INTERNATIONAL STANDARD

ISO 11704

Second edition
2018-11

Water quality — Gross alpha and gross beta activity — Test method using liquid scintillation counting

Qualité de l'eau — Activités alpha globale et bêta globale — Méthode d'essai par comptage des scintillations en milieu liquide

Reference number
ISO 11704:2018(E)

© ISO 2018



COPYRIGHT PROTECTED DOCUMENT

© ISO 2018

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
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

 © ISO 2018 – All rights reserved

Contents

Page

Foreword ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms, definitions, symbols and abbreviated terms **2**
3.1 Terms and definitions 2
3.2 Symbols and abbreviated terms 2
4 Principle **3**
5 Reagents and equipment **3**
6 Sampling **5**
7 Procedure **5**
7.1 Direct counting 5
7.2 Thermal preconcentration 5
7.3 Sample preparation 6
7.4 Liquid scintillation measurement 6
7.4.1 Preparation of alpha and beta calibration sources 6
7.4.2 Optimization of counting conditions 6
7.4.3 Blank sample preparation and measurement 7
7.4.4 Alpha and beta efficiencies 7
7.4.5 Sample measurement 8
8 Expression of results **8**
8.1 Calculation of activity per mass 8
8.2 Standard uncertainty 9
8.3 Decision threshold 10
8.4 Detection limit 10
8.5 Confidence limits 10
8.6 Quality control 11
9 Interference control **11**
9.1 Contamination 11
9.2 Ingrowth of radon 11
9.3 Loss of polonium 11
10 Test report **11**
Annex A (informative) **Set-up parameters and validation data** **13**
Annex B (informative) **Method performances under different conditions** **17**
Bibliography **18**

© ISO 2018 – 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*.

This second edition cancels and replaces the first edition (ISO 11704:2010), which has been technically revised. The main changes compared to the previous edition are as follows:

— 5.5.1 has been simplified;

— the application field of this document has been extended to emergency situations;

— slightly different counting conditions have been suggested;

— Annexes A and B have been added.

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.

© ISO 2018 – 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 water bodies 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[4] and ISO 5667-20[5].

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 0,5 Bq/l for gross alpha activity and 1 Bq/l for gross beta activity.

NOTE 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].

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][8].

Usually, the test methods can be adjusted to measure the activity concentration of the radionuclide(s) in either waste waters 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, waste waters/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 waste waters and liquid effluents with specific modifications that could increase the overall uncertainty, detection limit and threshold.

© ISO 2018 – All rights reserved

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).

An International Standard on a test method of gross alpha and gross beta 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 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.

© ISO 2018 – All rights reserved

Water quality — Gross alpha and gross beta activity — 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.

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 determination of gross alpha and gross beta activity concentration for alpha- and beta-emitting radionuclides using liquid scintillation counting (LSC).

The method is applicable to all types of waters with a dry residue of less than 5 g/l and when no correction for colour quenching is necessary.

Gross alpha and gross beta activity measurement is not intended to give an absolute determination of the activity concentration of all alpha- and beta-emitting radionuclides in a test sample, but is a screening analysis to ensure particular reference levels of specific alpha and beta emitters have not been exceeded. This type of determination is also known as gross alpha and beta index. Gross alpha and beta analysis is not expected to be as accurate nor as precise as specific radionuclide analysis after radiochemical separations.

The method covers non-volatile radionuclides below 80 °C, since some gaseous or volatile radionuclides (e.g. radon and radioiodine) can be lost during the source preparation.

The method is applicable to test samples of drinking water, rain water, surface and ground water as well as cooling water, industrial water, domestic and industrial waste water after proper sampling and test sample preparation (filtration when necessary and taking into account the amount of dissolved material in the water).

The method described in this document is applicable in the event of an emergency situation, because the results can be obtained in less than 4 h by directly measuring water test samples without any treatment.

