

DRAFT INTERNATIONAL STANDARD

ISO/DIS 13165-3

ISO/TC 147/SC 3

Secretariat: AFNOR

Voting begins on:
2014-06-16

Voting terminates on:
2014-09-16

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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Reference number
ISO/DIS 13165-3:2014(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 13165-3 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

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 co-precipitation 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 origin human-made origin:

- Natural radionuclides, including potassium 40, and those of the thorium and uranium decay series, in particular radium 226, radium 228, uranium 234, uranium 238, 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 some 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 discharged 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.

Drinking-water may 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 may be required by national authorities.

The need of a standard on a test method of radium 226 activity concentrations in water samples is justified for test laboratory 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 (Reference [11]).

NOTE The guidance level is the activity concentration (rounded to the nearest order of magnitude) with an intake of 2 l.day⁻¹ of drinking water for 1 year that results in an effective dose of 0,1 mSv.year⁻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 co-precipitation and gamma spectrometry

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 International Standard 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 gamma spectrometry.

The method described is suitable for determination of soluble ^{226}Ra activity concentrations greater than $0,02 \text{ Bq l}^{-1}$ in all types of water.

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

2 Normative references

The following referenced documents are indispensable for the application 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 921:1997, *Nuclear energy — Vocabulary*

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples*

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

ISO 10703: 2007, *Water quality — Determination of the activity concentration of radionuclides — Method by high resolution gamma-ray spectrometry*

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

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

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purpose of this document, the terms and definitions defined in the different parts of ISO 80000-10, and the following apply.

3.1.1 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]

3.1.2 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

3.1.3 working standard

standard which, usually calibrated against a reference standard, is used routinely to calibrate or check material measures, measuring instruments or reference materials. It 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 purpose of this document, 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 sulphate, 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 spectrum and in the background and calibration spectrum, respectively;
n_g, n_{g0}, n_{gs}	number of counts in the gross area of the peak considered, at energy E, in the spectrum and in the background and calibration spectrum, respectively;
n_b, n_{b0}, n_{bs}	number of counts in the background of the peak considered, at energy E, in the spectrum and in the background and calibration spectrum, 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 ;
$u_{rel}(x)$	relative uncertainty: $u(x)/x$;
U	expanded uncertainty calculated by $U = k u(c_A)$ with $k = 2$, in becquerels per litre;
c_A^*	decision threshold, in becquerels per litre;
$c_A^\#$	detection limit, in becquerels per litre;
$C_A^\triangleleft, C_A^\triangleright$	lower and upper confidence limits, of each radionuclide, in becquerels per litre.

4 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 [12][13][14].

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 can be followed by a pre-concentration, co-precipitation and accumulation of decay products of ^{226}Ra .

After filtration and acidification of the water sample, the ^{226}Ra is co-precipitated 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.

The ^{226}Ra activity concentration of the precipitate is measured by gamma spectrometry using a hyper pure germanium detector with low background.

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 co-precipitation step.

Note that the presence of other gamma emitters, such as ^{235}U , in the precipitate can interfere with the quantification of ^{214}Pb and ^{214}Bi activity.

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), 65 % or 69 %

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

5.1.3 Nitric acid solution (HNO_3) diluted to 18 %.

5.1.4 Carrier solution of $10 \text{ g l}^{-1} \text{ Ba}^{2+}$

Dissolve 19 g of barium nitrate $\text{Ba}(\text{NO}_3)_2$ or 14,3 g of barium carbonate BaCO_3 in one litre of solution (5.1.2).

It is recommended to use a $\text{Ba}(\text{NO}_3)_2$ or BaCO_3 reagent with a low ^{226}Ra activity concentration.

5.1.5 Concentrated sulfuric acid solution (H₂SO₄) (98 %)

5.1.6 Cellulose ester filter of porosity 0,45 μm

5.2 Equipment

Usual laboratory equipment and, in particular, the following

5.2.1 Peristaltic or membrane pump

5.2.2 Magnetic bar stirrer

5.2.3 Centrifuge and tubes

5.2.4 Precision balance

5.2.5 Drying oven

5.2.6 Gamma spectrometry system with low background capability

6 Sampling

The method can be used on water sample of several tens of litres.

6.1 Sample collection

The sampling conditions shall comply with ISO 5667-3.

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.

It is important that the laboratory receives a water sample container that has not been damaged to avoid the sample modification during its transport and storage.

6.2 Sample storage

If required, the water sample shall be 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 filtration is carried immediately on collection and before acidification otherwise radioactive material already adsorbed on to the suspended particulate material can be desorbed.

The analysis shall be performed as soon as possible.

7 Procedures

7.1 Sample preparation

- a) To prevent adsorption effects and proliferation of algae, the water sample is acidified to pH 1 with nitric acid (5.1.1). 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.