



**SLOVENSKI STANDARD**  
**oSIST prEN ISO 13165-3:2023**  
**01-maj-2023**

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**Kakovost vode - Radij Ra-226 - 3. del: Preskusna metoda s soobarjanjem in gama spektrometrijo (ISO/DIS 13165-3:2023)**

Water quality - Radium-226 - Part 3: Test method using coprecipitation and gamma-spectrometry (ISO/DIS 13165-3:2023)

Wasserbeschaffenheit - Radium-226 - Teil 3: Verfahren mittels Kopräzipitation und Gammaskpektrometrie (ISO/DIS 13165-3:2023)

Qualité de l'eau - Radium 226 - Partie 3: Méthode d'essai par co-précipitation et spectrométrie gamma (ISO/DIS 13165-3:2023)

**Ta slovenski standard je istoveten z: prEN ISO 13165-3**

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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 — Radium-226 —

### Part 3: Test method using coprecipitation and gamma- spectrometry

*Qualité de l'eau — Radium 226 —**Partie 3: Méthode d'essai par coprécipitation et spectrométrie gamma*

ICS: 17.240; 13.060.60

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## ISO/DIS 13165-3:2023(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](http://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](http://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](http://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 13165-3:2016), which has been technically revised.

The main changes are as follows:

- complete revision of the introduction
- the principal of measurement ([Clause 5](#)) was expanded
- the instrument verification (formerly [subclause 9.3](#)) was deleted
- formulae for the coverage intervals according to ISO 11929 series were updated
- requirements of the test report were 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](http://www.iso.org/members.html).

## Introduction

Radionuclides are present throughout the environment; thus, water bodies (e.g., surface waters, ground waters, sea waters) contain radionuclides, which can be of either natural or anthropogenic origin:

- naturally-occurring radionuclides, including  $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{40}\text{K}$  and those originating from the thorium and uranium decay series (see [Annex A](#)), in particular  $^{210}\text{Pb}$ ,  $^{210}\text{Po}$ ,  $^{222}\text{Rn}$ ,  $^{226}\text{Ra}$ ,  $^{228}\text{Ra}$ ,  $^{227}\text{Ac}$ ,  $^{231}\text{Pa}$ ,  $^{234}\text{U}$ , and  $^{238}\text{U}$ , can be found in water bodies due to either natural processes (e.g. desorption from the soil, runoff by rain water) or released from technological processes involving naturally occurring radioactive materials (e.g. mining, mineral processing, oil, gas, and coal production, water treatment and the production and use of phosphate fertilisers);
- anthropogenic radionuclides such as  $^{55}\text{Fe}$ ,  $^{59}\text{Ni}$ ,  $^{63}\text{Ni}$ ,  $^{90}\text{Sr}$ ,  $^{99}\text{Tc}$ , transuranic elements (e.g., Np, Pu, Am, and Cm), and some gamma emitting radionuclides such as  $^{60}\text{Co}$  and  $^{137}\text{Cs}$  can also be found in natural waters. Small quantities of anthropogenic radionuclides can be discharged from nuclear facilities to the environment as a result of authorized routine releases. The radionuclides present in liquid effluents are usually controlled before being discharged to the environment [\[1\]](#) and water bodies. Anthropogenic radionuclides used in medical and industrial applications can be released to the environment after use. Anthropogenic radionuclides are also found in waters due to contamination from fallout resulting from above-ground nuclear detonations and accidents such as those that have occurred at the Chernobyl and Fukushima nuclear facilities.

Radionuclide activity concentrations in water bodies can vary according to local geological characteristics and climatic conditions and can be locally and temporally enhanced by releases from nuclear facilities during planned, existing, and emergency exposure situations.[\[2\]\[3\]](#) Some drinking water sources can thus contain radionuclides at activity concentrations that could present a human health risk. The World Health Organization (WHO) recommends to routinely monitor radioactivity in drinking waters [\[4\]](#) and to take proper actions when needed to minimize the health risk.

