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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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Contents

Foreword

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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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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.

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, ^{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 (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), ^{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 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 $Bq \cdot l^{-1}$ 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 $Bq \cdot kg^{-1}$ 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.

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.

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 ng·kg^{-1} to 0,5 ng·kg^{-1} (0,1 Bq·kg^{-1} to 0,3 Bq·kg^{-1}), which is much lower than the WHO criteria for safe consumption of drinking water (100 Bq·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 $^{99\text{m}}$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

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	$\mathrm{Bq\cdot kg^{-1}}$	*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
$\overline{N_0}$	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	$\mathrm{N\text{-}N_0}$
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,..., U = k · u
μ	Standard uncertainty	—	Uncertainty of a term such as mass, counts, etc.
$\mu[\mathrm{C}]$	Standard uncertainty of the activity concentration	$\mathrm{Bq\cdot kg^{-1}}$	Standard uncertainty associated with the activity concentration result
$\mu[\mathrm{\rho}]$	Standard uncertainty of the mass concentration	$\mathrm{g\cdot kg^{-1}}$	Standard uncertainty associated with the mass concentration result
ρ	Mass concentration	$\mathrm{g\cdot kg^{-1}}$	Analyte mass for a given radionuclide per sample unit mass
ρ_{A}	Mass concentration of the analyte (^{99}Tc) standard solution	$\mathrm{g\cdot g^{-1}}$	Analyte mass for a given radionuclide per sample unit volume of the standard solution
ρ_{T}	Mass concentration of the tracer solution	$\mathrm{g\cdot g^{-1}}$	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 ± 0,1 × 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, ^{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 contain as little ^{99}Mo as possible[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[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}MoH$^+$ and ^{99}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).

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 mol·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 mol·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 interferents using one of the methods presented in the Annexes A, B or C.

For $^{95\text{m}}$Tc, $^{97\text{m}}$Tc and $^{99\text{m}}$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}MoH$^+$ and ^{99}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 usually employed. A defined quantity of the tracer solution is added to each sample and the mass of solution added (m_{Ts}) is recorded. The mass of tracer (m_T) added to each sample can be calculated using Formula (1):

$$m_T = \rho_T \, m_{Ts} \tag{1}$$

The uncertainty on m_T can be calculated using Formula (2):

$$\mu(m_T) = m_T \sqrt{\mu_{rel}^2(\rho_T) + \mu_{rel}^2(m_{Ts})} \tag{2}$$

To calculate the measurement bias and prepare control or spiked samples, which contains the tracer, a known amount of ^{99}Tc shall be added to the sample. For this purpose, a solution of known concentration of ^{99}Tc (ρ_A), ideally with great precision, is needed. The mass of ^{99}Tc solution added is recorded. The mass of ^{99}Tc added (m_A), can be calculated using Formula (3):

$$m_A = \rho_A \, m_{As} \tag{3}$$

The uncertainty on m_A can be calculated using Formula (4):

$$\mu(m_A) = m_A \sqrt{\mu_{rel}^2(\rho_A) + \mu_{rel}^2(m_{As})} \tag{4}$$

8.1.2 Measurement bias

The measurement bias is a correction factor that corrects for all the measurement deviations between the tracer and the analyte. It includes correction for the mass bias and the variation of signal intensity between the tracer and the analyte. When a stable tracer or internal standards is used, it also corrects for the fact that only one isotope of the element is used for the measurement.

The measurement bias (α) is first determined by measuring with the ICP-MS instrument the number of counts (cps) obtained for ^{99}Tc (N_{net}) and for the tracer (N_{netT}) using a solution containing a known quantity of ^{99}Tc (m_A) and tracer (m_T). The measurement bias is determined using Formula (5):

$$\alpha = \left(m_A \cdot N_{net_T}\right) / \left(m_T \cdot N_{net}\right) \tag{5}$$

And the uncertainty on the measurement bias is determined using Formula (6):

$$\mu(\alpha) = \alpha \sqrt{\mu_{rel}^2(m_A) + \mu_{rel}^2(m_T) + \mu_{rel}^2(N_{net}) + \mu_{rel}^2(N_{netT})} \tag{6}$$

