



SLOVENSKI STANDARD
oSIST prEN ISO 13164-4:2022
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Kakovost vode - Radon Rn-222 - 4. del: Preskusna metoda s štetjem z dvofaznim tekočinskim scintilatorjem (ISO/DIS 13164-4:2022)

Water quality - Radon-222 - Part 4: Test method using two-phase liquid scintillation counting (ISO/DIS 13164-4:2022)

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Qualité de l'eau - Radon 222 - Partie 4: Méthode d'essai par comptage des scintillations en milieu liquide à deux phases (ISO/DIS 13164-4:2022)

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ICS:

13.060.60	Preiskava fizikalnih lastnosti vode	Examination of physical properties of water
17.240	Merjenje sevanja	Radiation measurements

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Water quality — Radon-222 —

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

Qualité de l'eau — Radon 222 —

Partie 4: Méthode d'essai par comptage des scintillations en milieu liquide à deux phases

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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms, definitions and symbols	1
3.1 Terms and definitions.....	1
3.2 Symbols.....	2
4 Principle	2
5 Reagents and apparatus	2
5.1 Reagents.....	2
5.2 Apparatus.....	3
6 Sampling	3
6.1 General.....	3
6.2 Sampling with source preparation “on site”.....	3
6.3 Sampling without “on site” source preparation.....	3
7 Instrument set up and calibration	4
7.1 Preparation of calibration sources.....	4
7.2 Optimization of counting conditions.....	4
7.3 Detection efficiency.....	4
7.4 Blank sample preparation and measurement.....	5
8 Sample preparation and measurement	5
9 Expression of results	6
9.1 Calculation of activity per unit of mass.....	6
9.2 Standard uncertainty.....	6
9.3 Decision threshold.....	6
9.4 Detection limit.....	7
9.5 Limits of the coverage intervals.....	7
9.5.1 Limits of the probabilistically symmetric coverage interval.....	7
9.5.2 The shortest coverage interval.....	8
9.6 Calculations using the activity concentration.....	8
10 Interference control	8
11 Quality control	8
12 Test report	8
Annex A (informative) Set-up parameters and validation data	10
Bibliography	14

ISO/DIS 13164-4:2022(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).

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

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

This second edition cancels and replaces the first edition (ISO 13164-4:2015), which has been technically revised.

The main changes are as follows:

— xxx xxxxxxxx xxx xxxx

A list of all the parts in the ISO 13164 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

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

- natural radionuclides, including ^{40}K , ^3H , ^{14}C , and those originating from the thorium and uranium decay series, in particular ^{226}Ra , ^{228}Ra , ^{234}U , ^{238}U , ^{210}Po and ^{210}Pb can be found in water for natural reasons (e.g. desorption from the soil and washoff by rain water) or can be released from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or phosphate fertilizers production and use);
- human-made radionuclides such as transuranium elements (americium, plutonium, neptunium, curium), ^3H , ^{14}C , ^{90}Sr , and gamma emitting radionuclides can also be found in natural waters. Small quantities of these radionuclides are discharged from nuclear fuel cycle facilities into the environment as a result of authorized routine releases. Some of these radionuclides used for medical and industrial applications are also released into the environment after use. Anthropogenic radionuclides are also found in waters as a result of past fallout contaminations resulting from the explosion in the atmosphere of nuclear devices and accidents such as those that occurred in Chernobyl and Fukushima.

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

The radionuclides present in liquid effluents are usually controlled before being discharged into the environment^[2] and water bodies. Drinking waters are monitored for their radioactivity as recommended by the World Health Organization (WHO)^[3] so that proper actions can be taken to ensure that there is no adverse health effect to the public. Following these international recommendations, national regulations usually specify radionuclide authorized concentration limits for liquid effluent discharged to the environment and radionuclide guidance levels for waterbodies and drinking waters for planned, existing and emergency exposure situations. Compliance with these limits can be assessed using measurement results with their associated uncertainties as specified by ISO/IEC Guide 98-3^[4] and ISO 5667-20^[5].