National regulations usually specify the activity concentration limits that are authorized in drinking waters, water bodies, and liquid effluents to be discharged to the environment. These limits can vary for planned, existing, and emergency exposure situations. As an example, during either a planned or existing situation, the WHO guidance level for  $^{226}\text{Ra}$  in drinking water is  $1 \text{ Bq}\cdot\text{l}^{-1}$ , see NOTE. Compliance with these limits is assessed by measuring radioactivity in water samples and by comparing the results obtained, with their associated uncertainties, as specified by ISO/IEC Guide 98-3[\[5\]](#) and ISO 5667-20[\[6\]](#).

NOTE The guidance level calculated in Reference [\[4\]](#) is the activity concentration that, with an intake of  $2 \text{ l}\cdot\text{d}^{-1}$  of drinking water for one year, results in an effective dose of  $0,1 \text{ mSv}\cdot\text{a}^{-1}$  to members of the public. This is an effective dose that represents a very low level of risk to human health and which is not expected to give rise to any detectable adverse health effects[\[4\]](#).

This document contains method(s) to determine  $^{226}\text{Ra}$  in water samples. It has been developed to support laboratories that need either a certification or accreditation to determine  $^{226}\text{Ra}$  in water samples. A certification or accreditation are sometimes required by local and national authorities as well as some customers. The certification and accreditation are provided by an independent body

The method described in this document can be used for various types of waters (see [Clause 1](#)). Minor modifications such as sample volume and counting time can be made if needed to ensure that the characteristic limit, decision threshold, detection limit, and uncertainties are below the required limits. This can be done for several reasons such as emergency situations, lower national guidance limits, and operational requirements.

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 3:

# Test method using coprecipitation and gamma-spectrometry

**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 part of ISO 13165 specifies the determination of radium-226 ( $^{226}\text{Ra}$ ) activity concentration in all types of water by coprecipitation followed by gamma-spectrometry (see ISO 18589-3[Z]).

The method described is suitable for determination of soluble  $^{226}\text{Ra}$  activity concentrations greater than  $0,02 \text{ Bq l}^{-1}$  using a sample volume of up to 100 l of any water type.

For water samples smaller than a volume of 1 l, direct gamma-spectrometry can be performed following ISO 10703 with a higher detection limit.

**NOTE** This test method can be adapted to determine other naturally occurring isotopes of radium, such as  $^{223}\text{Ra}$ ,  $^{224}\text{Ra}$ , and  $^{228}\text{Ra}$  if the respective ingrowth periods are taken into account.

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

ISO 10703, *Water quality — Determination of the activity concentration of radionuclides — Method by high resolution gamma-ray spectrometry*

ISO 11929 (all parts), *Determination of the characteristic limits (decision threshold, detection limit and limits of the confidence interval) for measurements of ionizing radiation — Fundamentals and application*

ISO 12749 (all parts), *Nuclear energy — Vocabulary*

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*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 12749 and ISO 80000-10 and the following apply.

## ISO/DIS 13165-3:2023(E)

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/>

**3.1 activity**  
number of spontaneous nuclear disintegrations occurring in a given quantity of material during a suitably small interval of time divided by that interval of time

**3.2 reference standard**  
standard, generally having the highest metrological quality available at a given location or in a given organisation, from which measurements made there are derived

**3.3 working standard**  
standard which, usually calibrated against a *reference standard* (3.2), is used routinely to calibrate or check material measures, measuring instruments or reference materials. It can be used as a solution of known *activity* (3.1) concentration obtained by precise dilution or dissolution of a reference standard

## 4 Symbols

For the purposes of this document, the following symbols apply.

