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Kakovost vode - Določevanje aktivnosti ogljika C-14 - Metoda štetja s tekočinskim scintilatorjem

Water quality - Determination of carbon 14 activity - Liquid scintillation counting method

Qualité de l'eau - Détermination de l'activité volumique du carbone 14 - Méthode par comptage des scintillations en milieu liquide

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Water quality — Determination of carbon 14 activity — Liquid scintillation counting method

Qualité de l'eau — Détermination de l'activité volumique du carbone 14 — 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 13162 was prepared by Technical Committee ISO/TC 147, *Water quality*.

Introduction

The carbon 14 (^{14}C) 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 ^{14}C have been released into the environment. Due to the substantial proportion of ^{14}C in the human internal dose contribution, monitoring of ^{14}C activity concentrations in the environment is necessary in order to follow its circulation in the hydrosphere and biosphere. ^{14}C is the second radionuclide (~3 500 Bq) to contribute to the human body natural radioactivity, behind ^{40}K (~6 000 Bq).

Water quality — Determination of carbon 14 activity — Liquid scintillation counting method

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 International Standard specifies the conditions for the determination of ^{14}C activity concentration in samples of environmental water or of ^{14}C-containing water using liquid scintillation counting.

The method is applicable to the analysis of any organic molecule soluble in water that is well mixed with the scintillation cocktail. It does not apply to micelles or "large" particles (lipids, fulvic acid, humic acid, etc.) that are inadequately mixed with the scintillation cocktail and the water. Some beta energy is lost without any excitation of the scintillation cocktail and the results are underestimated. The method is not applicable to the analysis of organically bound ^{14}C, whose determination requires additional chemical processing (such as chemical oxidation, combustion).

It is possible to determine ^{14}C activity concentrations below 10^6 Bq l^{-1} without any sample dilution.

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: Preservation and handling of water samples*

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

ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Symbols, definitions, units, and abbreviations

For the purposes of this document, the symbols, definitions, units, and abbreviations defined in ISO 80000-10, ISO 11929, ISO/IEC Guide 98-3 and the following apply.

A	Activity of the calibration source, in becquerels
c_A	Activity concentration, in becquerels per litre
c_A^*	Decision threshold, in becquerels per litre
$c_A^\#$	Detection limit, in becquerels per litre
$c_A^\triangleleft$, $c_A^\triangleright$	Lower and upper limits of the confidence interval, in becquerels per litre
f_q	Quench factor
m	Mass of test sample, in kilograms
r_0	Background count rate, in reciprocal seconds
r_g	Sample count rate, in reciprocal seconds
r_s	Count rate of the calibration sample, in reciprocal seconds
t_0	Background counting time, in seconds
t_g	Sample counting time, in seconds
t_s	Counting time of the calibration sample, in seconds
U	Expanded uncertainty, calculated by $U = k\,u(c_A)$ with k = 1, 2, …, in becquerels per litre
$u(c_A)$	Standard uncertainty associated with the measurement result, in becquerels per litre
V	Volume of test sample, in litres
α	Activity per mass, in becquerels per kilogram
β_{max}	Maximum energy for the beta emission, in kiloelectronvolts
ε	Detection efficiency
ρ	Mass density of the sample, in kilograms per litre

4 Principle

The scintillation phenomenon results from interaction of ionizing radiation with solvents and compounds exhibiting fluorescence (scintillators). Both solvents and scintillators constitute the scintillation cocktail. The scintillation mixture is achieved by adding the scintillation cocktail to the test sample in order to obtain a homogeneous mixture.

The test sample is mixed with the scintillation cocktail in a counting vial to obtain a homogeneous medium. Electrons emitted by ^{14}C 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 electric pulses allows the determination of the test sample activity, after correcting for the background count rate and detection efficiency.

In order to determine the background, 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, in accordance with the activities to be measured.

The detection efficiency is determined with a calibration sample that is prepared with a standard of aqueous ^{14}C, or a dilution of this standard with reference water, measured under the same conditions as the test sample.

The sample (blank, test, calibration) and the measurement conditions shall be:

— same type of counting vial;

— same filling geometry;

— same scintillation cocktail;

— same ratio between test sample and scintillation cocktail;

— temperature stability of the detection apparatus;

— value of quench-indicating parameter included in calibration curve.

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 International Standard only provides a ^{14}C equivalent activity for the sample.

Examples of methods of sample pretreatment are described in Annexes C and D.

Concerning quench correction, if particular conditions of chemical quenching affect the measurement results, it is recommended that a quench curve be established. It is important to choose the chemical quenching agent in accordance with the supposed type of quenching observed in the sample.

5 Reagents and equipment

5.1 Reagents

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

5.1.1 Reference water for the blank

The reference water for the blank should be as free as possible of chemical or radioactive impurities.

The reference water may have a low ^{14}C activity concentration, in becquerels per litre, at the time t at which 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 next paragraph). Determine (see final paragraph) the ^{14}C activity concentration ($t = 0$), in becquerels per litre, of this water and note the date ($t = 0$) 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 l^{-1}, water with a very low activity concentration is necessary as reference water.