8.1.3 Sample mass concentration

The test sample mass concentration (ρ) in ^{99}Tc is calculated using Formula (7):

$$\rho = \left(\alpha \cdot m_T \cdot N_{net}\right) / \left(m \cdot N_{netT}\right) \tag{7}$$

The uncertainty on the sample mass concentration is calculated using Formula (8):

$$\mu(\rho) = \rho \sqrt{\mu_{rel}^2(m_T) + \mu_{rel}^2(N_{net}) + \mu_{rel}^2(N_{netT}) + \mu_{rel}^2\,\alpha + \mu_{rel}^2(m)} \tag{8}$$

8.1.4 Detection limit

The detection limit (DL) corresponds to 3 times the standard deviation (s_{N0}) associated with the measurement obtained from 10 test portions of a blank sample which has passed through all the steps of the method. The standard deviation on the measured counts is converted in g per kg of sample using Formula (9):

$$DL = \left(\alpha \cdot m_{\mathrm{T}} \cdot \left(\overline{N_0} + 3 \cdot S_{\mathrm{N}_0} \right) \right) / \left(m \cdot N_{\mathrm{netT}} \right) \tag{9}$$

8.1.5 Limit of quantification

The limit of quantification (LOQ) for a given mass, can be evaluated as 10 times the standard deviation (s_{N0}) associated with the measurement obtained for 10 test portions of the blank, which corresponds to 10/3 of the DL as expressed in Formula (10):

$$LOQ = 10/3 \cdot DL \tag{10}$$

8.2 Using $^{95\mathrm{m}}$Tc, $^{97\mathrm{m}}$Tc or $^{99\mathrm{m}}$Tc as a recovery tracer

8.2.1 Calculation of activity of tracer, mass of analyte and mass of internal standard added

The sample activity is determined using a recovery tracer, which corrects for losses during the sample preparation. The instrumental deviations during the measurement are corrected using an internal standard. The activity concentration of the tracer solution (C_{TS}) and the internal standard mass concentration (ρ_{IS}) shall be known, ideally with great precision. A certified solution is usually employed for $^{95\mathrm{m}}$Tc and $^{97\mathrm{m}}$Tc. A defined amount of activity of the tracer solution (C_{TS}) is added to each sample and the mass of the tracer solution added (m_{Ts}) is recorded. $^{99\mathrm{m}}$Tc is usually obtained from a $^{99\mathrm{m}}$Mo generator, thus the activity concentration of $^{99\mathrm{m}}$Tc in the solution prepared by the laboratory shall be determined after its purification before its addition to the samples. The activity of the tracer (C_{T}) added to each sample can be calculated using Formula (11):

$$C_{\mathrm{T}} = C_{\mathrm{Ts}} \cdot m_{\mathrm{Ts}} \tag{11}$$

Note that the activity of tracer added is calculated at a reference date. Consider the isotope decay when calculating the recovery obtained from the gamma spectrometry method.

The uncertainty on C_{T} can be calculated using Formula (12):

$$\mu\left(C_{\mathrm{T}}\right) = C_{\mathrm{T}} \sqrt{\mu_{\mathrm{rel}}^2\left(C_{\mathrm{Ts}}\right) + \mu_{\mathrm{rel}}^2\left(m_{\mathrm{Ts}}\right)} \tag{12}$$

To calculate the measurement bias from the internal standard and prepare control solutions, a known amount of ^{99}Tc (m_{A}) shall be added to the solution. For this purpose, a solution of know mass concentration of ^{99}Tc (ρ_{A}), ideally with great precision, is needed. The mass of the analyte solution added (m_{AS}) is recorded. The mass of ^{99}Tc (m_{A}) can be calculated using Formula (13):

$$m_{\mathrm{A}} = \rho_{\mathrm{A}} \cdot m_{\mathrm{As}} \tag{13}$$

The uncertainty on m_{A} can be calculated using Formula (14):

$$\mu\left(m_{\mathrm{A}}\right) = m_{\mathrm{A}} \sqrt{\mu_{\mathrm{rel}}^2\left(\rho_{\mathrm{A}}\right) + \mu_{\mathrm{rel}}^2\left(m_{\mathrm{As}}\right)} \tag{14}$$

