
Kakovost vode - Radon Rn-222 - 4. del: Preskusna metoda s štetjem z dvofaznim tekočinskim scintilatorjem (ISO 13164-4:2015)

Water quality - Radon-222 - Part 4: Test method using two-phase liquid scintillation counting (ISO 13164-4:2015)

Wasserbeschaffenheit - Radon-222 - Teil 4: Verfahren mittels zweistufiger Flüssigszintillationszählung

Qualité de l'eau - Radon 222 - Partie 4: Méthode d'essai par comptage des scintillations en Milieu liquide à deux phases (ISO 13164-4:2015)

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ISO 13164-4:2015(E)

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](#).

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

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

- *Part 1: General principles*
- *Part 2: Test method using gamma-ray spectrometry*
- *Part 3: Test method using emanometry*
- *Part 4: Test method using two-phase liquid scintillation counting*

Introduction

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

- Natural radionuclides, including potassium-40, and those originating from the thorium and uranium decay series, in particular radium-226, radium-228, uranium-234, uranium-238, and lead-210, can be found in water for natural reasons (e.g. desorption from the soil and wash-off by rain water) or can be released from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or phosphate fertilizer production and use).
- Human-made radionuclides such as transuranium elements (americium, plutonium, neptunium, curium), tritium, carbon-14, strontium-90, and some gamma-emitting radionuclides can also be found in natural waters as a result of authorized routine releases into the environment in small quantities in the effluent discharged from nuclear fuel cycle facilities. They are also released into the environment following their use in unsealed form in medicine or industrial applications. They are also found in the water as a result of past fallout resulting from explosion in the atmosphere of nuclear devices and accidents such as those that occurred in Chernobyl and Fukushima.

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

Standard test methods for radon-222 activity concentrations in water samples are needed by test laboratories carrying out such measurements in fulfilment of national authority requirements.

Laboratories may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

The radon activity concentration in surface water is very low, usually below 1 Bq l⁻¹. In groundwater, the activity concentration varies from 1 Bq l⁻¹ up to 50 Bq l⁻¹ in sedimentary rock aquifers, from 10 Bq l⁻¹ up to 300 Bq l⁻¹ in wells, and from 100 Bq l⁻¹ up to 1 000 Bq l⁻¹ in crystalline rocks. The highest activity concentrations are normally measured in rocks with high concentration of uranium (see Reference [9]).

High variations in the activity concentrations of radon in aquifers have been observed. Even in a region with relatively uniform rock types, some well water can exhibit radon activity concentration greatly higher than the average value for the same region. Significant seasonal variations have also been recorded (see ISO 13164-1:2013, Annex A).

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

Guidance on radon in drinking water supplies provided by WHO in 2008 suggests that controls should be implemented if the radon concentration of drinking water for public water supplies exceeds 100 Bq l⁻¹. It is also recommended that any new, especially public, drinking water supply using groundwater should be tested prior to being used for general consumption and that if the radon concentration exceeds 100 Bq l⁻¹, treatment of the water source should be undertaken to reduce the radon levels to well below that level (see Reference [10]).

This part of ISO 13164 is one of the series dealing with the measurement of the activity concentration of radionuclides in water samples.

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

Water quality — Radon-222 —

Part 4:

Test method using two-phase liquid scintillation counting

WARNING — Persons using this part of ISO 13164 should be familiar with normal laboratory practice. This part of ISO 13164 does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this part of ISO 13164 be carried out by suitably qualified staff.

1 Scope

This part of ISO 13164 describes a test method for the determination of radon-222 (^{222}Rn) activity concentration in non-saline waters by extraction and liquid scintillation counting.

The radon-222 activity concentrations, which can be measured by this test method utilizing currently available instruments, are at least above $0,5 \text{ Bq l}^{-1}$ for a 10 ml test sample and a measuring time of 1 h.

This test method can be used successfully with drinking water samples and it is the responsibility of the laboratory to ensure the validity of this test method for water samples of untested matrices.

[Annex A](#) gives indication on the necessary counting conditions to meet the required detection limits for drinking water monitoring.

