SLOVENSKI STANDARD
oSIST prEN ISO 11704:2017

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Kakovost vode - Merjenje skupne alfa in skupne beta aktivnosti - Metoda štetja s tekočinskim scintilatorjem

Water quality - Gross alpha and gross beta activity - Test method using liquid scintillation counting

Wasserbeschaffenheit - Bestimmung der Gesamt-Alpha- und Gesamt-Beta-Aktivität in nicht-salzhaltigem Wasser - Verfahren mit dem Flüssigszintillationszähler

Qualité de l'eau - Activités alpha globale et bêta globale - Méthode d'essai par comptage des scintillations en milieu liquide

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Qualité de l'eau — Activités alpha globale et bêta globale — 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.

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

This second edition cancels and replaces the first edition (2010), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

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 runoff 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);
- anthropogenic radionuclides such as transuranium elements (e.g. americium, plutonium, neptunium, and curium), ^{3}H, ^{14}C, ^{90}Sr, and some gamma emitting radionuclides can also be found in natural waters. Small quantities of these radionuclides may be 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 may also be released into the environment after use. Anthropogenic radionuclides are also found in waters as a result of past fallout contaminations resulting from the above ground detonation 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] Such control and monitoring can enable to take proper actions to ensure that there is no adverse health effects to the public. Following these international recommendations, radionuclide authorized concentration limits for liquid effluent discharged to the environment and radionuclide guidance levels for waterbodies and drinking waters are usually specified by national regulations for planned, existing, and emergency exposure situations. Compliance with these limits can be assessed using measurement results with their associated uncertainties as requested by ISO/IEC Guide 98-3 and ISO 5667-20.[4]

Depending of the exposure situation, the limits and guidance levels that would result in an action to reduce health risk differ. As an example, during planned or existing situation, the WHO1) guidance for screening levels in drinking water is 0,5 Bq/l for gross alpha activity and 1 Bq/l for gross beta activity. In the event of a nuclear emergency, the WHO Codex Guideline Levels2) mentioned that the activity concentration might not be greater than N_b Bq/l for radionuclide XX.

Thus, the test method may need to be adjusted depending if it is applied for either a planned-existing or an emergency situation since during emergency situations a large number of samples needs to be rapidly characterized. The test methods could be adapted so that its performance in term of characteristic limits, decision threshold, and detection limit, and the uncertainties ensure that the

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

2) 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 guideline levels 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 .document containing values of activity concentration in foods, including liquid beverages, that would result in an effective dose of 1 mSv/a for members of the public (infant and adult). The guideline levels have been checked against age-dependent ingestion dose coefficients defined as committed effective doses per unit intake for each radionuclide, which are taken from the “International Basic Safety Standards” [5]. The Codex GLs are included in the General Standard for Contaminants and Toxins in Food and Feeds [6].

radionuclide activity concentration test results permit the verification that they are below the guidance levels required by national authority for either planned-existing situations or an emergency situation. [6][7][8]

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 comply with national regulations in verifying that before their discharge, wastewaters/liquid effluent radioactive activity concentrations are lower than the authorized limits.

The test method(s) described in this standard 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 *ad hoc* part of ISO 5667).

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, and may be required 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.

DRAFT INTERNATIONAL STANDARD **ISO/DIS 11704:2017(E)**

Water quality — Gross alpha and gross beta activity — Test method using liquid scintillation counting

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 according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method for the determination of gross alpha and gross beta activity concentration for alpha- and beta-emitting radionuclides using liquid scintillation counting (LSC).

The method is applicable to all types of waters with a dry residue of less than 5 g/l and when no correction for colour quenching is necessary.

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

The method covers non-volatile radionuclides below 80 °C, since some gaseous or volatile radionuclides (e.g. radon and radioiodine) may be lost during the source preparation.

The method is applicable to test sample of drinking water, rainwater, surface and ground water as well as cooling water, industrial water, domestic and industrial wastewater after proper sampling and test sample preparation (filtration when necessary and taking into account the amount of dissolved material in the water).

The method described in this standard is applicable in the event of an emergency situation, because the results can be obtained in less than 4 h by directly measuring water test samples without any treatment.