For the preparation of the solution used to determine the measurement bias, a known amount of internal standard (m_{IS}) shall be added to the sample. For this purpose, a solution of know concentration of internal standard (ρ_{IS}), ideally with great precision, is needed. The mass of internal standard solution (m_{ISS}) added is recorded. The mass of internal standard added (m_{IS}) can be calculated using Formula (15):

$$m_{IS} = \rho_{IS} \cdot m_{ISS} \tag{15}$$

The uncertainty on m_{IS} can be calculated using Formula (16):

$$\mu(m_{IS}) = m_{IS}\sqrt{\mu_{rel}^2(\rho_{IS}) + \mu_{rel}^2(m_{ISS})} \tag{16}$$

8.2.2 Purification step recovery

The chemical recovery (R_c) is determined by counting the tracer by gamma spectroscopy. The chemical recovery (R_c) is obtained by dividing the measured activity by the expected activity taking into account the decay of the tracer.

8.2.3 Measurement bias

The measurement bias is a correction factor that corrects for all the measurement deviations between the tracer and the analyte. It includes correction for the mass bias and the variation of signal intensity between the tracer and the analyte. When a stable tracer or internal standards is used, it also corrects for the fact that only one isotope of the element is used for the measurement.

The measurement bias (α) is first determined by measuring with the ICP-MS instrument the number of counts (cps) obtained for ^{99}Tc (N_{net}) and for the internal standard (N_{netIS}) using a solution containing a known quantity of ^{99}Tc ($\mathrm{m_A}$) and internal standard (m_{IS}). The measurement bias is determined using Formula (17):

$$\alpha = \left(m_A \cdot N_{net_{IS}}\right) / \left(m_{IS} \cdot N_{net}\right) \tag{17}$$

And the uncertainty on the measurement bias is determined using Formula (18):

$$\mu(\alpha) = \alpha\sqrt{\mu_{rel}^2(m_A) + \mu_{rel}^2(m_{IS}) + \mu_{rel}^2(N_{net}) + \mu_{rel}^2(N_{netIS})} \tag{18}$$

8.2.4 Sample mass concentration

The sample mass concentration (ρ) in ^{99}Tc is calculated using Formula (19):

$$\rho = \left(\alpha \cdot m_{IS} \cdot N_{net}\right) / \left(R_c \cdot m \cdot N_{net_{IS}}\right) \tag{19}$$

The uncertainty on the sample mass concentration is calculated using Formula (20):

$$\mu(\rho) = \rho\sqrt{\mu_{rel}^2(m_{IS}) + \mu_{rel}^2(N_{net}) + \mu_{rel}^2(N_{netIS}) + \mu_{rel}^2(\alpha) + \mu_{rel}^2(m) + \mu_{rel}^2(R_c)} \tag{20}$$

8.2.5 Detection limit

The detection limit (*DL*) corresponds to 3 times the standard deviation (s_{N0}) associated with the measurement obtained from 10 test portions of a blank sample which has passed through all the steps of the method. The standard deviation on the measured counts is converted in g per kg of sample using Formula (21):

$$DL = \left(\alpha \cdot m_{\mathrm{IS}} \cdot \left(\overline{N_0} + 3 \cdot S_{N_0}\right)\right) / \left(m \cdot N_{\mathrm{netIS}}\right) \tag{21}$$

8.2.6 Limit of quantification

The limit of quantification (*LOQ*) for a given mass, can be evaluated as 10 times the standard deviation (s_{N0}) associated with the measurement obtained for 10 test portions of the blank, which corresponds to 10/3 of the *DL* as express in Formula (22):

$$LOQ = 10/3 \cdot DL \tag{22}$$

8.2.7 Conversion of mass concentration to activity concentration

The mass concentration (ρ), detection limit (*DL*), and limit of quantification (*LOQ*) can be converted to activity concentration (C) by multiplying the values obtained with the specific activity of ^{99}Tc (C_{s}) as in Formulae (23), (24), (25).

$$C = \rho \cdot C_{\mathrm{s}} \tag{23}$$

$$DL_{\mathrm{C}} = DL \cdot C_{\mathrm{s}} \tag{24}$$

$$LOQ_{\mathrm{C}} = LOQ \cdot C_{\mathrm{s}} \tag{25}$$

$$C_s = 6{,}3 \pm 0{,}2 \times 10^8\ \mathrm{Bq} \cdot \mathrm{g}^{-1}$$

The uncertainty of the activity concentration is calculated using Formula (26):

$$\mu(C) = C\sqrt{\mu_{\mathrm{rel}}^2(\rho) + \mu_{\mathrm{rel}}^2(C_{\mathrm{s}})} \tag{26}$$

There is no need to calculate the uncertainties on the *DL*, DL_{c}, *LOQ*, LOQ_{c} because they are estimated values.

