INTERNATIONAL STANDARD

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Water quality — Carbon 14 — Test method using liquid scintillation counting

Qualité de l'eau — Carbone 14 — Méthode d'essai par comptage des scintillations en milieu liquide

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 230, *Water analysis*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 13162:2011), which has been technically revised. The main changes compared to the previous edition are as follows:

- Introduction developed;
- Scope updated;
- References updated;
- Sample preparation revised.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 ^{40}K, ^{3}H, ^{14}C, and those originating from the thorium and uranium decay series, in particular ^{226}Ra, ^{228}Ra, ^{234}U, ^{238}U, ^{210}Po and ^{210}Pb 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 fertilizers production and use);
- human-made radionuclides such as transuranium elements (americium, plutonium, neptunium, curium), ^{3}H, ^{14}C, ^{90}Sr, and gamma emitting radionuclides can also be found in natural waters. Small quantities of these radionuclides are discharged from nuclear fuel cycle facilities into the environment as a result of authorized routine releases. Some of these radionuclides used for medical and industrial applications are also released into the environment after use. Anthropogenic radionuclides are also found in waters as a result of past fallout contaminations 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 characteristics and climatic conditions 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.

The radionuclides present in liquid effluents are usually controlled before being discharged into the environment[2] and water bodies. Drinking waters are monitored for their radioactivity as recommended by the World Health Organization (WHO)[3] so that proper actions can be taken to ensure that there is no adverse health effect to the public. Following these international recommendations, national regulations usually specify radionuclide authorized concentration limits for liquid effluent discharged to the environment and radionuclide guidance levels for waterbodies and drinking waters for planned, existing, and emergency exposure situations. Compliance with these limits can be assessed using measurement results with their associated uncertainties as specified by ISO/IEC Guide 98-3 and ISO 5667-20 [4].

Depending on the exposure situation, there are different limits and guidance levels that would result in an action to reduce health risk. As an example, during a planned or existing situation, the WHO guidelines for guidance level in drinking water is 100 Bq·l^{-1} for ^{14}C activity concentration.

NOTE 1 The guidance level is the activity concentration with an intake of 2 l/d of drinking water for one year that results in an effective dose of 0,1 mSv/a for members of the public. This is an effective dose that represents a very low level of risk and which is not expected to give rise to any detectable adverse health effects [3].

In the event of a nuclear emergency, the WHO Codex Guideline Levels[5] mentioned that the activity concentration might not be greater than 10 000 Bq·l^{-1} for ^{14}C in foods other than for infant foods.

NOTE 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 GLs 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 for members of the public (infant and adult) [5].

Thus, the test method can be adapted so that the characteristic limits, decision threshold, detection limit and uncertainties ensure that the radionuclide activity concentrations test results can be verified to be below the guidance levels required by a national authority for either planned/existing situations or for an emergency situation [6][7].

Usually, the test methods 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 verify that, before their discharge, wastewaters/liquid effluent radioactive activity concentrations do not exceed authorized limits.

The test method(s) described in this document may be used during planned, existing and emergency exposure situations as well as for wastewaters and liquid effluents 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 relevant part of the ISO 5667 series).

This document has been developed to support the need of test laboratories carrying out these measurements, that are sometimes required by national authorities, as they may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

This document 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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Water quality — Carbon 14 — Test method using liquid scintillation counting

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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 determine the applicability of any other restrictions.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies a method for the measurement of ^{14}C activity concentration in all types of water samples by liquid scintillation counting (LSC) either directly on the test sample or following a chemical separation.

The method is applicable to test samples of supply/drinking water, rainwater, surface and ground water, marine water, as well as cooling water, industrial water, domestic, and industrial wastewater.

The detection limit depends on the sample volume, the instrument used, the sample counting time, the background count rate, the detection efficiency and the chemical recovery. The method described in this document, using currently available liquid scintillation counters and suitable technical conditions, has a detection limit as low as 1 Bq·l^{-1}, which is lower than the WHO criteria for safe consumption of drinking water (100 Bq·l^{-1}). ^{14}C activity concentrations can be measured up to 10^6 Bq·l^{-1} without any sample dilution.

It is the user's responsibility to ensure the validity of this test method for the water samples tested.

