
**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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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*

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).

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.

5.2 Equipment

Laboratory equipment, such as pipettes and balances, shall be employed that enables the expected/agreed data quality objectives to be achieved, as well as the quantification of the uncertainty attached to the measurement.

NOTE Control of the quantity of liquid scintillation cocktail used in source preparation is essential to achieve consistent data quality.

5.2.1 Liquid scintillation counter

Liquid scintillation counter, preferably with an automatic sample transfer. Operation at constant temperature is recommended following the manufacturer's instructions. Depending on the limit of detection to be reached, a liquid scintillation counter with a low-level configuration may be needed. The method specified in this International Standard relates to the widely used liquid scintillation counters with vials that hold about 20 ml. When other vials are used with appropriate counters, the described method shall be adapted accordingly.

5.2.2 Counting vials

Different types of scintillation vials exist, manufactured using a range of materials. The most common are glass vials and polyethylene vials. Glass vials allow visual inspection of the scintillation medium, but have an inherent background, due to the presence of ^{40}K . However, some organic solvents contained in scintillation cocktails diffuse through the polyethylene, accelerating the degradation of the mixture.

Other types of vials exist:

- glass vials with low level of ^{40}K , will exhibit a lower background than "normal" glass vials;
- for the determination of very low tritium concentration, the use of polytetrafluoroethylene (PTFE) vials or polyethylene vials with an inner layer of PTFE on the inside vial wall is strongly recommended. Diffusion of organic solvents is then slower through PTFE than through polyethylene. These vials are used for long counting times with very low-level activity to be measured.

Generally, the vials are single use. If the vial is reused, it is necessary to apply an efficient cleaning procedure.

To prevent interfering luminescence, the counting vials should be kept in the dark and should not be exposed to direct sunlight or fluorescent light, particularly just before use.

Toluene-based scintillation solutions may physically distort polyethylene and should therefore not be used in combination with polyethylene counting vials. Diffusion of organic solvents into and through the polyethylene walls is also a serious drawback of polyethylene vials.

6 Sampling and samples

6.1 Sampling

Conditions of sampling shall conform to ISO 5667-1.

It is not advised to acidify the sample due to the high chemical quench caused by acids, and the potential presence of tritium in the acid, as specified in ISO 5667-3.

It is important that the laboratory receive a representative sample, unmodified during the transport or storage and in an undamaged container. It is recommended to use a glass flask and to fill it to the maximum, to minimize tritium exchange with the atmospheric moisture.

For low level activity measurements, it is important to avoid any contact between sample and atmosphere during the sampling.

6.2 Sample storage

If required, the sample shall be stored in compliance with ISO 5667-3. If the storage duration exceeds that specified in ISO 5667-3, it is advisable to store the samples in glass flasks.

7 Procedure

7.1 Sample preparation

7.1.1 Direct procedure

Measurement of the test sample is generally performed on raw water without removal of suspended matter. If the activity of a filtered or centrifuged sample is to be measured, the removal of suspended matter shall be performed as soon as possible after the sampling (see ISO 5667-3).

7.1.2 Distillation

Examples of distillation procedures are given in Annexes B, D and E.

Distillation shall avoid isotopic fractionation (see Reference [12]).

Distillation or any other physico-chemical treatment of water is not appropriate for simultaneous measurement of tritium and ^{14}C .

7.2 Preparation of the sources to be measured

Known quantity of test sample and scintillation cocktail are introduced into the counting vial.

After closing the vial, it shall be thoroughly shaken to homogenize the mixture.

The vial identification shall be written on the top of the vial stopper. The storage time depends upon the scintillation mixture, the mixture stability and the nature of the sample. It is recommended to perform the measurement as soon as any photoluminescence or static electricity effects have become negligible, e.g. after 12 h.

In order to reduce photoluminescence effects, it is recommended that the above mentioned operations take place in dimmed light (preferably light from an incandescent source or red light); in addition, direct sunlight or fluorescent light should be avoided.

7.3 Counting procedure

The measurement conditions (measurement time, blank sample, number of cycles or repetitions) will be defined according to the uncertainty and detection limit to be achieved.

7.3.1 Control and calibration

Statistical control of the detection system shall be monitored by measurement of suitable reference background and reference sources usually provided by the equipment supplier, for example in compliance with ISO 8258 [13].

The measurement of the blank sample is performed before each analysis or each series of sample measurement in representative conditions of each type of measurement (Clause 4).

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.