into the degassing cell. A pump is used to circulate the air in the system, bubbling it through the degassing cell to degas the radon dissolved in the water. An ionization chamber of suitable volume continuously measures the activity concentration of the radon in the air within the system and monitors any changes (see Figure B.1).



a) b) c)

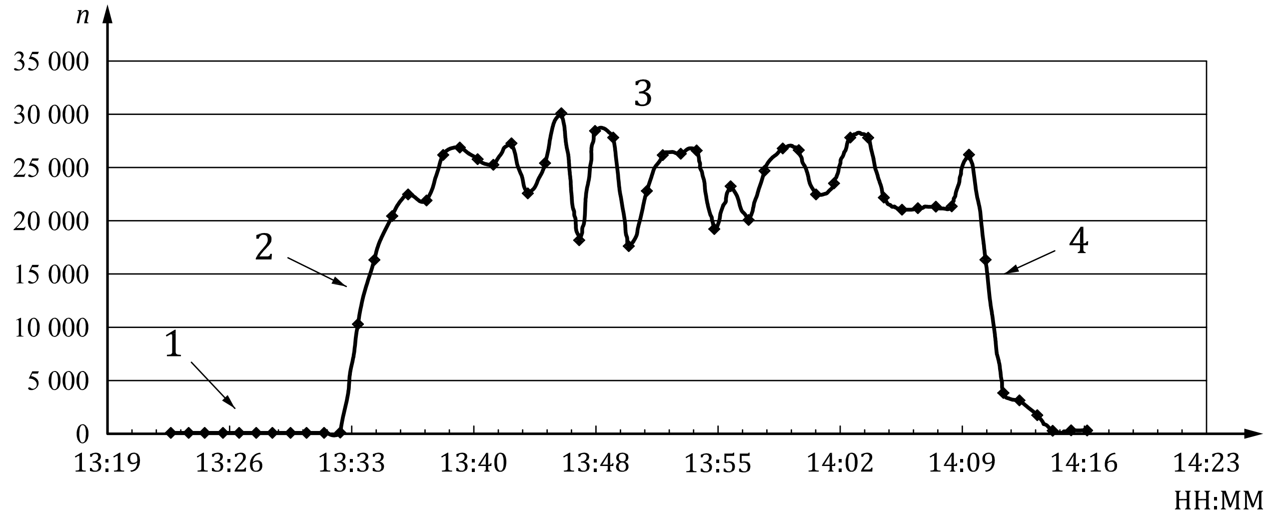
Key

1	water sample	5	buffer cell
2	syringe with the test sample	6	pump
3	ionization chamber	7	degassing system
4	degassing cell	8	air circulation

Figure B.1 — Principle of measuring radon in water by degassing and detection using an ionization chamber

The radon content increases gradually during degassing until a steady value is reached (diffusion mode). This value can then be used to calculate the activity concentration of the radon in the air, *c* (see Figure B.2). Opening the closed loop allows the air to be purged from the system.

The background noise of the ionization chamber is measured before the sample measurement is made.



Key

1	background measuring phase	*n*	number of counts
2	degassing phase	HH:MM	time of day
3	radon activity concentration measuring phase		
4	purging phase		

Figure B.2 — Change in the activity concentration of radon in the air within the system

B.4 Sampling

B.4.1 Objective

The aim of the sampling process is to obtain a representative sample of the water in the environment of concern collected over a short period of time (less than 1 h).

B.4.2 Conditions

As a precaution against later problems with the sample, two samples are taken in identical borosilicate glass containers.

The samples should be taken over a short period of time corresponding to the time taken for the containers to fill completely. The presence of any air in the container gives rise to an inaccurate result due to the degassing of the radon. The containers should be immediately sealed with a self-healing rubber stopper.

B.4.3 Test sample

The test sample consists of 0,1 l of water, taken through the self-healing rubber stopper by means of a hypodermic syringe. The volume of the sample is verified by conversion of the mass value obtained by weighing.

