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Measurement of radioactivity in the environment — Air: Radon-222 —

Part 9:
Test methods for exhalation rate of building materials

Mesurage de la radioactivité dans l'environnement — Air: Radon 222 —

Partie 9: Méthode de détermination du flux d'exhalation des matériaux de construction

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Foreword

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

ISO 11665 consists of the following parts, under the general title *Measurement of radioactivity in the environment — Air: Radon 222*

- *Part 1: Origins of radon and its short-lived decay products and associated measurement methods*
- *Part 2: Integrated measurement method for determining average potential alpha energy concentration of its short-lived-decay products*
- *Part 3: Spot measurement method of the potential alpha energy concentration of its short-lived decay products*
- *Part 4: Integrated measurement method for determining average activity concentration using passive sampling and delayed analysis*
- *Part 5: Continuous measurement method of the activity concentration*
- *Part 6: Spot measurement method of the activity concentration*
- *Part 7: Accumulation method for estimating surface exhalation rate*
- *Part 8: Methodologies for initial and additional investigations in buildings*
- *Part 9: Test methods for exhalation rate of building materials*
- *Part 10: Determination of diffusion coefficient in waterproof materials using activity concentration measurement*
- *Part 11: Test method for soil gas with sampling at depth*

Introduction

Radon isotopes 222, 220 and 219 are radioactive gases produced by the disintegration of radium isotopes 226, 224 and 223, which are decay products of uranium-238, thorium-232 and uranium-235 respectively, and are all found in the earth's crust. Solid elements, also radioactive, followed by stable lead are produced by radon disintegration.[1]

When disintegrating, radon emits alpha particles and generates solid decay products, which are also radioactive (polonium, bismuth, lead, etc.). The potential effects on human health of radon lie in its decay products rather than the gas itself. Whether or not they are attached to atmospheric aerosols, radon decay products can be inhaled and deposited in the bronchopulmonary tree to varying depths according to their size.

Radon is today considered to be the main source of human exposure to natural radiation. The UNSCEAR (2006) report[8] suggests that, at the international level, radon accounts for around 52 % of global average exposure to natural radiation. The radiological impact of isotope 222 (48 %) is far more significant than isotope 220 (4 %), while isotope 219 is considered negligible. For this reason, references to radon in this part of ISO 11665 refer only to radon-222.

Radon activity concentration can vary from one to multiple orders of magnitude over time and space. Exposure to radon and its decay products varies tremendously from one area to another, as it depends firstly on the amount of radon emitted by the soil and the building materials in each area and, secondly, on the degree of containment and weather conditions in the areas where individuals are exposed.

As radon tends to concentrate in enclosed spaces like houses, the main part of the population exposure is due to indoor radon. Soil gas is recognized as the most important source of residential radon through infiltration pathways. A secondary source is the radon exhalation from building materials.

The radon atoms in materials are produced by the disintegration of the radium-226 contained in the mineral grains of the material. Some of these atoms reach the interstitial spaces between the grains: this is the phenomenon of emanation. Some of these atoms produced by emanation reach the material's surface by diffusion and convection: this is the phenomenon of exhalation.

Values of the radon-222 surface exhalation rate observed for building materials vary from not detectable up to 5 mBq.m^{-2}.s^{-1}.[4],[5]

NOTE The origin of radon-222 and its short-lived decay products in the atmospheric environment and other measurement methods are described generally in ISO 11665-1.

Measurement of radioactivity in the environment — Air: Radon-222 —

Part 9:
Test methods for exhalation rate of building materials

1 Scope

This standard specifies a method for the determination of the free radon exhalation rate of a batch of mineral based building materials. The standard only refers to ^{222}Rn exhalation determination using two test methods: Liquid Scintillation Counting (LSC) and gamma ray spectrometry (Annex A and Annex B)

The exhalation of thoron (^{220}Rn) does not affect the test result when applying the determination methods described in this part of the standard.

2 Normative references

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

ISO 11665-1, *Measurement of radioactivity in the environment — Air: radon-222 — Part 1: Origins of radon and its short-lived decay products and associated measurement methods*

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

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11665-1 and the following apply.

3.1.1
batch
quantity of material that is regarded as a unit and for which it is assumed that it has uniform characteristics or an amount of fresh concrete produced under uniform conditions and which has the same strength and environmental class or which has the same composition

3.1.2
building material
product that is made of one or more materials and possibly admixtures and which has characteristics that meet previously set requirements after a formation process which may have been supplemented with a curing process if required

Note 1 to entry: The curing process, in which a chemical reaction occurs, may take place under ambient conditions (cold binding products), under elevated temperature (baked products) or under elevated temperature and pressures (autoclaved products).

3.1.3

building material laboratory sample

sample or sub-sample(s) of the *building material* (3.1.2) received by the laboratory

3.1.4

building material test sample

building material (3.1.2) sample that is either the laboratory sample or has been prepared from the laboratory sample used to determine the radon exhalation

3.1.5

adsorbent test sample

sample of adsorbent material, such as silica gel or charcoal, used to trap the radon exhaled from the *building material test sample* (3.1.4)

Note 1 to entry: This sample is used for testing.

3.1.6

free volume

volume of the exhalation vessel reduced by the volume of the *building material test sample* (3.1.4)

3.1.7

radon standard

solution of ^{226}Ra with a defined activity which can be traced to the primary standard or a source of radon emanation with a defined radon emanation rate respectively

3.1.8

ventilation rate

rate at which the *free volume* (3.1.6) is refreshed

Note 1 to entry: The ventilation rate can be calculated by dividing the volume flow rate (m^3/s) by the *free volume* (3.1.6) (m^3).

3.2 Symbols

For the purposes of this document, the symbols given in ISO 11665-1 and the following apply. See also the symbols specific to Annex B, given in the annex.

Symbol	Name of quantity
$A_{\mathrm{Ra,s}}$	^{226}Ra activity of the radon standard, in becquerel
A_{Ra}	^{226}Ra activity, in becquerel
F_{c}	Calibration factor
$\overline{F_{\mathrm{c}}}$	Average calibration factor
i	Subscript of the determination for the i^{th} counting measurement
m_{g}, m_0	Number of repeated counting measurements of the same kind: test sample and background, respectively
$n_{\mathrm{g,i}}, n_{0,i}$	Number of counts in the i^{th} measurement of the m counting measurements of the gross area of the peak of the adsorbent test sample and of the background spectrum, respectively
$\overline{n}_{\mathrm{g}}, \overline{n}_0$	Mean value of the number of counts of the m counting measurements of the adsorbent test sample and of the blank sample, respectively

Symbol	**Name of quantity**
$n_{\mathrm{g,Pb,i}}$, $n_{\mathrm{0,Pb,i}}$	Number of counts in the peak of the adsorbent test sample spectrum and of the blank spectrum for the i^{th} measurement of the m counting measurements, respectively at the energy line of ^{210}Pb
$\overline{n}_{\mathrm{g,Pb}}$, $\overline{n}_{\mathrm{0,Pb}}$	Mean value of the number of counts of the m counting measurements of the adsorbent test sample and of the blank sample, respectively in the gross area of the peak at the energy line of ^{210}Pb
$n_{\mathrm{g,Bi,i}}$, $n_{\mathrm{0,Bi,i}}$	Number of counts in the peak of the adsorbent test sample spectrum and of the blank spectrum for the i^{th} measurement of the m counting measurements, respectively at the energy line of ^{214}Bi
$\overline{n}_{\mathrm{g,Bi}}$, $\overline{n}_{\mathrm{0,Bi}}$	Mean value of the number of counts of the m counting measurements of the adsorbent test sample and of the blank sample, respectively in the gross area of the peak at the energy line of ^{214}Bi
R_{g}, R_0	Gross counting rate as the result of radon and/or radon decay products on the adsorbent test sample and of the blank, respectively, in per second
$\overline{R}_{\mathrm{g}}$, $\overline{R}_0$	Mean value of the m measurements of the gross counting rate as the result of radon and/or radon decay products on the adsorbent test sample and of the blank, respectively, in per second Counting rate in per second
$R_{\mathrm{g,Pb}}$, $R_{\mathrm{0,Pb}}$	Gross counting rate of the adsorbent test sample and of the blank, respectively, for ^{210}Pb, in per second
$R_{\mathrm{g,Bi}}$, $R_{\mathrm{0,Bi}}$	Gross counting rate of the adsorbent test sample and of the blank, respectively, for ^{214}Bi, in per second
t_{a}	Duration between the start and the end of the adsorption step, in seconds
t_{g}, t_0	Counting duration for the measurement of the background and the blank, respectively, in seconds
t_{c}	Counting duration of the adsorbent test sample, in seconds
t_{w}	Duration between the end of the adsorption period and the start of the count, in seconds
U_r	Expanded relative uncertainty, calculated by $U = k \cdot u\ (a)$ with $k = 2$, ...
V	Free volume to which the radon exhales, in cubic metres
V_{p}	Volume of the building material test sample, in cubic metres
λ_{Rn}	Radon decay constant, in per second
λ_{v}	Ventilation rate, in per second
k	Coverage factor
ϕ_{f}	Free radon exhalation rate, in per second
$\overline{\phi}_{\mathrm{f}}$	Mean value of the free radon exhalation rate, in becquerel per second
$\mu(\overline{\phi}_{\mathrm{f}})$	Standard uncertainty of the free radon exhalation rate, in becquerel per second

Symbol	**Name of quantity**
$\bar{\phi}_{\mathrm{f,Pb}}$	Free radon exhalation rate for ^{210}Pb, in becquerel per second
$\bar{\phi}_{\mathrm{f,Bi}}$	Free radon exhalation rate for ^{214}Bi, in becquerel per secon
$\bar{\phi}_{\mathrm{f}}^{*}$	Decision threshold, in becquerel per cubic meters, associated to the free radon exhalation rate
$\bar{\phi}_{\mathrm{f}}^{\#}$	Detection limit, in becquerel per cubic meters, associated to the free radon exhalation rate
$\bar{\phi}_{\mathrm{f,Pb}}^{*}$, $\bar{\phi}_{\mathrm{f,Bi}}^{*}$	Decision threshold, in becquerel per cubic meters, associated to the free radon exhalation rate for ^{210}PB and ^{214}Bi respectively
$\bar{\phi}_{\mathrm{f,Pb}}^{\#}$, $\bar{\phi}_{\mathrm{f,Bi}}^{\#}$	Detection limit, in becquerel per cubic meters, associated to the free radon exhalation rate for ^{210}PB and ^{214}Bi respectively

All symbols belonging to the countings performed on the test samples, blanks and reference samples are indicated by subscripts g, 0 and r, respectively.

In each case, arithmetic averaging over *m* countings of the same kind carried out with the same preselected measurement duration, *t* (time preselection), is denoted by an overline.

Thus, for example, for *m* counting results, n_i $(i=1,...,m\,;\ m>1)$, which are obtained in such a way and shall be averaged, the mean value, $\bar{n}$, and its uncertainty, $u^2(\bar{n})$, of the values, n_i, are given by

$$\bar{n} = \frac{1}{m}\sum_{i=1}^{m} n_i \ ; \quad u^2(\bar{n}) = \frac{1}{m}\left(\bar{n} + \frac{m-1}{m-3}\bar{n} + \frac{1}{m-3}\sum_{i=1}^{m}(n_i-\bar{n})^2\right)$$

4 Principle

The building material test sample (3.1.4) is conditioned at a temperature of (20 ± 2) °C and (50 ± 5) % relative humidity. After conditioning, the building material test sample (3.1.4) is placed in an exhalation vessel where the radon exhalation takes place.

The free radon exhalation rate is determined by flushing the exhaled radon from the free volume (3.1.6) using nitrogen and trapping it on an adsorbent material (purge and trap method) such as silica gel and charcoal. The radon content of these adsorbent materials is quantified using Liquid Scintillation Counting (LSC) for silica gel as described in the main text or using gamma ray spectrometry for charcoal as described in Annex A and Annex B.

