
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 coprecipitation et spectrométrie gamma
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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 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 13165 consists of the following parts, under the general title *Water quality — Radium-226*:

- *Part 1: Test method using liquid scintillation counting*
- *Part 2: Test method using emanometry*
- *Part 3: Test method using coprecipitation and gamma-spectrometry*

Introduction

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

Natural radionuclides, including potassium-40, and those of 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 releases 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 gamma emitters radionuclides can also be found in natural waters as they can be authorized to be routinely released into the environment in small quantities in the effluent discharge from nuclear fuel cycle facilities and following their use in unsealed form in medicine or industry. They are also found in the water due to the past fallout of the explosion in the atmosphere of nuclear devices and those following the Chernobyl and Fukushima accident.

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 for medical and industrial applications. They are also found in the water as a result of past fallout contamination resulting from the 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 can be required by some national authorities.

The need of a standard on a test method of radium-226 activity concentrations in water samples is justified for test laboratories carrying out these measurements, required sometimes by national authorities, as they may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

Radium-226 activity concentration can vary widely according to local geological and climatic characteristics and ranges from 0,001 Bq·l⁻¹ in surface waters up to 50 Bq·l⁻¹ in natural groundwaters; the guidance level for radium 226 in drinking water as recommended by WHO is 1 Bq·l⁻¹ (see Reference [13]).

NOTE The guidance level is the activity concentration (rounded to the nearest order of magnitude) with an intake of 2 l·d⁻¹ of drinking water for 1 year that results in an effective dose of 0,1 mSv·y⁻¹ for members of the public, an effective dose that represents a very low level of risk that is not expected to give rise to any detectable adverse health effect.

This International Standard is one of a series on determination of the activity concentration of radionuclides in water samples.

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Water quality — Radium-226 —

Part 3:

Test method using coprecipitation and gamma-spectrometry

WARNING — Persons using this part of ISO 13165 should be familiar with normal laboratory practice. This part of ISO 13165 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 ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this part of ISO 13165 be carried out by suitably qualified staff.

1 Scope

This part of ISO 13165 specifies the determination of radium-226 (^{226}Ra) activity concentration in all types of water by coprecipitation followed by gamma-spectrometry (see ISO 18589-3).

The method described is suitable for determination of soluble ^{226}Ra activity concentrations greater than $0,02 \text{ Bq l}^{-1}$ using a sample volume of 1 l 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. [ISO 13165-3:2016](https://standards.iteh.ai/catalog/standards/sist/c2f7c0e1-f158-4fcb-9db2-c5c369bd8d76/iso-13165-3-2016)

NOTE This test method also allows other isotopes of radium, ^{223}Ra , ^{224}Ra , and ^{228}Ra , to be determined.

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

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 80000-10 and the following apply.

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

[SOURCE: ISO 921:1997, 23]

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 part of ISO 13165, the symbols defined in ISO 11929 and ISO 80000-10, and the following apply.

Table 1 — List of symbols

V	Volume of the test sample in litres
A	Activity of each radionuclide in calibration source, at the calibration time, in becquerels
C_A, C_{Ac}	Activity concentration of ^{226}Ra , without and with corrections, expressed in becquerels per litre
M_{sulfate}	Mass of barium sulfate, in grams
M_{nitrate}	Mass of barium nitrate, in grams
$M_{\text{carbonate}}$	Mass of barium carbonate, in grams
t_g	Sample spectrum counting time, in seconds
t_0	Background spectrum counting time, in seconds
t_s	Calibration spectrum counting time, in seconds
n_N, n_{N0}, n_{Ns}	Number of counts in the net area of the peak considered, at energy E , in the sample, background and calibration spectra, respectively
n_g, n_{g0}, n_{gs}	Number of counts in the gross area of the peak considered, at energy E , in the sample, background and calibration spectra, respectively
n_b, n_{b0}, n_{bs}	Number of counts in the background of the peak considered, at energy E , in the sample, background and calibration spectra, respectively
ε	Efficiency of the detector at energy E , at actual measurement geometry
R_C	Chemical yield
P	Probability of the emission of a gamma ray with energy, E , of each radionuclide, per decay, i.e. the branching ratio or the number of γ -quanta per decay
$u(C_A)$	Standard uncertainty of activity concentration, in becquerels per litre
$u(x)$	Standard uncertainty of measurand x
$u_{\text{rel}}(x)$	Relative uncertainty: $u(x)/x$
U	Expanded uncertainty calculated by $U = k u(C_A)$ with k usually equals 2, in becquerels per litre
C_A^*	Decision threshold, in becquerels per litre

Table 1 (continued)

$c_A^{\#}$	Detection limit, in becquerels per litre
$C_A^{\triangleleft}, C_A^{\triangleright}$	Lower and upper confidence limits, of each radionuclide, in becquerels per litre

5 Principle of the measurement

Measurement of ^{226}Ra activity concentration in water is carried out in two separate steps: a sample preparation step, followed by a measurement by gamma-spectrometry. [13][14][15]

The sample preparation consists of a filtration of the water sample (see ISO 5667-3) to ensure the measurement of the activity concentration of the ^{226}Ra soluble fraction. The filtration is followed by a pre-concentration, coprecipitation, and accumulation of decay products of ^{226}Ra .

After filtration and acidification of the water sample, the ^{226}Ra is coprecipitated into sulfate using a carrier such as barium nitrate or carbonate. 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 ^{133}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 ^{226}Ra activity concentration of the precipitate is measured by gamma-spectrometry using a high purity germanium detector with low system background.

