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Water quality — Plutonium, americium, curium and neptunium — Test method using alpha spectrometry

Qualité de l'eau — Plutonium, americium, curium and neptunium — Méthode d'essai par spectrométrie alpha

~~Second edition~~

~~Date: 2022-12-23~~

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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~~ 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~~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 ~~on~~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~~Organization (WTO) principles in the Technical Barriers to Trade (TBT~~)~~), see ~~the following URL: www.iso.org/iso/foreword.html~~www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 13167:2015), which has been technically revised.

The main ~~change is the~~ changes are as follows:

— addition of a description for determination of bias in the chemical recoveries of americium and curium.

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 —~~ Naturally-occurring radionuclides, including ^{3}H, ^{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 the production and use of phosphate fertilisers~~);~~).

~~anthropogenic —~~ 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 Chornobyl 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 ~~could~~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 ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Am, ^{243}Cm, ^{244}Cm, ^{237}Np in drinking water is 1 Bq·l^{-1}[3]. For ^{242}Cm the GL is 10 Bq·l^{-1}.Bq·l^{-1}[3]. 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 1 If the value is not specified in Annex 6 of Reference [4], the value has been calculated using the formula provided in Reference [4] and the dose coefficient data from References [7] and [8].

NOTE 2 The guidance level calculated in Reference [4] is the activity concentration that, with an intake of 2 l·d^{-1} of drinking water for one year, results in an effective dose of 0,1 mSv·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]~~.~~

This document contains methods to determine ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Am, ^{242}Cm, ^{243}Cm, ^{244}Cm, ^{237}Np in water samples. It has been developed to support laboratories that need either a certification or accreditation to determine these nuclides in water samples. A certification or accreditation are sometimes required by local and national authorities as well as some customers. The certification and accreditation are provided by an independent body.

The methods described in this document can be used for various types of waters (see Scope section).. 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.

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Water quality — Plutonium, americium, curium and neptunium — 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 essential that tests conducted according to this test method be carried out by suitably trained staff.

1 Scope

This document specifies a test method for measuring actinides (^{238}Pu, $^{239+240}$Pu, ^{241}Am, ^{242}Cm, $^{243+244}$Cm and ^{237}Np) in water samples by alpha spectrometry following a chemical separation.

This method can be used for any type of environmental study or monitoring after appropriate sampling and handling, and test sample preparation.

The detection limit of the test method is 5 × 10−3 to 5 × 10−4 Bq l^{-1} for a volume of test portion between 0,1 l to 5 l with a counting time of two to ten days. This is lower than the WHO criteria for safe consumption of drinking water (1 or 10) Bq·l^{-1} depending on radionuclide[4].

The methods described in this document ~~is~~are applicable in the event of an emergency situation.

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

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

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 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste water*

~~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 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*

3 Terms, definitions and symbols

3.1 Terms and definitions

No terms and definitions are listed in this document.

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

For the purposes of this document, the symbols given in ISO/IEC Guide 98-3, ISO/IEC Guide 99 ISO 80000-10, ISO 11929-1 and the following shall apply.

Symbol	Description	Unit
A	Activity of the tracer added	Bq
α	Probability of the false positive decision	-
β	Probability of the false negative decision	-
c_A	Activity concentration of the measurand measured in the sample	Bq·l-1
c_A^*	Decision threshold of the measurand	Bq·l-1
$c_A^\#$	Detection limit of the measurand	Bq·l-1
$c_A^\triangleleft$, $c_A^\triangleright$	Lower and upper limits of the probabilistically symmetric coverage interval of the measurand, respectively	Bq·l-1
$c_A^<$, $c_A^>$	Lower and upper limits of the shortest coverage interval of the measurand, respectively	Bq·l-1
$\tilde{c}_A$	Possible or assumed true quantity values of the measurand	Bq·l-1
c_{AT}	Activity concentration of the tracer solution at the moment of separation	Bq·l-1
ε	Counting efficiency	-

Field Code Changed

Field Code Changed

Field Code Changed

Field Code Changed

Symbol	Description	Unit
~~f~~ f	Correction factor for possible bias for curium isotopes using ^{243}Am as a tracer or for ^{237}Np using ^{236}Pu as a tracer. For plutonium isotopes or for ^{241}Am, f is equal to 1	-
Φ	Distribution function of the standardized normal distribution; $\Phi(k_p) = p$ applies	-
$1-\gamma$	Probability for the coverage interval of the measurand	-
k_p	Quantiles of the standardized normal distribution for the probabilities p (for instance $p = 1-\alpha$, $1-\beta$ or $1-\gamma/2$)	-
~~λ~~ λ	Decay constant of the isotope (ex: ~~λ_{215Po}~~ $\lambda_{^{215}Po}$ is the decay constant of ^{215}Po)	
m	Sample mass	kg
~~m_{ST}~~ m_{ST}	Mass of tracer solution	g
N_0	Number of counts measured of the background on the alpha spectrum for a given time in the region of interest of the measurand.	Counts
N_{0T}	Number of counts measured of the background on the alpha spectrum for a given time in the region of interest of the tracer.	Counts
N_g	Number of counts measured on the alpha spectrum for a given time in the region of interest of the measurand.	Counts
N_T	Number of counts measured on the alpha spectrum for a given time in the region of interest of the tracer.	Counts
~~P_α~~ P_α	Probability of the isotope decaying by alpha particle emission (branching ratio)	-
r_0	Background count rate in the region of interest of the measurand	Bq
r_{0T}	Background count rate in the tracer region of interest of the tracer	Bq
R	Total recovery	-
R_c	Chemical recovery	-
r_g	Gross count rate in the region of interest of the measurand	Bq
r_{net}	Net count rate of the measurand	Bq
r_{netT}	Net count rate of the tracer	Bq
r_T	Gross count rate in the region of interest of the tracer	Bq
$t_{1/2}$	Radiological half-life of the isotope of interest	s
t_0	Counting time of the background by alpha spectrometry	s
t_1	Time elapsed between separation and counting	s
t_g	Sample counting time by alpha spectrometry	s
U	Expanded uncertainty	-
u	Standard uncertainty	-

Field Code Changed

Field Code Changed