SLOVENSKI STANDARD
oSIST prEN ISO 9698:2017

01-maj-2017

Kakovost vode - Tritij - Preskusna metoda štetja s tekočinskim scintilatorjem (ISO/DIS 9698:2017)

Water quality - Tritium - Test method using liquid scintillation counting (ISO/DIS 9698:2017)

Wasserbeschaffenheit - Tritium - Verfahren mit dem Flüssigszintillationszähler (ISO/DIS 9698:2017)

Qualité de l'eau - Tritium - Méthode d'essai par comptage des scintillations en milieu liquide (ISO/DIS 9698:2017)

Ta slovenski standard je istoveten z: **prEN ISO 9698**

ICS:

13.060.60	Preiskava fizikalnih lastnosti vode	Examination of physical properties of water

oSIST prEN ISO 9698:2017 **en,fr,de**

DRAFT INTERNATIONAL STANDARD
ISO/DIS 9698

ISO/TC **147**/SC **3** Secretariat: **AFNOR**

Voting begins on:
2017-03-10

Voting terminates on:
2017-06-01

Water quality — Tritium — Test method using liquid scintillation counting

Qualité de l'eau — Tritium — Méthode d'essai par comptage des scintillations en milieu liquide

ICS: 13.060.60; 13.280

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 9698:2017(E)

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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 on 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 the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC147 Water quality, subcommittee SC 3, Radioactivity measurements.

This third edition cancels and replaces the second 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 potassium 40, tritium, carbon 14, and those originating from the thorium and uranium decay series, in particular radium 226, radium 228, uranium 234, uranium 238, lead 210, 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), tritium, carbon 14, strontium 90 and gamma emitting radionuclides can also be found in natural waters as a result of authorized routine releases into the environment in small quantities in the effluent discharged from nuclear fuel cycle facilities. Radionuclides are also released into the environment following their use in unsealed form for medical and industrial applications. They are also found in the water as a result of past fallout contamination 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 and climatic characteristics 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.

Radioactivity of liquid effluent are usually controlled before they are discharged into the environment[2] and water bodies and drinking water are monitored for their radioactivity content as recommended by the World Health Organization (WHO).[3] It enables to take proper actions to ensure that there is no adverse health effect 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 WHO[1] guidance level for tritium in drinking water is 10 000 Bq/l. In the event of a nuclear emergency, the WHO Codex Guideline Levels[2] mentioned that the activity concentration might not be greater than 1 000 for infant foods and 10 000 Bq/l for foods other than infant foods, both for organically bound tritium.

Thus, the test method has 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 screened. The test methods shall be adapted so that their characteristic limits, decision threshold and detection limit, and the uncertainties ensure the verification that the radionuclide activity

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[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] 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].

concentrations are below the guidance levels required by national authority for either planned-existing situations or an emergency situation[6][7][8].

Usually, the test method 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 effluent 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 tritium 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 International Standard 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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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 the conditions for the determination of tritium activity concentration in samples of marine, surface, ground, rain drinking waters or of tritiated water ([^{3}H] H_2O) in effluents using liquid scintillation counting.

The choice of the analytical procedure (either with or without distillation of the water sample prior to determination), depends on the aim of the measurement and the sample characteristics[20],[21],[22].

Direct measurement of a raw water sample using liquid scintillation counting[9] has to consider the potential presence of other beta emitter radionuclides. To avoid interference with these radionuclides when they are detected, the quantification of tritium is performed following the sample treatment by distillation.[23],[24],[25],[26] The Annexes B, D and E describe three distillation procedures.

The method is not directly applicable to the analysis of organically bound tritium; its determination requires additional chemical processing of the sample (such as chemical oxidation or combustion).

With suitable technical conditions, the detection limit may be as low as 1 Bq l^{-1}. Tritium activity concentrations below 10^6 Bq l^{-1} can be determined without any sample dilution.

A prior enrichment step can significantly lower the limit of detection[27],[28].

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

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/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

ISO/IEC Guide 99:2007, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

3 Terms and definitions

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

3.1

effluent

water or wastewater discharged from a containing space such as a treatment plant, industrial process or lagoon

3.2

industrial wastewater

water discharged after being used in, or produced by, an industrial process, and which is of no further immediate value to that process

3.3

filtration

treatment process whereby water is passed through a porous layer of material in order to remove particulate matter

4 Symbols and abbreviations

β_{max}	Maximum energy for the beta emission, in keV
V	Volume of test sample, in litre
m	Mass of test sample, in kilogram
ρ	Density of the sample, in kilogram per litre
c_A	Activity concentration, in becquerel per litre
a	Activity per unit of mass, in becquerel per kilogram
A	Activity of the calibration source, in becquerel
t_0	Background counting time, in second
t_g	Sample counting time, in second
t_s	Calibration counting time, in second
r_0	Background count rate, per second
r_g	Sample count rate, per second
r_s	Calibration count rate, per second
ε	Detection efficiency
f_q	Quench factor
$u(c_A)$	Standard uncertainty associated with the measurement result; in becquerel per litre
U	Expanded uncertainty, calculated by $U = k \cdot u\left(c_A\right)$ with k= 1, 2,..., in becquerel per litre
c_A^*	Decision threshold, in becquerel per litre
$c_A^\#$	Detection limit, in becquerel per litre
$c_A^\triangleleft, c_A^\triangleright$	Lower and upper limits of the confidence interval, in becquerel per litre

5 Principle

The test portion is mixed with the scintillation cocktail in a counting vial to obtain a homogeneous medium. Electrons emitted by tritium transfer their energy to the scintillation medium. Molecules excited by this process return to their ground state by emitting photons that are detected by photodetectors.

The electric pulses emitted by the photodetectors are amplified, sorted (in order to remove random events) and analyzed by the electronic systems and the data analysis software. The count rate of these photons allows the determination of the test portion activity, after correcting for the background count rate and detection efficiency.

In order to determine the background count rate, a blank sample is prepared in the same way as the test portion. The blank sample is prepared using a reference water of the lowest activity available, also sometimes called “dead water”.

In order to determine the detection efficiency, it is necessary to measure a water sample having a known tritium activity under conditions that are identical to those used for the test sample. This water shall be a dilution of this mixture produced with the reference water.

The conditions to be met for the blank sample, the test portion and the calibration source are:

- same scintillation cocktail;
- same type of counting vial;
- same filling geometry;
- same ratio between test portion and scintillation cocktail;
- temperature stability of the detection equipment;
- value of quench indicating parameter included in calibration curve.

If particular conditions of chemical quenching affect the measurement results, it is recommended to correct the counting data using a quench curve. It is important to choose the chemical quenching agent according to the supposed type of quenching observed in the sample. This method is not applicable to colour quenched samples.

For high activity and highly quenched samples or colour quenched samples, it may be practical to use an internal standard method, as described in Annex C.

6 Reagents and equipment

Use only reagents of recognized analytical grade.

6.1 Reagents

6.1.1 Water for the blank

The water used for the blank shall be as free as possible of chemical impurities to avoid quenching, of radioactive impurities[22] and with an activity concentration of tritium negligible in comparison with the activities to be measured.

For example, a water sample with a low tritium activity concentration can be obtained from (deep) subterranean water kept in a well-sealed borosilicate glass bottle in the dark at controlled temperature (ISO 5667-3). This blank water sample shall be kept physically remote from any tritium containing material.