



SLOVENSKI STANDARD
oSIST prEN ISO 22125-2:2018
01-oktober-2018

Kakovost vode - Tehnecij Tc-99 - 2. del: Preskusna metoda z masno spektrometrijo z induktivno sklopljeno plazmo (ICP-MS) (ISO/DIS 22125-2:2018)

Water quality - Technetium-99 - Part 2: Test method using inductively coupled plasma mass spectrometry (ICP-MS) (ISO/DIS 22125-2:2018)

Wasserbeschaffenheit - Technetium 99 - Teil 2: Verfahrens mittels Massenspektrometrie und induktiv gekoppeltem Plasma (ICP-MS) (ISO/DIS 22125-2:2018)

Qualité de l'eau - Technétium-99 - Partie 2: Méthode d'essai par spectrométrie de masse couplée à un plasma induit (ISO/DIS 22125-2:2018)

Ta slovenski standard je istoveten z: prEN ISO 22125-2

ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
-----------	---------------------------------	--

oSIST prEN ISO 22125-2:2018

en,fr,de

DRAFT INTERNATIONAL STANDARD

ISO/DIS 22125-2

ISO/TC 147/SC 3

Secretariat: AFNOR

Voting begins on:
2018-08-02Voting terminates on:
2018-10-25

Water quality — Technetium-99 —

Part 2: Test method using inductively coupled plasma mass spectrometry (ICP-MS)

*Qualité de l'eau — Technétium-99 —**Partie 2: Méthode d'essai par spectrométrie de masse couplée à un plasma induit*

ICS: 13.060.60; 17.240

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN ISO 22125-2:2020

<https://standards.iteh.ai/catalog/standards/sist/4e1d9768-0130-4b47-bdf2-4aff648f9df5/sist-en-iso-22125-2-2020>

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

This document is circulated as received from the committee secretariat.



Reference number
ISO/DIS 22125-2:2018(E)

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN ISO 22125-2:2020

<https://standards.iteh.ai/catalog/standards/sist/4e1d9768-0130-4b47-bdf2-4aff648f9df5/sist-en-iso-22125-2-2020>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2018

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Symbols	2
4 Principle	3
5 Sampling, handling, and storage	4
6 Procedure	4
6.1 Sample preparation for measurement.....	4
6.2 Sample measurement.....	4
7 Quality assurance and quality control program	5
7.1 General.....	5
7.2 Variables that could influence the measurement.....	5
7.3 Instrument verification.....	5
7.4 Contamination.....	5
7.5 Interference control.....	5
7.6 Method verification.....	5
7.7 Demonstration of analyst capability.....	6
8 Hazards	6
9 Expression of results	6
9.1 When Re, ⁹⁷ Tc, or ⁹⁸ Tc is used as a recovery tracer.....	6
9.1.1 Calculation of mass of tracer and analyte added.....	6
9.1.2 Mass bias.....	7
9.1.3 Sample mass concentration.....	7
9.1.4 Detection limit.....	7
9.1.5 Limit of quantification.....	7
9.2 When ^{95m} Tc, ^{97m} Tc or ^{99m} Tc is used as a recovery tracer.....	8
9.2.1 Calculation of activity of tracer, mass of analyte, and mass of internal standard added.....	8
9.2.2 Purification step recovery.....	8
9.2.3 Mass bias.....	8
9.2.4 Sample mass concentration.....	9
9.2.5 The detection limit.....	9
9.2.6 Limit of quantification.....	9
9.2.7 Conversion of mass concentration to activity concentration.....	9
9.2.8 Conversion of mass concentration to volume unit.....	10
10 Test report	10
Annex A (informative) Method 1 - TEVA resin	11
Annex B (informative) Method 2- TRU resin	14
Annex C (informative) Method 3 - Anion exchange resin	17
Bibliography	20

ISO/DIS 22125-2:2018(E)

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*.

A list of all the parts in the ISO 22125- series can be found on the ISO website. [standards.itih.ai](http://www.iso.org/iso/standards/standards.itih.ai)
[SIST EN ISO 22125-2:2020](http://www.iso.org/iso/standards/standards.itih.ai)
[bdf2-4aff648f9df5/sist-en-iso-22125-2-2020](http://www.iso.org/iso/standards/standards.itih.ai)

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 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 and ISO 5667-20^[4].

