SLOVENSKI STANDARD
oSIST prEN ISO 13165-2:2021

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Kakovost vode - Radij Ra-226 - 2. del: Preskusna metoda z emanometrijo (ISO/DIS 13165-2:2021)

Water quality - Radium-226 - Part 2: Test method using emanometry (ISO/DIS 13165-2:2021)

Wasserbeschaffenheit - Radium 226 - Teil 2: Verfahren mittels Emanometrie (ISO/DIS 13165-2:2021)

Qualité de l'eau - Radium 226 - Partie 2: Méthode d'essai par émanométrie (ISO/DIS 13165-2:2021)

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Part 2: **Test method using emanometry**

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

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*.

A list of all parts in the ISO 13165 series can be found on the ISO website.

This second edition cancels and replaces the first edition (ISO 13165-2:2014), which has been technically revised.

The main changes compared to the previous edition are as follows:

- new common introduction
- introduction of the shortest coverage interval in accordance with the new ISO 11929 series
- modification of the test report.

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, ^{3}H, ^{14}C, and those originating from the thorium and uranium decay series, in particular ^{226}Ra, ^{228}Ra, ^{234}U, ^{238}U 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), ^{3}H, ^{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[13] and ISO 5667-20[4].

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 guideline for guidance level in drinking water is 1 Bq/l for ^{226}Ra activity concentration.

NOTE 1 The guidance level is the activity concentration with an intake of 2 l/d of drinking water for one year that results in an effective dose of 0,1 mSv/a 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[5] mentioned that the activity concentrations might be greater.

NOTE 2 The Codex guidelines levels (GLs) apply to radionuclides contained in food 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)[5].

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[6][7].

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. 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 the ISO 5667 series).

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 — Radium-226 — Part 2: Test method using emanometry

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of any other restrictions.

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

1 Scope

This document specifies the determination of radium-226 (^{226}Ra) activity concentration in all types of water by emanometry.

The method specified is suitable for the determination of the soluble, suspended, and total ^{226}Ra activity concentration in all types of water with soluble ^{226}Ra activity concentrations greater than 0,02 Bq l^{-1}.

In water containing high activity concentrations of ^{228}Th, interference from ^{220}Rn decay products can lead to overestimation of measured levels (see Figure A.2).

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

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

ISO 11929-1, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 1: Elementary applications*

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 and the following apply.

3.1.1
reference measurement standard
measurement standard designated for the calibration of other measurement standards for quantities of a given kind in a given organization or at a given location

3.1.2
working measurement standard
measurement standard that is used routinely to calibrate or verify measuring instruments or measuring systems

Note 1 to entry: A working measurement standard can be used as a solution of known activity concentration obtained by precise dilution or dissolution of a reference standard.

3.2 Symbols

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

c_A	^{226}Ra activity concentration in water	$Bq.l^{-1}$
c_A^*	Decision threshold	$Bq.l^{-1}$
$c_A^\#$	Detection limit	$Bq.l^{-1}$
$c_A^\triangleleft, c_A^\triangleright$	Lower and upper limits of the probabilistically symmetric coverage interval	$Bq.l^{-1}$
$c_A^<, c_A^>$	Lower and upper limits of the shortest coverage interval	$Bq.l^{-1}$
f_a	Correction factor for ingrowth of ^{222}Rn in the bubbler	
f_d	Correction factor for the decay of ^{222}Rn in the detection volume	
n	Number of counting cycle	
n_α	Number of alpha-emitters present in the cell per becquerel of radon after a waiting time period between the filling time and the counting time of the cell (n_α is approximately 3 at a waiting time of 3 h for 1 Bq of radon)	
N_0	Number of background counts	
N	Number of gross counts	
t_c	Counting time (common to N, N_0)	s
t_i	Time of the different steps of the measurement procedure, i = 0,1 and 2	s
U	Expanded uncertainty calculated by $U = ku(c_A)$ with $k = 2$	$Bq.l^{-1}$
$u(c_A)$	Standard uncertainty associated with the measurement result	$Bq.l^{-1}$
V	Volume of the test sample, in litre	l
ε	total efficiency including degassing efficiency and counting efficiency of the system for a count carried out with a radioactive equilibrium between ^{222}Rn and its short-lived decay products, in pulses per second per becquerel	

λ	decay constant of the ^{222}Rn	s^{-1}

4 Principle

This test method is based on the emanation and scintillation counting of ^{222}Rn, a gaseous daughter product of ^{226}Ra, from a solution (see Reference [8]).

The measurement of ^{226}Ra activity concentration in water is carried out following two separate steps, the sample preparation followed by the measurement of the test sample.

Preparation consists of:

— dissolution when total or particulate radium is to be assayed;

— filtration when soluble radium is to be measured.

It is followed by pre-concentration, if necessary, and an accumulation of decay products without an initial separation.

After filtration and acidification, the test sample is placed in a bubbler (see Figure B.1) and stored for ingrowth of ^{222}Rn.

After a suitable ingrowth period, the radon gas is removed from the solution by purging with the radon-free gas and transferring it to a scintillation cell, whose internal surface is coated with silver-activated zinc sulfide, ZnS(Ag) (see Figure C.1)(see Reference [10]).

The alpha-particles produced by the decay of ^{222}Rn and its short-lived decay products (^{218}Po, ^{214}Po) transfer their energy as they pass through the scintillation medium. As they return to their ground state, the excitation electrons in the scintillation medium emit photons from the ZnS(Ag) coating that can be detected by a photomultiplier (PMT). The photomultiplier converts the photons into electrical pulses that are then counted. The pulse count is directly proportional to the activity concentration of radon and its decay products present in the scintillation cell.

The soluble ^{226}Ra activity concentration is calculated, taking into account the known steady state between ^{226}Ra and ^{222}Rn after transferring ^{222}Rn into a scintillation cell.

Given its high power of emanation, radon can also escape from particles suspended in water. In the case of the analysis of raw water, it is therefore advisable to dissolve the particulate fraction (see Reference [9]).

5 Reagents and equipment

5.1 Reagents

Unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity and no undesirable radioactivity.

5.1.1 Concentrated nitric acid solution, HNO_3.

5.1.2 Dilute nitric acid solution, less than or equal to 100 g l^{-1}, with no alpha-radioactivity.

5.1.3 Reference solution of ^{226}Ra.