International Standard

ISO 13165-3

Water quality — Radium-226 —

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

Qualité de l'eau — Radium-226 —

Partie 3: Méthode d'essai par coprécipitation et spectrométrie gamma

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Contents

Page

Foreword

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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 document 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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This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 230, *Water analysis,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 13165-3:2016), which has been technically revised.

The main changes are as follows:

— the introduction has been completely revised;

— the principal of measurement in Clause 5 has been expanded;

— the instrument verification subclause (formerly 9.3) has been deleted;

— formulae for the coverage intervals according to ISO 11929 series have been updated;

— requirements of the test report have 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

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}H, ^{14}C, ^{40}K, and those originating from the thorium and uranium decay series (see Annex A), in particular ^{210}Pb, ^{210}Po, ^{222}Rn, ^{226}Ra, ^{228}Ra, ^{227}Ac, ^{231}Pa, ^{234}U, and ^{238}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}Fe, ^{59}Ni, ^{63}Ni, ^{90}Sr, ^{99}Tc, transuranic elements (e.g. Np, Pu, Am, Cm) and some gamma emitting radionuclides, such as ^{60}Co, ^{137}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 into 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 Chornobyl 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 can 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}Ra in drinking water is 1 Bq·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 l·d^{-1} of drinking water for one year, results in an effective dose of 0,1 mSv·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 support laboratories, which need to determine ^{226}Ra in water samples.

The method described in this document can be used for various types of waters (see Clause 1). Minor modifications to, for example, the sample volume and the 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 deals with the measurement of the activity concentration of radionuclides in water samples.

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Part 3:
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WARNING — Persons using this document should be familiar with normal laboratory practices. 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 are carried out by suitably trained staff.

1 Scope

This document specifies a method to determine radium-226 (^{226}Ra) activity concentration in all types of water by coprecipitation followed by gamma-ray spectrometry (see ISO 20042[7]).

The method covers the measurement of soluble ^{226}Ra activity concentrations greater than 0,002 Bq·l^{-1} using a sample volume of up to 100 l of any water type.

For water samples with a volume of less than a volume of 1 l, direct gamma-ray spectrometry can be performed following ISO 10703 but with a higher detection limit. The typical detection limit for samples of 1 l to 5 l is in the range of 0,002 to 0,000 40 Bq·l^{-1}[8].

NOTE   This test method can be adapted to determine other naturally occurring isotopes of radium, such as ^{223}Ra, ^{224}Ra and ^{228}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 — Gamma-ray emitting radionuclides — Test method using high resolution gamma-ray spectrometry*

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

ISO 12749-1, *Nuclear energy — Vocabulary — Part 1: General terminology*

ISO 12749-2, *Nuclear energy, nuclear technologies, and radiological protection — Vocabulary — Part 2: Radiological protection*

ISO 12749-3, *Nuclear energy, nuclear technologies, and radiological protection — Vocabulary — Part 3: Nuclear installations, processes and technologies*

ISO 12749-4, *Nuclear energy, nuclear technologies, and radiological protection — Vocabulary — Part 4: Dosimetry for radiation processing*

ISO 12749-5, *Nuclear energy, nuclear technologies, and radiological protection — Vocabulary — Part 5: Nuclear reactors*

ISO 12749-6, *Nuclear energy, nuclear technologies, and radiological protection — Vocabulary — Part 6: Nuclear medicine*

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 12749-1, ISO 12749-2, ISO 12749-3, ISO 12749-4, ISO 12749-5, ISO 12749-6, ISO 80000-10 and the following 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/

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

4 Symbols

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

A	Activity of each radionuclide in calibration source, at the time of calibration	Bq
c_A	Activity concentration of ^{226}Ra, without and with corrections	Bq·l^{-1}
c_A^*	Decision threshold, without and with corrections	Bq·l^{-1}
$c_A^\#$	Detection limit, without and with corrections	Bq·l^{-1}
$\tilde{c}_A$	Possible or assumed true quantity values of the measurand	Bq·l^{-1}
$c_A^\triangleleft$	Lower limit of the probabilistically symmetric coverage interval of the measurand	Bq·l^{-1}
$c_A^\triangleright$	Upper limit of the probabilistically symmetric coverage interval of the measurand	Bq·l^{-1}
$c_A^<$	Lower limit of the shortest coverage interval of the measurand	Bq·l^{-1}
$c_A^>$	Upper limit of the shortest coverage interval of the measurand	Bq·l^{-1}
ε_E	Detection efficiency at energy, E, at actual measurement geometry	—
f_E	Correction factor considering all necessary corrections	—
k_p	Quantiles of the standardised normal distribution for the probabilities p (for instance $p = 1 - \alpha$, $p = 1 - \beta$ or $p = 1 - \gamma / 2$)	—
k_q	Quantiles of the standardised normal distribution for the probabilities q (for instance $q = 1 - \alpha$, $q = 1 - \beta$ or $q = 1 - \gamma / 2$)	—