Depending of 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 guidelines for guidance level in drinking water is $100 \text{ Bq} \cdot \text{l}^{-1}$ for ^{99}Tc activity concentration

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^[6] mentioned that the activity concentration might not be greater than $10\,000 \text{ Bq} \cdot \text{kg}^{-1}$ for ^{99}Tc .

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.

ISO/DIS 22125-2:2018(E)

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 of ^{99}Tc activity concentrations 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.

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN ISO 22125-2:2020

<https://standards.iteh.ai/catalog/standards/sist/4e1d9768-0130-4b47-bdf2-4aff648f9df5/sist-en-iso-22125-2-2020>

Water quality — Technetium-99 —

Part 2:

Test method using inductively coupled plasma mass spectrometry (ICP-MS)

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 ensure compliance with any national regulatory conditions.

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 method for the measurement of ^{99}Tc in all types of waters by inductively coupled plasma mass spectrometry (ICP-MS).

The method is applicable to test samples of supply/drinking water, rainwater, surface and ground water, as well as cooling water, industrial water, domestic, and industrial wastewater. A filtration of the test sample is necessary.

The detection limit depends on the sample volume and the instrument used. The method described in this standard, using currently available ICP-MS, has a detection limit of approximately $0,2 \text{ ng}\cdot\text{kg}^{-1}$ to $0,5 \text{ ng}\cdot\text{kg}^{-1}$ ($0,1 \text{ Bq}\cdot\text{kg}^{-1}$ to $0,3 \text{ Bq}\cdot\text{kg}^{-1}$), which is much lower than the WHO criteria for safe consumption of drinking water ($100 \text{ Bq}\cdot\text{l}^{-1}$). The method presented in this standard is not intended for the determination of ultra-trace amount of ^{99}Tc .

The mass concentration values in this document are expressed by sample mass unit instead of sample volume unit as it is usually the case in similar standards. The reason is that ^{99}Tc is measured in various matrix types such as fresh water or sea water, which have significant density differences. The mass concentration values can be easily converted to sample volume unit by measuring the sample volume. However, it increases the uncertainty on the mass concentration result.

The method described in this standard is applicable in the event of an emergency situation, but not if $^{99\text{m}}\text{Tc}$ is present at quantities that could cause interference.

The analysis of Tc adsorbed to suspended matter is not covered by this method.

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/DIS 22125-2:2018(E)

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 10703, *Water quality — Determination of the activity concentration of radionuclides — Method by high resolution gamma-ray spectrometry*

ISO 11929, *Determination of the characteristic limits (decision threshold, detection limit and limits of the confidence interval) for measurements of ionizing radiation — Fundamentals and application*

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

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

ISO 20042, *Measurement of radioactivity - Gamma emitting radionuclides - Generic test method using gamma spectrometry*

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

3 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	Term	Unit symbol	Definition
C	Activity concentration	Bq·kg ⁻¹	Corresponding to the activity concentration ρ measured for a given radionuclide
ρ	Mass concentration	g·kg ⁻¹	Analyte mass for a given radionuclide per sample unit mass
DL	Detection limit in mass concentration	g·kg ⁻¹	DL is the lowest mass concentration that can be considered statistical different from a blank sample.
LOQ	Limit of quantification in mass concentration	g·kg ⁻¹	LOQ is the lowest mass concentration that can be quantified with statistical certainty
DL _C	Detection limit in activity concentration	Bq·kg ⁻¹	DL is the lowest activity concentration that can be considered statistical different from a blank sample.
LOQ _C	Limit of quantification in activity concentration	Bq·kg ⁻¹	LOQ is the lowest activity concentration that can be quantified with statistical certainty
U	Expanded uncertainty		Product of the standard uncertainty and the coverage factor k with k = 1, 2, ..., U = k · u
μ	Standard uncertainty		Uncertainty of a term such as mass, counts, etc.
$\mu[C]$	Standard uncertainty of the activity concentration	Bq·kg ⁻¹	Standard uncertainty associated with the activity concentration result
$\mu[\rho]$	Standard uncertainty of the mass concentration	g·kg ⁻¹	Standard uncertainty associated with the mass concentration result
s	Standard deviation	counts	
m _T	Tracer mass	g	Mass of the tracer added to the blank and sample
m _{IS}	Internal standard mass	g	Mass of the internal standard added to the blank and sample
m _{TS}	Tracer solution mass	g	Mass of the tracer solution added to a blank sample or a sample
m _{ISS}	Internal standard solution mass	g	Mass of the internal standard solution added to a blank sample or a sample