It is the laboratory's responsibility to ensure the suitability of this test 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, *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: Guidance on the preservation and handling of water samples*

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

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*

3 Symbols, definitions and units

For the purposes of this document, the definitions, symbols and abbreviations defined in ISO 80000-10, and the following apply.

a_α, a_β	Alpha and beta activity per mass	Bq g^{-1}
a^*	Decision threshold	Bq g^{-1}
$a^\#$	Detection limit	Bq g^{-1}
$a^\triangleleft, a^\triangleright$	Lower and upper limits of the confidence interval	Bq g^{-1}
A_α, A_β	Activity of the alpha and beta emitter certified reference solution used for the α and β calibration sources	Bq
m	Mass of the test sample	g
m_1	Mass of initial sample subject to heating or possibly concentration	g
m_2	Mass of heated or concentrated sample	g
m_3	Mass of heated or concentrated sample transferred in the vial	g
$m_{S\alpha}$, $m_{S\beta}$	Mass of alpha and beta emitters certified reference solutions, respectively	g
$r_{g\alpha}$, $r_{g\beta}$	Sample gross count rate, from the alpha and beta windows, respectively	s^{-1}
$r_{0\alpha}$, $r_{0\beta}$, r_{0T}	Blank count rate, from the alpha, beta and total windows, respectively	s^{-1}
$r_{S\alpha,\alpha}$, $r_{S\alpha,\beta}$, $r_{S\alpha,T}$	Count rate of the alpha calibration source in the alpha, beta and total window	s^{-1}
$r_{S\beta,\alpha}$, $r_{S\beta,\beta}$, $r_{S\beta,T}$	Count rate of the beta calibration source in the alpha, beta and total window	s^{-1}
t_g	Sample counting time	s
t_0	Blank counting time	s
$t_{s\alpha}$, $t_{s\beta}$	Counting time of α and β calibration sources	s
$u\ (a)$	Standard uncertainty associated with the measurement result	Bq g^{-1}
U	Expanded uncertainty, calculated from $U = ku\ (a)$, where $k = 1, 2 \ldots$	Bq g^{-1}
$\tilde{u}(\tilde{a}_\alpha)$	Standard uncertainty of a_α as a function of its true value	Bq g^{-1}

ε_α, ε_β	Counting efficiency for alpha and beta, respectively	—
$\tau_\alpha(\chi_{\alpha\,\beta})$	Alpha interference — Fraction of counts observed in the beta window with respect to the total number of counts measured by the counter when an alpha emitter is measured	—
$\tau_\beta(\chi_{\beta\,\alpha})$	Beta interference — Fraction of counts observed in the alpha window with respect to the total number of counts measured by the counter when a beta emitter is measured	—

4 Principle

Gross alpha and beta activity concentrations are determined by using liquid scintillation counting of a water sample mixed with a scintillation cocktail.

Gross alpha and beta determinations are not absolute determinations of the sample radioactive contents, but relative determinations referred to a specific alpha or beta emitter which constitutes the standard calibration sources. These types of determinations are also known as the alpha and beta index and are usually employed as screening parameters for first assessment of total radioactive content.

The aqueous sample is acidified using nitric acid and heated. Subsequently, water with low salt content can be thermally concentrated by slow evaporation to improve the method sensitivity. An aliquot of sample is transferred into a liquid scintillation vial with scintillation cocktail; scintillations from the vial are then counted by equipment with an alpha and beta discrimination device.

The counter is previously optimized with respect to an alpha and beta discriminator setting and then calibrated against alpha and beta emitter certified reference solutions. In data evaluation, no correction for chemical quenching is applied, since the procedure is designed to provide samples with a constant quenching level.

The method does not account for ^{222}Rn and its daughters of short half-life and it is not suitable for ^{3}H measurement.

When suspended matter is present in significant quantities, a filtration step is required before acidification.

5 Reagents and equipment

All reagents shall be of recognized analytical grade, except for the scintillation cocktail, and shall not contain any detectable alpha and beta activity, except for the radioactive certified reference solutions.

5.1 **Nitric acid**, $c(\text{HNO}_3)$ = 15,8 mol/l, ρ = 1,42 g/ml, mass fraction $w(\text{HNO}_3)$ = 70 %.

5.2 **Water**, ISO 3696, grade 3.

Deionized water can contain detectable amounts of ^{222}Rn and short half-life decay products. It is therefore strongly recommended to boil water under vigorous stirring and let it stand for one day before use. Alternatively, use nitrogen flushing for about 1 h for a 2 l sample.

5.3 Scintillation cocktail

Commercially available scintillation cocktails suitable for alpha and beta discrimination (e.g. diisopropylnaphthalene-based cocktails).

5.4 Volatile organic solvents

Methanol or ethanol.