SLOVENSKI STANDARD
SIST ISO 11704:2013

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Kakovost vode - Merjenje skupne alfa in skupne beta koncentracije aktivnosti v neslanih vodah - Metoda štetja s tekočinskim scintilatorjem

Water quality - Measurement of gross alpha and beta activity concentration in non-saline water - Liquid scintillation counting method

Qualité de l'eau - Mesurage des activités alpha globale et bêta globale des eaux non salines - Méthode de comptage par scintillation liquide

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17.240	Merjenje sevanja	Radiation measurements

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INTERNATIONAL STANDARD

ISO 11704

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Water quality — Measurement of gross alpha and beta activity concentration in non-saline water — Liquid scintillation counting method

Qualité de l'eau — Mesurage des activités alpha globale et bêta globale des eaux non salines — Méthode de comptage par scintillation liquide

Reference number
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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 11704 was prepared by Technical Committee ISO/TC 147, *Water quality*.

Water quality — Measurement of gross alpha and beta activity concentration in non-saline water — Liquid scintillation counting method

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 in waters for radionuclides which are not volatile at 80 °C. Radon isotopes and their decay products of short half life are not included in the determination.

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

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 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 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, as well as the following symbols, definitions and units, 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}
$a^\lhd, a^\rhd$	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	—
$\tau_\alpha(\chi_{\alpha\to\beta})$	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	—
$\tau_\beta(\chi_{\beta\to\alpha})$	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.

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 and ^{14}C 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, $c(HNO_3) = 15{,}8$ mol/l, $\rho = 1{,}42$ g/ml, mass fraction $w(HNO_3) = 70$ %.

5.2 Water, ISO 3696[1], 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.

5.5 Certified reference solutions. A calibration laboratory establishes traceability of its own calibration sources and measuring instruments to the International System of Units (SI) by means of an unbroken chain of calibrations or comparisons linking them to relevant certified reference solutions of the SI units of measurement. The link to the SI units may be achieved with respect to national certified reference materials. These may be primary realizations of the SI units, or agreed representations of SI units based on fundamental physical constants, or they may be secondary materials which are materials certified by another national metrology institute. When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability, and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or statement of compliance with an identified metrological specification.

NOTE Calibration laboratories fulfilling the requirements of this International Standard are considered to be competent. A calibration certificate bearing an accreditation body logo from a calibration laboratory accredited to this International Standard, for the calibration concerned, is sufficient evidence of traceability of the calibration data reported.

In general, the experimental parameters (efficiency, alpha and beta optimum discrimination) depend on alpha and beta energies, thus the choice of alpha and beta emitter certified reference solutions will depend on knowledge of the type of radioactive contaminant likely to be present in the waters being tested (see ISO 9696[4] and Reference [11]).

5.5.1 Alpha emitter certified reference solution. The alpha emitter certified reference solution shall not contain any unexpected detectable alpha and beta activity.