5 Reagents and equipment

5.1 Reagents

a) **Ice water.**

b) **Potassium hydroxide**, KOH, solid (pellets);

c) **Radon standard** (3.1.7).

d) **Scintillation cocktail.**

NOTE 1 A cocktail based on toluene has, in practice, been found to be the most suitable.

e) **Silica gel** with a particle size of 1 mm to 3 mm and dried at 105 °C until a constant mass is obtained.

A constant mass is obtained when the mass of the last weighing does not deviate by more than 0,5 % of the mass of the previous weighing when weighing with an intervening period of at least 24 h.

f) **Nitrogen, N_2, gas**.

NOTE 2 By the blank determination, any radon content of the gas and the influence of this on the result is taken into account;

g) **Nitrogen, N_2, liquid.**

5.2 Equipment for sample preparation

h) **Conditioning room** in which the temperature can be set to a value of (20 ± 2) °C and the relative humidity can be set to a value of (50 ± 5) %.

i) **Calibrated length measuring instrument**, with a reading uncertainty of maximum 1 mm.

j) **Calibrated weighing apparatus**, with a measuring range of minimum 1,5 times the mass of the building material test sample (3.1.4), and reading uncertainty of a maximum 0,01 %.

k) **Relative-humidity meter**, with a measuring range of 40 % to 60 %, measurement uncertainty of maximum 3 %, and reading uncertainty of maximum 1 %.

l) **Thermometer**, with a measuring range of 15 °C to 25 °C, measurement uncertainty of maximum of 1 °C, and reading uncertainty of a maximum of 0,5 °C.

m) **Saw**.

5.3 Equipment for procedure

a) **Adsorption column**. Glass U tube of sufficient length and diameter, able to contain approximately 5 g silica gel.

b) **Dewar flask**.

c) **Drying column**, comprising a glass U tube of sufficient length and diameter, able to contain 20 g of KOH pellets.

d) **Exhalation vessel**, in which one or more adsorbent test samples (3.1.5) can be placed without touching each other or the walls of the vessel and which can be sealed airtight. The dimensions of the vessel shall be so that adequate flushing of the free volume (3.1.6) is possible. Ensure that the volume of the exhalation vessel has at least twice the volume of the adsorbent test sample (3.1.5).

The material used to manufacture the exhalation vessel shall not release radon. The vessel shall have an inlet and an outlet to allow flushing of the free volume (3.1.6) with nitrogen, and shall be provided with a thermometer and a relative-humidity meter. The ingoing volume flow shall be distributed over various inlet points to ensure that the whole inner space of the vessel is flushed uniformly. Ensure that there are no dead corners in this inner space. Volume flow rate meters shall be mounted in the lines used to supply and exhaust the nitrogen.

e) **Gas washing bottle(s)**. At least one, of volume 150 ml to 200 ml.

f) **Calibrated length measuring instrument** with a reading uncertainty of maximum 1 mm.

g) **Glass vials**, to be used as sample holders for the liquid scintillation counter; volume 20 ml.

h) **Relative-humidity meter** with a measuring range of 40 % to 60 %, measurement uncertainty of maximum 3 % in absolute terms, and reading uncertainty of at most 1 %.

i) **Round bottom flasks or gas washing bottles** of sufficient volume to ensure the ^{226}Ra solution can be flushed.

j) **Liquid scintillation counter**, preferably with a sample changer and the option of setting windows and displaying pulse height spectra.

k) **Thermometer** with a measuring range of 15 °C to 25 °C, measurement uncertainty of maximum 1 °C, and reading uncertainty of maximum 0,5 °C.

l) **Chronometer**, with reading uncertainty of maximum 1 s.

m) **Connection tubes**, together with valves if required.

n) **Plastic tubes**.

o) **Volume flow rate meter** of accuracy such that the actual volume flow rate does not deviate by more than 1 % from the value set during the test, with a measurement uncertainty of maximum 2 % of the measured value, and a reading uncertainty of 1 % of the measured value.

5.4 Test bench

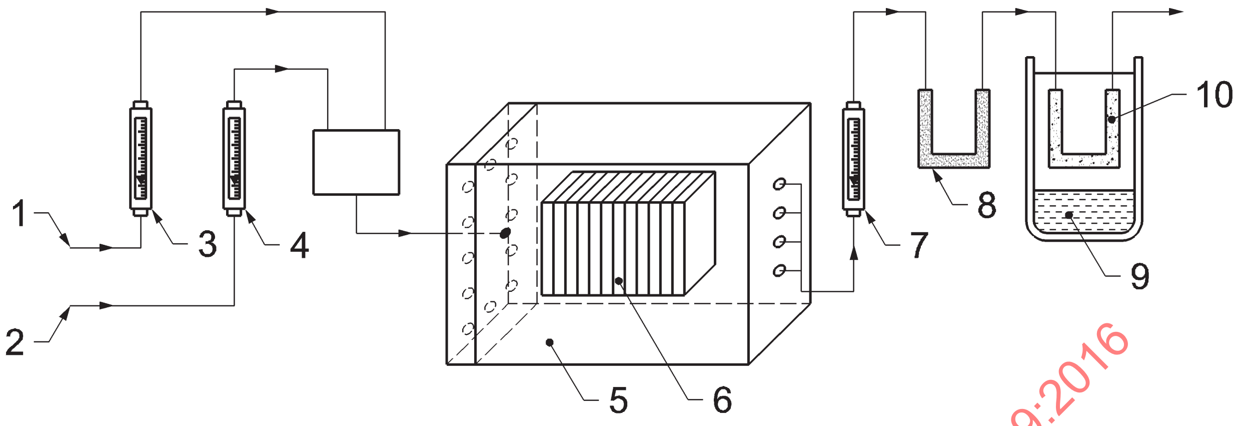
Set up the test bench as specified in Figure 1 with the components described in 5.3.

The components are connected with plastic tubes [5.3 n)]. The tubes that may be in contact with radon, that is, downstream from the exhalation vessel [5.3 d)], shall be as short as possible. Split the nitrogen supply into two parts.

Pass one section over one or more gas washing bottles [5.3 e)] filled with water to ensure that this volume flow can reach a relative humidity of at least 50 %. The ratio between the dry and the humidified volume flows can be changed through the valves mounted on the supply lines. Fit the relative-humidity meter [5.3 h)] in the exhalation vessel [5.3 d)], seal the vessel hermetically and start the nitrogen volume flow. Check after 1 h to ensure that:

— the volume flow rates in the supply and exhaust line(s) are the same within the reading accuracies (A + B = C) ;

— the relative-humidity meter is (50 ± 5) %. If this is not the case, the ratio of both volume flows shall be modified using the valves.



Key

1	volume flow of nitrogen with relative humidity of 0 %	6	sample
2	volume flow of nitrogen with relative humidity of 100 %	7	volume flow rate meter C [5.3 o)]
3	volume flow rate meter A [5.3 o)]	8	KOH pellets [5.1 b)]
4	volume flow rate meter B [5.3 o)]	9	liquid nitrogen [5.1 g)]
5	exhalation vessel [5.3 d)]	10	silica gel [5.1 e)]

Figure 1 — Schematic representation of test bench

6 Building material test sample preparation

6.1 General

The samples prepared for testing during the different steps of the measuring process are shown in Figure 2.

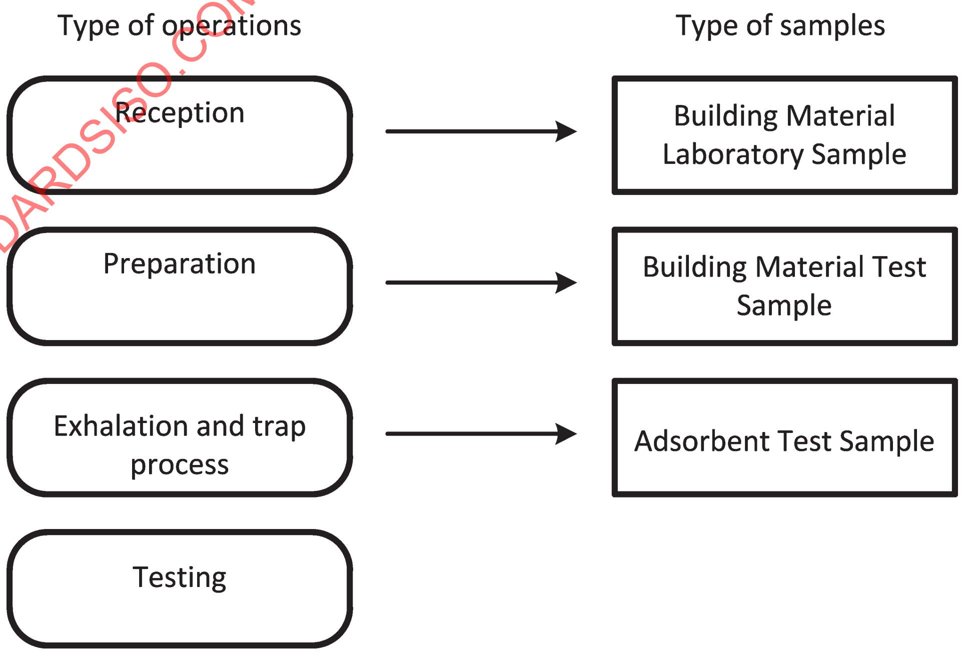


Figure 2 — Steps of measuring process with correspondent samples prepared

6.2 Number and dimensions

6.2.1 General

Two building material test sample preparation procedures are used depending on the types of building material laboratory samples received:

a) building materials (3.1.2) with a defined product-geometry, these are called end products;

b) fluid intermediate materials that require curing.

For solid intermediate materials that have no specific product geometry, for example granular materials, a building material test sample (3.1.4) can be prepared using a tray that is open from the top side. The material used to manufacture the tray shall not release radon. No advice is provided on the dimensions of the tray; however, do ensure that requirements on the volume of the building material test samples (3.1.4) as stated in the standard are satisfied.

For these building material laboratory samples (3.1.3) ensure that the total volume of the building material test samples (3.1.4) that are tested simultaneously are at most half the volume of the exhalation vessel.

Determine, for each building material test sample (3.1.4), the mass with an accuracy of three decimal places in kg, the dimensions with an accuracy of three decimal places in metres (m), and, if required, the external surface with an accuracy of three decimal places in square metres (m^2) if that surface cannot easily be derived from the dimensions.

NOTE The total volume of the building material test samples (3.1.4) is not prescribed in this standard as the volume becomes greater, the material shows a higher exhalation rate and with a constant adsorption time, the uncertainty of the exhalation rate decreases. In general, building material test samples (3.1.4) with a volume in the range of 3 dm^3 to 10 dm^3 are sufficient.

6.2.2 End product

For end products with defined product geometry take at least three building material test samples (3.1.4) from the building material laboratory sample (3.1.3). The building material test sample can be sawed from a laboratory sample that is too large for the exhalation vessel (blocks, panels, units).

6.2.3 Fluid intermediate materials

For fluid intermediate materials that require curing, for example concrete under paste form; take at least three representative samples of the paste during pouring of the building product.

Prepare a building material test sample (3.1.4) from each of the paste samples using a cube-shaped template, which measures 150 mm × 150 mm × 150 mm with a tolerance of no more than 0,5 %, and allow the concrete to cure for 28 days.

6.3 Conditioning

6.3.1 End products

Store the building material test samples (3.1.4), after preparation, in the conditioning room. Set the temperature and the relative humidity of the conditioning room at (20 ± 2) °C and (50 ± 5) %. Wait until the moisture content is in equilibrium with the set conditions. This equilibrium is achieved when the mass of the building material test samples (3.1.4) over a period of seven days deviates by less than 0,07 % from the value determined during the previous measurement. Once this is the case, testing can start. If the moisture content is not in equilibrium, the test shall start 12 weeks after conditioning has begun.

