SLOVENSKI STANDARD
oSIST prEN ISO 22908:2018

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Kakovost vode - Radij Ra-226 in Ra-228 - Preskusna metoda štetja s tekočinskim scintilatorjem (ISO/DIS 22908:2018)

Water quality - Radium 226 and radium 228 - Test method using liquid scintillation counting (ISO/DIS 22908:2018)

asserbeschaffenheit - Radium 226 und Radium 228 - Verfahren mit dem Flüssigszintillationszähler (ISO/DIS 22908:2018)

Qualité de l'eau - Radium 226 et radium 228 - Méthode d'essai par comptage des scintillations en milieu liquide (ISO/DIS 22908:2018)

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Qualité de l'eau — Radium 226 et radium 228 — Méthode d'essai par comptage des scintillations en milieu liquide

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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).

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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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *radioactivity measurements*.

Introduction

Radioactivity from several naturally-occurring and anthropogenic sources is present throughout the environment. Thus, water bodies (e.g. 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);

— human-made 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 installation during planned, existing, and emergency exposure situations**[1]**. Drinking-water may thus contain radionuclides at activity concentrations which could 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 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 of the exposure situation, there are different limits and guidance levels that would result in an action to reduce health risk. As an example, during a planned or existing situation, the WHO guidelines for guidance level in drinking water are 1 Bq/l and 0,1 Bq/l, for ^{226}Ra and ^{228}Ra activity concentrations, respectively.

NOTE 1 The guidance level is the activity concentration with an intake of 2 l/d of drinking water for one year that results in an effective dose of 0,1 mSv/a 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**[3]**.

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

NOTE 2 The Codex guidelines levels (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**[5][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 could 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).

An International Standard on a test method of for ^{226}Ra and ^{228}Ra activity concentrations in water samples is justified for test laboratories carrying out these measurements, required sometimes by national authorities, as laboratories may have 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.

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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 ensure compliance with any national regulatory conditions.

IMPORTANT — It is essential that tests conducted according to this test method be carried out by suitably trained staff.

1 Scope

The document specifies the determination of radium-226 (^{226}Ra) and radium-228 (^{228}Ra) activity concentrations in drinking water samples by chemical separation of radium and its measurement using liquid scintillation counting. ^{226}Ra and ^{228}Ra are present in the environment as radionuclides from the ^{238}U and ^{232}Th decay series, as shown in Annex A.

The test method applies to the analysis of 0,5 kg of drinking water containing less than 100 mg/kg barium. If the barium concentration is higher than 100 mg/kg, it is recommended to reduce the volume of the test sample to be analysed so that the total content of barium in the sample does not exceed 50 mg.

Activity concentrations of ^{226}Ra and ^{228}Ra can vary widely according to local geological and climatic characteristics. ^{226}Ra activity concentration range from some mBq/l in surface waters up to several tens of Bq/l in some natural groundwaters[8]; the guidance level for ^{226}Ra in drinking water as recommended by WHO is 1 Bq/l[3][9]. ^{228}Ra activity concentration range from a few mBq/l in surface waters up to several Bq/l in some natural groundwaters[8]; the guidance level for ^{228}Ra in drinking water as recommended by WHO is 0,1 Bq/l[3][9].

Activity concentrations of ^{226}Ra and ^{228}Ra which can be measured by this test method utilizing currently available liquid scintillation counters goes down to 0,01 Bq/kg for ^{226}Ra and 0,06 Bq/kg for ^{228}Ra for a 0,5 kg sample mass and a 1 h counting time in a low background liquid scintillation counter.[10]

NOTE Adjustment of the test sample mass and counting time can lead to lower detection limits. As an example, a limit of detection of 0,04 Bq/kg can be achieved for ^{228}Ra using a 0,5 kg test sample and a 2 h counting time; similarly a limit of detection of 0,02 Bq/kg can be achieved for ^{228}Ra using a 1 kg test sample and a 2 h counting time.

The test method can be used for the fast detection of contamination of drinking water by radium in emergency situations or for routine environmental monitoring purposes.

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 8258, *Shewhart control charts*

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 17025:2017, *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 Symbols, definitions and units

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

u(x) uncertainty in quantity x.

A_x certified massic activity of the analyte in the certified standard solution at the reference date, in becquerels per kilogram

A_x^t massic activity of the analyte in the quality control sample at the reference date, in becquerels per kilogram

a_x massic activity of the analyte in the test sample at the sampling date, in becquerels per kilogram

a^* decision threshold of the analyte, in becquerels per kilogram

$a^\#$ detection limit of the analyte, in becquerels per kilogram

$a^\triangleleft$, $a^\triangleright$ lower and upper limits of the confidence interval, in becquerels per kilogram

c_a activity concentration of the analyte in the test sample at the sampling date, in becquerels per litre

C_x target massic activity of the analyte in the quality control sample prepared for the validation of the procedure, in becquerels per kilogram

$m_{s\text{-}x}$ mass of the certified standard solution taken for the analysis of the analyte, in kilograms

m_{t-x} mass of the quality control sample taken for the analysis of the analyte, in kilograms

m_s mass of the test sample, in kilograms

n_x^s net count rates of the analyte in the certified standard solution, in reciprocal seconds

n_x^t net count rates of the analyte in the quality control sample, in reciprocal seconds

n_x net count rates of the analyte in the test sample, in reciprocal seconds

PI precision index, in per cent

R_L reproducibility limit, in becquerels per kilogram

r_L repeatability limit, in becquerels per kilogram

r_{g-x} gross count rate of the analyte in the test sample, in reciprocal seconds

$r_{0\text{-}x}$ gross count rate of the analyte in the blank sample, in reciprocal seconds

