SLOVENSKI STANDARD
SIST ISO 9696:2010

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Kakovost vode - Merjenje skupne alfa aktivnosti v neslanih vodah - Metoda robustnega vira

Water quality - Measurement of gross alpha activity in non-saline water - Thick source method

Qualité de l'eau - Mesurage de l'activité alpha globale des eaux non salines - Méthode par source concentrée

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ICS:

13.060.60	Preiskava fizikalnih lastnosti vode	Examination of physical properties of water
17.240	Merjenje sevanja	Radiation measurements

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INTERNATIONAL STANDARD

ISO 9696

Second edition
2007-11-15

Water quality — Measurement of gross alpha activity in non-saline water — Thick source method

Qualité de l'eau — Mesurage de l'activité alpha globale des eaux non salines — Méthode par source concentrée

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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 9696 was prepared by Technical Committee ISO/TC 147, *Water quality*.

This second edition cancels and replaces the first edition (ISO 9696:1992), which has been technically revised.

Water quality — Measurement of gross alpha activity in non-saline water — Thick source 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 in accordance with this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method for the determination of gross alpha activity in non-saline waters for alpha-emitting radionuclides which are not volatile at 350 °C. It is possible to determine supported volatile radionuclides measured to an extent determined by half-life, matrix retention (of the volatile species) and the duration of measurement (counting time).

The method is applicable to raw and potable waters.

The range of application depends on the amount of suspended matter in the water and on the performance characteristics (background count rate and counting efficiency) of the counter.

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 31-9, *Quantities and units — Part 9: Atomic and nuclear physics*

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

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

Guide to the expression of uncertainty in measurement (GUM), BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML

3 Symbols, definitions and units

For the purposes of this document, the definitions, symbols and abbreviations defined in ISO 31-9, as well as the following symbols, apply.

V_t	Volume of the water sample, in litres
V	Volume of test sample, in litres, equivalent to the mass of solid on the planchet
m	Mass, in milligrams, of ignited residue from volume V
m_r	Mass of the residue deposited on the planchet sample, in milligrams
A	Alpha activity of the calibration source, in becquerels
c_A	Alpha activity concentration, in becquerels per litre
t_0	Background counting time, in seconds
t_g	Sample counting time, in seconds
r_0	Background count rate, per second
r_g	Sample gross count rate, per second
r_s	Calibration count rate, per second
ε	Counting efficiency of the specified calibration source
S	Area of the planchet, in square millimetres
$u(c_A)$	Standard uncertainty associated with the measurement result, in becquerels per litre
U	Expanded uncertainty calculated by $U = k \cdot u(a)$ with k = 1, 2.., in becquerels per litre
c_A^*	Decision threshold, in becquerels per litre
$c_A^\#$	Detection limit, in becquerels per litre
$c_A^\triangleleft, c_A^\triangleright$	Lower and upper limits of the confidence interval, in becquerels per litre

4 Principle

Gross alpha radioactivity is determined by using proportional counting or solid scintillation counting on water residue deposited on a planchet. Due to the strong absorption of the residue deposit, it is considered that the alpha emission from the surface is proportional to the alpha activity of the deposit.

Gross alpha determination is not an absolute determination of the sample alpha radioactive content, but a relative determination referring to a specific alpha emitter which constitutes the standard calibration source. This type of determination is also known as alpha index.

The sample is acidified to stabilize it, evaporated almost to dryness, converted to the sulfate form and then ignited at 350 °C. A portion of the residue is transferred to a planchet and the alpha activity measured by counting in an alpha-particle detector or counting system previously calibrated against an alpha-emitting standard and the alpha activity concentration calculated.

5 Reagents and equipment

5.1 Reagents

Except for the certified reference solution, all reagents shall be of recognized analytical grade and shall not contain any measurable alpha activity.

NOTE A method for preparing reagent blanks to check for endemic radioactivity or contamination is given in Clause 7.

5.1.1 Water, distilled or deionized, for all applications.

5.1.2 Certified reference solution.

A calibration laboratory establishes traceability of its own measurement standards and measuring instruments to the International System of Units (SI) by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. The link to SI units may be achieved by reference to national measurement standards. National measurement standards may be primary standards, which are primary realizations of the SI units or agreed representations of SI units based on fundamental physical constants, or they may be secondary standards which are standards calibrated 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 a statement of compliance with an identified metrological specification.

The choice of alpha standard will depend on knowledge of the type of radioactive contaminant likely to be present in the waters being tested. In general, this amounts to a choice between naturally occurring and man-made alpha emitters.

Among standard solutions of artificial alpha-emitting radionuclides, ^{241}Am and ^{239}Pu are commonly used. When ^{239}Pu is used, the presence of ^{241}Pu impurity shall be taken into account as it leads to growth of ^{241}Am in prepared standard solutions of sources. When ^{241}Am is used, the potential interferences of its gamma emission, or alpha-beta crosstalk shall be taken into account (see Reference [1]).

NOTE 1 A uranium compound of certified natural or known isotopic composition has one arguable advantage, in that its specific activity can be calculated from established physical constants and isotopic abundance data which are independent of the calibration procedures of a particular organization.

NOTE 2 Furthermore, since the energies of the alpha emissions from uranium isotopes are less than those from the artificial transuranic nuclides, the use of a uranium standard tends to give a high result for transuranics. Some authorities prefer to err on the high side in situations where the true composition is unknown.

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

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

5.1.5 Volatile organic solvents.

Methanol and acetone.

5.1.6 Vinyl acetate.

5.1.7 Calcium sulfate, $CaSO_4$.

As calcium salts may contain trace amounts of ^{226}Ra and/or ^{210}Pb, checks for the presence of these nuclides shall be made.