6.3.2 Fluid intermediate materials

Store the building material test samples (3.1.4) prepared for example from concrete in the conditioning room following the curing period of 28 days. Set the temperature and the relative humidity of the conditioning room at (20 ± 2) °C and (50 ± 5) %. Wait until the moisture content is in equilibrium with the set conditions before starting the exhalation step. The equilibrium is achieved when the mass of the building material test sample (3.1.4) over a period of seven days deviates by less than 0,07 % from the value determined during the previous measurement.

Conditioning, shall not last longer than 8 weeks. If the moisture content is not in equilibrium after 8 weeks, the exhalation step shall start between 8 and 12 weeks after the curing period of 28 days.

NOTE The age of the building material test sample (3.1.4) may influence the radon exhalation. For example, it has been demonstrated that the free radon exhalation rate of certain types of concrete may decrease by approximately 50 % over a period of 6 years when stored at a relative humidity of approximately 50 %.

7 Measurement

7.1 General

For clarification, Figure 3 presents the successive different steps of the measuring process with their respective duration symbol.

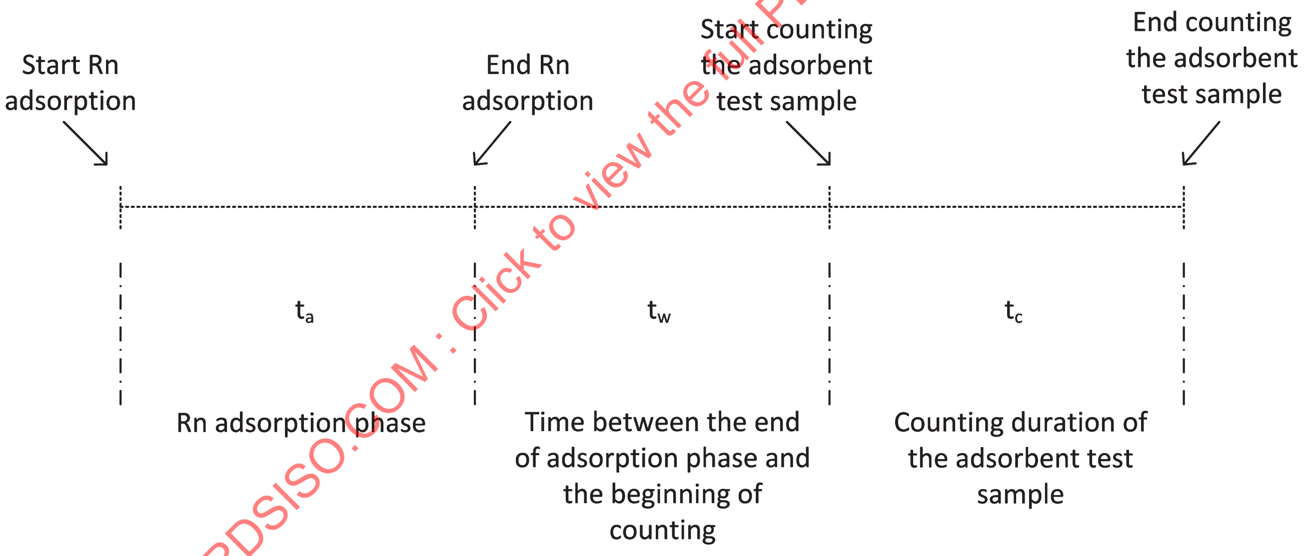


Figure 3 — Successive different steps of measuring process with their respective duration symbols

7.2 Set up of test bench

7.2.1 Choice of volume flow rate

Choose the volume flow rate of the nitrogen gas [5.1 f)] so that the following relationship is satisfied under all conditions:

$$\frac{V_\mathrm{p}}{V} \cdot \frac{\lambda_\mathrm{Rn}}{\lambda_\mathrm{Rn} + \lambda_\mathrm{v}} < 0{,}1 \qquad (1)$$

If a higher volume flow rate is chosen at a later stage, the activities described in 7.2.3 and 7.2.4 shall again be performed. Therefore, the choice should be carefully considered.

NOTE The drying functioning of the KOH column also depends on the adsorption time and the flow rate. 20 g of KOH [5.1 b)] has been found sufficient for a flow rate of 600 ml/min and an adsorption time of 30 min at a relative humidity of 50 %.

7.2.2 Determination of amount of adsorbent material

The minimum required amount of the silica gel [5.1 e)] used as adsorbent material is determined to ensure that all radon is adsorbed on the silica gel in the adsorption column [5.3 a)]. This mass of silica gel for the adsorbent material test sample (3.1.5) is determined in accordance with 7.3 using two adsorption columns [5.2 a)] placed in series. After the adsorption period, the radon activity concentration in the silica gel of the second column shall not be more than 1 % of the radon activity concentration in the silica gel in the first column. When higher percentage is measure, the amount of silica gel [5.1 e)] is increased and the determination is repeated.

Once the silica gel test sample [5.1 e)] has been transferred to a glass vial [5.2 g)] containing scintillation cocktail [5.1. d)], the adsorbed radon is slowly released into the scintillation cocktail [5.1 d)]. The rate at which this takes place, depends *inter alia* on the particle size of the silica gel used [5.1 e)].

It is not necessary to shake the glass vial [5.2 g)].

The radon adsorption capacity of the silica gel [5.1 e)] may differ per batch (3.1.1). If a new batch (3.1.1) is used, the previously determined amount shall be reassessed.

The required amount of silica gel [5.1 e)] depends on the volume flow rate and the adsorption period. The correct amount shall be determined empirically. If the volume flow rate of the adsorption period is increased, the required amount of silica gel [5.1 e)] shall be determined again. It was found that a mass of 4 g to 5 g was sufficient when the adsorption period is 30 min and the volume flow rate is 600 ml/min.

7.2.3 Determination of minimum desorption duration

The minimum desorption duration is the duration at the end of which the counting rate has reached a value of at least 99 % of the maximum counting rate. To determine this duration, place the (first) adsorbent material test sample [5.2 g)] into the LSC immediately after the addition of the scintillation cocktail [5.1 d)] and count it intermittently at short intervals over a period of approximately 24 h.

NOTE Figure 4 includes an example of a desorption curve. The minimum desorption time is approximately 16 h in this example when considering the 99 % condition.

7.2.4 LSC procedure

7.2.4.1 General

To determine the radon activity concentration of a test portion, a measuring range (energy window) that matches the β energies of 110 keV to 600 keV is suitable.

It is however, recommended to optimize this setting for individual counting apparatus which may result in a higher counting efficiency. For this optimization, use an adsorbent material test sample (3.1.5) with a high radon activity concentration obtained, for example, in accordance with 7.2.4.2. The counting efficiency varies, from 1,5 (Bq.s)$^{-1}$ radon-222 to 2,8 (Bq.s)$^{-1}$ radon-222.

In this standard, no information is provided regarding the optimization method for the setting of the LSC, this information can be found in the manual provided by the manufacturer.

ISO 19361 describes the conditions for measuring the activity of beta emitter radionuclides by liquid scintillation counting and can be used to determine the measuring parameters according to the intended use of the measurement results and the associated data quality objectives.

7.2.4.2 Determination of calibration factor

Prepare a solution with an activity of approximately 50 Bq from the ^{226}Ra standard solution. Use a round bottom flask [5.2 i)].

Flush the liquid with at least 10 times its volume at the chosen volume flow rate (7.2.1).

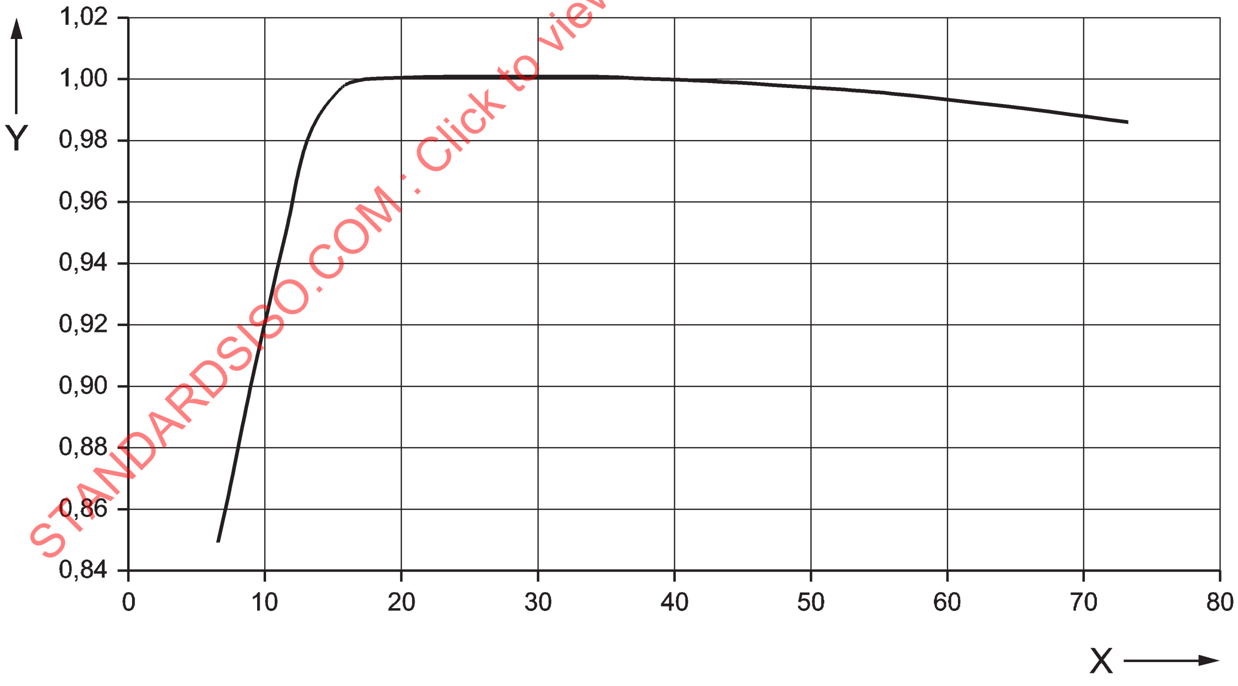
Connect the round bottom flask [5.2 i)] to the drying [5.2 c)] and adsorption columns [5.3 a)].

Start the nitrogen volume flow and adjust it to the tested volume flow rate (see 7.2.2).

Perform the steps 8) to 14) described in 7.3. Repeat for another 4 different adsorption durations.

Prepare a blank adsorbent test sample in accordance with 7.3 16).

Analyse the adsorbent material test sample (3.1.5) in accordance with 7.3.



Key

Y relative activity

X time (h)

Figure 4 — Example of a desorption curve: Evolution of the activity (%) with time (h)

7.3 Measurement procedure

Perform the following steps consecutively:

1) Place the building material test sample (3.1.4) in the exhalation vessel [5.3 d)].

   The building material test sample (3.1.4) shall be set up centrally in the exhalation vessel [5.3 d)] on supports that are at least 1 cm high. If multiple building material test samples (3.1.4) are being analysed simultaneously, the distance between the samples shall be at least 1 cm. The distance to the side walls of the exhalation vessel shall also be at least 1 cm.

2) Place the thermometer [5.2 l)] and the relative-humidity meter [5.3 h)] in the exhalation vessel [5.3 d)].

3) Seal the vessel [5.2 d)] hermetically.

4) Start the nitrogen volume flow and adjust it to the tested volume flow rate (7.2.2).

5) Check whether the incoming and outgoing volume flow rates are equal. If this is not the case, find and resolve the cause of the leak.

6) Flush the free volume (3.1.4) of the exhalation vessel [5.3 d)] at least five times using nitrogen gas [5.3 f)]. It is necessary to flush the volume to obtain a constant supply of radon to the adsorbent material. On the one hand, the radon present in the free volume (3.1.6) is gradually removed and, on the other hand, radon activity is built up due to release from the building material test sample (3.1.4).

