



SLOVENSKI STANDARD

SIST ISO 9698:2013

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Nadomešča:
SIST ISO 9698:2010

Kakovost vode - Določevanje koncentracije aktivnosti tritija - Metoda štetja s tekočinskimi scintilatorjem

Water quality - Determination of tritium activity concentration - Liquid scintillation counting method

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Qualité de l'eau - Détermination de l'activité volumique du tritium - Méthode par comptage des scintillations en milieu liquide

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**Water quality — Determination of tritium
activity concentration — Liquid
scintillation counting method**

*Qualité de l'eau — Détermination de l'activité volumique du tritium —
Méthode par comptage des scintillations en milieu liquide*

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ISO 9698:2010(E)**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 9698 was prepared by Technical Committee ISO/TC 147, *Water quality*.

This second edition cancels and replaces the first edition (ISO 9698:1989), which has been technically revised.

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Introduction

The tritium present in the environment is of natural origin and man made. As a result of atmospheric nuclear weapon testing, emissions from nuclear engineering installations, and the application and processing of isotopes, relatively large amounts of tritium have been released to the environment. Despite the low dose factor associated to tritium, monitoring of tritium activity concentrations in the environment is necessary in order to follow its circulation in the hydrosphere and biosphere.

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Water quality — Determination of tritium activity concentration — Liquid scintillation counting method

WARNING — This International 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 environmental water or of tritiated water ($[^3\text{H}]\text{H}_2\text{O}$) 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 (see References [1], [2], [3]).

Direct measurement of a raw water sample using liquid scintillation counting 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 will be performed following the sample treatment by distillation (see References [4], [5], [6], [7]). Three distillation procedures are described in Annexes B, D and E.

The method is not applicable to the analysis of organically bound tritium; its determination requires additional chemical processing (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 (see References [8], [9]).

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 5667-1, *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 80000-10, *Quantities and units — Part 10: Atomic and nuclear physics*

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

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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 Symbols, definitions and units

For the purposes of this document, the definitions, symbols and units defined in ISO 80000-10, ISO/IEC Guide 98-3 and ISO/IEC Guide 99, as well as the following symbols, apply.

β_{\max}	Maximum energy for the beta emission, in kilo-electronvolts
V	Volume of test sample, in litres
m	Mass of test sample, in kilograms
ρ	Mass density of the sample, in grams per litre
c_A	Activity concentration, in becquerels per litre
a	Activity per unit of mass, in becquerels per kilogram
A	Activity of the calibration source, in becquerels
t_0	Background counting time, in seconds
t_g	Sample counting time, in seconds
t_s	Calibration counting time, in seconds
n	Number of repetitions
r_{0i}	Background count rate in the repetition i , per second
r_0	Mean background count rate for i repetitions, per second
r_{gi}	Sample count rate in the repetition i , per second
r_g	Mean sample count rate for i repetitions, per second
r_s	Calibration count rate, per second
ε	Detection efficiency
ε_q	Efficiency measured for each of the working standards to elaborate the quench curve
f_q	Quench factor
$u(c_A)$	Standard uncertainty associated with the measurement result, in becquerels per litre
U	Expanded uncertainty, calculated by $U = k \cdot u(c_A)$ with $k = 1, 2, \dots$, in becquerels per litre
c_A^*	Decision threshold, in becquerels per litre
$c_A^\#$	Detection limit, in becquerels per litre
$c_A^{<}, c_A^{>}$	Lower and upper limits of the confidence interval, in becquerels per litre

4 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 analysed 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".

The detection efficiency is determined with a sample of a standard of aqueous tritium (calibration source), or a dilution of this standard with water for the blank, measured in the same conditions as the test portion.

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

- 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.

IMPORTANT — Quench correction: If the measurement results are affected by particular conditions of chemical quenching, it is recommended that a quench curve be established. It is important to choose a suitable chemical quenching agent for the type of quenching suspected in the sample.

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

5 Reagents and equipment

Use only reagents of recognized analytical grade.

5.1 Reagents

5.1.1 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 (see Reference [10]) 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 a controlled temperature (ISO 5667-3). This blank water sample shall be kept physically remote from any tritium-containing material (see Clause 4, important notice). Determine the tritium activity concentration ($t = 0$), in Bq l^{-1} , of this water and note the date ($t = 0$) of this determination (see Clause 4, Note).

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It is advisable to keep an adequate quantity of blank water in stock and to make small working amounts from it for immediate use, as required. Contamination with tritium (e.g. from water vapour in the air and from tritium sources such as luminous watches and gas chromatographs) or other radioactive species should be avoided.

As the activity is becoming non-negligible for activities around 1 Bq l^{-1} , it is necessary to use a blank water measured to ensure the "absence" of tritium. The tritium activity concentration in the blank water can be determined by enrichment followed by liquid scintillation counting or from the measurement of ^3He by mass spectrometry. Preferably use blank water with a tritium activity concentration of less than $0,5 \text{ Bq l}^{-1}$.

When the volume of blank water is sufficiently large, e.g. 10 l to 20 l, and well-sealed, tritium activity concentration will remain stable for years, although it is advisable to determine the tritium activity concentration at predetermined intervals, e.g. every year.

5.1.2 Calibration source solution

In order to avoid cross-contamination, prepare, in a suitable location which is remote from the area where the tritium analyses are to be carried out, weigh and pour into a weighed volumetric flask (for example, 100 ml) the requisite quantity of a concentrated tritium ($[^3\text{H}]\text{H}_2\text{O}$) standard solution, so that the tritium activity concentration will generate sufficient counts to reach the required measurement uncertainty after dilution with blank water and thorough mixing. Calculate the tritium activity concentration of the resulting calibration source solution ($t = 0$). Note the date at which the standard solution was made up ($t = 0$).

The tritium activity concentration of the calibration source solution at time t at which the samples are measured must be corrected for radioactive decay.

5.1.3 Scintillation solution iTeh STANDARD PREVIEW

The scintillation cocktail is chosen according to the characteristics of the sample to be analysed and according to the properties of the detection equipment (see Reference [11]).

It is recommended to use a good hydrophilic scintillation cocktail, especially for the measurement of usual environmental water.

The characteristics of the scintillation cocktail must allow the mixture to be homogeneous and stable.

For the direct measurement of raw waters containing particles in suspension, it is recommended that a scintillation cocktail leading to a gel-type mixture be used.

It is recommended to:

- store in the dark and, particularly just before use, avoid exposure to direct sunlight or fluorescent light in order to prevent interfering luminescence;
- comply with the storage conditions specified by the scintillation cocktail supplier.

The mixtures (scintillation cocktail and test sample) should be disposed of as chemical waste, and, depending on the radioactivity, may require disposal as radioactive waste.

5.1.4 Quenching agent

The following are examples of chemical quenching agents: nitric acid, acetone, organochloride compounds, nitromethane.

NOTE Some quenching agents are dangerous or toxic.