**Table 1 — List of symbols**

$V$	Volume of the test sample	l
$A$	Activity of each radionuclide in calibration source, at the calibration time	Bq
$c_A$	Activity concentration of $^{226}\text{Ra}$ , without and with corrections	$\text{Bq}\cdot\text{l}^{-1}$
$M_{\text{sulfate}}$	Mass of barium sulfate	g
$M_{\text{nitrate}}$	Mass of barium nitrate	g
$M_{\text{carbonate}}$	Mass of barium carbonate	g
$t_g$	Sample spectrum counting time	s
$t_0$	Background spectrum counting time	s
$n_{N,E}, n_{NO,E}$	Number of counts in the net area of the peak considered, at energy $E$ , in the sample, background, respectively	
$n_{g,E}, n_{g0,E}$	Number of counts in the gross area of the peak considered, at energy $E$ , in the sample, background, respectively	
$n_{b,E}, n_{b0,E}$	Number of counts in the background of the peak considered, at energy $E$ , in the sample, background, respectively	
$\varepsilon_E$	Detection efficiency at energy, $E$ , at actual measurement geometry	
$f_E$	Correction factor considering all necessary corrections	
$R_C$	Chemical yield	
$P_E$	Probability of the emission of a gamma-ray with energy $E$ of each radionuclide, per decay	
$\lambda$	Decay constant of each radionuclide	$\text{s}^{-1}$
$u(c_A)$	Standard uncertainty associated with the measurement result (without and with corrections) and with fitting efficiency curve respectively	$\text{Bq}\cdot\text{l}^{-1}$
$U$	Expanded uncertainty calculated with $k = 2$ .	$\text{Bq}\cdot\text{l}^{-1}$
$c_A^*$	Decision threshold, without and with corrections	$\text{Bq}\cdot\text{l}^{-1}$

Table 1 (continued)

$c_A^\#$	Detection limit, without and with corrections	Bq·l <sup>-1</sup>
$c_A^<, c_A^>$	Lower and upper limits of the probabilistically symmetric coverage interval	Bq·l <sup>-1</sup>
$c_A^{<}, c_A^{>}$	Lower and upper limits of the shortest coverage interval	Bq·l <sup>-1</sup>

## 5 Principle of the measurement

Measurement of <sup>226</sup>Ra activity concentration in water is carried out in two separate steps: a sample preparation step, followed by a measurement by gamma-spectrometry<sup>[8][9]</sup>.

After filtration and acidification of the water sample (see ISO 5667-3), to ensure the measurement of the activity concentration of the <sup>226</sup>Ra soluble fraction, the <sup>226</sup>Ra is coprecipitated as its sulfate salt using a barium carrier. The radium and barium sulfates are then washed, dried, and weighed. The chemical yield is obtained gravimetrically.

For water samples that may have an existing concentration of soluble barium, another tracer should be used to avoid a chemical yield above 100 %.

When a radiometric tracer, such as <sup>133</sup>Ba is used as an internal standard to assess the recovery yield, then this recovery yield is used instead of  $R_c$  in the [Formulae \(2\)](#) and [\(4\)](#).

The <sup>226</sup>Ra activity concentration of the precipitate is measured by gamma-spectrometry using a high purity germanium detector with low system background.

Secular equilibrium between radon and its progeny is a prerequisite when measuring <sup>226</sup>Ra using <sup>214</sup>Bi and <sup>214</sup>Pb gamma energies. The equilibrium is reached four weeks after the last coprecipitation step. The equilibrium condition necessitates the sample container to be air-tight.

The presence of other gamma emitters, such as <sup>235</sup>U, in the precipitate can interfere with the quantification of <sup>226</sup>Ra activity.

**NOTE** Since the coprecipitation described in this test method doesn't discriminate against specific radium isotopes, it can be adapted to determine other naturally occurring isotopes of radium, such as <sup>223</sup>Ra, <sup>224</sup>Ra, and <sup>228</sup>Ra if the respective ingrowth periods are taken into account. For this method, however, the equilibrium time of 4 weeks does not allow for determination of <sup>223</sup>Ra, <sup>224</sup>Ra or <sup>228</sup>Ra.

## 6 Reagents and equipment

### 6.1 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 (ISO 3696) [\[10\]](#).

**6.1.1 Concentrated nitric acid solution (HNO<sub>3</sub>), 14,5 – 15,5 mol·l<sup>-1</sup>.**

**6.1.2 Nitric acid solution (HNO<sub>3</sub>), diluted to 0,4 mol·l<sup>-1</sup>.**

**6.1.3 Nitric acid solution (HNO<sub>3</sub>), diluted to 3,2 mol·l<sup>-1</sup>.**

**6.1.4 Carrier solution, 10,0 g·l<sup>-1</sup>·Ba<sup>2+</sup>.**

Slowly dissolve barium nitrate Ba(NO<sub>3</sub>)<sub>2</sub> (19,0 g) or barium carbonate BaCO<sub>3</sub> (14,3 g) in one litre of nitric acid solution ([6.1.2](#)). The salt mass shall be weighed accurately (better than 1 %) and recorded.