Kakovost vode - Skupna beta aktivnost - Preskusna metoda robustnega vira (ISO 9697:2018)

Water quality - Gross beta activity - Test method using thick source (ISO 9697:2018)

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Qualité de l'eau - Activité bêta globale - Méthode d'essai par source épaisse (ISO 9697:2018)

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Water quality — Gross beta activity — Test method using thick source

Qualité de l'eau — Activité bêta globale — Méthode d'essai par source épaisse

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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 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.

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

This fourth edition cancels and replaces the third edition (ISO 9697:2015), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— the title has been changed from "Gross beta activity in non-saline water" to "Gross beta activity";

— the Introduction has been reworded;

— Formulae (10) and (11) have been corrected to replace ± by α in the index of r;

— the units have been corrected so that mm^2 and mol/l are used throughout.

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

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 on 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 is 0,5 Bq/l for gross alpha activity and 1 Bq/l for gross beta activity.

NOTE   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].

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.

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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 gross alpha and gross beta 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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Water quality — Gross beta activity — Test method using thick source

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably trained staff.

1 Scope

This document specifies a test method for the determination of gross beta activity concentration in non-saline waters. The method covers non-volatile radionuclides with maximum beta energies of approximately 0,3 MeV or higher. Measurement of low energy beta emitters (e.g. ^{3}H, ^{228}Ra, ^{210}Pb, ^{14}C, ^{35}S and ^{241}Pu) and some gaseous or volatile radionuclides (e.g. radon and radioiodine) might not be included in the gross beta quantification using the test method described in this document.

This test method is applicable to the analysis of raw and drinking waters. The range of application depends on the amount of total soluble salts in the water and on the performance characteristics (background count rate and counting efficiency) of the counter used.

It is the laboratory's responsibility to ensure the suitability of this method for the water samples tested.

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 3696:1987, *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 5667-14, *Water quality — Sampling — Part 14: Guidance on quality assurance and quality control of environmental water sampling and handling*

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

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

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

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3   Terms, definitions, symbols and units

No terms and definitions are listed in this document.

For the purposes of this document, the symbols and designations given in ISO 80000-10, ISO 11929, ISO/IEC Guide 98-3, ISO/IEC Guide 99 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

A	beta activity, in becquerels, of the calibration source
c_A	beta activity concentration, in becquerels per litre
c_A^*	decision threshold, in becquerels per litre
$c_A^\#$	detection limit, in becquerels per litre
$c_A^<, c_A^>$	lower and upper limits of the confidence interval, in becquerels per litre
m	mass, in milligrams, of ignited residue from volume, V
m_r	mass, in milligrams, of the sample residue deposited on the planchet
r_0	background count rate, per second
$r_{0\alpha}$	background count rate, per second, from the alpha window
r_g	sample gross count rate, per second
$r_{g\alpha}$	sample gross count rate, per second, from the alpha window
r_s	calibration count rate of the beta source, per second
$r_{s\alpha}$	calibration count rate of the alpha source, per second, from the alpha window
S	surface area, in square millimetres, of the planchet
t_0	background counting time, in seconds
t_g	sample counting time, in seconds
t_s	calibration count time of the beta source, in seconds
$t_{s\alpha}$	calibration count time of the alpha source, in seconds
$u(c_A)$	standard uncertainty, in becquerels per litre, associated with the measurement result
U	expanded uncertainty, in becquerels per litre, calculated from $U = ku(c_A)$, with k = 1, 2 …
V	volume, in litres, of test sample equivalent to the mass of solid on the planchet
V_t	volume, in litres, of the water sample

ε	counting efficiency for the specified radioactive standard
ρ_s	source thickness, in milligrams per square millimetre, of the sample residue deposited on the planchet
χ	alpha-beta cross-talk, percentage of alpha count going into the beta window from the alpha calibration source

4 Principle

Gross beta measurement is not intended to give an absolute determination of the activity concentration of all beta-emitting radionuclides in a test sample, but rather a screening analysis to ensure particular reference levels of specific beta emitters have not been exceeded. This type of determination is also known as gross beta index. Gross beta analysis is not expected to be as accurate nor as precise as specific radionuclide analysis after radiochemical separations.

The sample, taken, handled and preserved as specified in ISO 5667-1, ISO 5667-3 and ISO 5667-14, is evaporated to almost dryness, converted to the sulfate form, and ignited at 350 °C. A portion of the residue is transferred onto a planchet and the beta activity measured by counting in an appropriate counting assembly, which is calibrated against a suitable beta calibration source, such as potassium-40 (^{40}K) or strontium-90/yttrium-90 (^{90}Sr + ^{90}Y) in equilibrium.

If simultaneous gross alpha and beta measurements are required on the same water sample, the procedure specified in this document is common to that of ISO 9696[8]. However, to simultaneously measure gross alpha activity, the counting source thickness should be below 0,1 mg/ mm2[9][10].

A performance criteria example is given in Annex A.

5 Reagents and equipment

5.1 Reagents

All reagents shall be of recognized analytical grade and shall not contain any detectable beta activity.

NOTE   A method for preparing reagent blanks to check for the absence of any endemic beta radioactivity or contamination is given in Clause 7.

5.1.1   Water, conforming to ISO 3696:1987, grade 3.

5.1.2   Calibration source, the choice of beta calibration source depends on the knowledge of the type of radioactive contaminant likely to be present in the waters being tested. Among calibration source of beta-emitting radionuclides, ^{90}Sr and ^{40}K are commonly used.

NOTE   The beta activity of ^{40}K in natural potassium is 27,9 Bq g^{-1}, i.e. 14,5 Bq g^{-1} in potassium chloride[3].

5.1.3   Nitric acid, $c(HNO_3)$ = 8 mol/l.

5.1.4   Sulfuric acid, $c(H_2SO_4)$ = 18 mol/l, ρ = 1,84 g/ml, mass fraction $w(H_2SO_4)$ = 95 %.

5.1.5   Volatile organic solvents, methanol or acetone.

5.1.6   Calcium sulfate, $CaSO_4$.

5.1.7   Vinyl acetate, $[(C_4H_6O_2)n]$.