7) Determine the temperature [5.2 k)] and the relative humidity [5.2 h)]. Stop the flushing procedure if the temperature falls outside the range of 18 °C to 22 °C or if the relative humidity falls outside the range of 45 % to 55 %.

8) Weigh 20 g of KOH [5.1. b)] and transfer it to the drying column [5.3 c)].

9) Weigh the required mass of silica gel [5.1. e)] and transfer it to the adsorption column [5.3 a)]. Fill the Dewar flask [5.3 b)] with liquid nitrogen [5.3 g)] and cool the adsorption column [5.3 a)] for at least 10 min. Ensure that the column remains above the level of the liquid.

10) Pass the outgoing nitrogen volume flow during a certain duration over the drying column [5.3 c)] and the (constantly cooled) adsorption column [5.3 a)] that are connected in series. Determine the adsorption duration using a chronometer [5.3 l)]. The duration shall not exceed the maximum tested value (7.2.3).

    NOTE The duration of the adsorption period depends on a number of factors e.g. the size of the building material test sample (3.1.4), the expected free radon exhalation rate and the sensitivity of the analysis method. If the adsorption duration is too short, the result may fall below the detection limit.

11) Disconnect the adsorption column once the adsorption duration has finished;

12) Cool the adsorption column [5.3 a)] using ice water [5.1 a)] to ensure pressure build-up does not occur in the glass vial [5.2 g)] with scintillation cocktail [5.1 d)]. It is, in practice, sufficient to put the adsorption column [5.2 a)] in ice water [5.1 a)] for at least 5 min.

13) Fill a glass vial [5.2 g)] with scintillation cocktail [5.1. d)]. The required volume of scintillation cocktail [5.1 d)] depends on the volume of the applied vials [5.2 g)] and the volume of silica gel [5.1 e)]. The head space in the vial after the addition of the silica gel [5.1 e)] shall be approximately 1 ml. Since the silica gel [5.1 e)] of one batch (3.1.1) always assumes a fixed volume, the volume of scintillation cocktail [5.1 d)] (of this silica gel batch) is fixed.

14) Remove the adsorption column [5.3 a)] from the ice water [5.1 a)], dry the outside using a tissue, transfer the content to the vial [5.2 g)], seal the vial [5.2 g)] immediately and determine its counting rate (7.2.3).

15) Repeat steps 1) up to and including 14) for the next building material test sample (3.1.4) or steps 8) up to and including 14) for a multiple determination of the same building material test sample (3.1.4);

16) Weigh the required mass of silica gel [5.1 e)], (7.2.3) for the blank and transfer it immediately and without performing any other step to a vial [5.2 g)] with scintillation cocktail [5.1 d)]. Include at least one blank per counting series. Strictly speaking, the blank is obtained by performing the steps 2) up to and including 14) without there being a building material test sample (3.1.4) present. In practice, however, no difference has been found between both types of blanks. If a difference can be demonstrated, this may indicate that the used nitrogen gas is not radon-free [5.1 f)]. The blank shall always be performed in accordance with activities 2) up to and including 14), should this be the case.

17) Put the glass vials including one or more blanks in the LSC.

18) Only start the count following the desorption duration for which a minimum value has been determined in 7.2.3.

19) Count the adsorbent material test sample (3.1.5) for at least 1 h.

20) Determine the (average) counting rate of the adsorbent material test samples (3.1.5).

8 Expression of results

8.1 General

These measurements are cases where in addition to the Poisson uncertainties of the counting measurements unknown influences add to the uncertainties.

Random influences due to, for instance, sample treatment and instruments cause measurement deviations, which can be different from sample to sample. In such cases, the counting results, n_i , of the counting measurements on several samples of a radioactive material to be examined, on several blanks of a radioactively labelled blank material, and on several reference samples of a standard reference material are therefore respectively averaged to obtain suitable estimates, x_1 and x_2, of the input quantities, X_1 and X_2 and the associated standard uncertainties, $u(X_1)$ and $u(X_2)$, respectively. Accordingly, X_1 shall be considered as the mean gross count rate and X_2 as the mean background count rate. Therefore, the measurand, Y, with the sought-after true value, $\tilde{y}$, shall also be taken as an averaged quantity, for instance as the mean net count rate or mean activity of the samples.

8.2 Free exhalation rate

The free exhalation rate is calculated using the following model of evaluation:

$$\begin{aligned}\bar{\varphi}_f &= \frac{\bar{R}_g - \bar{R}_0}{\bar{F}_c \cdot t_a} \cdot \frac{\lambda_{Rn} + \lambda_v}{\lambda_v} \cdot \frac{\lambda_{Rn} \cdot t_a}{1 - e^{-\lambda_{Rn} \cdot \mathrm{t}_a}} \cdot \frac{\lambda_{Rn} \cdot t_g}{e^{-\lambda_{Rn} \cdot \mathrm{t}_w} - e^{-\lambda_{Rn} \cdot (\mathrm{t}_w + \mathrm{t}_c)}} \\ &= \frac{\bar{n}_g / t_g - \bar{n}_0 / t_0}{\bar{F}_c \cdot t_a} \cdot \frac{\lambda_{Rn} + \lambda_v}{\lambda_v} \cdot \frac{\lambda_{Rn} \cdot t_a}{1 - e^{-\lambda_{Rn} \cdot t_a}} \cdot \frac{\lambda_{Rn} \cdot t_g}{e^{-\lambda_{Rn} \cdot t_w} - e^{-\lambda_{Rn} \cdot (t_w + t_g)}}\end{aligned} \quad (2)$$

Note that subscript g is used for the gross measurements of the adsorbent test sample.

For easier use the model is written as

$$\begin{aligned}\overline{\varphi}_{\mathrm{f}} &= (\overline{R}_{\mathrm{g}} - \overline{R}_0)\cdot\frac{1}{\overline{\overline{F}}_{\mathrm{c}}}\cdot\frac{1}{t_{\mathrm{a}}}\cdot\frac{\lambda_{\mathrm{Rn}}+\lambda_{\mathrm{v}}}{\lambda_{\mathrm{v}}}\cdot\frac{\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}{1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}}\cdot\frac{\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{g}}}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w}}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_{\mathrm{w}}+t_{\mathrm{g}}\right)}} = (\overline{R}_{\mathrm{g}} - \overline{R}_0)\cdot w \\ &= (\overline{n}_{\mathrm{g}}/t_{\mathrm{g}} - \overline{n}_0/t_0)\cdot\frac{1}{\overline{\overline{F}}_{\mathrm{c}}}\cdot\frac{\lambda_{\mathrm{Rn}}+\lambda_{\mathrm{v}}}{\lambda_{\mathrm{v}}}\cdot\frac{\lambda_{\mathrm{Rn}}}{1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}}\cdot\frac{\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{g}}}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w}}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_{\mathrm{w}}+t_{\mathrm{g}}\right)}} = (\overline{n}_{\mathrm{g}}/t_{\mathrm{g}} - \overline{n}_0/t_0)\cdot w\end{aligned} \tag{3}$$

with

$$\overline{n}_{\mathrm{g}} = \frac{1}{m_{\mathrm{g}}}\sum_{i=1}^{m_{\mathrm{g}}} n_{\mathrm{g},i}\,;\quad \overline{n}_0 = \frac{1}{m_0}\sum_{i=1}^{m_0} n_{0,i}$$

and

$$w = \frac{1}{\overline{\overline{F}}_{\mathrm{c}}}\cdot\frac{\lambda_{\mathrm{Rn}}+\lambda_{\mathrm{v}}}{\lambda_{\mathrm{v}}}\cdot\frac{\lambda_{\mathrm{Rn}}}{1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}}\cdot\frac{\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{g}}}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w}}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_{\mathrm{w}}+t_{\mathrm{g}}\right)}}$$

8.3 Standard uncertainty

The standard uncertainty of the free exhalation rate is calculated by

$$u^2(\overline{\varphi}_{\mathrm{f}}) = \left(u^2(\overline{n}_{\mathrm{g}})/t_{\mathrm{g}}^2 + u^2(\overline{n}_0)/t_0^2\right)\cdot w^2 + \overline{\varphi}_{\mathrm{f}}^2\cdot u_{\mathrm{rel}}^2(w) \tag{4}$$

with

$$u^2(\overline{n}_{\mathrm{g}}) = \frac{1}{m_{\mathrm{g}}}\left(\overline{n}_{\mathrm{g}} + \frac{m_{\mathrm{g}}-1}{m_{\mathrm{g}}-3}\cdot\overline{n}_{\mathrm{g}}\right) + \frac{1}{m_{\mathrm{g}}-3}\sum_{i=1}^{m_{\mathrm{g}}}(n_{\mathrm{g},i}-\overline{n}_{\mathrm{g}})^2$$

$$u^2(\overline{n}_0) = \frac{1}{m_0}\left(\overline{n}_0 + \frac{m_0-1}{m_0-3}\cdot\overline{n}_0\right) + \frac{1}{m_0-3}\sum_{i=1}^{m_0}(n_{0,i}-\overline{n}_0)^2$$

Neglecting the uncertainties of the time measurements and of the decay constants, the relative uncertainty of w is

$$u_{\mathrm{rel}}(w) = u_{\mathrm{rel}}(\overline{F_{\mathrm{c}}})$$

In 7.2.4 it is specified that the calibration factor has to be determined at least five times:

$$\begin{aligned}\bar{F}_\mathrm{c} &= \frac{\bar{R}_\mathrm{g}-\bar{R}_0}{A_\mathrm{Ra}}\cdot\frac{\lambda_\mathrm{Rn}\cdot t_\mathrm{a}}{1-e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{a}}}\cdot\frac{\lambda_\mathrm{Rn}\cdot t_\mathrm{g}}{e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{w}}-e^{-\lambda_\mathrm{Rn}\cdot\left(t_\mathrm{w}+t_\mathrm{g}\right)}}\\ &= \frac{\bar{n}_\mathrm{g}/t_\mathrm{g}-\bar{n}_0/t_0}{A_\mathrm{Ra}}\cdot\frac{\lambda_\mathrm{Rn}\cdot t_\mathrm{a}}{1-e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{a}}}\cdot\frac{\lambda_\mathrm{Rn}\cdot t_\mathrm{g}}{e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{w}}-e^{-\lambda_\mathrm{Rn}\cdot\left(t_\mathrm{w}+t_\mathrm{g}\right)}}\end{aligned} \tag{5}$$

where

$$A_\mathrm{Ra} = A_\mathrm{Ra,s}\cdot\lambda_\mathrm{Rn}\cdot t_\mathrm{a}$$

The uncertainty of the calibration factor is calculated by

$$u_\mathrm{rel}^2(\bar{F}_\mathrm{c}) = u_\mathrm{rel}^2(\bar{n}_\mathrm{g}/t_\mathrm{g}-\bar{n}_0/t_0)+u_\mathrm{rel}^2(A_\mathrm{Ra})$$

with

$$u_\mathrm{rel}^2(\bar{n}_\mathrm{g}/t_\mathrm{g}-\bar{n}_0/t_0) = \frac{u^2(\bar{n}_\mathrm{g})/t_\mathrm{g}^2+u^2(\bar{n}_0)/t_0^2}{(\bar{n}_\mathrm{g}/t_\mathrm{g}-\bar{n}_0/t_0)^2}$$

and

$$u^2(\bar{n}_\mathrm{g}) = \frac{1}{m_\mathrm{g}}\left(\bar{n}_\mathrm{g}+\frac{m_\mathrm{g}-1}{m_\mathrm{g}-3}\cdot\bar{n}_\mathrm{g}\right)+\frac{1}{m_\mathrm{g}-3}\sum_{i=1}^{m_\mathrm{g}}(n_{\mathrm{g},i}-\bar{n}_\mathrm{g})^2$$

$$u^2(\bar{n}_0) = \frac{1}{m_0}\left(\bar{n}_0+\frac{m_0-1}{m_0-3}\cdot\bar{n}_0\right)+\frac{1}{m_0-3}\sum_{i=1}^{m_0}(n_{0,i}-\bar{n}_0)^2$$

8.4 Decision threshold

For a zero-free exhalation rate one expects $\bar{n}_\mathrm{g}=\bar{n}_0$ and obtains

$$\tilde{u}^2(0) = 2\cdot w^2\cdot u^2(\bar{n}_0)/t_0^2 \tag{6}$$

and the decision threshold

$$\bar{\varphi}_\mathrm{f}^* = k_{1-\alpha}\cdot w\cdot\sqrt{2}\cdot u(\bar{n}_0)/t_0 \tag{7}$$

8.5 Detection limit

The detection limit has to be calculated by the interpolation method of ISO 11929 since no knowledge exists about the standard uncertainty as a function of the true value of the free exhalation rate.

