SLOVENSKI STANDARD
oSIST ISO 20899:2019

01-september-2019

Kakovost vode - Plutonij in neptunij - Preskusna metoda masne spektrometrije z induktivno sklopljeno plazmo (ICP/MS)

Water quality - Plutonium and neptunium - Test method using ICP-MS

Qualité de l'eau - Plutonium et neptunium - Méthode d'essai par ICP-MS

Ta slovenski standard je istoveten z: **ISO 20899:2018**

ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
17.240	Merjenje sevanja	Radiation measurements

oSIST ISO 20899:2019 **en**

INTERNATIONAL STANDARD

ISO 20899

First edition
2018-09

Water quality — Plutonium and neptunium — Test method using ICP-MS

Qualité de l'eau — Plutonium et neptunium — Méthode d'essai par ICP-MS

Reference number
ISO 20899:2018(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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, *Radioactivity measurements*.

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, ^{3}H, ^{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), ^{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 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 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 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[4] and ISO 5667-20[5].

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 guidelines for guidance level in drinking water is 1 Bq/l activity concentration for ^{239}Pu, ^{240}Pu and ^{241}Pu.

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 (GLs)[7] states that the activity concentration might not be greater than 1 Bq/l and 10 Bq/l for ^{239}Pu, ^{240}Pu, ^{241}Pu, respectively, for infant and for others than infant.

NOTE 2 The Codex 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)[7].

Thus, the test method can be adapted so that the characteristic limits, decision threshold, detection limit and uncertainties ensure that the test results of the radionuclide activity concentrations 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][8].

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).

An International Standard on a test method for ^{239}Pu, ^{240}Pu, ^{241}Pu and ^{237}Np concentration in water samples is justified for test laboratories carrying out these measurements, required sometimes by national authorities, as laboratories 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 and neptunium — Test method using ICP-MS

WARNING — Persons using this document should be familiar with normal laboratory practice. 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.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document specifies methods used to determine the concentration of plutonium and neptunium isotopes in water by inductively coupled plasma mass spectrometry (ICP-MS) (^{239}Pu, ^{240}Pu, ^{241}Pu and ^{237}Np). The concentrations obtained can be converted into activity concentrations of the different isotopes[9].

Due to its relatively short half-life and ^{238}U isobaric interference, ^{238}Pu can hardly be measured by this method. To quantify this isotope, other techniques can be used (ICP-MS with collision-reaction cell, ICP-MS/MS with collision-reaction cell or chemical separation). Alpha spectrometry measurement, as described in ISO 13167[10], is currently used[11].

This method is applicable to all types of water having a saline load less than 1 g·l^{-1}. A dilution of the sample is possible to obtain a solution having a saline load and activity concentrations compatible with the preparation and the measurement assembly.

A filtration at 0,45 µm is needed for determination of dissolved nuclides. Acidification and chemical separation of the sample are always needed.

The limit of quantification depends on the chemical separation and the performance of the measurement device.

This method covers the measurement of those isotopes in water in activity concentrations between around[12][13]:

— 1 mBq·l^{-1} to 5 Bq·l^{-1} for ^{239}Pu, ^{240}Pu and ^{237}Np;

— 1 Bq·l^{-1} to 5 Bq·l^{-1} for ^{241}Pu.

In both cases, samples with higher activity concentrations than 5 Bq·l^{-1} can be measured if a dilution is performed before the chemical separation.

It is possible to measure ^{241}Pu following a pre-concentration step of at least 1 000.

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

ISO 17294-1:2004, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines*

ISO 17294-2:2016, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of selected elements including uranium isotopes*

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

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

3 Terms, definitions, symbols and abbreviated terms

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

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 http://www.electropedia.org/

Term	**Symbol or abbreviated term**	**Unit symbol**	**Definition**
Mass concentration	ρ	µg·l^{-1}	Analyte mass for a given radionuclide per sample unit volume.
Mass concentration of the internal standard solution	ρ_T	µg·l^{-1}	Mass of internal standard radionuclide element per unit volume of the internal standard solution.
Internal standard mass	m_T	µg	Mass of the isotope dilution tracer added
Standard uncertainty	$u(\rho)$	µg·l^{-1}	Standard uncertainty associated with the measurement result
Expanded uncertainty	$U(x)$		Product of the standard uncertainty and the coverage factor k with $k = 1, 2, \ldots$, $U = k \cdot u$
Standard uncertainty	$u(x)$		Standard uncertainty associated with the measurement result of x
Detection limit	DL	µg·l^{-1}	DL is the lowest amount of an analyte that is detectable using an instrument, as determined by repeated measurement of a regent blank.
Limit of quantification	LOQ	µg·l^{-1} mBq.l^{-1}	LOQ is the smallest concentration of an analyte in the test sample which can be determined with a fixed precision.
Standard deviation of the blank	s_{No}		Standard deviation of replicates of the blank.
Instrumental limit of quantification	LOQ$_{\text{ins}}$	Counts.s^{-1}	LOQ$_{\text{ins}}$ is the LOQ expressed in counts rate for the chosen mass on charge ration (m/z), due to the blank and the instrument.
Instrumental detection limit	IDL	Counts.s^{-1}	IDL is the DL expressed in counts rate for the chosen mass on charge ration (m/z).
Volume of the sample	V	l	
Background	N_0	Counts.s^{-1}	Counts rates for a given mass in the blank solution.
Counts	N	Counts.s^{-1}	Gross counts rates: uncorrected counts rate of a measurement.
Net counts	N_{net}	Counts.s^{-1}	N-N_0