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Water quality — Gamma-ray emitting radionuclides — Test method using high resolution gamma-ray spectrometry

Qualité de l'eau — Radionucléides émetteurs gamma — Méthode d'essai par spectrométrie gamma à haute résolution

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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 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*, 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 third edition cancels and replaces the second edition (ISO 10703:2007), which has been technically revised.

The main changes compared to the previous edition are as follows:

— new common Introduction;

— Scope enlarged to emergency situation and to wastewater, upper dead time increase to 10 %;

— the sample storage conditions are in compliance with ISO 5667-3 (see Clause 10);

— modification of the reference source for calibration (see 6.2);

— calibration efficiency determination by Monte Carlo method (see 11.2.3);

— complete revision of the pulse pile up and dead time;

— complete revision of the true coincidence summing subclause (see 12.1.4);

— addition of the correction factor for dead time and pile up (see 12.1.2);

— introduction of the shortest coverage interval in accordance with the new ISO 11929 series (see 12.5.2);

— modification of the test report (see Clause 13).

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.

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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, 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 fertilizer 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 the 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[7]. 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[8]. Water bodies and drinking waters are monitored for their radioactivity content as recommended by the World Health Organization (WHO)[9] 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 water bodies 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.

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 10 Bq·l^{-1} for $^{134/137}$Cs and ^{131}I activity concentration, 1 Bq·l^{-1} for ^{241}Am and 0,1 Bq·l^{-1} for ^{210}Pb.

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[9].

In the event of a nuclear emergency, the WHO Codex guideline levels[10] mentioned that the activity concentration for infant food might not be greater than 1 000 Bq·kg^{-1} for $^{134/137}$Cs, 100 Bq·kg^{-1} for ^{131}I and 1 Bq·kg^{-1} for ^{241}Am. For food other than infant food, the activity concentration might not be greater than 1 000 Bq·kg^{-1} for $^{134/137}$Cs, 100 Bq·kg^{-1} for ^{131}I and 10 Bq·kg^{-1} for ^{241}Am.

NOTE 2 The Codex guidelines levels (GLs) apply to radionuclides contained in food 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 food, and are based on an intervention exemption level of 1 mSv in a year for members of the public (infant and adult)[10].

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[11][12].

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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 discharge 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 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 may be used for water samples after proper sampling, sample handling, and test sample preparation (see the relevant part of the ISO 5667 series).

This document has been developed to answer the need of test laboratories carrying out these measurements, that are sometimes required by national authorities, as they 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 — Gamma-ray emitting radionuclides — Test method using high resolution gamma-ray spectrometry

WARNING — Persons using this document should be familiar with normal laboratory practice. 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 be carried out by suitably trained staff.

1 Scope

This document specifies a method for the physical pre-treatment and conditioning of water samples and the determination of the activity concentration of various radionuclides emitting gamma-rays with energies between 40 keV and 2 MeV, by gamma-ray spectrometry according to the generic test method described in ISO 20042.

The method is applicable to test samples of drinking water, rainwater, surface and ground water as well as cooling water, industrial water, domestic and industrial wastewater after proper sampling, sample handling, and test sample preparation (filtration when necessary and taking into account the amount of dissolved material in the water). This method is only applicable to homogeneous samples or samples which are homogeneous via timely filtration.

The lowest limit that can be measured without concentration of the sample or by using only passive shield of the detection system is about $5 \cdot 10^{-2}$ Bq/l for e.g. ^{137}Cs[1)]. The upper limit of the activity corresponds to a dead time of 10 %. Higher dead times may be used but evidence of the accuracy of the dead-time correction is required.

Depending on different factors, such as the energy of the gamma-rays, the emission probability per nuclear disintegration, the size and geometry of the sample and the detector, the shielding, the counting time and other experimental parameters, the sample may require to be concentrated by evaporation if activities below $5 \cdot 10^{-2}$ Bq/l need to be measured. However, volatile radionuclides (e.g. radon and radioiodine) can be lost during the source preparation.

This method is suitable for application in emergency situations.

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 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste water*

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

1) The sample geometry: 3l Marinelli beaker; detector: GE HP N relative efficiency 55 % ; counting time: 18h.

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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.

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

3.1
blank sample
container of an identical composition to the one used for the water test sample, filled with demineralized water

3.2
dead time
time during spectrum acquisition (real time) during which pulses are not recorded or processed

Note 1 to entry: Dead time is expressed in percent, calculated as follows: real time minus live time, all divided by real time then multiplied by 100

3.3
dead time correction
correction to be applied to the observed number of pulses to take into account the number of pulses lost during the *dead time* (3.2)

3.4
decay constant
λ
quotient of the rate at which a population of radioactive atoms decreases because of radioactive decay by the size of that population of radioactive atoms

Note 1 to entry: It can also be expressed as the quotient of dP by dt,

$$\lambda = \frac{\mathrm{d}P}{\mathrm{d}t} = -\frac{1}{N}\frac{\mathrm{d}N}{\mathrm{d}t}$$

where

dP is the probability of a given nucleus undergoing a spontaneous nuclear transition from that energy state in the time interval dt;

N is the number of nuclei of concern existing at time t.

Note 2 to entry: The time required for half of the original population of radioactive atoms to decay is called the half-life. The relationship between the half-life, $T1/2$, and the decay constant is given by $T1/2 = \ln(2)/\lambda$.

3.5
efficiency
ratio of the number of gamma photons detected in the full energy peak to the number of the same type emitted by the radiation source in the same time interval, under stated conditions of detection

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3.6
energy resolution
measure, at a given energy, of the smallest difference between the energy of two gamma-rays which can be distinguished by the apparatus used for *gamma-ray spectrometry* (3.10)

Note 1 to entry: The energy resolution is measured as the full width at half maximum height of the energy peak (FWHM).

3.7
full energy peak
peak of spectral response corresponding to the total absorption of the photon energy in the detector by the photoelectric effect or by consecutive photon interactions of pair production (only for photon energy >1 022 keV), Compton scattering and photoelectric absorption

3.8
gamma cascade
two or more different gamma-photons emitted successively from one nucleus when it de-excites through one or more intermediate energy levels

3.9
gamma radiation
electromagnetic radiation emitted in the process of nuclear transition or particle annihilation

3.10
gamma-ray spectrometry
method of measuring gamma-rays yielding the energy spectrum of the *gamma radiation* (3.9)

3.11
pile-up
processing by a radiation spectrometer of pulses resulting from the simultaneous absorption of particles, or photons, originating from different decaying nuclei, in the radiation detector

Note 1 to entry: As a result, pulses are counted as one single particle or photon with an energy between the individual energies and the sum of these energies.

3.12
transition probability
probability of de-excitation of the nucleus occurring by one specific transition at a given energy state to a less energetic state or to the ground state

4 Symbols

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

V	Volume of the water sample for test	l
A	Activity of each radionuclide in calibration source, at the calibration time	Bq
c_A , $c_{A,c}$	Activity concentration of each radionuclide, without and with corrections	$Bq \cdot l^{-1}$
t_g	Test sample spectrum counting time	s
t_0	Background spectrum counting time	s
t_i	Time between the reference time and the start of the measuring time	s
t_S	Calibration spectrum counting time	s
$n_{N,E}$, $n_{N0,E}$, $n_{Ns,E}$	Number of counts in the net area of the peak, at energy E, in the test sample spectrum, in the background spectrum and in the calibration spectrum, respectively	

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