B.5 Transfer of the radon from the water phase to the air phase

The test sample is injected into the degassing cell. The air present in the system is circulated by a pump so that it bubbles through the water in the cell and degasses the radon dissolved in the water. The air flow rate is fixed at 1 l min^{-1}.

B.6 Detection and counting

The degassed radon enters the detection volume in the ionization chamber. The radon emits alpha-particles with an energy of 5,5 MeV. As these particles move through the air in the detection volume, they give up all or part of this energy creating pairs of ions. On collection, these ions generate an electric current (ionization current) which can be used to determine the radon activity concentration.

B.7 Measurement procedure

The measurement procedure is as follows:

a) determine the background of the ionization chamber by measuring the radon activity concentration in the measuring system before injecting the water;

b) choose and identify the location of the sampling point;

c) take one or more water samples;

d) record the location, date and time of the sampling;

e) take a test portion using a syringe;

f) measure the water temperature;

g) inject the water into the degassing cell;

h) close the measuring system;

i) start the pump in order to circulate the air in the measuring system;

j) stop the pump after running for 15 min;

k) measure the variation of the radon activity concentration in the air of the measuring system;

l) determine the activity concentration by calculation.

B.8 Expression of results

B.8.1 General

The radon activity concentration, c_A, is obtained from Formula (1). This yields Formula (B.1):

$$c_A = (c - c_0)\omega = (I - I_0)\omega_{\mathrm{ic}}\omega \quad \text{(B.1)}$$

According to ISO 11665-5[2] $c$and $c_0$are given by the following expressions:

$$c = I\,\omega_{\mathrm{ic}} \quad \text{and} \quad c_0 = I_0\omega_{\mathrm{ic}} \quad \text{(B.2)}$$

where

$$\omega_{\mathrm{ic}} = F_{\mathrm{c}} f_p f_T \quad \text{(B.3)}$$

in which

$$F_{\mathrm{c}} = \frac{1}{V_{\mathrm{ch}}}\frac{\omega_{\mathrm{a}}}{E_{\mathrm{m}}e} \quad \text{(B.4)}$$

$$f_p = \frac{p_\mathrm{e}}{p_\mathrm{m}} \tag{B.5}$$

$$f_T = \frac{T_\mathrm{m}}{T_\mathrm{e}} \tag{B.6}$$

In practice, it is possible that the measured temperature, T_m, and pressure, p_m, do not differ significantly from the calibration values, T_e, and p_e. Therefore Formula (A.1) can be simplified.

Formula (2) gives ω.

B.8.2 Standard uncertainty

The standard uncertainty of c_A is obtained from Formula (5). This yields Formula (B.7):

$$u(c_A) = \sqrt{(I + I_0)k_I \omega^2 \omega_\mathrm{ic}^2 + c_A^2 \left[u_\mathrm{rel}^2(\omega) + u_\mathrm{rel}^2(\omega_\mathrm{ic})\right]} \tag{B.7}$$

The relative standard uncertainty of ω is calculated using Formula (6).

The relative standard uncertainty of $\omega_\mathrm{ic}$is calculated using Formula (B.8).

$$u_\mathrm{rel}^2(\omega_\mathrm{ic}) = u_\mathrm{rel}^2(F_\mathrm{c}) + u_\mathrm{rel}^2(f_p) + u_\mathrm{rel}^2(f_T) \tag{B.8}$$

The standard uncertainties of the calibration temperature, T_e, and the calibration pressure, p_e, are neglected.

$u^2(I) = k_I I$ and $u^2(I_0) = k_I I_0$

where k_I is a constant provided by the manufacturer.