Using the result of a measurement of the free exhalation rate, $\bar{\varphi}_\mathrm{f,1}$, and its associated standard uncertainty, $u(\bar{\varphi}_\mathrm{f,1})$, the linear interpolation leads to the approximation for the detection limit:

$$\bar{\varphi}_\mathrm{f}^\# = a+\sqrt{a^2+(k_{1-\beta}^2-k_{1-\alpha}^2)\cdot\tilde{u}^2(0)} \tag{8}$$

with

$$a = k_{1-\alpha} \cdot \tilde{u}(0) + \frac{1}{2}\left\{\left(k_{1-\beta}^{2} / \bar{\varphi}_{\mathrm{f},1}\right)\left[u^{2}(\bar{\varphi}_{\mathrm{f},1}) - \tilde{u}^{2}(0)\right]\right\} \tag{9}$$

9 Test report

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

a) reference to this part of ISO 11665;

b) measurement method;

c) sampling method;

d) number of test samples and identification of the sample;

e) characteristics of the building material test samples (mass, dimensions and external surface); storage duration in the conditioning room (in days);

f) variation of the mass of the building material test samples (decrease or increase) during the storage duration (in g);

g) units in which the results are expressed;

h) test result, $\overline{\varphi_\mathrm{f}} \pm u_r(\overline{\varphi_\mathrm{f}})$ or $\overline{\varphi_\mathrm{f}} \pm U_r$, with the associated k value;

i) any deviations from this method, and details of all circumstances which could have affected the result.

Complementary information may be provided, such as the following:

j) purpose of measurement;

k) probabilities α, β and (1-γ);

l) the decision threshold and the detection limit; depending on the customer request, there are different ways to present the result:

  - when the free exhalation rate is compared with the decision threshold (see ISO 11929), the result of the measurement shall be expressed as ≤ $\bar{\phi}_\mathrm{f}^{*}$ when the result is below the decision threshold;
  - when the free exhalation rate is compared with the detection limit, the result of the measurement can be expressed as ≤ $\bar{\phi}_\mathrm{f}^{\#}$ 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.

Annex A
(normative)

Method for determination of free radon exhalation rate of mineral-based building materials — Total count determination using gamma-ray spectrometry

A.1 General

The following clauses of the main body of this part of ISO 11665 apply to Annex A: 1, 2, 3, 5, 9 and 10.

For the purpose of Annex A, the following symbols. in addition to those given in Clause 3, apply.

Symbol	**Name of quantity**
P_{Rn}	Source strength of the radon emanation source in Becquerel per second
$t_{w,0}$	Duration between the end of the adsorption phase and the start of the count of the blank, in seconds
$t_{a,0}$	Duration between the start and the end of the adsorption step of the test portion of the blank, in seconds

A.2 Principle

The building material test sample (3.1.4) is conditioned at (20 ± 2) °C and (50 ± 5) % relative humidity. After conditioning of the building material test sample (3.1.4), the building material test sample (3.1.4) is placed in an exhalation vessel so that radon exhalation takes place towards the free volume (3.1.6) of the exhalation vessel.

The free radon exhalation rate is determined by flushing the radon activity concentration using nitrogen from the free volume (3.1.6) during a defined period and by collecting it on an adsorbent material (purge and trap method).

The ventilation rate (3.1.8) is chosen sufficiently larger than the radon decay rate to ensure that the radon exhalation rate approximates the free radon exhalation rate. The humidity of the gas, the volume flow rate and the temperature are kept at constant values while flushing. After the free volume (3.1.6) has been flushed at least five times, the radon activity concentration of the free volume (3.1.6) and of the outgoing nitrogen volume flow is be constant. Next, the nitrogen volume flow is passed over an adsorbent material used to collect the radon.

The activity of the adsorbed radon decay products is determined using an integral measurement with a semi-conductor gamma-ray spectrometer [A.3.2 e)] after a defined adsorption duration. The free radon exhalation rate is calculated starting from this.

The test method consists, consecutively, of

- sampling and preparation of building material test samples in accordance with 6,
- performing the test in accordance with A.3, and
- processing results in accordance with A.4.

A.3 Reagents and equipment

A.3.1 Reagents

a) **Activated charcoal** reactivated for at least 12 h at 110 °C;

b) **Demineralized water** of volume 0,5 l to 1,0 l.

The volume will depend on the volume of the gas washing bottle that is used to humidify the nitrogen volume flow.

c) **Source of radon emanation** of certified source strength ($Bq.s^{-1}$).

d) **Silica gel** [5.1 e)].

e) **Nitrogen**, N_2, gas.

f) **Nitrogen, N_2, liquid** of volume 5 l to 10 l.

The volume will depend on the volume of the Dewar flasks [A.3.1 b)] used.

A.3.2 Equipment

a) **Adsorption columns (2)**. Glass U tube of sufficient length and diameter. The tube should be able to contain approximately 17 g of activated charcoal.

b) **Dewar flasks (2)** with a volume of a few litres.

c) **Drying column** of volume 3 l to 6 l.

d) **Exhalation vessel** [5.3 d)].

e) **Gamma-ray spectrometer**.

f) **Gas washing bottles (2)** of volume 500 ml to 1 000 ml.

g) **Calibrated length measuring instrument** [5.2 i)].

h) **Sample containers** of geometry suitable for putting on a gamma-ray spectrometer, sealable, made of polystyrene, and of volume sufficiently large to contain the total volume of activated charcoal from the adsorption column.

i) **Relative-humidity meters (3)** with a measuring range of 0 % to 99 %, measurement uncertainty of maximum 0,5 %, and reading uncertainty of maximum 0,5 %.

j) **Thermometer** [5.2 l)].

k) **Chronometer** [5.3 l)].

l) **Connection tubes,** together with valves, if required [5.3 m)].

m) **Volume flow rate meter** with a measuring range of 0 $ml.min^{-1}$ to 1 000 $ml.min^{-1}$, measurement uncertainty of maximum 1 %, and reading uncertainty of maximum 1 % [5.3 o)].

n) **Volume flow regulators (2)** with a measuring range of 0 $ml.min^{-1}$ to 500 $ml.min^{-1}$, measurement uncertainty of maximum 1 %, and reading uncertainty of maximum 1 %.

p) **Adhesive tape**.

A.3.3 Test bench

Create a measuring assembly as specified in Figure A.1 with the components described in A.3.2.

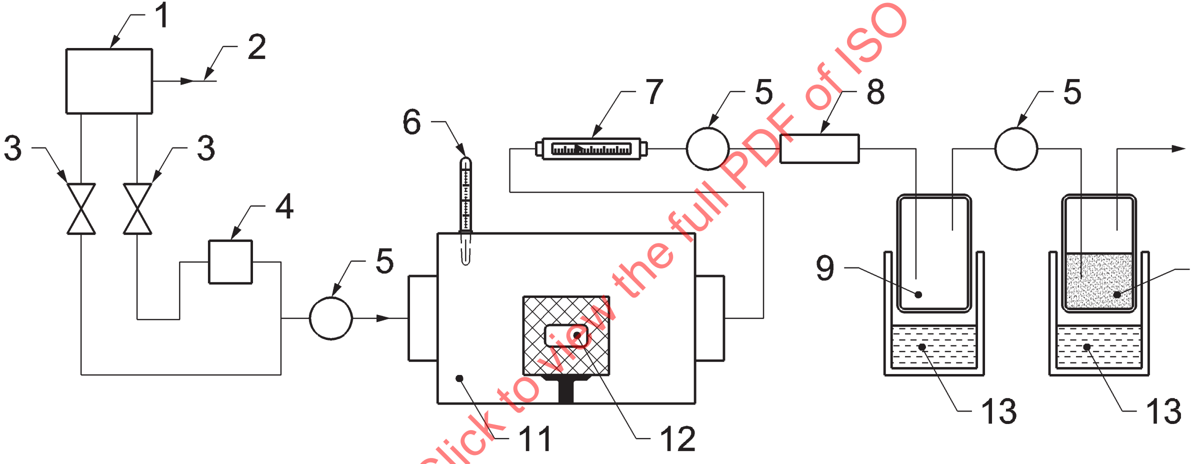
The components are connected using tubes and fittings [A.3.2 l)]. Fill the gas washing bottle [A.3.2 f)] for humidifying the nitrogen volume flow with water [A.3.2 b)] and start the nitrogen volume flow. Adjust the volume flow regulators [A.3.2 n)] so that the relative-humidity meter [A.3.2 i)] in the nitrogen volume flow that enters into the exhalation vessel [5.3 d)] reads (50 ± 5) %.

Check whether the sum of the regulated volume flows (within the reading accuracies) equals the value that is specified by the volume flow rate meter [A.3.2 m)] that is included in the volume flow that leaves the exhalation vessel.

Check whether the value specified by the relative-humidity meter [A.3.2 i)] in the outgoing nitrogen volume flow matches within an accuracy of 2 % the value of the incoming nitrogen volume flow and that both correspond to a value of (50 ± 5) %.

Check whether the value specified by the relative-humidity meter [A.3.2 i)] in the outgoing nitrogen volume flow after the cold trap is smaller than 1 %.

If one of the above checks is negative, the problem should be traced and resolved.



Key

1 nitrogen gas [A.3.1 e)]
2 dry nitrogen volume flow
3 volume flow rate regulator [A.3.2 n)]
4 humidifier (gas washing bottle filled with water) [A.3.2 f)] with [A.3.1 b)]
5 relative-humidity meter [A.3.2 i)]
6 thermometer [5.2 l)]
7 volume flow rate meter [A.3.2 m)]
8 drying column (silica gel) [A.3.2 c)] with [5.3 e)]
9 cold trap (gas washing bottle connected in the opposite direction) [A.3.2 f)]
10 adsorption column [A.3.2 a)] filled with activated charcoal [A.3.1 a)]
11 exhalation vessel [5.3 d)]
12 building material test sample
13 liquid nitrogen [A.3.1 f)]

Figure A.1 — Schematic representation of test bench

A.4 Test procedure

A.4.1 Set-up of test bench

a) Select the volume flow rate (7.2.1).

b) Determine the amount of activated charcoal [A.3.1 a)].

The minimum required amount of activated charcoal should be determined so that all radon is adsorbed in the adsorption column [A.3.2 a)]. Determine this in accordance with 7.2.2 using two adsorption columns [A.3.2 a)] placed in series and with a radon exhalation source [A.3.1 c)] for which the source strength (Bq.s^{-1}) is greater than that of the test samples to be measured later. The adsorption period for the determination should be the same or longer than the period for building material test samples to be measured later. After adsorption, no radon should be present in the second column [A.3.2 a)] (the quotient of the net counting rates recalculated towards the reference time of the test portions made from the second column [A.3.2 a) and the first column [A.3.2 a) respectively, should be smaller than 0,01). If this is not the case, the amount of activated charcoal [A.3.1 a)] should be increased and the procedure described above should be repeated.

The required amount of activated charcoal [A.3.1 a)] depends on the volume flow rate. The correct amount should be determined empirically. If the volume flow rate is increased, the required amount of activated charcoal [A.3.1 a)] should be determined again.

NOTE It has been found that a mass of activated charcoal of 17 g is sufficient for adsorption durations up to and including 100 h using a volume flow rate of 400 ml.min^{-1} and source strength of 0,04 Bq.s^{-1}.

