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Water quality — Thorium 232 —

Part 1: Test method using alpha spectrometry

Qualité de l'eau — Thorium 232 —

Partie 1: 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 document 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).

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

A list of all parts in the ISO 4722 series can be found on the ISO website.

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.

Introduction

Radionuclides are present throughout the environment; thus, water bodies (e.g. surface waters, ground waters, sea waters) contain radionuclides, which can be of either natural or anthropogenic origin.

- Naturally-occurring radionuclides, including ^3H , ^{14}C , ^{40}K and those originating from the thorium and uranium decay series, in particular ^{210}Pb , ^{210}Po , ^{222}Rn , ^{226}Ra , ^{228}Ra , ^{227}Ac , ^{231}Pa , ^{234}U and ^{238}U , can be found in water bodies due to either natural processes (e.g. desorption from the soil, runoff by rain water) or released from technological processes involving naturally occurring radioactive materials (e.g. mining, mineral processing, oil, gas, and coal production, water treatment, and production and use of phosphate fertilisers).
- Anthropogenic radionuclides such as ^{55}Fe , ^{59}Ni , ^{63}Ni , ^{90}Sr , ^{99}Tc , transuranic elements (e.g. Np, Pu, Am and Cm) and some gamma emitting radionuclides such as ^{60}Co and ^{137}Cs can also be found in natural waters. Small quantities of anthropogenic radionuclides can be discharged from nuclear facilities to the environment as a result of authorized routine releases. The radionuclides present in liquid effluents are usually controlled before being discharged to the environment^[1] and water bodies. Anthropogenic radionuclides used in medical and industrial applications can be released to the environment after use. Anthropogenic radionuclides are also found in waters due to contamination from fallout resulting from above-ground nuclear detonations and accidents such as those that have occurred at the Chernobyl and Fukushima nuclear facilities.

Radionuclide activity concentrations in water bodies can vary according to local geological characteristics and climatic conditions and can be locally and temporally enhanced by releases from nuclear facilities during planned, existing and emergency exposure situations.^{[2][3]} Some drinking water sources can thus contain radionuclides at activity concentrations that can present a human health risk. The World Health Organization (WHO) recommends to routinely monitor radioactivity in drinking waters^[4] and to take proper actions when needed to minimize the health risk.

National regulations usually specify the activity concentration limits that are authorized in drinking waters, water bodies and liquid effluents to be discharged to the environment. These limits can vary for planned, existing, and emergency exposure situations. As an example, during either a planned or existing situation, the WHO guidance level for ^{232}Th in drinking water is $1 \text{ Bq}\cdot\text{l}^{-1}$, see NOTE. Compliance with these limits is assessed by measuring radioactivity in water samples and by comparing the results obtained, with their associated uncertainties, as specified by ISO/IEC Guide 98-3^[5] and ISO 5667-20^[6].

NOTE The guidance level calculated in Reference ^[4] is the activity concentration that, with an intake of $2 \text{ l}\cdot\text{d}^{-1}$ of drinking water for one year, results in an effective dose of $0,1 \text{ mSv}\cdot\text{a}^{-1}$ to members of the public. This is an effective dose that represents a very low level of risk to human health and which is not expected to give rise to any detectable adverse health effects^[4].

In the event of a nuclear emergency, the WHO Codex Guideline Levels^[7] states that the activity concentration can possibly be not greater than 1 Bq/l and 10 Bq/l for ^{232}Th , respectively, for infants and other than infants.

This document contains a method to determine ^{232}Th in water samples. The test method has been developed to support laboratories to determine ^{232}Th in water samples.

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

The methods described in this document can be used for various types of waters (see [Clause 1](#)). Minor modifications such as sample volume and counting time can be made if needed to ensure that the characteristic limit, decision threshold, detection limit and uncertainties are below the required limits. This can be done for several reasons such as emergency situations, lower national guidance limits and operational requirements.

