INTERNATIONAL STANDARD

ISO 13162

Second edition
2021-06

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

This document is a preview generated by EVS

Reference number
ISO 13162:2021(E)

© ISO 2021

This document is a preview generated by EVS



COPYRIGHT PROTECTED DOCUMENT

© ISO 2021

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

Contents

Page

Foreword ... **iv**
Introduction ... **v**
1 Scope ... **1**
2 Normative references ... **1**
3 Terms, definitions, symbols and abbreviations ... **2**
4 Principle ... **3**
5 Sampling and storage ... **4**
5.1 Sampling ... 4
5.2 Sample storage ... 4
6 Reagents and equipment ... **4**
6.1 Reagents ... 4
6.1.1 Reference water for the blank ... 4
6.1.2 Calibration source solution ... 4
6.1.3 Scintillation solution ... 5
6.1.4 Quenching agent ... 5
6.2 Equipment ... 5
7 Procedure ... **5**
7.1 Sample preparation ... 5
7.2 Preparation of the counting vial ... 5
7.3 Counting procedure ... 6
7.4 Calibration and verification ... 6
7.5 Measurement conditions ... 7
8 Expression of results ... **7**
8.1 General ... 7
8.2 Calculation of activity concentration without sample preparation ... 7
8.3 Calculation of activity concentration with sample preparation ... 8
8.4 Decision threshold ... 8
8.5 Detection limit ... 9
8.6 Limits of the coverage intervals ... 9
8.6.1 Limits of the probabilistically symmetric coverage interval ... 9
8.6.2 Limits of the shortest coverage interval ... 9
8.7 Calculations using the activity per mass ... 10
9 Test report ... **10**
Annex A (informative) **Extraction of total carbon by precipitation of calcium carbonate** ... **12**
Annex B (informative) **Extraction of total carbon: absorption counting** ... **15**
Annex C (informative) **Internal standard method** ... **18**
Annex D (informative) **Numerical applications** ... **20**
Bibliography ... **22**

This document is a preview generated by EVS

© ISO 2021 – All rights reserved

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.

This document is a preview generated by EVS

 © ISO 2021 – All rights reserved

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 $Bq{\cdot}l^{-1}$ 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 $Bq{\cdot}l^{-1}$ 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.

This document is a preview generated by EVS

© ISO 2021 – All rights reserved

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.

This document is a preview generated by EVS

© ISO 2021 – All rights reserved

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 $Bq{\cdot}l^{-1}$, which is lower than the WHO criteria for safe consumption of drinking water (100 $Bq{\cdot}l^{-1}$). ^{14}C activity concentrations can be measured up to 10^6 $Bq{\cdot}l^{-1}$ 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)*

This document is a preview generated by EVS

© ISO 2021 – All rights reserved

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	$kg \cdot l^{-1}$
c_A	Activity concentration	$Bq \cdot l^{-1}$
a	Activity per unit of mass	$Bq \cdot kg^{-1}$
A	Activity of the calibration source	Bq
A_s	Activity of the internal standard solution	Bq
n	Number of counting	-
N_o	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	s^{-1}
r_g	Test sample count rate	s^{-1}
r_s	Calibration count rate	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	$Bq \cdot l^{-1}$
u_{rel}	Relative standard uncertainty	-
$\tilde{c}_A$	Possible or assumed true quantity values of the measurand	$Bq \cdot l^{-1}$
$\tilde{u}(\tilde{c}_A)$	Standard uncertainty of $\tilde{c}_A$	$Bq \cdot l^{-1}$
α, β	Probability of a false positive and false negative decision, respectively	-

This document is a preview generated by EVS

© ISO 2021 – All rights reserved