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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*

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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](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

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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](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)).

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](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee 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.

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](http://www.iso.org/members.html).

## Introduction

Radioactivity from several naturally-occurring and anthropogenic sources is present throughout the environment. Thus, water bodies (such as 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 content 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 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 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>[5]</sup> mentioned that the activity concentration on contaminated food 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>[5]</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 can 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 answer the need of test laboratories carrying out these measurements, that 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.

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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 determine the applicability of any other restrictions.

**IMPORTANT** — It is absolutely essential that tests conducted according to this document 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 document, using currently available LSC instruments, 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 ( $100 \text{ Bq l}^{-1}$ )<sup>[3]</sup>. 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 document 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 differences in density. 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 document 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*

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 Terms, definitions and symbols

#### 3.1 Terms and definitions

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For the purposes of this document, the terms and definitions given in ISO 80000-10, ISO 11929, ISO/IEC Guide 98-3 and ISO/IEC Guide 99 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/>

#### 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, ISO/IEC Guide 99 and the following apply.

Symbol	Term	Unit
$c_A^*$	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>
$c_{A\rho}^\#$	detection limit in mass concentration	g·kg <sup>-1</sup>
A	activity of the calibration source	Bq
$A_T$	tracer activity	Bq
$A_{Tm}$	tracer activity measured	Bq
$c_A$	activity concentration	Bq·kg <sup>-1</sup>
$c_m$	mass concentration	g·kg <sup>-1</sup>
$C_s$	specific activity	Bq·g <sup>-1</sup>



Symbol	Term	Unit
DPM	disintegrations per minute	
$f_q$	quench factor	
$m$	test sample mass	kg
$m_1$	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
$m_{TB}$	mass of tracer added to the reagent blank for the calculation of $r_b$	g
$m_{te}$	empty container mass of the eluate	g
$m_{tf}$	full container mass of the eluate	g
$m_{Tm}$	tracer mass measured	g
$m_{TS}$	tracer solution mass	g
$r_b$	reagent blank count rate	counts·s <sup>-1</sup>
$R_c$	chemical recovery	
$r_g$	sample count rate	counts·s <sup>-1</sup>
$R_m$	mass ratio	
$r_s$	calibration count rate	counts·s <sup>-1</sup>
$r_{sp}$	spiked reagent blank count rate for $r_o$ calculation	counts·s <sup>-1</sup>
$r_T$	<sup>99</sup> Tc count rate from the tracer	counts·s <sup>-1</sup>
$r_{us}$	unspiked reagent blank count rate for $r_o$ calculation	counts·s <sup>-1</sup>
SQPE	spectral quench parameter of the external standard	
$t_0$	background counting time	s
TDCR	triple to double counts ratio	
$t_g$	sample counting time	s
$t_s$	calibration counting time	s
tSIE	transformed spectral index of the external standard	
$\tilde{u}(\tilde{C}_A)$	characteristic limits	Bq·kg <sup>-1</sup>
$U$	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>
$V$	test sample volume	l
$\varepsilon$	detection efficiency	
$\rho$	sample density	kg·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 (approximately 6 % yield<sup>[8]</sup>) with a maximum beta-energy of (294 ± 1) keV and a half-life of (2,1 ± 0,1) × 10<sup>5</sup> years<sup>[9]</sup>.

To determine <sup>99</sup>Tc in water, a water sample is collected, filtered, acidified, and oxidized (see [Clause 5](#)). 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<sup>[8]</sup>. 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<sup>[10][11]</sup>; thus Re cannot be used as a recovery tracer when the method includes a vaporization 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.

Rhenium can be measured for example by:

- ICP-OES according to ISO 11885<sup>[12]</sup>
- AAS according to ISO 15586<sup>[13]</sup>
- UV-visible spectroscopy<sup>[14][15]</sup>
- When  $^{99m}\text{Tc}$  or  $^{95m}\text{Tc}$  is used as a chemical recovery tracer, the chemical recovery is determined by gamma spectrometry<sup>[8]</sup>. Enough activity of  $^{99m}\text{Tc}$  or  $^{95m}\text{Tc}$  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.