The adsorption capacity of the activated charcoal [A.3.1 a)] may differ per batch. If a new batch is used, the previously determined amount should be reassessed.

A.4.2 Gamma-ray spectrometer procedure

a) **Setting up the gamma-ray spectrometer** [A.3.2 e)]

Set a lower and an upper limit for the measuring range (energy window). The lower limit should be higher than 50 keV and the upper limit may, in general, be equated with the maximum energy that can still be determined with the spectrometer.

The lower limit of the gamma-ray spectrometer [A.3.2 e)] is chosen at a higher value than 50 keV to exclude the 46,5 keV line of ^{210}Pb.

b) **Determination of counting efficiency**

The following steps should be performed to generate test portions for the counting efficiency determination:

1) place the emanation source [A.3.1 c)] in the exhalation vessel;

2) perform the activities A.4.3 2) up to and including 13).

Analyse the test portion in accordance with the procedure described in A.4.3 15) to 17) after performing these activities. At least five test portions should be generated and analysed for the counting efficiency determination. The adsorption periods of these test portions should be different and should be representative for the adsorption period that is used during the measurement of test samples.

c) **Background determination**

The required mass of activated charcoal [A.3.1 a)] is weighed and placed directly into a sample container [A.3.2 h)] for generating a test portion for the background determination. This sample container [A.3.2 h)] is closed and placed in the gamma-ray spectrometer [A.3.2 e)]. The counting rate is determined in the measuring range (energy window) set in accordance with a).

d) **Blank determination**

The following activities should be performed for generating a blank:

1) check whether the exhalation vessel [5.3 d)] is empty;

2) perform the A.4.3 2) up to and including 13).

Analyse the test portion in accordance with the procedure described in A.4.3 15) to 17) after performing these steps.

A.4.3 Measurement procedures

Perform the following steps for generating an adsorbent test, test sample of a building material test sample:

1) See 7.3, 1).

2) Seal the vessel [5.3 d)] hermetically.

3) Start the nitrogen volume flow and set this to the tested volume flow (see 7.2.2) and also set the incoming relative humidity to (50 ± 5) %.

4) See 7.3 5).

5) See 7.3 6).

6) See 7.3 7).

7) Fill the Dewar flask [A.3.2 b)] of the cold trap with liquid nitrogen [A.3.1 f)]. Put the cold trap in the nitrogen vapour in this Dewar flask [A.3.2 b)] (ensure that the trap remains above the level of the liquid). Check whether the value specified by the relative-humidity meter [A.3.2 i)] in the outgoing nitrogen volume flow after the cold trap is smaller than 1 %.

8) Weigh the required [A.4.1 c)] mass of activated charcoal and transfer this to the adsorption column [A.3.2 c)].

9) Fill the Dewar flask [A.3.2 b)] of the adsorption column [A.3.2 a)] with liquid nitrogen [A.3.1 f)]. Put the adsorption column [A.3.2 a)] in the nitrogen vapour in this Dewar flask [A.3.2 b)] (ensure that the column remains above the level of the liquid). Pass the dry nitrogen volume flow through the activated charcoal [A.3.1 a)] and cool the adsorption column [A.3.2 a)] for at least 15 min.

10) Stop the dry nitrogen volume flow and pass the outgoing volume flow of the cold trap through the continuously cooled adsorption column [A.3.2 a)]. Determine the adsorption time with the chronometer [A.3.2 k)]. The adsorption time may not be longer than the maximum tested value (see A.4.2 c)].

NOTE The duration of the adsorption process depends on the expected free radon exhalation rate and the counting efficiency of the used gamma-ray spectrometer [A.3.2 e)]. If the adsorption duration is too short, the result may fall below the detection limit.

11) Stop the nitrogen supply through the adsorption column [A.3.2 a)] at the end of the adsorption duration.

12) Transfer the activated charcoal [A.3.1 a)] from the adsorption column [A.3.2 a)] to the sample container. The activated charcoal [A.3.1 a)] reaches room temperature after 10 min and the sample container [A.3.2 h)] is closed with a lid and sealed with adhesive tape [A.3.2 p)].

13) Homogenize the sample by shaking and apply a waiting time of at least 3 h. The sample is now ready for analysis.

14) Repeat steps 1) up to and including 13) for a new building material test sample or activities 2) up and including 13) for a new determination of the same building material test sample.

Analyse the adsorbent test sample(s) (3.1.5) in accordance with the procedure described in 15) to 17) after performing these steps.

15) Place test portion *n* in the gamma-ray spectrometer.

16) Determine the gross counting rate in the set measuring range (energy window) for a minimum of 1 h (see A.4.3).

17) Calculate the net counting rate by subtracting the measured background counting rate (see A.4.3).

A.5 Expression of results

A.5.1 Free exhalation rate

The free exhalation rate is calculated by the model of evaluation:

$$\bar{\varphi}_\mathrm{f} = \left[\bar{R}_\mathrm{g} \cdot \frac{\lambda_\mathrm{Rn} \cdot t_\mathrm{g}}{e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{w}} - e^{-\lambda_\mathrm{Rn} \cdot (t_\mathrm{w} + t_\mathrm{g})}} - \bar{R}_0 \cdot \frac{\lambda_\mathrm{Rn} \cdot t_0}{e^{-\lambda_\mathrm{Rn} \cdot t_{\mathrm{w},0}} - e^{-\lambda_\mathrm{Rn} \cdot (t_0 + t_{\mathrm{w},0})}} \cdot \frac{1 - e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{a}}}{1 - e^{-\lambda_\mathrm{Rn} \cdot t_{\mathrm{a},0}}} \right] \cdot \left[\frac{\lambda_\mathrm{Rn} + \lambda_\mathrm{v}}{\lambda_\mathrm{v}} \cdot \frac{\lambda_\mathrm{Rn} \cdot t_\mathrm{a}}{\left(1 - e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{a}}\right)} \right] \cdot \frac{1}{\bar{F}_\mathrm{c} \cdot t_\mathrm{a}}$$

$$= \left[\frac{\bar{n}_\mathrm{g}}{t_\mathrm{g}} \cdot \frac{\lambda_\mathrm{Rn} \cdot t_\mathrm{g}}{e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{w}} - e^{-\lambda_\mathrm{Rn} \cdot (t_\mathrm{w} + t_\mathrm{g})}} - \frac{\bar{n}_0}{t_0} \cdot \frac{\lambda_\mathrm{Rn} \cdot t_0}{e^{-\lambda_\mathrm{Rn} \cdot t_{\mathrm{w},0}} - e^{-\lambda_\mathrm{Rn} \cdot (t_0 + t_{\mathrm{w},0})}} \cdot \frac{1 - e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{a}}}{1 - e^{-\lambda_\mathrm{Rn} \cdot t_{\mathrm{a},0}}} \right] \cdot \left[\frac{\lambda_\mathrm{Rn} + \lambda_\mathrm{v}}{\lambda_\mathrm{v}} \cdot \frac{\lambda_\mathrm{Rn} \cdot t_\mathrm{a}}{\left(1 - e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{a}}\right)} \right] \cdot \frac{1}{\bar{F}_\mathrm{c} \cdot t_\mathrm{a}}$$

$$= \left[\bar{n}_\mathrm{g} \cdot \frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{w}} - e^{-\lambda_\mathrm{Rn} \cdot (t_\mathrm{w} + t_\mathrm{g})}} - \bar{n}_0 \cdot \frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn} \cdot t_{\mathrm{w},0}} - e^{-\lambda_\mathrm{Rn} \cdot (t_0 + t_{\mathrm{w},0})}} \cdot \frac{1 - e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{a}}}{1 - e^{-\lambda_\mathrm{Rn} \cdot t_{\mathrm{a},0}}} \right] \cdot \left[\frac{\lambda_\mathrm{Rn} + \lambda_\mathrm{v}}{\lambda_\mathrm{v}} \cdot \frac{\lambda_\mathrm{Rn}}{\left(1 - e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{a}}\right)} \right] \cdot \frac{1}{\bar{F}_\mathrm{c}}$$

with

$$\bar{n}_\mathrm{g} = \frac{1}{m_\mathrm{g}} \sum_{i=1}^{m_\mathrm{g}} n_{\mathrm{g},i} \, ; \quad \bar{n}_0 = \frac{1}{m_0} \sum_{i=1}^{m_0} n_{0,i}$$

For easier use the model is written as

$$\bar{\varphi}_\mathrm{f} = \left[\frac{\bar{n}_\mathrm{g}}{t_\mathrm{g}} \cdot a - \frac{\bar{n}_0}{t_0} \cdot b \right] \cdot c$$

with

$$a = \frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{w}} - e^{-\lambda_\mathrm{Rn} \cdot (t_\mathrm{w} + t_\mathrm{g})}}$$

$$b = \frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn} \cdot t_{\mathrm{w},0}} - e^{-\lambda_\mathrm{Rn} \cdot (t_0 + t_{\mathrm{w},0})}} \cdot \frac{1 - e^{-\lambda_\mathrm{Rn} \cdot t_\mathrm{a}}}{1 - e^{-\lambda_\mathrm{Rn} \cdot t_{\mathrm{a},0}}}$$

$$c=\left[\frac{\lambda_{\mathrm{Rn}}+\lambda_{\mathrm{v}}}{\lambda_{\mathrm{v}}}\cdot\frac{\lambda_{\mathrm{Rn}}}{\left(1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}\right)}\right]\cdot\frac{1}{\overline{F}_{\mathrm{c}}}$$

to obtain

$$\overline{\varphi}_{\mathrm{f}}=\left[\frac{\overline{n}_{\mathrm{g}}}{t_{\mathrm{g}}}-\frac{\overline{n}_0}{t_0}\cdot\frac{b}{a}\right]\cdot a\cdot c=\left[\frac{\overline{n}_{\mathrm{g}}}{t_{\mathrm{g}}}-\frac{\overline{n}_0}{t_0}\cdot d\right]\cdot w$$

Neglecting the uncertainties of the decay constants and the time measurements, the product $a \cdot c = w$ is the calibration factor without producing correlations when calculating the uncertainties.

$$w=\frac{\lambda_{\mathrm{Rn}}}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w}}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_{\mathrm{w}}+t_{\mathrm{g}}\right)}}\cdot\left[\frac{\lambda_{\mathrm{Rn}}+\lambda_{\mathrm{v}}}{\lambda_{\mathrm{v}}}\cdot\frac{\lambda_{\mathrm{Rn}}}{\left(1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}\right)}\right]\cdot\frac{1}{\overline{F}_{\mathrm{c}}}$$

The relative uncertainty of w is

$$u_{\mathrm{rel}}(w)=u_{\mathrm{rel}}(\overline{F}_{\mathrm{c}})$$

$\frac{b}{a}=d$ is a background modifying factor with zero uncertainty.

Thus is obtained a model of evaluation which corresponds to the general model according to ISO 11929.

The standard uncertainty of the free exhalation rate is calculated by

$$u^2(\overline{\varphi}_{\mathrm{f}})=(u^2(\overline{n}_{\mathrm{g}})/t_{\mathrm{g}}^2+d^2\cdot u^2(\overline{n}_0)/t_0^2)\cdot w^2+\overline{\varphi}_{\mathrm{f}}^2\cdot u_{\mathrm{rel}}^2(w)$$

with

$$u^2(\overline{n}_{\mathrm{g}})=\frac{1}{m_{\mathrm{g}}}\left(\overline{n}_{\mathrm{g}}+\frac{m_{\mathrm{g}}-1}{m_{\mathrm{g}}-3}\cdot\overline{n}_{\mathrm{g}}\right)+\frac{1}{m_{\mathrm{g}}-3}\sum_{i=1}^{m_{\mathrm{g}}}(n_{\mathrm{g},i}-\overline{n}_{\mathrm{g}})^2$$

$$u^2(\overline{n}_0)=\frac{1}{m_0}\left(\overline{n}_0+\frac{m_0-1}{m_0-3}\cdot\overline{n}_0\right)+\frac{1}{m_0-3}\sum_{i=1}^{m_0}(n_{0,i}-\overline{n}_0)^2$$

A.4.2 specifies that at least five test portions should be generated and analysed for the calibration factor determination.

