SLOVENSKI STANDARD
oSIST prEN ISO 13165-1:2023

01-december-2023

Kakovost vode - Radij Ra-226 - 1. del: Preskusna metoda s štetjem s tekočinskim scintilatorjem (ISO 13165-1:2022)

Water quality - Radium-226 - Part 1: Test method using liquid scintillation counting (ISO 13165-1:2022)

Wasserbeschaffenheit - Radium-226 - Teil-1: Verfahren mit dem Flüssigszintillationszhler (ISO 13165-1:2022)

Qualité de l'eau - Radium-226 - Partie 1: Méthode d'essai par comptage des scintillations en milieu liquide (ISO 13165-1:2022)

Ta slovenski standard je istoveten z: **prEN ISO 13165-1**

ICS:

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

oSIST prEN ISO 13165-1:2023 **en,fr,de**

INTERNATIONAL STANDARD

ISO 13165-1

Second edition
2022-11

Water quality — Radium-226 —

Part 1: Test method using liquid scintillation counting

Qualité de l'eau — Radium-226 —

Partie 1: Méthode d'essai par comptage des scintillations en milieu liquide

Reference number
ISO 13165-1:2022(E)

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Published in Switzerland

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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).

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 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 13165-1:2013), which has been technically revised.

The main changes are as follows:

— the introduction has been updated;

— the list of symbols has been updated;

— the expression of results has been updated;

— the test report has been updated;

— the validation data has been updated;

A list of all parts in the ISO 13165 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 (such as 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, ^{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).

— Anthropogenic 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 installations during planned, existing and emergency exposure situations.[1] Drinking water can thus contain radionuclides at activity concentrations which can 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 content 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 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 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 (GL) 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[7].

In the event of a nuclear emergency, the WHO Codex Guideline Levels[5] mentioned that the activity concentration can be greater.

NOTE 2 The Codex 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)[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 can 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 answer the need of test laboratories carrying out these measurements, that are sometimes required by national authorities, as they can be required 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 1: Test method using 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 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 absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies the determination of radium-226 (^{226}Ra) activity concentration in non-saline water samples by extraction of its daughter radon-222 (^{222}Rn) and its measurement using liquid scintillation analysis.

The test method described in this document, using currently available scintillation counters, has a detection limit of approximately 50 mBq·l^{-1}. This method is not applicable to the measurement of other radium isotopes.

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 and sampling techniques*

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

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

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

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 80000-10, ISO/IEC Guide 98-3 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/

4 Symbols and units

For the purposes of this document, the symbols given in Table 1, ISO 80000-10 and ISO/IEC Guide 98-3 apply.

Table 1 — Symbols

Symbol	Description	Unit
a	Massic activity of the sample at the measuring time	$Bq \cdot g^{-1}$
a_S	Massic activity of the ^{226}Ra standard solution at the measuring time	$Bq \cdot g^{-1}$
a^*	Decision threshold for the massic alpha-activity	$Bq \cdot g^{-1}$
$a^\#$	Detection limit for the massic alpha-activity	$Bq \cdot g^{-1}$
$a^\triangleleft$, $a^\triangleright$	Lower and upper limits of the probabilistically symmetric coverage interval	$Bq \cdot g^{-1}$
$a^<$, $a^>$	Lower and upper limits of the shortest coverage interval	$Bq \cdot g^{-1}$
c	Concentration	$mol \cdot l^{-1}$
c_A	Activity concentration	$Bq \cdot l^{-1}$
k	Coverage factor	—
m	Mass of the test sample	g
m_1	Mass of initial sample subject to heating or possibly concentration	g
m_2	Mass of heated or concentrated sample	g
m_3	Mass of heated or concentrated sample transferred in the vial	g
m_S	Mass of ^{226}Ra standard solution used for the preparation of the calibration sample	g
p	Probability (for instance $p = 1 - \alpha$, $1- \beta$ or $1- \gamma /2$)	—
q	Probability	—
r_0	Blank sample count rate in the alpha-window	s^{-1}
r_g	Sample gross count rate in the alpha-window	s^{-1}
r_S	Count rate of the calibration sample in the alpha-window	s^{-1}
t_0	Blank sample counting time	s
t_g	Sample counting time	s
t_S	Calibration sample counting time	s
$u(a)$	Standard uncertainty associated with the measurement result	$Bq \cdot l^{-1}$
$\tilde{u}(\tilde{a})$	Standard uncertainty of a as a function of its true value	—
u_{rel}	Relative standard uncertainty	—
u_c	Combined uncertainty	—
U	Expanded uncertainty, calculated using $U = ku(a)$, with $k = 1, 2, \ldots$	$Bq \cdot l^{-1}$
w	Factor equal to $1/\varepsilon\, m$	—
ε	Alpha-efficiency, relative	—
ρ	Density	$g \cdot l^{-1}$
Φ	Distribution function of the standardized normal distribution	—
ω	Auxiliary quantity	—
γ	Coverage interval probability	—

5 Principle

The massic activity of ^{226}Ra is indirectly determined by isolating its progeny ^{222}Rn by liquid scintillation counting (LSC). ^{222}Rn is in secular equilibrium with its parent, ^{226}Ra, after 30 d (99,56 %). ^{222}Rn is extracted from the aqueous solution using a scintillation cocktail, within the scintillation vial, that is immiscible in water (see References [8], [9], [10] and [11]).