$^{95m}\text{Tc}$  or  $^{99m}\text{Tc}$  should completely decay before measuring the sample by LSC. It can take several days for  $^{99m}\text{Tc}$  and several months for  $^{95m}\text{Tc}$  depending of the initial quantity added. The tracer  $^{99m}\text{Tc}$  is usually preferred to  $^{95m}\text{Tc}$  due to a faster decay and also because commercial  $^{95m}\text{Tc}$  standard solutions may contain a significant amount of  $^{99}\text{Tc}$ <sup>[8]</sup>.

$^{99}\text{Tc}$  is chemically purified from potential interferents, which consist of any isotope that can cause the liquid scintillator to emit light in the region of interest (ROI) of  $^{99}\text{Tc}$ . Different methods for the purification of  $^{99}\text{Tc}$  are presented in the Annexes A to C.

After removal of the potential interferents, the chemical recovery ( $R_c$ ) is determined. The purified sample is mixed with the scintillation cocktail in a counting vial to obtain a homogenous medium. The vial is counted by LSC.

## 5 Sampling and storage

Sampling, handling, and storage of the water shall be done as specified in ISO 5667-1, ISO 5667-3 and ISO 5667-10 and guidance is given for the different types of water in References [16] to [23]. It is important that the laboratory receives a sample that is truly representative and has not been damaged or modified during transportation or storage.

The sample is filtered to remove suspended matter using a 0,45  $\mu\text{m}$  filter. A smaller pore size filter can also be used, but the filtration might be more tedious and time consuming. Technetium (VII) is not strongly adsorbed to plastic or glass container, but it can be reduced by the organic matter in the sample to technetium oxide ( $\text{TcO}_2$ ). After filtration, the sample is acidified with nitric acid ( $\text{HNO}_3$ ) to 0,01  $\text{mol}\cdot\text{l}^{-1}$   $\text{HNO}_3$ . Then, hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) is added to maintain Tc as  $\text{TcO}_4^-$  and reduce its adsorption to the container. An addition of  $\text{H}_2\text{O}_2$  to bring the sample to a concentration of 0,02  $\text{mol}\cdot\text{l}^{-1}$  is recommended for the sample.

## 6 Procedure

### 6.1 Sample preparation for measurement

Filter, acidify, and oxidize the samples and a blank sample prepared with ultrapure water as specified in Clause 5. A minimum of 1 blank sample is required for all the methods presented. However, the average of several blanks can be used. Also, measuring blank samples at regular interval enables to rapidly detect a background issue when measuring the samples (see quality assurance and quality control program in Clause 7). Add the tracer to the sample.

Purify the sample from potential interferents. Purification methods are described in the informative Annexes A to C. An equivalent method can be used but shall follow all the criteria stated in this document.

Determine the chemical recovery.

Measure the  $^{99}\text{Tc}$  in the sample by LSC.

## 6.2 Sample measurement

Measure  $^{99}\text{Tc}$  in the samples by LSC by following the instructions provided by the instrument manufacturer and the steps described in ISO 19361.

## 7 Quality assurance and quality control program

### 7.1 General

Quality control operations shall meet the requirements of ISO/IEC 17025. Measurement methods shall be performed by suitably skilled staff under a quality assurance program.

### 7.2 Variables that could influence the measurement

Special care shall be taken in order to limit as much as possible the influence of parameters that may bias the measurement and lead to a non-representative result. Failure to take sufficient precautions may require corrective factors to be applied to the measured result. The variables affecting each measurement method are discussed in ISO 13164-2<sup>[24]</sup> and ISO 13164-3<sup>[25]</sup>.

Influencing variables can affect the following stages of the measurement process: sampling, transportation and storage, reagents, transfer, and the activity measurement.

The presence of luminescence prevents the proper measurement of the samples. To reduce the luminescence, the samples are left in the dark for a few hours before counting them. If a luminescence peak is observed, wait a few more hours until no luminescence is observed and re-count the samples.

### 7.3 Instrument verification

Major instrument parameters (efficiency, background) shall be periodically verified within a quality assurance program established by the laboratory and in accordance with the manufacturer's instructions.

### 7.4 Contamination

Verify for contamination of the reagents through the periodic performance of reagent blank analysis. Laboratory procedures shall ensure that laboratory and equipment contamination as well as sample cross contamination is avoided.

### 7.5 Interference control

It is the user's responsibility to ensure that all potential interferents have been removed. The removal of potential interferents is limited by the decontamination factor of the method and the instrumental capabilities.

### 7.6 Method verification

A periodic verification of the method accuracy should be performed. This may be accomplished by:

- participating in intercomparison exercises;