$m_{\text{carbonate}}$	Mass of barium carbonate	g
m_{nitrate}	Mass of barium nitrate	g
m_{sulfate}	Mass of barium sulfate	g
$n_{\text{b},E}$	Number of counts in the background of the peak considered, at energy E, in the sample	—
$n_{\text{b0},E}$	Number of counts in the background of the peak considered, at energy E, in the background	—
$n_{\text{bs},E}$	Number of counts in the background of the peak considered, at energy E, in the standard	—
$n_{\text{g},E}$	Number of counts in the gross area of the peak considered, at energy E, in the sample	—
$n_{\text{g0},E}$	Number of counts in the gross area of the peak considered, at energy E, in the background	—
$n_{\text{gs},E}$	Number of counts in the gross area of the peak considered, at energy E, in the standard	—
$n_{\text{N},E}$	Number of counts in the net area of the peak considered, at energy E, in the sample	—
$n_{\text{N0},E}$	Number of counts in the net area of the peak considered, at energy E, in the background	—
$n_{\text{Ns},E}$	Number of counts in the net area of the peak considered, at energy E, in the standard	—
P_E	Probability of the emission of a gamma-ray with energy E of each radionuclide, per decay	—
R_{c}	Chemical yield	—
t_0	Background spectrum counting time	s
t_{g}	Sample spectrum counting time	s
t_s	Standard spectrum counting time	s
U	Expanded uncertainty calculated with $k = 2$	Bq·l⁻¹
$u(c_A)$	Standard uncertainty associated with the measurement result	Bq·l⁻¹
$\tilde{u}(\tilde{c}_A)$	Standard uncertainty of the estimator c_A as a function of an assumed true value $\tilde{c}_A$ of the measurand	Bq·l⁻¹
$u(\text{fit})$	Standard uncertainty associated with fitting efficiency curve	Bq·l⁻¹
V	Volume of the test sample	l
Φ	Distribution function of the standardized normal distribution	—
w	Estimated value for the calibration factor	—
ω	Auxiliary quantity	—

5 Principle

Measurement of ²²⁶Ra activity concentration in water is carried out in two separate steps: a sample preparation step, followed by a measurement using gamma-ray spectrometry[9],[10].

After filtration and acidification of the water sample (see ISO 5667-3), to ensure that only the soluble fraction of the ²²⁶Ra is measured, the ²²⁶Ra is coprecipitated as its sulfate salt using a barium carrier. Radium and barium sulfates are then washed, dried and weighed. The chemical yield is obtained gravimetrically.

For water samples that can 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, the recovery yield is used instead of the chemical yield, R_c, in Formulae (2) and (5). A correction of counting losses due to coincidence is then required for the determination of ^{133}Ba.

The ^{226}Ra activity concentration of the precipitate is measured by gamma-ray spectrometry using a high purity germanium detector.

Secular equilibrium between ^{222}Rn and its progenies is a prerequisite when measuring ^{226}Ra using ^{214}Bi and ^{214}Pb gamma rays. The equilibrium is reached four weeks after the last coprecipitation step. The sample container shall be air-tight to achieve equilibrium conditions.

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

NOTE   Since the coprecipitation described in this test method does not allow for the determination of specific radium isotopes, it can be adapted to determine other naturally occurring isotopes of radium, such as ^{223}Ra, ^{224}Ra and ^{228}Ra, if the respective ingrowth periods are taken into account. For this method, however, the equilibrium time of four weeks does not allow for the determination of ^{223}Ra, ^{224}Ra or ^{228}Ra.

6   Reagents and equipment

6.1   Reagents

Unless otherwise stated, use only reagents of recognized analytical grade.

6.1.1   Distilled or demineralised water, with a resistivity of more than 18,2 MΩ·cm at 25 °C and total organic carbon less than 1 μg·l^{-1}.

6.1.2   Concentrated nitric acid solution, $c(HNO_3)$ = 14,5 mol·l^{-1} to 15,5 mol·l^{-1}.

6.1.3   Nitric acid solution, $c(HNO_3)$ = 0,4 mol·l^{-1}.

6.1.4   Nitric acid solution, $c(HNO_3)$ = 3,2 mol·l^{-1}.

6.1.5   Carrier solution, $c(Ba^{2+})$ = 10,0 g·l^{-1}.

Slowly dissolve barium nitrate $Ba(NO_3)_2$ (19,0 g) or barium carbonate $BaCO_3$ (14,3 g) in one litre of nitric acid solution (6.1.3). The salt mass shall be weighed accurately (better than 1 %) and recorded.

It is recommended to use a $Ba(NO_3)_2$ 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.

6.1.6   Concentrated sulfuric acid solution, $c(H_2SO_4)$ = 18,3 mol·l^{-1}.

6.1.7   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.