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

NOTE 1 The guidance level is the activity concentration with an intake of 2 l d⁻¹ of drinking water for one year that results in an effective dose of 0,1 mSv a⁻¹ for members of the public. This is an effective dose that represents a very low level of risk and which is not expected to give rise to any detectable adverse health effects^[3].

In the event of a nuclear emergency, the WHO Codex Guideline Levels^[6] mentioned that the activity concentration might not be greater than XBq l⁻¹ for ^{226}Ra .

NOTE 2 The Codex guidelines levels (GLs) apply to radionuclides contained in foods destined for human consumption and traded internationally, which have been contaminated following a nuclear or radiological emergency. These GLs apply to food after reconstitution or as prepared for consumption, i.e. not to dried or concentrated foods, and are based on an intervention exemption level of 1 mSv in a year for members of the public (infant and adult)^[6].

Thus, the test method can be adapted so that the characteristic limits, decision threshold, detection limit and uncertainties ensure that the radionuclide activity concentrations test results can be verified to be below the guidance levels required by a national authority for either planned/existing situations or for an emergency situation^{[7],[8]}.

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

ISO/DIS 13164-4:2022(E)

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

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

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

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^[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^[10]).

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.

Nevertheless, in circumstances where high radon concentrations might be expected in drinking-water, it is prudent to measure for radon and, if high concentrations are identified, consider whether measures to reduce the concentrations present are justified^[1].

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

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

Water quality — Radon-222 —

Part 4:

Test method using two-phase liquid scintillation counting

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety 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 document be carried out by suitably qualified staff.

1 Scope

This document 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.

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2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

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

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, definitions and symbols

3.1 Terms and definitions

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

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

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <https://www.electropedia.org/>

ISO/DIS 13164-4:2022(E)

3.2 Symbols

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

a	massic activity of the sample	$\text{Bq}\cdot\text{g}^{-1}$
a_S	massic activity of the standard solution at the measuring time	$\text{Bq}\cdot\text{g}^{-1}$
a^*	decision threshold for the total massic activity	$\text{Bq}\cdot\text{g}^{-1}$
$a^\#$	detection limit for the total massic activity	$\text{Bq}\cdot\text{g}^{-1}$
$a^<, a^>$	Lower and upper limits of the probabilistically symmetric coverage interval	$\text{Bq}\cdot\text{g}^{-1}$
$a^<, a^>$	Lower and upper limits of the shortest coverage interval	$\text{Bq}\cdot\text{g}^{-1}$
c_A	activity concentration	$\text{Bq}\cdot\text{l}^{-1}$
m	mass of the test sample	g
m_S	mass of standard solution used for the preparation of the counting standard	g
r_0	blank sample count rate	s^{-1}
r_g	sample gross count rate	s^{-1}
r_S	count rate of the standard in the counting window (alpha + beta)	s^{-1}
t_0	blank sample counting time	s
t_g	test sample counting time	s
t_S	calibration sample counting time	s
$u(a)$	standard uncertainty associated with the measurement result	$\text{Bq}\cdot\text{g}^{-1}$
U	expanded uncertainty, calculated using $U = ku(a)$, with $k = 2$	$\text{Bq}\cdot\text{g}^{-1}$
w	coefficient equal to $1/(\varepsilon m)$	g^{-1}
ε	total efficiency	
ρ	density	$\text{g}\cdot\text{l}^{-1}$

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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. [11] [12] [13] [14]

The aqueous sample is drawn with 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.

²²⁶Ra 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 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 ⁴⁰K content.

6 Sampling

6.1 General

Sampling, handling and storage of the water samples shall be done as specified in ISO 5667-1 and ISO 5667-3.

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 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. Sampling time shall be recorded to calculate decay correction.

6.3 Sampling without “on site” source preparation

Attach a plastic tube to a faucet with a proper fitting. Insert the other end of the tube in a wide-mouth borosilicate bottle (5.2.2). Allow a steady water stream to flow out and overflow the bottle for approximately 2 min. Adjust flux to avoid turbulence, bubbles, and empty volumes both in the tube and