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Water quality — Uranium isotopes — Test method using alpha-spectrometry

Qualité de l'eau — Isotopes de l'uranium — Méthode d'essai par spectrométrie alpha

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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, *Radiological methods.*

This second edition cancels and replaces the first edition (ISO 13166:2014), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— update of the common introduction;

— update of the text considering the new ISO 11929 series published in 2019.

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.

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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, and ^{210}Pb, 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), ^{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 the 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 water is monitored for its radioactivity content as recommended by the World Health Organization (WHO)[3] so that proper actions can be taken to ensure that there is no adverse health effects to the public. Following these international recommendations, national regulation 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 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 guidance level in drinking water for ^{238}U and ^{234}U is 10 and 1 $Bq \cdot l^{-1}$, respectively. The provisional guideline value for the concentration of uranium in drinking water is 30 $\mu g \cdot l^{-1}$ based on its chemical toxicity, which is predominant compared with its radiological toxicity.

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 be greater.

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].

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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 answer 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 family 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 — Uranium isotopes — Test method using alpha-spectrometry

WARNING — Persons using this document should be familiar with normal laboratory practices. 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 the conditions for the determination of uranium isotope activity concentration in samples of environmental water (including sea waters) using alpha-spectrometry and ^{232}U as a yield tracer.

A chemical separation is required to separate and purify uranium from a test portion of the sample.

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 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 11929-3, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 3: Applications to unfolding methods*

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

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

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

3 Terms, definitions and symbols

For the purposes of this document, the terms, definitions, and symbols given in ISO 80000-10, ISO 11929-1 and the following apply.

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

— IEC Electropedia: available at http://www.electropedia.org/

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— ISO Online browsing platform: available at http://www.iso.org/obp

Table 1 — Symbols and definitions

Symbol	Definition
A	activity of ^{232}U tracer added, in becquerel, at the date of measurement
c_A	activity concentration of ^{238}U or ^{235}U or ^{234}U, 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 probabilistically symmetric coverage interval, in becquerels per litre
$c_A{}^<$, $c_A{}^>$	lower and upper limits of the shortest coverage interval, in becquerels per litre
R	total measurement yield
r_0, r_{0T}	background count rate per second for the uranium analytes and tracer in the respective regions of interest (ROI) of the blank sample spectrum
R_c	chemical yield
r_g, r_{gT}	gross count rate per second for the uranium analytes and tracer in the respective regions of interest (ROI) of the test sample spectrum
t_0	background counting time, in seconds
t_g	sample counting time, in seconds
U	expanded uncertainty, calculated by $U = ku(c_A)$ with $k = 1, 2 \ldots$, 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
ε	counting efficiency

4 Principle

The test sample is mixed with an aliquot of ^{232}U tracer, followed by equilibration of the sample prior to analysis. Chemical isolation of uranium is achieved by a concentration step (e.g. a precipitation) followed by a specific separation step (e.g. using ion exchange chromatography).

The detection limit for measurement of a test portion of about 500 ml is approximately 5 mBq · l^{-1} with a counting time of about 200 000 s.

Natural radionuclides such as ^{230}Th, ^{226}Ra and ^{228}Th can be present in water and can interfere with the counting of uranium isotopes if no chemical separation is carried out to remove these radionuclides from the test portion. Plutonium isotopes can also interfere, if present with detectable activities in the sample.

The measured thin source is prepared by electrodeposition or coprecipitation and measured by alpha-spectrometry using a grid chamber or a semiconductor-type apparatus. Measurements rely on the interaction of alpha-particles with the detecting medium. This interaction creates a charge, which is amplified and output as a voltage pulse proportional to the deposited energy of the incoming alpha-particle.

The electric pulse from the detector is analysed by the electronic systems. Data analysis software provides a spectrum, in which the number of pulses (counts) recorded in each energy interval is shown.

The analysis of the count rates in the uranium isotopes alpha-energy windows allows the determination of the test sample activity concentration for ^{238}U, ^{235}U and ^{234}U, after correcting for the blank count rate, volume of the test sample and the total measurement yield (chemical yield and detection efficiency).

The chemical yield and detection efficiency are not necessarily determined separately, but are determined together by measuring the total measurement yield from the net count rate of ^{232}U, added as a chemical yield tracer.

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