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 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste water*

ISO 11929-1, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 1: Elementary applications*

ISO 19361, *Measurement of radioactivity — Determination of beta emitters activities — Test method using liquid scintillation counting*

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) — 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, definitions, symbols and abbreviations

For the purposes of this document, the definitions, symbols and abbreviations given in ISO/IEC Guide 99:2007, ISO/IEC Guide 98-3:2008, ISO 80000-10, and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

β_{max}	Maximum energy for the beta emission	keV
V	Volume of laboratory sample	l
m	Mass of laboratory sample	kg
ρ	Density of the sample	$\mathrm{kg \cdot l^{-1}}$
c_A	Activity concentration	$\mathrm{Bq \cdot l^{-1}}$
a	Activity per unit of mass	$\mathrm{Bq \cdot kg^{-1}}$
A	Activity of the calibration source	Bq
A_s	Activity of the internal standard solution	Bq
n	Number of counting	-
N_0	Number of the counted pulses for the background	-
t_0	Background counting time, in second	s
N_g	Number of the counted pulses for the sample	-
t_g	Sample counting time	s
t_s	Calibration counting time	s
r_0	Background count rate	$\mathrm{s^{-1}}$
r_g	Test sample count rate	$\mathrm{s^{-1}}$
r_s	Calibration count rate	$\mathrm{s^{-1}}$
ε	Detection efficiency	-
Q	Quench parameter	-
f_q	Quench factor	-
ε_q	Counting efficiency at quench parameter Q	-
R_c	Chemical recovery	-
m_{TC}	Mass of total carbon in the sample	kg
m_{PC}	Mass of carbon in the precipitate	kg
m_{CC}	Mass of carbon in the carrier	kg
m_{SC}	Mass of sample carbon in the precipitate	kg
$u(c_A)$	Standard uncertainty associated with the measurement result	$\mathrm{Bq \cdot l^{-1}}$
u_{rel}	Relative standard uncertainty	-
$\tilde{c}_A$	Possible or assumed true quantity values of the measurand	$\mathrm{Bq \cdot l^{-1}}$
$\tilde{u}(\tilde{c}_A)$	Standard uncertainty of $\tilde{c}_A$	$\mathrm{Bq \cdot l^{-1}}$
α, β	Probability of a false positive and false negative decision, respectively	-

$k_{1-\alpha}$	Quantile of the standardized normal distribution for the probability 1- α	-
$k_{1-\beta}$	Quantile of the standardized normal distribution for the probability 1- β	-
c_A^*	Decision threshold	$Bq \cdot l^{-1}$
$c_A^\#$	Detection limit	$Bq \cdot l^{-1}$
$c_A^{\triangleleft}, c_A^{\triangleright}$	Lower and upper limits of the probabilistically symmetric coverage interval	$Bq \cdot l^{-1}$
$c_A^{<}, c_A^{>}$	Lower and upper limits of the shortest coverage interval	$Bq \cdot l^{-1}$
$\gamma/2$	Probability of the measurand being smaller than $c_A^{\triangleleft}$ or larger than $c_A^{\triangleright}$	-
$1-\gamma$	Probability for the coverage interval of the measurand	-
Φ	Distribution function of the standardized normal distribution	-
ω	Distribution function of the standardized normal distribution of $c_A / u(c_A)$	-
k_p, k_q	Quantiles of the standardized normal distribution for the probabilities p and q, respectively	-
U	Expanded uncertainty, calculated by $U = k \cdot u(c_A)$ with $k = 1, 2, ...$	$Bq \cdot l^{-1}$

4 Principle

The method described is for measurement of ^{14}C in water samples by direct liquid scintillation counting. The general principles of this method are described in ISO 19361.

This direct determination is applicable to the analysis of water samples that can produce a homogeneous mixture between the test portion and a suitable scintillation cocktail.

The direct LSC method does not apply to waters containing micelles or large organic molecules (e.g. lipids, fulvic acid, humic acid, etc.) that do not form homogeneous mixtures with scintillation cocktails. In these cases, there is a risk that the beta particles could be attenuated. This reduces the counting efficiency of the system and hence the results can be underestimated. For these samples, the determination of ^{14}C requires additional chemical processing (such as chemical oxidation or combustion). Examples of methods of chemical preparation are described in Annexes A and B.

The choice of the analytical procedure (either with or without chemical preparation of the water sample prior to determination) depends on the aim of the measurement and the sample characteristics [17][18][19][20].

A prerequisite for the direct determination of ^{14}C in a water sample is the absence of, or a negligible contribution from, other beta-emitting radionuclides, such as ^{90}Sr and Ra isotopes. When the radionuclide content of the sample is unknown, the method specified in this document only provides a ^{14}C equivalent activity for the sample.