S_r standard deviation of repeatability, in becquerels per kilogram

S_R standard deviation of reproducibility, in becquerels per kilogram

$T_{s\text{-}x}$ counting time of the analyte in the test sample, in seconds

$t_{0\text{-}x}$ counting time of the analyte in the blank, in seconds

$t_{s\text{-}x}$ time interval between measurement date and reference date of the analyte in the certified standard solution, in seconds

$t_{t\text{-}x}$ time interval between measurement date and reference date of the analyte in the quality control sample, in seconds

t_x time interval between measurement date and sampling date of the analyte in the test sample, in seconds

$u(a)$ standard uncertainty associated with the measurement result; in becquerels per kilogram

U expanded uncertainty, calculated using $U = ku(a)$, with $k = 1, 2, \ldots$ in becquerels per kilogram

w factor equal to $1/\varepsilon_x m_s$

ε_x counting efficiency of the analyte

ε_x^c overall efficiency of the analyte in the quality control sample

λ_x decay constant of the analyte, in reciprocal seconds

$\overline{X}_x$ mean of all measured values of the analyte in the quality control sample for the validation of the procedure, in becquerels per kilogram

δ relative bias of the method, in per cent

ρ density, in kilograms per litre

4 Principle

Barium co-precipitation is used as a method of separation for radium due to the very similar chemical properties of barium and radium. The exploitation of the ability of barium to react with an excess of sulfate ions to produce a precipitate allows the quantitative analysis of environmental activity concentrations of radium in water. The inclusion of a lead carrier allows the removal of ^{210}Pb from solution, which increases the accuracy of ^{228}Ra measurement, as ^{210}Pb can produce a spectral interference. The removal of ^{210}Pb is achieved by lowering the pH of the solution to re-precipitate barium sulfate using acetic acid in which lead sulphate is soluble. This allows ^{210}Pb to remain in solution and therefore be removed.

The source preparation is achieved by suspending the barium sulfate precipitate in the EDTA solution. Barium sulfate is insoluble in water, alkalis and acids, but EDTA increases the solubility due to the complexation of barium and the speciation effect. The EDTA molecule inhibits barium sulfate nucleation. This enables the use of a naphthalene-based scintillation cocktail to gain better spectral resolution than with the use of a gel-forming cocktail.

The flow chart of the procedure is given in Annex B.

Massic activities of ^{226}Ra and ^{228}Ra in the sample are calculated from net count rates of the sample, sample amount and the overall efficiency that can be obtained from spiked sample with known activities of ^{226}Ra and ^{228}Ra, and that shows the ability of the method to extract radium (chemical recovery) as well as the ability (counting efficiency) of the instrument to detect it.

5 Reagents and equipment

5.1 Reagents

All reagents shall be of recognized analytical grade and, except for 5.1.12, 5.1.13 and 5.1.14, shall not contain any detectable alpha- and beta-activity.

5.1.1 Laboratory water, distilled or deionized, complying with ISO 3696, grade 3.

5.1.2 Lead carrier solution prepared using 2,397 g lead nitrate, 0,5 ml nitric acid solution (5.1.4) and made up to 100 ml with laboratory water (5.1.1).

5.1.3 Barium carrier solution prepared using 2,836 g barium chloride, 0,5 ml nitric acid solution (5.1.4) and made up to 100 ml laboratory water (5.1.1).

5.1.4 Nitric acid solution, $c(HNO_3)$ = 15,8 mol/l, ρ = 1,42 g/ml, mass fraction $w(HNO_3)$ = 70 %.

5.1.5 Hydrochloric acid solution, $c(HCl)$ = 10,2 mol/l, ρ = 1,16 g/ml, mass fraction $w(HCl)$ = 32 %.

5.1.6 Sulfuric acid solution, $c(H_2SO_4)$ =9,2 mol/l, ρ = 1,84 g/ml, mass fraction $w(H_2SO_4)$ = 98 %.

5.1.7 Ammonia solution, $c(NH_3)$ = 13,4 mol/l, ρ = 0,91 g/ml, mass fraction $w(NH_3)$ = 25 %.

5.1.8 Glacial acetic acid solution, $c(CH_3COOH)$ = 16,8 mol/l, ρ = 1,05 g/ml, mass fraction $w(CH_3COOH)$ = 96 %.

5.1.9 Ethylenediaminetetraacetic acid (EDTA), M(EDTA) = 292,2 g/mol.

NOTE Within the whole document, an EDTA solution warmed up within the 60-80°C temperature range is considered as a hot EDTA solution.

5.1.10 Analytical grade ammonium sulphate, $M((NH_4)_2SO_4)$ = 132,1 g/mol.

5.1.11 Scintillation cocktail, commercially available scintillation cocktails, water immiscible and suitable for alpha and beta discrimination (e.g. diisopropylnaphthalene-based cocktails).

5.1.12 ^{226}Ra and ^{228}Ra standard solutions

^{226}Ra and ^{228}Ra standard solutions shall be provided with calibration certificates containing at least the activity concentration, measurement uncertainty and/or statement of compliance with an identified metrological specification.

5.1.13 Alpha emitter standard solution (^{241}Am or ^{210}Po or ^{242}Pu)

The alpha emitter standard solution shall be provided with calibration certificate containing at least the activity concentration, measurement uncertainty and/or statement of compliance with an identified metrological specification.

5.1.14 Beta emitter standard solution (^{90}Sr/^{90}Y or ^{36}Cl)

The beta emitter standard solution shall be provided with calibration certificate containing at least the activity concentration, measurement uncertainty and/or statement of compliance with an identified metrological specification.

