
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 (ICP-MS)*

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Published in Switzerland

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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).

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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. (standards.iteh.ai)

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.
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<https://standards.iteh.ai/catalog/standards/sist/57b91dd-4e18-4723-9fe0-i8854734-6c8f-201252-iso>
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.

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 origin.

- 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 (such as desorption from the soil and washoff by rain water) or can be released from technological processes involving naturally occurring radioactive materials (such as 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 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}Tc ^[3] 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^[5] mentioned that the activity concentration in contaminated food 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)^[5].

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.

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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).

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 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 determine the applicability of any other restrictions.

IMPORTANT — It is absolutely 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 water 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 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 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 ($100 \text{ Bq}\cdot\text{l}^{-1}$)^[3]. The method presented in this document 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 differences in density. 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 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.

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 (all parts), *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 Terms, definitions and symbols

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3.1 Terms and definitions

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 symbol	Definition
α	Measurement bias	—	α is a constant which allows to correct for the signal intensity bias between the tracer or the internal standard and the analyte
C	Activity concentration	Bq·kg ⁻¹	Corresponding to the activity concentration ρ measured for a given radionuclide
C _s	Specific activity	Bq·g ⁻¹	Activity corresponding to one gram of the radionuclide
DL	Detection limit in mass concentration	g·kg ⁻¹	DL is the lowest mass concentration that can be considered statistically different from a blank sample.
DL _C	Detection limit in activity concentration	Bq·kg ⁻¹	DL is the lowest activity concentration that can be considered statistically 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 statistically certainty

Symbol	Term	Unit symbol	Definition
LOQ_C	Limit of quantification in activity concentration	$Bq \cdot kg^{-1}$	LOQ is the lowest activity concentration that can be quantified with statistically certainty
m	Sample mass	kg	Mass of the water sample
m/z	Mass on charge ratio	—	Mass on charge ratio measured by the ICP-MS
m_A	Analyte mass	g	Mass of analyte added to a spiked solution
m_{As}	Analyte solution mass	g	Mass of the analyte solution added to a control sample or for measurement calculation
m_{IS}	Internal standard mass	g	Mass of the internal standard added to the blank and sample
m_{ISS}	Internal standard solution mass	g	Mass of the internal standard solution added to a blank sample or a sample
m_T	Tracer mass	g	Mass of the tracer added to the blank and sample
m_{TB}	Reagent blank tracer mass	g	Mass of tracer added to the reagent blank for the calculation of N_{net}
m_{TS}	Tracer solution mass	g	Mass of the tracer solution added to a blank sample or a sample
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_{netIS}	Net counts of the internal standard	counts	At the internal standard mass
N_{netT}	Net counts of the tracer	counts	At the tracer mass
N_{sp}	spiked reagent blank count	counts	spiked reagent blank count rate for N_{net} calculation
N_T	^{99}Tc counts from the tracer	counts	^{99}Tc present in the tracer as impurities
N_{us}	Unspiked reagent blank	counts	Unspiked reagent blank count rate for N_{net} calculation
R_c	Chemical recovery	—	Recovery of the purification step obtained by gamma measurement
S	Standard deviation	counts	
s_{N0}	Standard deviation	counts	Standard deviation associated with the measurement obtained from 10 test portions of a blank sample
U	Expanded uncertainty	—	Product of the standard uncertainty and the coverage factor k with $k = 1, 2, \dots$, $U = k \cdot u$
μ	Standard uncertainty	—	Uncertainty of a term such as mass, counts, etc.
$\mu[C]$	Standard uncertainty of the activity concentration	$Bq \cdot kg^{-1}$	Standard uncertainty associated with the activity concentration result
$\mu[\rho]$	Standard uncertainty of the mass concentration	$g \cdot kg^{-1}$	Standard uncertainty associated with the mass concentration result
ρ	Mass concentration	$g \cdot kg^{-1}$	Analyte mass for a given radionuclide per sample unit mass
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
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

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 (approximately 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 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, $^{95\text{m}}\text{Tc}$, $^{97\text{m}}\text{Tc}$, ^{97}Tc , ^{98}Tc , and $^{99\text{m}}\text{Tc}$.

$^{95\text{m}}\text{Tc}$ and $^{99\text{m}}\text{Tc}$ are the easiest Tc isotopes to be obtained commercially. ^{97}Tc and ^{98}Tc are not currently commercially available. The isotopes $^{95\text{m}}\text{Tc}$, $^{97\text{m}}\text{Tc}$, and $^{99\text{m}}\text{Tc}$ 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 $^{99\text{m}}\text{Tc}$, the standard should contain as little ^{99}Mo as possible^[10]. The activity of $^{95\text{m}}\text{Tc}$ and $^{99\text{m}}\text{Tc}$ 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^[8]. 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 an evaporation step.