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”.

To determine the detection efficiency, it is necessary to measure a water sample having a known ^{14}C 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, or a water with a traceable ^{14}C activity usable without dilution.

Where chemical quenching can affect the measurement results, it is necessary to correct the counting data using a quench curve (see 7.4).

5 Sampling and storage

5.1 Sampling

Conditions of sampling and handling shall conform to ISO 5667-1, ISO 5667-3 and ISO 5667-10. Guidance is given for the different types of water in References [8] to [15].

The samples shall not be acidified to avoid the destruction of the carbonic equilibrium (CO_3^{2-}, HCO_3^-, H_2CO_3), as specified in ISO 5667-3. Basification of the sample is recommended, for example between pH 8 and 9.

It is important that the laboratory receives a representative sample, unmodified during transport or storage and in an undamaged container. It is recommended that a glass container filled to the maximum is used to minimize ^{14}C exchange with atmospheric CO_2.

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

5.2 Sample storage

If required, the sample shall be stored in accordance with ISO 5667-3 for carbon dioxide. If the storage duration exceeds that specified in ISO 5667-3, it is advisable to store the samples in glass containers.

6 Reagents and equipment

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

6.1 Reagents

6.1.1 Reference water for the blank

The reference water for the blank should be as free as possible of chemical or radioactive impurities, although it may have a low ^{14}C activity concentration, at the time the samples are measured.

For example, obtain water with a ^{14}C activity concentration as low as possible, e.g. (deep) subterranean water. Distil the water. Keep the distillate in a well-sealed borosilicate glass bottle in the dark at a temperature as constant as possible. This reference water shall be kept physically remote from any ^{14}C-containing material (see 6.1.2). Determine (7.4) the ^{14}C activity concentration of this water, in becquerel per litre, and note the date of this determination.

It is advisable to keep an adequate quantity of reference water in stock and to draw off small working volumes from it for immediate use, as required. Contamination with ^{14}C (e.g. from CO_2 in the air) or other radioactive species should be avoided.

For measurement of activity concentrations close to 1 $Bq \cdot l^{-1}$, water with a very low activity concentration is necessary as reference water.

6.1.2 Calibration source solution

In order to avoid cross-contamination, prepare the calibration source solution in a suitable location which is well removed from the area where the ^{14}C analyses are to be carried out. Transfer a known amount of ^{14}C aqueous standard solution into a volumetric flask (e.g. of capacity 100 ml). Make up to the mark with blank reference water and mix well. The calibration source solution shall have sufficient ^{14}C activity such that, when used to prepare counting sources, a suitable count rate to reach the required measurement uncertainty is obtained. Calculate the ^{14}C activity concentration of the resulting calibration source solution, in becquerel per litre. Note the date at which the standard solution was made up, to monitor the ageing of the solution.

It is recommended to select the standard source container size so as to minimize the volume of air above the solution and therefore minimize the exchange of ^{14}C with the atmosphere, when opening the container.

6.1.3 Scintillation solution

Chose the scintillation cocktail according to the characteristics of the test sample to be analysed (e.g. precipitate or alkaline) and according to the properties of the detection equipment [21][22].

It is recommended to use a hydrophilic scintillation cocktail for the direct measurement of environmental water or waste water.

The characteristics of the scintillation cocktail shall ensure the mixture is homogeneous and stable at the given mixing ratio and at the temperature of the counting system.

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

It is recommended to:

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

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

6.1.4 Quenching agent

Examples of chemical quenching agents (non-acid): organochloride compounds, nitromethane.

NOTE Some quenching agents are dangerous or toxic.

6.2 Equipment

Use the equipment specified in ISO 19361.

7 Procedure

7.1 Sample preparation

For the direct determination of ^{14}C in a raw sample, measurement of the test sample is generally performed without removal of any suspended matter if the sample has low levels of such material. 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 sampling.

For the measurement of high alkaline samples (pH above 9) with a low-level activity of ^{14}C, extract the carbon from the sample (for example, see Annex A).

7.2 Preparation of the counting vial

Known quantities of test sample and scintillation cocktail are transferred into a counting vial.

After closing the vial, shake thoroughly to homogenize the mixture.

The sample identification shall be written on the top of the vial lid. The sample may require storage before counting to allow photoluminescence or static electricity effects to decay. The storage