DRAFT INTERNATIONAL STANDARD ISO/DIS 13168

ISO/TC **147**/SC **3** Secretariat: **AFNOR**

Voting begins on
2013-07-03

Voting terminates on
2013-10-03

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Water quality — Simultaneous determination of tritium and carbon 14 activities — Test method using liquid scintillation counting

Qualité de l'eau — Détermination simultanée des activités volumiques du tritium et du carbone 14 — Méthode par comptage des scintillations en milieu liquide

ICS 13.060.60; 13.280

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

Pour accélérer la distribution, le présent document est distribué tel qu'il est parvenu du secrétariat du comité. Le travail de rédaction et de composition de texte sera effectué au Secrétariat central de l'ISO au stade de publication.

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

ISO 13168 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

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 potassium 40, and those originating from the thorium and uranium decay series, in particular radium 226, radium 228, uranium 234, uranium 238, lead 210, can be found in water for natural reasons (e.g. desorption from the soil and wash-off 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 fertilizer production and use);

- human-made radionuclides, such as transuranium elements (americium, plutonium, neptunium, curium), tritium, carbon 14, strontium 90 and gamma emitting radionuclides can also be found in natural waters as a result of authorized routine releases into the environment in small quantities in the effluent discharged from nuclear fuel cycle facilities. They are also released into the environment following their used in unsealed form for medical and industrial applications. They are also found in the water as a result of past fallout contamination resulting from the explosion in the atmosphere of nuclear devices and accidents such as those that occurred in Chernobyl and Fukushima.

Drinking-water may thus contain radionuclides at activity concentrations which could present a risk to human health. In order to assess the quality of drinking-water (including mineral waters and spring waters) with respect to its radionuclide content and to provide guidance on reducing health risks by taking measures to decrease radionuclide activity concentrations, water resources (groundwater, river, lake, sea, etc.) and drinking water are monitored for their radioactivity content as recommended by the World Health Organization [WHO].

An international standard on a test method of simultaneous measurement of tritium and carbon 14 concentrations in water samples is justified for test laboratory 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.

Tritium and carbon 14 natural activity concentration can vary according to local geological and climatic characteristics, at a level below 5 Bq/L and below 0,1 Bq/L respectively. These radioactivity levels can be locally enhanced by nuclear installation authorized discharges of low level radioactive effluent into the environment. The guidance level for tritium and carbon 14 in drinking water as recommended by WHO is 10 000 and 100 Bq/L respectively [1].

NOTE The guidance level is the activity concentration with an intake of 2 litres/day of drinking water for 1 year, that results in an effective dose of 0,1 mSv/year for members of the Public, an effective dose that represents a very low level of risk that is not expected to give rise to any detectable adverse health effect.

This standard is one of a set of International Standards on test methods dealing with the measurement of the activity concentration of radionuclides in water samples.

Water quality — Simultaneous determination of tritium and carbon 14 activities — Test method using liquid scintillation counting

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This 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 document applies to liquid scintillation counters and requires the preparation of a scintillation source obtained by mixing the sample and a hydrophilic scintillation cocktail.

This document describes the conditions for measuring the activity of pure Beta emitters by liquid scintillation, these conditions being adapted to the simultaneous measurement of tritium and carbon 14 present in water samples taken from the environment.

This document is applicable to all types of waters having an activity concentration ranging from 5 Bq.l^{-1} to 10^{6} Bq.l^{-1} (upper limit of the liquid scintillation counters for direct counting). It is possible to produce a dilution in order to obtain a solution having an activity compatible with the measuring instrument.

This document is applicable to both raw and filtered waters according to EN 872.

The measurement range is related to the methodology being used: nature of test portion, preparation of the scintillator - test portion mixture, measuring assembly.

Furthermore, the measurement range depends upon the activity levels of other interfering nuclides in the sample.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 programs 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 11929, *Determination of the characteristic limits (decision threshold, detection limit and limits of the confidence interval) for measurements of ionizing radiation - Fundamentals and application*.

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

ISO/IEC Guide 98-3:2008, *Guide to the expression of uncertainty in measurement*.

ISO/IEC Guide 99:2007, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*.

EN 872, *Water quality — Determination of suspended solids — Method by filtration through glass fibre filters*.