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

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

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

3 Terms and definitions and symbols

3.1 Terms and definitions

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

3.2 Symbols and abbreviated terms

For the purposes of this document, symbols and abbreviations defined in ISO 80000-10, as well as the following symbols, apply.

a massic activity of the sample, in becquerels per gram

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a_S	massic activity of the standard solution at the measuring time, in becquerels per gram
a^*	decision threshold for the total massic activity, in becquerels per gram
$a^\#$	detection limit for the total massic activity, in becquerels per gram
$a^<, a^>$	lower and upper limits of the confidence interval, in becquerels per gram
c_A	activity concentration, in becquerels per litre
m	mass of the test sample, in grams
m_S	mass of standard solution used for the preparation of the counting standard, in grams
r_0	blank sample count rate, in reciprocal seconds
r_g	sample gross count rate, in reciprocal seconds
r_S	count rate of the standard in the counting window (alpha + beta), in reciprocal seconds
t_0	blank sample counting time, in seconds
t_g	test sample counting time, in seconds
t_S	calibration sample counting time, in seconds
$u(a)$	standard uncertainty associated with the measurement result; in becquerels per gram
U	expanded uncertainty, calculated using $U = ku(a)$, with $k = 2$, in becquerels per gram
w	coefficient equal to $1/(\epsilon m)$, in reciprocal gram
ϵ	total efficiency
ρ	density, in grams per litre

4 Principle

^{222}Rn is extracted from aqueous solution by means of a scintillation cocktail not miscible with water (without emulsifier) inside the scintillation vial and counted as the equilibrium with its short lived decay products is reached.[1] [2] [3] [4]

The aqueous sample is drawn by the mean of a gas-tight syringe from inside the water volume (i.e. well below surface) to avoid radon losses during sampling and transferred into a scintillation vial containing the desired amount of scintillation cocktail. For the same reason, the water sample is injected below the cocktail surface. The vial is tightly capped, shaken and kept for 3 h preferably in the dark and at controlled temperature. The sample is then counted by a liquid scintillation counter. Either total counts (alpha + beta) or alpha only counts are considered. In these conditions ^{222}Rn and its short lived progeny (^{218}Po , ^{214}Pb , ^{214}Bi , and ^{214}Po) are measured.

5 Reagents and apparatus**5.1 Reagents**

All reagents shall be of recognized analytical grade and, except for 5.1.4, shall not contain any detectable alpha and beta activity.

5.1.1 **Water**, distilled or deionized, complying with ISO 3696, grade 3.

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

5.1.2 **Scintillation cocktail**, commercially available scintillation cocktails, not water miscible.

5.1.3 **Ethanol**, 95 %.

5.1.4 **Radium standard solution**.

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

5.2 **Apparatus**

5.2.1 **Balance**.

5.2.2 **Wide-mouth glass sample bottles**, volume from 500 ml to 1 l.

5.2.3 **Wide-mouth Erlenmeyer flask**, volume from 500 ml to 1 l.

5.2.4 **Gas-tight syringe**.

5.2.5 **Liquid scintillation counter**, preferably with thermostated counting chamber and preferably ultra-low level counter to achieve better detection limits.

5.2.6 **Polyethylene scintillation vials**, PTFE coated, volume 20 ml.

5.2.7 **Glass scintillation vials**, low potassium glass, volume 20 ml.

NOTE PTFE coated polyethylene vials are the best choice since they prevent both the diffusion of the cocktail into the wall of the vial, radon loss and the absorption of radon from the external environment. Glass vials exhibit a considerably higher background due to potassium-40 content.

6 **Sampling**

6.1 **General**

Since radon is easily desorbed from water sample, care should be taken to avoid analyte losses during the sampling.

6.2 **Sampling with source preparation “on site”**

Attach a plastic tube to a faucet with a proper fitting. Insert the other end of the tube in a wide-mouth Erlenmeyer flask (5.2.3). Allow a steady water stream to get out and overflow the flask for approximately 2 min. Adjust the flux to avoid turbulence, bubbles, and empty volumes both in the tube and in the flask.

Draw the water sample aliquot with a gas-tight syringe (5.2.4) inserting the needle well below the surface.