For each of these determinations the corresponding calibration factor is calculated as given in

$$\overline{F}_{\mathrm{c}}=\left[\overline{R}_{\mathrm{g}}\cdot\frac{\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{g}}}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w}}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_{\mathrm{w}}+t_{\mathrm{g}}\right)}}-\overline{R}_0\cdot\frac{\lambda_{\mathrm{Rn}}\cdot t_0}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w},0}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_0+t_{\mathrm{w},0}\right)}}\cdot\frac{1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}}{1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a},0}}}\right]\cdot\left[\frac{\lambda_{\mathrm{Rn}}+\lambda_{\mathrm{v}}}{\lambda_{\mathrm{v}}}\cdot\frac{\lambda_{\mathrm{Rn}}}{P_{\mathrm{Rn}}\cdot\left(1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}\right)}\right]$$

$$=\left[\frac{\overline{n}_{\mathrm{g}}}{t_{\mathrm{g}}}\cdot\frac{\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{g}}}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w}}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_{\mathrm{w}}+t_{\mathrm{g}}\right)}}-\frac{\overline{n}_0}{t_0}\cdot\frac{\lambda_{\mathrm{Rn}}\cdot t_0}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w},0}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_0+t_{\mathrm{w},0}\right)}}\cdot\frac{1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}}{1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a},0}}}\right]\cdot\left[\frac{\lambda_{\mathrm{Rn}}+\lambda_{\mathrm{v}}}{\lambda_{\mathrm{v}}}\cdot\frac{\lambda_{\mathrm{Rn}}}{P_{\mathrm{Rn}}\cdot\left(1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}\right)}\right]$$

$$=\left[\overline{n}_{\mathrm{g}}\cdot\frac{\lambda_{\mathrm{Rn}}}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w}}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_{\mathrm{w}}+t_{\mathrm{g}}\right)}}-\overline{n}_0\cdot\frac{\lambda_{\mathrm{Rn}}}{e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{w},0}}-e^{-\lambda_{\mathrm{Rn}}\cdot\left(t_0+t_{\mathrm{w},0}\right)}}\cdot\frac{1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}}{1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a},0}}}\right]\cdot\left[\frac{\lambda_{\mathrm{Rn}}+\lambda_{\mathrm{v}}}{\lambda_{\mathrm{v}}}\cdot\frac{\lambda_{\mathrm{Rn}}}{P_{\mathrm{Rn}}\cdot\left(1-e^{-\lambda_{\mathrm{Rn}}\cdot t_{\mathrm{a}}}\right)}\right]$$

with

$$u^2(\bar{n}_\mathrm{g}) = \frac{1}{m_\mathrm{g}}\left(\bar{n}_\mathrm{g} + \frac{m_\mathrm{g}-1}{m_\mathrm{g}-3}\cdot\bar{n}_\mathrm{g}\right) + \frac{1}{m_\mathrm{g}-3}\sum_{i=1}^{m_\mathrm{g}}(n_{\mathrm{g},i}-\bar{n}_\mathrm{g})^2$$

$$u^2(\bar{n}_0) = \frac{1}{m_0}\left(\bar{n}_0 + \frac{m_0-1}{m_0-3}\cdot\bar{n}_0\right) + \frac{1}{m_0-3}\sum_{i=1}^{m_0}(n_{0,i}-\bar{n}_0)^2$$

The relative standard uncertainty $u_\mathrm{rel}(\bar{F}_\mathrm{c})$ associated with the factor $\bar{F}_\mathrm{c}$ is calculated by

$$u_\mathrm{rel}^2(\bar{F}_\mathrm{c}) = u_\mathrm{rel}^2\left(\bar{n}_\mathrm{g}\cdot\frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{w}} - e^{-\lambda_\mathrm{Rn}\cdot(t_\mathrm{w}+t_\mathrm{g})}} - \bar{n}_0\cdot\frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{w,0}} - e^{-\lambda_\mathrm{Rn}\cdot(t_0+t_\mathrm{w,0})}}\cdot\frac{1-e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{a}}}{1-e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{a,0}}}\right) + u_\mathrm{rel}^2(P_\mathrm{Rn})$$

$$u_\mathrm{rel}^2(\bar{F}_\mathrm{c}) = \frac{u^2(\bar{n}_\mathrm{g})\cdot\left(\frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{w}} - e^{-\lambda_\mathrm{Rn}\cdot(t_\mathrm{w}+t_\mathrm{g})}}\right)^2 + u^2(\bar{n}_0)\cdot\left(\frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{w,0}} - e^{-\lambda_\mathrm{Rn}\cdot(t_0+t_\mathrm{w,0})}}\cdot\frac{1-e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{a}}}{1-e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{a,0}}}\right)^2}{\left(\bar{n}_\mathrm{g}\cdot\frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{w}} - e^{-\lambda_\mathrm{Rn}\cdot(t_\mathrm{w}+t_\mathrm{g})}} - \bar{n}_0\cdot\frac{\lambda_\mathrm{Rn}}{e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{w,0}} - e^{-\lambda_\mathrm{Rn}\cdot(t_0+t_\mathrm{w,0})}}\cdot\frac{1-e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{a}}}{1-e^{-\lambda_\mathrm{Rn}\cdot t_\mathrm{a,0}}}\right)^2} + u_\mathrm{rel}^2(P_\mathrm{Rn})$$

A.5.2 Decision threshold

For a zero-free exhalation rate, expected is

$$\frac{\bar{n}_\mathrm{g}}{t_\mathrm{g}} = \frac{\bar{n}_0}{t_0}\cdot d$$

and obtains

$$\tilde{u}^2(0) = 2\cdot w^2\cdot d^2\cdot u^2(\bar{n}_0)/t_0^2$$

and the decision threshold

$$\bar{\varphi}_\mathrm{f}^* = k_{1-\alpha}\cdot w\cdot\sqrt{2}\cdot d\cdot u(\bar{n}_0)/t_0$$

A.5.3 Detection limit

The detection limit has to be calculated by the interpolation method of ISO 11929 since no knowledge exists about the standard uncertainty as a function of the true value of the free exhalation rate.

Using the result of a measurement of the free exhalation rate, $\bar{\varphi}_\mathrm{f,1}$, and its associated standard uncertainty, $u(\bar{\varphi}_\mathrm{f,1})$, the linear interpolation leads to the approximation for the detection limit:

$$\bar{\varphi}_\mathrm{f}^\# = a + \sqrt{a^2 + (k_{1-\beta}^2 - k_{1-\alpha}^2)\cdot\tilde{u}^2(0)}$$

with

$$a = k_{1-\alpha}\cdot\tilde{u}(0) + \frac{1}{2}\left\{\left(k_{1-\beta}^2/\bar{\varphi}_\mathrm{f,1}\right)\left[u^2(\bar{\varphi}_\mathrm{f,1}) - \tilde{u}^2(0)\right]\right\}$$

Annex B
(normative)

Method for determination of free radon exhalation rate of mineral-based building materials — Determination by nuclide-specific gamma-ray spectrometry

B.1 General

The following clauses of the main body of this part of ISO 11665 apply to Annex B: 1, 2, 3, 5, 9 and 10.

For the purposes of Annex B, the symbols given in Clause 3 and the following apply.

Symbol	**Name of quantity**	**Unit**
P_{Rn}	source strength of the radon emanation source	$\mathrm{Bq} \cdot \mathrm{s}^{-1}$
R_{Bi}	net ^{214}Bi counting rate measured using a gamma-ray spectrometer for test portion *n* of the counting efficiency determination	s^{-1}
R_{Pb}	net ^{214}Pb counting rate measured using a gamma-ray spectrometer for test portion *n* of the counting efficiency determination	s^{-1}
$R_{0,\mathrm{Bi}}$	net ^{214}Bi counting rate measured using a gamma-ray spectrometer for the test portion of the blank	s^{-1}
$R_{0,\mathrm{Pb}}$	net ^{214}Pb counting rate measured using a gamma-ray spectrometer for the test portion of the blank	s^{-1}
$R_{\mathrm{bg,Bi}}$	background ^{214}Bi counting rate measured using the gamma-ray spectrometer for the test portion without adsorbed activity	s^{-1}
$R_{\mathrm{bg,Pb}}$	background ^{214}Pb counting rate measured using the gamma-ray spectrometer for the test portion without adsorbed activity	s^{-1}
$R_{\mathrm{min,Bi}}$	minimum detectable counting rate as a result of the ^{214}Bi radon decay products on the adsorbent material	s^{-1}
$R_{\mathrm{min,Pb}}$	minimum detectable counting rate as a result of the ^{214}Pb radon decay products on the adsorbent material	s^{-1}
$t_{w,0}$	duration between the end of the adsorption period and the start of the count of the blank	s
$t_{a,0}$	duration between the start and end of the adsorption period of the test portion of the blank	s
$t_{c,bg}$	Counting duration for measurement of the background	s
$u_r(\overline{F_{\mathrm{c,Bi}}})$	relative uncertainty for the ^{214}Bi line associated with the calibration factor	—
$u_r(\overline{F_{\mathrm{c,Pb}}})$	relative uncertainty for the ^{214}Pb line associated with the calibration factor	—

Symbol	Name of quantity	Unit
$u_r(\overline{R_{\mathrm{Bi}}})$	relative uncertainty for the ^{214}Bi line associated with the mean counting rate	—
$u_r(\overline{R_{\mathrm{Pb}}})$	relative uncertainty for the ^{214}Pb line associated with the mean counting rate	—
$u_r(R_{\mathrm{Bi}})$	relative uncertainty for the ^{214}Bi line associated with the counting rate	—
$u_r(R_{\mathrm{Pb}})$	relative uncertainty for the ^{214}Pb line associated with the counting rate	—
$u_r(\overline{\varphi_{\mathrm{f,Bi}}})$	relative uncertainty for the ^{214}Bi line associated with the free radon exhalation rate	—
$u_r(\overline{\varphi_{\mathrm{f,Pb}}})$	relative uncertainty for the ^{214}Pb line associated with the free radon exhalation rate	—
$F_{\mathrm{c,Bi}}$	calibration factor for the ^{214}Bi line	$\mathrm{s{\cdot}kg^{-1}}$
$F_{\mathrm{c,Pb}}$	calibration factor for the ^{214}Pb line	$\mathrm{s{\cdot}kg^{-1}}$
$\overline{F_{\mathrm{c,Bi}}}$	average calibration factor for the ^{214}Bi line	$\mathrm{s^{-1}{\cdot}kg^{-1}}$
$\overline{F_{\mathrm{c,Pb}}}$	average calibration factor for the ^{214}Pb line	$\mathrm{s^{-1}{\cdot}kg^{-1}}$
$\phi_{\mathrm{f,Bi}}$	free radon exhalation rate calculated using the ^{214}Bi counting rate	$\mathrm{Bq{\cdot}s^{-1}}$
$\phi_{f,Pb}$	free radon exhalation rate calculated using the ^{214}Pb counting rate	$\mathrm{Bq{\cdot}s^{-1}}$
$\overline{\varphi_{\mathrm{f,Bi}}}$	average free radon exhalation rate calculated using the ^{214}Bi counting rate	$\mathrm{(Bq.s)^{-1}}$
$\overline{\varphi_{\mathrm{f,Pb}}}$	average free radon exhalation rate calculated using the ^{214}Pb counting rate	$\mathrm{(Bq.s)^{-1}}$

B.2 Principle

The building material test sample (3.1.4) is conditioned at (20 ± 2) °C and (50 ± 5) % relative humidity. After conditioning of the building material test sample (3.1.4), the building material test sample (3.1.4) is placed in an exhalation vessel so that radon exhalation takes place towards the free volume (3.1.6) of the exhalation vessel.