The potential interferents for the measurement of ^{99}Tc by ICP-MS are removed chemically. The two main interferents are $^{98}\text{MoH}^+$ and $^{99}\text{Ru}^+$. Methods for the purification of ^{99}Tc are presented in detail in the [Annexes A](#) to [C](#).

Finally, ^{99}Tc is measured by ICP-MS and the mass or activity concentration of ^{99}Tc is calculated and reported (see [6.2](#) for more details).

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5 Sampling, handling 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 [\[13\]](#) to [\[20\]](#). 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 μm 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 could be reduced by the organic matter in the sample to technetium oxide (TcO_2). After filtration, the sample is acidified with nitric acid (HNO_3) to 0,01 $\text{mol}\cdot\text{l}^{-1}$ HNO_3 . Then, hydrogen peroxide (H_2O_2) is added to maintain Tc as TcO_4^- and reduce its adsorption to the container. An addition of H_2O_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, which contains the tracer, is required for all the method 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 (for quality assurance and quality control program, see [Clause 7](#)).

Add the tracer as specified in one of the purification methods described in [Annexes A to C](#). An equivalent method can be used but shall follow all the criteria enounced in this document.

Purify the samples from potential interferences using one of the methods presented in the [Annexes A, B or C](#).

For ^{95m}Tc , ^{97m}Tc and ^{99m}Tc tracers only, determine the recovery by gamma spectrometry and add the internal standard to the sample.

Measure the ^{99}Tc and tracer or internal standard signal intensity by ICP-MS.

6.2 Sample measurement

The ^{99}Tc and tracer or internal standard signal intensity are measured by the ICP-MS instrument. The mass concentrations of ^{99}Tc and the tracer or internal standard employed are measured. The instructions provided by the instrument manufacturer to use the ICP-MS and the steps described in ISO 17294-2 should be followed.

The detection sensitivity, the instrumental detection limit, and the measurement precision should be established for each analysis performed on the instrument. The interferences for the masses of interest should be reported in a separate table. The measurement bias should be determined for each measurement.

A rinsing sequence, which enables the signal intensity for the analyte and tracer or internal standard to return down to background level, shall be performed after each sample measurement. Memory effects often occur when measuring Tc and Re by ICP-MS. The sample introduction system may be rinsed using a solution of HNO_3 (e.g. 2 %) followed by water. The acid and water used are at least of ICP-MS grade. Then a blank solution should be processed to verify that all remaining Tc and Re have been removed from the system.

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.

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

7.3 Instrument verification

Major instrument parameters (detection efficiency, background signal) 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 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.

The main interferents for ^{99}Tc measurement by ICP-MS are $^{98}\text{MoH}^+$ and $^{99}\text{Ru}^+$. It is of good practice to monitor these interferents during the measurement step to evaluate their impact on m/z 99. Mo and Ru can be measured free of interferences at m/z 95 and 101, respectively. If Mo and/or Ru has an influence on m/z 99, the result obtained should be considered not valid, except if it is corrected. Since Mo and Ru have several natural isotopes, it is possible to use the natural abundance ratio to correct their influence on m/z 99. Such a correction affects the measurement precision and the detection limit of the method. It should only be used if necessary.

7.6 Method verification

A blank solution should be measured at constant interval in a sample sequence. The obtained value shall be subtracted from the measured sample values. If the blank value exceeds the expected background value (within measurement limits), follow the recommendations of the instrument manufacturer and improve the rinsing sequence. In addition, all the results of the measurement obtained before the failing blank and the last valid blank are considered invalid; thus, ideally a blank solution should be measured after each sample measurement.

A quality control solution should be measured at constant interval in a sample sequence. It is verified that the value of the concentration does not deviate from the expected value (within measurement limits). If the deviation exceeds the established measurement limits (optimum sensitivity, optimum stability), follow the recommendations of the instrument manufacturer and perform the optimization of the parameters again. In addition, all the results of the measurement obtained before the failing control and the last valid control are considered invalid; thus, ideally a control solution should be measured before each sample.

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

- participating in intercomparison exercises;
- analysing reference materials;
- analysing spiked samples.

The repeatability of the method should be verified (for example, by replicate measurements).

7.7 Demonstration of analyst capability

If an analyst has not performed this procedure before, a precision and bias test should be performed by running a duplicate measurement of a reference or spiked material. Acceptance limits should be defined by the laboratory.

A similar evaluation should be performed by the analysts who routinely apply this procedure, with a periodicity defined by the laboratory. Acceptance limits should be defined.

8 Expression of results

8.1 Using Re , ^{97}Tc , or ^{98}Tc as a recovery tracer

8.1.1 Calculation of mass of tracer and analyte added

The sample concentration is determined using a tracer, which corrects for losses during the sample preparation. The tracer also corrects for instrumental deviations during the measurement. The tracer solution concentration (ρ_{T}) shall be known, ideally with great precision. Certified solutions are