It is the laboratory's responsibility to ensure the suitability 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 3696, *Water for analytical laboratory use — Specification and test methods*

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

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

© ISO 2018 – All rights reserved

3 Terms, definitions, symbols and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 80000-10 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/

3.2 Symbols and abbreviated terms

For the purposes of this document, the symbols and abbreviated terms defined in ISO 80000-10 and the following apply.

a_α, a_β	Alpha and beta activity per mass	Bq g^{-1}
a^*	Decision threshold	Bq g^{-1}
$a^\#$	Detection limit	Bq g^{-1}
$a^<$, $a^>$	Lower and upper limits of the confidence interval	Bq g^{-1}
A_α, A_β	Activity of the alpha and beta emitter certified reference solution used for the α and β calibration sources	Bq
m	Mass of the test sample	g
m_1	Mass of initial sample subject to heating or possibly concentration	g
m_2	Mass of heated or concentrated sample	g
m_3	Mass of heated or concentrated sample transferred in the vial	g
$m_{S\alpha}$, $m_{S\beta}$	Mass of alpha and beta emitters certified reference solutions, respectively	g
$r_{g\alpha}$, $r_{g\beta}$	Sample gross count rate, from the alpha and beta windows, respectively	s^{-1}
$r_{0\alpha}$, $r_{0\beta}$, r_{0T}	Blank count rate, from the alpha, beta and total windows, respectively	s^{-1}
$r_{S\alpha,\alpha}$, $r_{S\alpha,\beta}$, $r_{S\alpha,T}$	Count rate of the alpha calibration source in the alpha, beta and total window	s^{-1}
$r_{S\beta,\alpha}$, $r_{S\beta,\beta}$, $r_{S\beta,T}$	Count rate of the beta calibration source in the alpha, beta and total window	s^{-1}
t_g	Sample counting time	s
t_0	Blank counting time	s
$t_{s\alpha}$, $t_{s\beta}$	Counting time of α and β calibration sources	s
$u\,(a)$	Standard uncertainty associated with the measurement result	Bq g^{-1}
U	Expanded uncertainty, calculated from $U = ku\,(a)$, where $k = 1, 2 \ldots$	Bq g^{-1}
$\tilde{u}(\tilde{a}_\alpha)$	Standard uncertainty of a_α as a function of its true value	Bq g^{-1}

© ISO 2018 – All rights reserved

ε_α, ε_β	Counting efficiency for alpha and beta, respectively	—
$\tau_\alpha(\chi_{\alpha\,\beta})$	Alpha interference — Fraction of counts observed in the beta window with respect to the total number of counts measured by the counter when an alpha emitter is measured	—
$\tau_\beta(\chi_{\beta\,\alpha})$	Beta interference — Fraction of counts observed in the alpha window with respect to the total number of counts measured by the counter when a beta emitter is measured	—

4 Principle

Gross alpha and beta activity concentrations are determined by using liquid scintillation counting of a water sample mixed with a scintillation cocktail.

Gross alpha and beta determinations are not absolute determinations of the sample radioactive contents, but relative determinations referred to a specific alpha or beta emitter which constitutes the standard calibration sources. These types of determinations are also known as the alpha and beta index and are usually employed as screening parameters for first assessment of total radioactive content.

The aqueous sample is acidified using nitric acid and heated. Subsequently, water with low salt content can be thermally concentrated by slow evaporation to improve the method sensitivity. An aliquot of sample is transferred into a liquid scintillation vial with scintillation cocktail; scintillations from the vial are then counted by equipment with an alpha and beta discrimination device.

The counter is previously optimized with respect to an alpha and beta discriminator setting and then calibrated against alpha and beta emitter certified reference solutions. In data evaluation, no correction for chemical quenching is applied, since the procedure is designed to provide samples with a relatively constant quench level.

The method does not account for ^{222}Rn and its short lived progeny and it is not suitable for ^{3}H measurement.

When suspended matter is present in significant quantities, a filtration step is required before acidification.

5 Reagents and equipment

All reagents shall be of recognized analytical grade, except for the scintillation cocktail, and shall not contain any detectable alpha and beta activity, except for the radioactive certified reference solutions.

5.1 Nitric acid, $c(HNO_3)$ = commercially available acid with mass fraction $w(HNO_3)$ = (65 to 70) %.

5.2 Water, ISO 3696, grade 3.

Deionized water can contain detectable amounts of ^{222}Rn and short lived progeny. It is therefore strongly recommended to boil water under vigorous stirring and let it stand for one day before use. Alternatively, use nitrogen flushing for about 1 h for a 2 l sample.

5.3 Scintillation cocktail.

Commercially available scintillation cocktails suitable for alpha and beta discrimination (e.g. diisopropylnaphthalene-based cocktails), water miscible.

5.4 Volatile organic solvents.

Methanol or ethanol.

© ISO 2018 – All rights reserved