INTERNATIONAL STANDARD

ISO 11665-9

First edition
2016-02-01

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

Reference number
ISO 11665-9:2016(E)

Contents

Page

Foreword

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

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

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

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

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

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
$A_{\text{Ra,s}}$	^{226}Ra activity of the radon standard, in becquerel
A_{Ra}	^{226}Ra activity, in becquerel
F_c	Calibration factor
$\overline{F_c}$	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
$n_{g,i}$, $n_{0,i}$	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
$\overline{n}_g$, $\overline{n}_0$	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
$n_{g,Pb,i}$, $n_{0,Pb,i}$	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
$\bar{n}_{g,Pb}$, $\bar{n}_{0,Pb}$	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
$n_{g,Bi,i}$, $n_{0,Bi,i}$	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
$\bar{n}_{g,Bi}$, $\bar{n}_{0,Bi}$	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
$\bar{R}_g$, $\bar{R}_0$	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
$R_{g,Pb}$, $R_{0,Pb}$	Gross counting rate of the adsorbent test sample and of the blank, respectively, for ^{210}Pb, in per second
$R_{g,Bi}$, $R_{0,Bi}$	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
$\bar{\phi}_f$	Mean value of the free radon exhalation rate, in becquerel per second
$\mu(\bar{\phi}_f)$	Standard uncertainty of the free radon exhalation rate, in becquerel per second

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

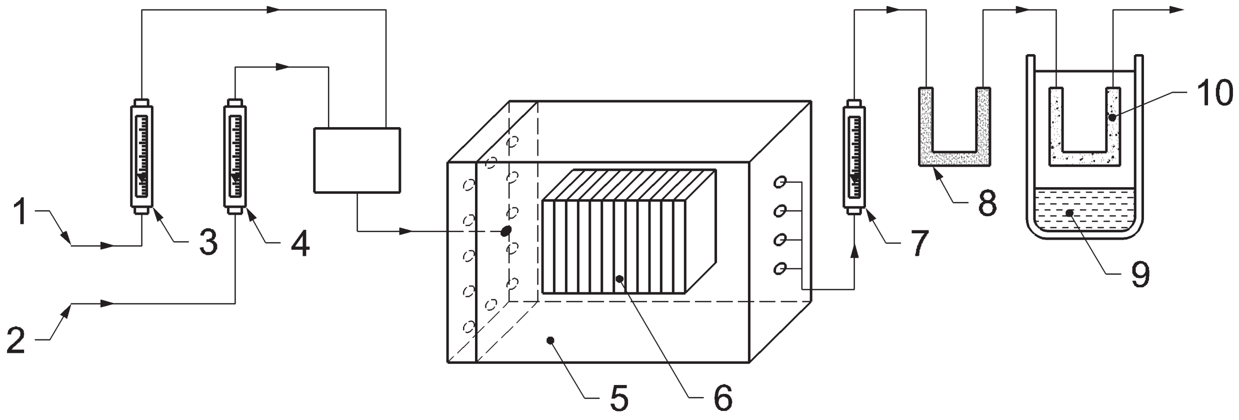
Set up the test bench as specified in Figure 1 with the components described in 5.3.

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.

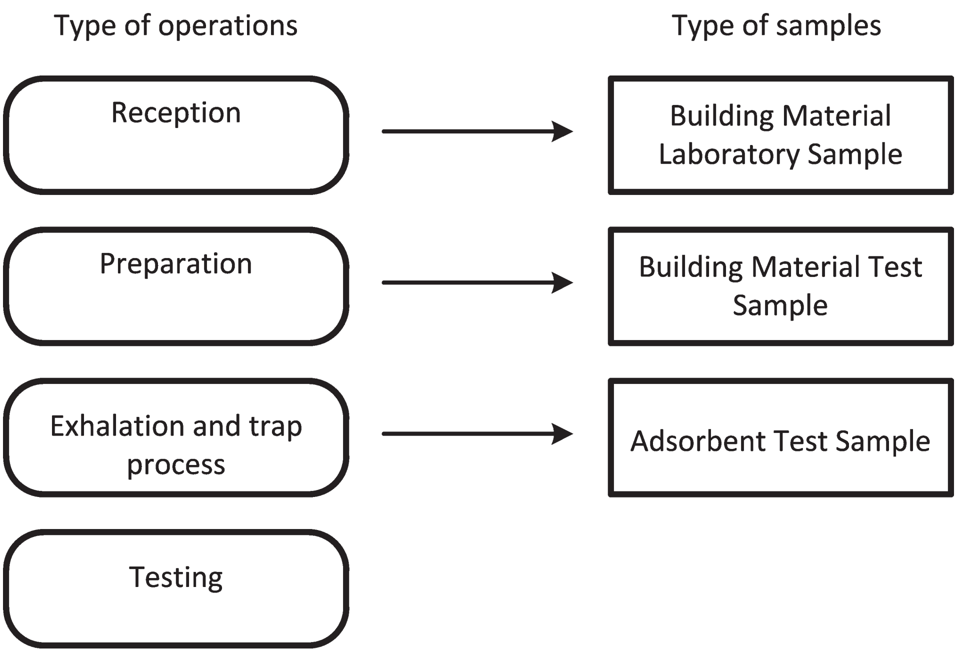


Figure 2 — Steps of measuring process with correspondent samples prepared