The calculation of the characteristic limits according to ISO 11929 requires the calculation of $\tilde{u}(\tilde{c}_A)$, i.e. the standard uncertainty of c_A as a function of its true value, as given in Formula (B.9):

$$\tilde{u}(\tilde{c}_A) = \sqrt{2k_I I_0 \omega_\mathrm{ic}^2 + \tilde{c}_A k_I \omega \omega_\mathrm{ic} + (\tilde{c}_A)^2 \left[u_\mathrm{rel}^2(\omega) + u_\mathrm{rel}^2(\omega_\mathrm{ic})\right]} \tag{B.9}$$

B.8.3 Decision threshold

The decision threshold, c_A^*, is obtained from the Formula (B.9) for $\tilde{c}_A = 0$ (see ISO 11929).

This yields Formula (B.10):

$$c_A^* = k_{1-\alpha} \tilde{u}(0) = k_{1-\alpha} \omega \omega_\mathrm{ic} \sqrt{2k_I I_0} \tag{B.10}$$

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

B.8.4 Detection limit

The detection limit, $c_A^\#$, is calculated as given in Formula (B.11) (see ISO 11929):

$$\begin{gathered} c_A^\# = c_A^* + k_{1-\beta} \tilde{u}\left(c_A^\#\right) \\ c_A^\# = c_A^* + k_{1-\beta} \sqrt{\omega^2 \omega_\mathrm{ic}^2 \times 2 \times k_I I_0 + \omega k_I \omega_\mathrm{ic} c_A^\# + \left(c_A^\#\right)^2 \left[u_\mathrm{rel}^2(\omega) + u_\mathrm{rel}^2(\omega_\mathrm{ic})\right]} \end{gathered} \tag{B.11}$$

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

The detection limit can be calculated by solving Formula (B.11) for $c_A^{\#}$ or, more simply, by iteration with a starting approximation $c_A^{\#} = 2c_A^*$ in terms of the right side of Formula (B.11).

With $k_{1-\alpha} = k_{1-\beta} = k$, $c_A^{\#}$ is given by:

$$c_A^{\#} = \frac{2c_A^* + k^2 \omega \omega_{\text{ic}} k_I}{1 - k^2 \left[u_{\text{rel}}^2(\omega) + u_{\text{rel}}^2(\omega_{\text{ic}}) \right]} \tag{B.12}$$

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

B.8.5 Example

The measurement of the radon activity concentration in a spring water at a sampling temperature of $T_{H_2O} = 10\,°\text{C}$ is performed.

The test portion of volume $V_{H_2O} = (0{,}1 \pm 0{,}0025)\ \text{l}$ is injected immediately into the degassing cell after sampling.

The air is circulated through the system by means of a pump, and the ionization chamber continuously measures the activity concentration of radon in the air in the system. The activity concentration of radon gradually increases as the dissolved radon degasses from the water until it reaches a steady value. The mean activity concentration calculated at this level is $c = 25\,000 \pm 250\ \text{Bq m}^{-3}$.

The activity concentration of radon in the air in the system before the water is injected is $c_0 = (20 \pm 10)\ \text{Bq m}^{-3}$.

The total volume of air in the measuring system before injecting the test sample is equal to:

$(1\,127 \times 10^{-6} \pm 0{,}5 \times 10^{-6})\ \text{m}^3$

$L = 0{,}35$ at $T_{H_2O} = 10\,°\text{C}$

$$f_p = \frac{1030}{1030 - 35} = 1{,}035$$

The radon activity concentration, in becquerel per litre, in the water is:

$$c_A = (25000 - 20) \times \left(0{,}35 + \frac{1127 - 100}{100} \right) \times 10^{-3} = 256$$

The standard uncertainty of c_A, in becquerel per litre, is calculated using Formula (B.7):

$u^2(c_A) = 8$

Lastly, the result, in becquerel per litre, is:

$c_A = 165 \pm 8$

The decision threshold, c_A^*, in becquerel per litre, is obtained from Formula (B.10):

$$c_A^* = k_{1-\alpha} \cdot \tilde{u}(0) = 0{,}25$$

The detection limit, $c_A^{\#}$, in becquerel per litre, is calculated by Formula (B.12):

$$c_A^{\#} = 4{,}65$$

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