3 Symbols, definitions and units

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

β_{max}	Maximum energy for the beta emission, in keV
V	Volume of test sample, in litre
m	Mass of test sample, in kilogram
ρ	Density of the sample, in kilogram per litre
c_A	Activity concentration, in becquerel per litre
a	Activity per unit of mass, in becquerel per kilogram
A_T, A_C	Activity of the calibration source, in becquerel, respectively for tritium and carbon 14
t_0	Background counting time, in second
t_g	Sample counting time, in second
t_s	Calibration counting time, in second
r_{0T}, r_{0C}	Mean background count rate, per second, respectively for tritium and carbon 14
r_{gT}, r_{gC}	Mean sample count rate, per second, respectively for tritium and carbon 14
r_{sT}, r_{sC}	Calibration count rate, per second, respectively for tritium and carbon 14
$r_{sC \to T}$	Calibration count rate, per second, for the interfering carbon 14 in the chosen window of the tritium energy range
ε_q	Detection efficiency for the quenching parameter q
ε	Detection efficiency for the lowest value of the quenching parameter
ε_T, ε_C	Detection efficiency, respectively for tritium and carbon 14
$\varepsilon_{C \to T}$	Detection efficiency for carbon 14 in the chosen window of the tritium energy range

χ	Correcting factor, for the interfering carbon 14 in the chosen window of the tritium energy range
f_{qT}, f_{qC}	Quench factor, respectively for tritium and carbon 14
$f_{\mathrm{qT}\rightarrow\mathrm{C}}$	Quench factor, for the interfering carbon 14 in the chosen window of the tritium energy range
$u(c_{\mathrm{A}})$	Standard uncertainty associated with the measurement result; in becquerel per litre
U	Expanded uncertainty, calculated by $U = k \cdot u(c_A)$ with k = 1, 2,…, in becquerel per litre
c_{A}^{*}	Decision threshold, in becquerel per litre
$c_{\mathrm{A}}^{\#}$	Detection limit, in becquerel per litre
$c_{\mathrm{A}}^{\triangleleft}$, $c_{A}^{\triangleright}$	Lower and upper limits of the confidence interval, in becquerel per litre

4 Principle

The test sample is mixed with the scintillation cocktail in a counting vial to obtain a homogeneous medium. Electrons emitted by the radionuclide 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 analyzed by the electronic systems and the data analysis software. The count rate of these photons allows the determination of the test sample 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 sample. The blank sample is prepared using a reference water of the lowest activity available, also sometimes called “dead water”.

In order to determine the detection efficiencies, it is necessary to measure a water having known tritium and carbon 14 activities under conditions that are identical to those used for the sample. This water shall be a mixture of certified radioactive sources or a dilution of this mixture produced with the reference water.

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

- same type of counting vial;
- same filling geometry;
- same ratio between test sample and scintillation cocktail;
- temperature stability of the detection equipment;
- value of quench indicating parameter included in calibration curve.

NOTE Quench correction: if particular conditions of chemical quenching affect the measurement results, it is recommended to correct the result using a quench curve. It is important to choose the chemical quenching agent according to the supposed type of quenching observed in the sample, but not acid if the carbon 14 in the standard solution is of a carbonated species. This method is not applicable to colour quenched samples.

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 [2] and with an activity concentration of tritium and carbon 14 negligible in comparison with the activities to be measured.

For example, a water sample with a low tritium and carbon 14 activity concentration can be obtained from (deep) subterranean water kept in a well-sealed borosilicate glass bottle in the dark at controlled temperature (ISO 5667-3). This blank water sample shall be kept physically remote from any tritium and carbon 14 containing material (Note 1). Determine the tritium and carbon 14 activity concentration (t = 0), in Bq/l, of this water and note the date (t = 0) of this determination (see Note 2).

NOTE 1 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) and carbon 14 (air CO_2) or other radioactive species should be avoided.

NOTE 2 As the activity is becoming non-negligible for activities around 1 Bq/l, it is necessary to use a blank water measured to ensure the “absence” of tritium and carbon 14. The tritium activity concentration in the blank water can be determined by enrichment followed by liquid scintillation counting or from the measurement of ^{3}He by mass spectrometry. Preferably use blank water with a tritium activity concentration of less than 0,5 Bq/l. The carbon 14 activity concentration in the blank water can be determined by techniques such as the synthesis of benzene or by accelerated mass spectrometry.

When the volume of blank water is sufficiently large, e.g. 10 l to 20 l, and well-sealed, tritium and carbon 14 activity concentrations remain stable for years, although it is advisable to determine these activity concentrations at predetermined intervals, e.g. every year.

5.1.2 Calibration source solutions

In order to avoid cross-contamination prepare, in a suitable location which is remote from the area where the 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 ($[^3H]H_2O$) standard solution and of a concentrated carbon 14 standard solution, so that the tritium and carbon 14 activity concentrations generate sufficient counts to reach the required measurement uncertainty after dilution with blank water and thorough mixing. Calculate the activity concentrations 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 the measurement time t of the samples shall be corrected for radioactive decay.

5.1.3 Scintillation solution

The scintillation cocktail is chosen according to the characteristics of the sample to be analyzed and according to the properties of the detection equipment [3].

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

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

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.