The free radon exhalation rate is determined by flushing the radon activity concentration using nitrogen from the free volume (3.1.6) during a defined period and by collecting it on an adsorbent material (purge and trap method).

The ventilation rate (3.1.8) is chosen sufficiently larger than the radon decay rate to ensure that the radon exhalation rate approximates the free radon exhalation rate. The humidity of the gas, the volume flow rate and the temperature are kept at constant values while flushing. After the free volume (3.1.6) has been flushed at least five times, the radon activity concentration of the free volume (3.1.6) and of the outgoing nitrogen volume flow is be constant. Next, the nitrogen volume flow is passed over an adsorbent material used to collect the radon.

The activity of the ^{214}Bi and ^{214}Pb adsorbed radon decay products is determined using semiconductor gamma spectrometry after a defined adsorption period. The free radon exhalation rate is calculated starting from this.

The test method consists of consecutively:

- sampling and preparing test samples in accordance with 6;
- performing the test in accordance with B.4;
- processing results in accordance with B.5.

B.3 Reagents and equipment

B.3.1 Chemicals

a) **Activated charcoal** with an active surface of 100 m^2/g to 1600 m^2/g, and reactivated for at least 16 h at 110 °C.

b) **Radon standard** (3.1.7).

c) **Demineralized water**.

d) **Silica gel** [5.1 e)].

e) **Nitrogen, N_2, gas**.

B.3.2 Equipment

a) **Adsorption column**. Stainless steel tube with a diameter of 3 cm to 5 cm and sufficient capacity.

The tube should be able to contain approximately 1 l of active charcoal.

b) **Drying column** of volume: 1 l to 3 l, manufactured from stainless steel or plastic, with a diameter of 5 cm to 10 cm.

c) **Conditioning room** (temperature) with specifications as per 5.2 h), and a temperature setting of 15 °C to 21 °C.

The temperature of the adsorption column should be constant at (18 ± 3) °C during the adsorption duration.

d) **Exhalation vessel** [5.3 d)].

e) **Gamma spectrometer**.

f) **Gas washing bottle or round bottom flask** of volume sufficient to ensure the ^{226}Ra solution can be flushed.

g) **Calibrated length measuring instrument** [5.2 i)].

h) **Plastic sample container** suitable for placing on the gamma spectrometer, and capable of holding the total volume of active carbon which is 1 000 ml.

i) **Reducing valve** of minimum range 100 kPa to 300 kPa.

j) **Relative-humidity meter with probe** [5.3 h)].

k) **Thermometer** [5.2 l)].

l) **Collectors (2)** of stainless steel or Perspex with a volume of 0,7 l to 2 l.

m) **Connection tubes**, plastic [5.3 m)].

n) **Volume flow rate meter** with a measuring range of at least 0 l/h to 8 l/h, measurement uncertainty of maximum 3 %, and reading uncertainty of maximum 2 %.

o) **Volume flow rate meter (adjustable)** with a measuring range of at least 0 l/h to 8 l/h, measurement uncertainty of maximum 3 %, and reading uncertainty of maximum 2 %.

p) **Washing bottle**, stainless steel or plastic, and of volume 1 l to 3 l.

q) **Beaker** of volume 2 l.

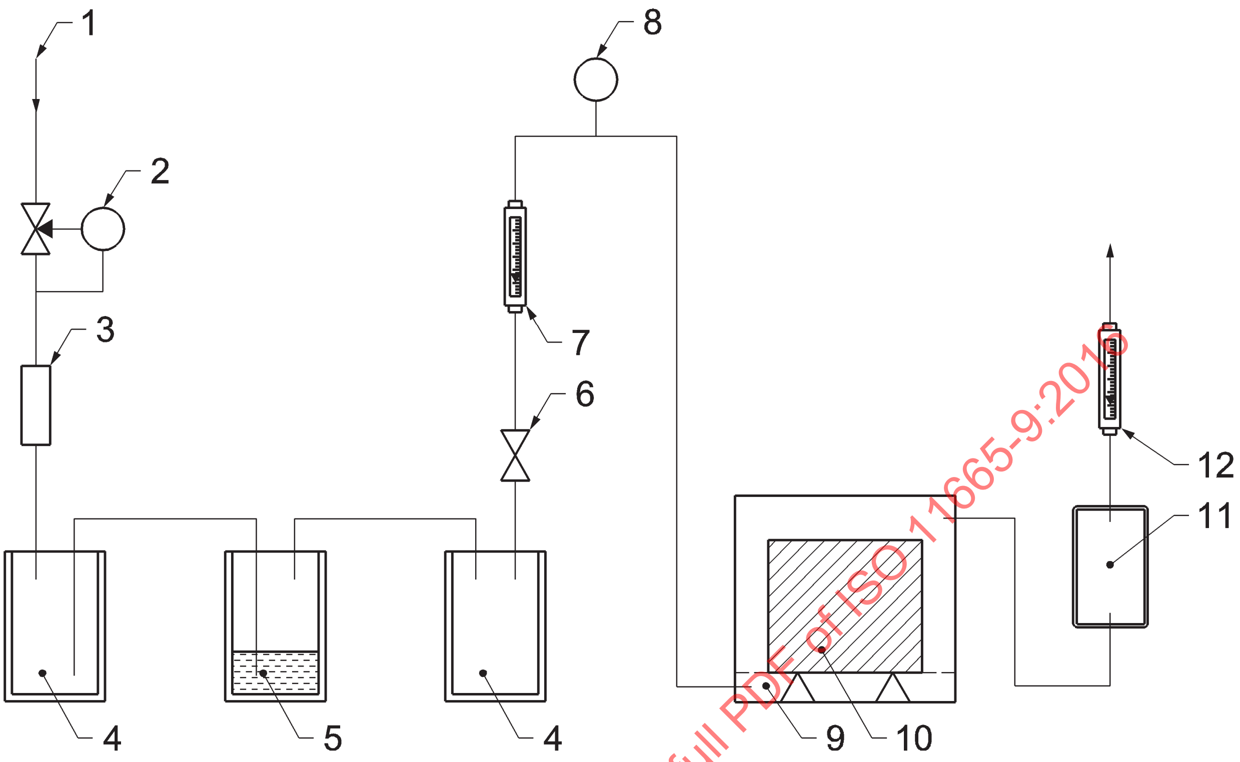
B.3.3 Test bench

Create a measuring assembly as specified in Figure B.1 with the components described in B.3.2.

The components are connected with plastic tubes [5.3 n)]. Fill the washing bottle [B.3.2 f)] with demineralized water [B.3.1 c)] (humidifier). Pass a dry nitrogen volume flow via a reducing valve [B.3.2 i)] (at approximately 200 kPa) over, successively, a drying column [B.3.2 b)] filled with silica gel [5.1 e)], a collector [B.3.2 l)], a humidifier and a collector [B.3.2 l)].

The collectors [B.3.2 l)] and the drying column [B.3.2 b)] with silica gel [5.1 e)] are used to prevent the moisture from spreading through tubes when suddenly pressure differences occur.

Pass the humidified nitrogen volume flow through the exhalation vessel [5.3 d)] via an adjustable gas volume meter [B.3.1 o)]. Check the relative humidity [B.3.2 j)] of the nitrogen volume flow, (50 ± 5) %, in the supply line to the exhalation vessel [5.3 d)] using an air-humidity meter [B.3.2 j)]. Adjust, if necessary, the pressure of the reducing valve [B.3.2 i)] until the relative humidity specified is (50 ± 5) %.



Key

1	nitrogen [B.3.2 e)]	7	volume flow rate meter 1 [B.3.2 n)]
2	volume flow rate meter (adjustable) [B.3.2 o)]	8	relative-humidity meter [B.3.2 j)]
3	drying column (silica gel) [B.3.2 b)] with [5.1 e)]	9	exhalation vessel [5.3 d)]
4	collector [B.3.2 l)]	10	building material test sample
5	humidifier (gas washing bottle filled with water) [B.3.2 f)] with [B.3.1 c)]	11	adsorption column filled with activated charcoal [B.3.2 a)] with [A.3.1 a)]
6	reducing valve [B.3.2 i)]	12	volume flow rate meter 2 [B.3.2 n)]

Figure B.1 — Schematic representation of test bench

B.4 Test procedure

B.4.1 Set-up of the test bench

a) **Select the volume flow rate** (7.2.1).

b) **Determine the amount of activated charcoal** [B.3.1. a)]

The minimum required amount of activated charcoal [B.3.1 a)] should be determined so that all radon is adsorbed in the adsorption column [B.3.2 a)]. Determine this in accordance with 7.3. using two adsorption columns [B.3.2 a)] placed in series and with a radon exhalation source [B.3.1 b)] for which the source strength (Bq.s^{-1}) is greater than that of the test samples to be measured later. The adsorption period for the determination should be the same or longer than the period for test samples to be measured later. After adsorption no radon should be present in the second column [B.3.2. a)] (the quotient of the net counting rates recalculated towards the reference time of the test portions made from the second column [B.3.2 a) and the first column [B.3.2 a) respectively, should be smaller than 0,01). If this is not the case, the amount of activated charcoal [B.3.1 a)] should be increased and the procedure described above should be repeated.

The required amount of activated charcoal [B.3.1 a)] depends on the volume flow rate (7.2.2). The correct amount should be determined empirically. If the volume flow rate is increased, the required amount of activated charcoal [B.3.1 a)] should be determined again.

NOTE It has been found that 1 l (approximately 400 g) of activated charcoal is sufficient for adsorption periods up to and including 14 days with a volume flow rate of 6 l/h.

The adsorption capacity of the activated charcoal may differ per batch. If a new batch is used, the previously determined amount should be reassessed.

c) **Determine the calibration factor**

1) prepare a solution with an activity of approximately 5 Bq to 50 Bq from the ^{226}Ra solution [5.3 c)]. Use a gas washing bottle or a round bottom flask as described in B.3.2 f);

2) Connect the gas washing bottle [B.3.2 f)] instead of the exhalation vessel [5.3 d)] downstream from the adjustable volume flow rate meter [B.3.2. o)] but do not yet connect it to the adsorption column [B.3.2 a)];

3) flush the volume at least 10 times at the chosen volume flow rate (7.2.2);

4) connect the gas washing bottle [B.3.2. f)] to the adsorption column [B.3.2 a)];

5) perform the activities B.4.3, 6) up to and including 11). Repeat this for another 4 different collection periods;

6) analyse the activated charcoal [B.3.1 a)] in accordance with B.4.1 b).

B.4.2 Gamma-ray spectrometer procedure

Set two measuring ranges (energy windows) with a lower and upper limit around 352 keV and 609 keV, respectively.

NOTE ^{214}Bi (609 keV) and ^{214}Pb (352 keV) energy lines are used.

a) **Background determination**

The required mass of activated charcoal [B.3.1 a)] is weighed and placed directly into a sample container [B.3.2 h)] for generating a test portion for the background determination. This sample container [B.3.2 h)] is closed and placed in the gamma spectrometer [B.3.2 e)]. The counting rate is determined in the measuring ranges (energy windows) set in accordance with a).

b) **Blank determination**

The steps B.4.3, 3) up to and including 12), should be performed for the blank determination.

B.4.3 Measurement procedure

Perform the following activities consecutively.

1) Place the test sample in the exhalation vessel [5.3 d)].

The test sample should be set up centrally in the exhalation vessel [5.3 d)] on supports that are at least 1 cm high. If multiple test samples are being analysed simultaneously, the distance between the samples should be at least 1 cm. The distance to the side walls of the exhalation vessel [5.3 d)] should also be at least 1 cm.

2) Place the probe of the relative-humidity meter [B.3.2 j)] in the inflow of the exhalation vessel [5.3 d)].

3) Seal the vessel [5.2 d)] hermetically.

4) Start the nitrogen volume flow and adjust it to the required volume flow rate and relative humidity.