The presence of other gamma emitters, such as ^{235}U , in the precipitate can interfere with the quantification of ^{226}Ra activity.

The assessment of the soluble ^{226}Ra activity concentration is carried out by the measurement of its decay products, ^{214}Pb and ^{214}Bi , at equilibrium reached four weeks after the last coprecipitation step.

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.

6.1.1 Concentrated nitric acid solution (HNO_3), 65 % or 69 %.

6.1.2 Nitric acid solution (HNO_3), diluted to 2,28 %.

6.1.3 Nitric acid solution (HNO_3), diluted to 18 %.

6.1.4 Carrier solution, $10,0 \text{ g}\cdot\text{l}^{-1}\cdot\text{Ba}^{2+}$.

Slowly dissolve barium nitrate $\text{Ba}(\text{NO}_3)_2$ (19,0 g) or barium carbonate BaCO_3 (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.

It is recommended to use a $\text{Ba}(\text{NO}_3)_2$ or BaCO_3 reagent with a low ^{226}Ra activity concentration. The presence of ^{226}Ra in the reagent shall be controlled using a blank sample prepared with distilled or demineralized water.

6.1.5 Concentrated sulfuric acid solution (H₂SO₄), 98 %.

6.1.6 Cellulose ester filter of porosity, 0,45 µm.

6.2 Equipment

Usual laboratory equipment and, in particular, the following:

6.2.1 Peristaltic or membrane pump.

6.2.2 Magnetic bar stirrer.

6.2.3 Centrifuge and tubes.

6.2.4 Precision balance.

6.2.5 Drying oven.

6.2.6 Gas-tight container.

6.2.7 Gamma-spectrometry system, with low background capability.

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7 Sampling

The method can be used on water samples up to 100 l.

[ISO 13165-3:2016](#)

7.1 Sample collection <https://standards.iteh.ai/catalog/standards/sist/c2f7c0e1-f158-4fcb-9db2-c5e569bd8d76/iso-13165-3-2016>

The sampling conditions shall comply with ISO 5667-3.

If required, the filtration is carried out during or immediately after collection and before acidification, otherwise, radioactive material already adsorbed onto the suspended particulate material can be desorbed.

It is recommended that plastic single-use type containers are used for sample collection.

The volume of the water sample to collect for measurement depends on the detection limits required by the customer.

7.2 Sample transport and storage

The water sample shall be transported and stored according to ISO 5667-3.

When pre-concentration is desired, acidify the sample to between pH 1 and pH 3 with HNO₃. Acidification of the water sample minimizes the loss of radioactive material from solution by adsorption.

The test shall be performed as soon as possible.

8 Procedures

8.1 Blank sample preparation

Prepare a blank with distilled or demineralized water by performing the operations described in step a) to i) in [8.2](#).

8.2 Sample preparation

- a) To prevent adsorption effects and proliferation of algae, the water sample is acidified to pH 1 with nitric acid (see 6.1.1). The water sample volume can be 1 l to 100 l. Acidification of the sample shall not be performed with hydrochloric acid as barium sulfate is partially soluble in it.
- b) Pour dropwise, precisely, 50 ml of carrier solution (see 6.1.4) into the solution to be analysed, with vigorous stirring. In the case of natural water rich in sulfate ions, such as seawater, the barium and radium have to be mixed before the sulfate is precipitated.
- c) Add 20 ml of concentrated H₂SO₄ (see 6.1.5) to precipitate the sulfate.
- d) Stop mixing.
- e) Leave the barium radium sulfate precipitate Ba(Ra)SO₄ to settle for about 24 h.
- f) Separate most of the supernatant, e.g. using the pump, and centrifuge the remainder at about 2 000 rpm for about 5 min or filter to recover the precipitate.
- g) Wash the precipitate with nitric acid (see 6.1.3) in the centrifuge tube, shake, and centrifuge again. Repeat steps f) and g) two times.
- h) Eliminate the supernatant.
- i) Rinse the precipitate with water (e.g. shaking and centrifuging each time).
- j) Dry the precipitate in an oven at 105 °C for at least 24 h.
- k) Weigh a gas-tight container.
- l) Place the precipitate in the weighed gas-tight container and tightly seal to prevent any loss of radon (see ISO 18589-3).
- m) Weigh the container.

8.3 Counting procedure

²²⁶Ra activity is assessed by gamma-spectrometry using the detection of emissions of ²¹⁴Pb and/or ²¹⁴Bi following an ingrowth period of approximately 4 weeks, during which, ²²²Rn reaches secular equilibrium with ²²⁶Ra (see ISO 18589-3).

Calibration shall be performed using the same geometry as the one used to count the sample precipitate.

In the case of counting before the equilibrium is reached, a correction for the subsequent accumulation of the decay products has to be applied.

The ²²⁶Ra activity concentration can be determined directly from its own energy emission at 186 keV, without the need for decay products ingrowth. The disadvantages of this technique relate primarily in that the emission probability of this gamma photopeak is weak (3,28 %), and furthermore by the presence of the interfering primary gamma emission of ²³⁵U at 185,7 eV that has a higher emission probability of 57,24 %. Assuming the equilibrium between ²²⁶Ra and ²¹⁴Bi, the theoretical detection limit of ²²⁶Ra can be improved using the energy line of ²¹⁴Bi at 609 keV to measure the ²²⁶Ra activity.

9 Quality assurance and quality control programme

9.1 General

Quality control operations shall meet the requirements of ISO/IEC 17025 and ISO 10703.