Symbol	Term	Unit symbol	Definition
m_{As}	Analyte solution mass	g	Mass of the analyte solution added to a control sample or for mass bias calculation
ρ_T	Mass concentration of the tracer solution	$g \cdot g^{-1}$ *	Tracer mass for a given radionuclide per sample unit volume of the tracer solution
ρ_A	Mass concentration of the analyte (^{99}Tc) standard solution	$g \cdot g^{-1}$ *	Analyte mass for a given radionuclide per sample unit volume of the standard solution
m	Sample mass	kg	Mass of the water sample
m_A	Analyte mass	g	Mass of analyte added to a spiked solution
N	Counts	counts	Number of counts directly obtained when performing the ICP-MS measurement for a sample at a given mass on charge ratio
N_0	Counts of the blank	counts	Number of counts directly obtained when performing the ICP-MS measurement for a blank at a given mass on charge ratio
\bar{N}_0	Average counts of blank samples	counts	Average number of counts directly obtained when performing the ICP-MS measurement for several blanks at a given mass on charge ratio
N_{net}	Net counts	counts	$N - N_0$
N_{netT}	Net counts of the tracer	counts	At the tracer mass
N_{netIS}	Net counts of the internal standard	counts	At the internal standard mass
C_s	Specific activity	$Bq \cdot g^{-1}$	Activity corresponding to one gram of the radionuclide
α	Bias per unit mass		α is a constant which allow to correct for the instrumental difference of counts efficiency between the tracer and the analyte
R_c	Chemical recovery		Recovery of the purification step obtained by gamma measurement
m/z	Mass on charge ratio		Mass on charge ratio measured by the ICP-MS
s_{N0}	Standard deviation	counts	Standard deviation associated with the measurement obtained from 10 test portions of a blank sample

4 Principle

Technetium is mainly an anthropogenic element, but trace amounts are found in uranium ores. It has no stable isotope. ^{99}Tc is a significant fission product of ^{235}U (~6% yield [8]) with a maximum beta-energy of (294 ± 1) keV and a half-life of $2,1 \pm 0,1 \times 10^5$ years [9].

To determine ^{99}Tc in water, a water sample is collected, filtered, acidified, and oxidized (see [clause 5](#) on sampling and storage).

A tracer is added before the chemical separation to take into account the losses of recovery during the purification step. Enough tracer is added to obtain a good statistical precision and be easily distinguished from a blank sample. The tracers that can be used are stable Re, ^{95m}Tc , ^{97m}Tc , ^{97}Tc , ^{98}Tc , and ^{99m}Tc .

^{95m}Tc and ^{99m}Tc are the easiest Tc isotopes to be obtained commercially. ^{97}Tc and ^{98}Tc are not currently commercially available. The isotopes ^{95m}Tc , ^{97m}Tc , and ^{99m}Tc have a short radiological half-life and cannot be used as an internal standard (IS) (they are not measured by ICP-MS) to correct the variation of signal by the ICP-MS instrument; thus, an internal standard such as ^{115}In is added before the measurement. When using ^{99m}Tc , the standard should be as pure of possible from ^{99}Mo [10]. The activity of ^{95m}Tc and ^{99m}Tc are measured by gamma spectrometry according to ISO 10703 and ISO 20042.

Stable Re is often used as a recovery tracer for Tc measurement due to its similar reactivity. It has the advantages of being easily available, stable, and can be measured by ICP-MS. Tc and Re do not behave similarly when heated in an acidic solution: Tc is more volatile [11,12]; thus, Re cannot be used as a recovery tracer when the method includes a vaporisation step.