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Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

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Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	

DRAFT INTERNATIONAL STANDARD

ISO/DIS 13167

ISO/TC 147/SC 3

Secretariat: **AFNOR**

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2022-02-16

Voting terminates on:
2022-05-11

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

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

ICS: 13.060.60; 17.240; 13.280

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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 on 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 the following URL: 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 changes are as follows:

— xxx xxxxxxxx xxx xxx

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

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 , ^3H , ^{14}C , and those originating from the thorium and uranium decay series, in particular ^{226}Ra , ^{228}Ra , ^{234}U , ^{238}U , ^{210}Po and ^{210}Pb can be found in water for natural reasons (e.g. desorption from the soil and washoff 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 fertilizers production and use);
- human-made radionuclides such as transuranium elements (americium, plutonium, neptunium, curium), ^3H , ^{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 a 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 installations 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 waters are monitored for their radioactivity as recommended by the World Health Organization (WHO)^[3] so that proper actions can be taken to ensure that there is no adverse health effect to the public. Following these international recommendations, national regulations usually specify radionuclide authorized concentration limits for liquid effluent discharged to the environment and radionuclide guidance levels for waterbodies 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 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 $1 \text{ Bq}\cdot\text{l}^{-1}$ ^[3]. For ^{242}Cm the guidance level is $10 \text{ Bq}\cdot\text{l}^{-1}$.

NOTE 1 The guidance level is the activity concentration with an intake of $2 \text{ l}\cdot\text{d}^{-1}$ of drinking water for one year that results in an effective dose of $0,1 \text{ mSv}\cdot\text{a}^{-1}$ 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 not be greater than $10 \text{ Bq}\cdot\text{l}^{-1}$ for ^{238}Pu , $^{239,240}\text{Pu}$ and ^{241}Am .

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)^[6].

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^{[5][6][7]}.

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 support the need of test laboratories carrying out these measurements, which 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 set of International Standards on test methods dealing with the measurement of the activity concentration of radionuclides in water samples.

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

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

The volume of the test portion required depends on the assumed activity of the sample and the desired detection limit.

The detection limit depends on the sample volume, the instrument used, the background count rate, the sample counting time, the detection efficiency, and the chemical yield. The detection limit of the test method is 5×10^{-3} to 5×10^{-4} Bq/l for a volume of the test portion of 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^[3].

The methods described in this document is applicable in the event of an emergency situation. For an emergency situation, it is preferable to reduce the counting time rather than the sample volume.

It is the user's responsibility to ensure the validity of this test method for the water samples tested.

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 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 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*

3 Terms, definitions and symbols

3.1 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological 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 and designations given in ISO 80000-10, ISO 11929, ISO/IEC Guide 98-3, and ISO/IEC Guide 99 and the following apply.

Symbol	Definition	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
$\widetilde{c_A}$	Possible or assumed true quantity values of the measurand	Bq·l ⁻¹
c_{A_T}	Activity concentration of the tracer solution at the moment of separation	Bq·l ⁻¹
ε	Counting efficiency	
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 $1-\gamma/2$)	
m	Sample mass	kg
m_{S_T}	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