



**SLOVENSKI STANDARD**  
**oSIST prEN ISO 22125-1:2018**  
**01-oktober-2018**

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**Kakovost vode - Tehnecij Tc-99 - 1. del: Preskusna metoda štetja s tekočinskim scintilatorjem (ISO/DIS 22125-1:2018)**

Water quality - Technetium-99 - Part 1: Test method using liquid scintillation counting (ISO/DIS 22125-1:2018)

Wasserbeschaffenheit - Technetium 99 - Teil 1: Verfahren mit dem Flüssigszintillationszähler (ISO/DIS 22125-1:2018)

Qualité de l'eau - Technétium-99 - Partie 1: Méthode d'essai par comptage des scintillations en milieu liquide (ISO/DIS 22125-1:2018)

**Ta slovenski standard je istoveten z: prEN ISO 22125-1**

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**ICS:**

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
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## Water quality — Technetium-99 —

### Part 1: Test method using liquid scintillation counting

*Qualité de l'eau — Technétium-99 —**Partie 1: Méthode d'essai par comptage des scintillations en milieu liquide*

ICS: 13.060.60; 17.240

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## ISO/DIS 22125-1:2018(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](http://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](http://www.iso.org/patents)).

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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](http://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 22125-1 series can be found on the ISO website [www.iso.org/83-483a-a092-83025435472a/sist-en-iso-22125-1-2020](http://www.iso.org/83-483a-a092-83025435472a/sist-en-iso-22125-1-2020)

## 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  $^{40}\text{K}$ ,  $^3\text{H}$ ,  $^{14}\text{C}$ , and those originating from the thorium and uranium decay series, in particular  $^{226}\text{Ra}$ ,  $^{228}\text{Ra}$ ,  $^{234}\text{U}$ ,  $^{238}\text{U}$ ,  $^{210}\text{Po}$  and  $^{210}\text{Pb}$  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),  $^3\text{H}$ ,  $^{14}\text{C}$ ,  $^{90}\text{Sr}$ , 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.<sup>[1]</sup> 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<sup>[2]</sup> and water bodies. Drinking waters are monitored for their radioactivity as recommended by the World Health Organization (WHO)<sup>[3]</sup> 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.<sup>[4]</sup>

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  $^{99}\text{Tc}$  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.<sup>[3]</sup>

In the event of a nuclear emergency, the WHO Codex Guideline Levels<sup>[6]</sup> mentioned that the activity concentration might not be greater than  $10\,000 \text{ Bq} \cdot \text{kg}^{-1}$  for  $^{99}\text{Tc}$ .

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).<sup>[6]</sup>

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.<sup>[5][6][7]</sup>

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.

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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 <sup>99</sup>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.

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# Water quality — Technetium-99 —

## Part 1:

## Test method using liquid scintillation counting

**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  $^{99}\text{Tc}$  in all types of waters by liquid scintillation counting (LSC).

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 after proper sampling and handling, and test sample preparation. 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 LSC counters, has a detection limit of approximately  $5 \text{ Bq}\cdot\text{kg}^{-1}$  to  $20 \text{ Bq}\cdot\text{kg}^{-1}$ , which is lower than the WHO criteria for safe consumption of drinking water ( $100 \text{ Bq}\cdot\text{l}^{-1}$ ). These values can be achieved with a counting time of 30 min for a sample volume varying between 14 ml to 40 ml. The method presented in this standard is not intended for the determination of ultra-trace amount of  $^{99}\text{Tc}$ .

The activity 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  $^{99}\text{Tc}$  is measured in various matrix types such as fresh water or sea water, which have significant density differences. The activity concentration values can be easily converted to sample volume unit by measuring the sample volume. However, it increases the uncertainty on the activity 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 and not if  $^{99\text{m}}\text{Tc}$  is used as a recovery tracer.

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 3696, *Water for analytical laboratory use — Specification and test methods*

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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 19361, *Measurement of radioactivity — Determination of beta emitters activities — Test method using liquid scintillation counting*

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
$c_A$	activity concentration	Bq·kg <sup>-1</sup>
$c_A^*$	decision threshold	Bq·kg <sup>-1</sup>
$c_A^\#$	detection limit	Bq·kg <sup>-1</sup>
$c_A^<, c_A^>$	lower and upper limits of the confidence interval	Bq·kg <sup>-1</sup>
$\tilde{\mu}(\tilde{C}_A)$	characteristic limits	Bq·kg <sup>-1</sup>
$R_c$	chemical recovery	
$r_g$	sample count rate	s <sup>-1</sup>
$r_s$	calibration count rate	s <sup>-1</sup>
$r_0$	background count rate	s <sup>-1</sup>
$r_b$	Reagent blank count rate	s <sup>-1</sup>
$t_g$	sample counting time	s
$t_s$	calibration counting time	s
$t_0$	background counting time	s
$U$	expanded uncertainty, calculated by $U = ku(c_A)$ with $k = 1, 2, \dots$ ,	Bq·kg <sup>-1</sup>
$u(c_A)$	standard uncertainty associated with the measurement result	Bq·kg <sup>-1</sup>
$A$	Activity of the calibration source	Bq
$\varepsilon$	detection efficiency	
$f_q$	Quench factor	
$m_{te}$	Empty tube mass of the eluate	g
$m_{tf}$	Full tube mass of the eluate	g

Symbol	Term	Unit symbol
$m_1$	Sub sample mass of the eluate for Tc measurement by LSC	g
$m_2$	Sub sample mass of the eluate for recovery measurement	g
$m_e$	Eluate mass	g
$m_T$	Tracer mass	g
$m_{TS}$	Tracer solution mass	g
$\rho$	Sample density	kg·l <sup>-1</sup>
$m$	Test sample mass	kg
$V$	Test sample volume	l
tSIE	Transformed External Standard Spectrum	
TDCR	Triple to Double Counts Ratio	
DPM	Disintegrations Per Minutes	
$R_m$	Mass ratio	
$C_s$	Specific activity	Bq·g <sup>-1</sup>
DL	Detection limit	Bq·kg <sup>-1</sup> or Bq·l <sup>-1</sup>
LOQ	Limit of quantification	Bq·kg <sup>-1</sup> Bq·l <sup>-1</sup>

## 4 Principle

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

To determine <sup>99</sup>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, <sup>95m</sup>Tc and <sup>99m</sup>Tc. 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 and stable. 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.

— When Re is used as a chemical recovery tracer, a sub-sample ( $m_2$ ) of known mass is taken before the LSC measurement for the recovery determination. It is recommended to complete the recovery determination before counting the sample.

Re can be measured for example by:

- ICP-OES according to ISO 11885 [13]
- AAS according to ISO 15586 [14]
- UV-visible spectroscopy [15][16]
- When <sup>99m</sup>Tc or <sup>95m</sup>Tc is used as a chemical recovery tracer, the chemical recovery is determined by gamma spectroscopy [17]. Enough activity of <sup>99m</sup>Tc or <sup>95m</sup>Tc is added to obtain 10 000 counts when counting the sample. The sample is directly placed in the gamma counter, without any sample pre-treatment. It is measured according to the instrument specifications and in accordance with ISO 10703 and ISO 20042.

<sup>95m</sup>Tc or <sup>99m</sup>Tc has to completely decay before measuring the sample by LSC. It can take several days for <sup>99m</sup>Tc and several months for <sup>95m</sup>Tc depending of the initial quantity added. The tracer <sup>99m</sup>Tc is usually preferred to <sup>95m</sup>Tc due to a faster decay and also because commercial <sup>95m</sup>Tc standard solutions may contain a significant amount of <sup>99</sup>Tc.[9]