Water quality — Thorium 232 —

Part 1: Test method using alpha spectrometry

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all 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 essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies the method and the conditions for the determination of ^{232}Th activity concentration in samples of environmental water (including sea waters) and waste waters before release to the environment using alpha spectrometry and ^{229}Th as a recovery tracer. A chemical separation allows to separate and purify thorium from a test portion of the sample.

The general principles outlined in this document can be applied for the analysis of other alpha-emitting thorium isotopes such as ^{228}Th and ^{230}Th in aqueous samples.

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

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

3 Terms, definitions and symbols

3.1 Terms and definitions

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

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

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <https://www.electropedia.org/>

3.2 Symbols

Symbols used in this document are listed in [Table 1](#).

Table 1 — Symbols

Symbol	Description	Unit
A	Activity of the ^{229}Th tracer added	Bq
c_A	Activity concentration of ^{232}Th measured in the sample	$\text{Bq}\cdot\text{l}^{-1}$
c_A^*	Decision threshold	$\text{Bq}\cdot\text{l}^{-1}$
$c_A^\#$	Detection limit	$\text{Bq}\cdot\text{l}^{-1}$
$c_A^<, c_A^>$	Lower and upper coverage limits	$\text{Bq}\cdot\text{l}^{-1}$
$c_A^{<}, c_A^{>}$	Lower and upper shortest coverage interval	$\text{Bq}\cdot\text{l}^{-1}$
\tilde{c}_A	Possible or assumed true quantity values of the measurand	$\text{Bq}\cdot\text{l}^{-1}$
k_p	Quantiles of the standardized normal distribution for the probabilities p (for instance, $p = 1-\alpha$, $p = 1-\beta$ or $p = 1-\gamma/2$)	—
p	Probability p , the probability for “success” in a binominal distribution	—
q	Probability q , the probability for “failure” in a binominal distribution	—
R	Total recovery	—
R_c	Chemical recovery	—
r_g	Gross count rate in the region of interest of ^{232}Th , the isotope to measure	s^{-1}
r_{gT}	Background count rate in the region of interest of the ^{232}Th , the isotope to measure	s^{-1}
r_T	Gross count rate in the region of interest of the ^{229}Th tracer	s^{-1}
r_{0T}	Background count rate in the region of interest of the ^{229}Th tracer	s^{-1}
r_0	Blank value or background value of the detector, if similar	s^{-1}
t_0	Counting time of the background by alpha spectrometry	s
t_g	Sample counting time by alpha spectrometry	s
U	Expanded uncertainty	—
u_c	Standard uncertainty	—
$u(c_A)$	Standard uncertainty of the activity concentration of ^{232}Th	$\text{Bq}\cdot\text{l}^{-1}$
$\tilde{u}(\tilde{c}_A)$	Standard uncertainty of the activity concentration of ^{232}Th	$\text{Bq}\cdot\text{l}^{-1}$
u_{rel}	Standard uncertainty of the estimator c_A as a function of its detection limit	—
w	standard variance	—
V	Sample volume	l
α	Probability of the false positive decision	—
β	Probability of the false negative decision	—
ε	Counting efficiency	—
Φ	Distribution function of the standardized normal distribution	
ω	Auxiliary quantity	

4 Principle

The test sample is mixed with an aliquot of ^{229}Th tracer, followed by equilibration of the sample prior to analysis, chemical isolation of thorium by a concentration step (e.g. a precipitation) and a specific separation step (e.g. using ion exchange chromatography).

Uranium isotopes, ^{210}Po , ^{237}Np , ^{238}Pu or ^{241}Am can be present in water and can interfere with the counting of ^{232}Th and the ^{229}Th tracer if no chemical separation is carried out to remove these radionuclides from the water sample.

The measured thin source is prepared by electrodeposition or co-precipitation 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 ^{232}Th alpha energy window allows the determination of the test sample activity concentration for ^{232}Th , after correcting for the blank count rate, the volume of the test sample and the total measurement recovery (chemical recovery and detection efficiency).

The chemical recovery and detection efficiency are not necessarily determined separately but are determined together by measuring the total measurement recovery from the net count rate of ^{229}Th , added as a chemical recovery tracer.

For quality control and to quantify potential impurities in the tracer solution, a blank sample shall be prepared using grade 3 laboratory water in compliance with ISO 3696 with the addition of tracer. The radioactive characteristics of the main thorium isotopes are given in [Table 2](#) (see References [8], [9] and [10]).

Table 2 — Characteristics of the main thorium isotopes

Thorium isotope	Half-life	Main alpha particle emission energy (uncertainty)	Intensity
	years (uncertainty)	keV	%
228	1,912 6 (9)	5 340,35 (22)	26,0
		5 423,24 (22)	73,4
229	7 880 (12)	4 845,3 (12)	56,2
		4 901,0 (12)	10,2
230	75 380 (30)	4 687,0 (15)	76,3
		4 620,5 (15)	23,4
232	$14,02 (6) \times 10^9$	4 011,2 (14)	21,0
		3 948,5 (14)	78,9

5 Chemical reagents and equipment

5.1 General

The chemical reagents and equipment used for chemical treatment and preparation of the source are described in [Annexes A](#) to [C](#), which give various options. Where there are choices, at least one of the options presented shall be used.

Use only reagents of recognized analytical grade.

5.2 Chemical reagents

5.2.1 Laboratory water, used as a blank, as free as possible of chemical or radioactive impurities (e.g. uranium or thorium isotopes), complying with ISO 3696, grade 3.

Unless otherwise stated, water refers to deionised water.

5.2.2 Tracer solution containing ^{229}Th can be used to determine the total recovery. It can also be used to calculate the chemical recovery. The solution is prepared by the dilution of a suitable standard that provides traceability to national and international standards. The tracer solution shall be homogeneous and stable.

The tracer solution concentration should be calculated to allow adding a small amount of this solution to be in the range of activity contained in the test portion. For example, the tracer solution concentration can be between $0,05 \text{ Bq}\cdot\text{g}^{-1}$ and $1 \text{ Bq}\cdot\text{g}^{-1}$.

It is recommended that the activity and the purity of the tracer solution dilution be checked before use and at regular intervals after preparation. This can be done, for example, by liquid scintillation counting, but an account needs to be taken of progeny radionuclide ingrowth. Performing a blank analysis with tracer is a potential way to identify any presence of thorium isotope analytes in the tracer.

5.3 Equipment

Usual laboratory apparatus and, in particular, the following.

5.3.1 Alpha spectrometer of the grid chamber (with higher detection recovery but lower resolution) or semiconductor type (with lower detection recovery, but higher resolution). Operation at constant temperature is recommended. Follow the manufacturer's instructions.

For semiconductor-type apparatus, the measurements using alpha spectrometry depend on the interaction of alpha particles with ion-implanted silicon. This interaction instantly changes the conductivity of the silicon, proportional to the energy of the incoming alpha particle. To achieve well-resolved spectra, the detection system needs to be maintained at a pressure $<1 \text{ Pa}$. Resolution can be further enhanced by increasing the distance between the source and the detector. However, it has to be noted that counting efficiency will decrease which increases the measurement time.

5.3.2 Pipette, suitable for the accurate transfer of (e.g. $100 \mu\text{l}$) ^{229}Th tracer solution with a total precision within $\pm 1 \%$.

5.3.3 Balance capable of achieving $\pm 0,1 \text{ mg}$ precision.

6 Sampling and samples

6.1 Sampling

Conditions of sampling shall conform to ISO 5667-1.

Filter the sample to remove solids and then acidify to pH less than 2 with nitric acid as soon as possible after sampling prior to analysis, as specified in ISO 5667-3. Acidification prior to filtration can result in leaching of thorium from the solid component of the sample.

It is important that the laboratory receive a representative sample, unmodified during transport or storage and in an undamaged container.

6.2 Sample storage

If the sample is not going to be analysed immediately, the sample shall be stored in accordance to ISO 5667-3.