8.2.8 Conversion of mass concentration to volume unit

To convert the concentration by sample mass to sample volume, the mass (m) and volume (V) of the sample shall be recorded when performing the measurement. The mass concentration by sample mass $\rho(\mathrm{g \cdot kg^{-1}})$ may be calculated by sample volume $\rho(\mathrm{g \cdot l^{-1}})$ using Formula (27):

$$\rho_{\left(\mathrm{g \cdot l^{-1}}\right)} = m/V \cdot \rho_{\left(\mathrm{g \cdot kg^{-1}}\right)} \tag{27}$$

And the uncertainty can be calculated using Formula (28):

$$\mu\left(\rho_{\left(\mathrm{g \cdot l^{-1}}\right)}\right) = \rho_{\left(\mathrm{g \cdot l^{-1}}\right)} \sqrt{\mu_{\mathrm{rel}}^2 \rho_{\left(\mathrm{g \cdot kg^{-1}}\right)} + \mu_{\mathrm{rel}}^2(m) + \mu_{\mathrm{rel}}^2(V)} \tag{28}$$

8.3 Correction for the presence of ^{99}Tc in the tracer

It is possible that an isotopic tracer of Tc contains a significant amount of ^{99}Tc that can be detected by ICP-MS in which case the ^{99}Tc contribution shall be removed. The contribution of ^{99}Tc coming from the tracer varies according to the chemical recovery and the amount of tracer added. To be able to calculate this contribution, 2 reagent blanks, with no tracer added, shall be prepared using the same method as for the samples. After separation, one of these method blanks is spiked with a known amount of the tracer (m_{TB}). The unspiked and spiked samples are then measured by ICP-MS. The subtraction of the spiked (N_{sp}) and unspiked (N_{us}) counts reagent blanks allows to determine the ^{99}Tc counts (N_T) from the tracer as in Formula (29):

$$N_T = N_{sp} - N_{us} \tag{29}$$

The contribution of ^{99}Tc from the tracer (N_T) varies as a function of the sample recovery (R_c), but not the instrumental background. The ^{99}Tc net counts (N_{net}) for each sample is determined using Formula (30):

$$N_{net} = N - \left(N_{us} + \left(R_c \cdot N_T \cdot m_T / m_{TB}\right)\right) \tag{30}$$

And the uncertainties on N_{net} is calculated using Formula (31):

$$\mu\left(N_{net}\right) = \sqrt{\left(\mu(N)\right)^2 + \left(\mu\left(N_{us}\right)\right)^2 + \left(\mu_{rel}^2\left(R_c\right) + \mu_{rel}^2\left(N_T\right) + \mu_{rel}^2\left(m_T\right) + \mu_{rel}^2\left(m_{TB}\right)\right)^2} \tag{31}$$

9 Test report

The test report shall conform to the requirements given in ISO/IEC 17025 and shall contain at least the following information:

a) a reference to this document, i.e. ISO 22125-2:2019;

b) reference to the measurement and evaluation procedure used;

c) all information necessary for the complete identification of the sample;

d) units in which the results are expressed;

e) test result, expressed as the mass concentrations and /or the activity concentrations expressed as:

- $\rho \pm k.u(\rho)$ or $\rho \pm U$ with the associated k value and units in which the results are expressed;

  and/or

- $C \pm k.u(C)$ or $C \pm U$ with the associated k value and units in which the results are expressed.

Complementary information can be provided such as:

f) limit of application;

g) any relevant information likely to affect and/or explaining the results.

Annex A
(informative)

Method 1 — TEVA[1] resin

A.1 Principle

The sample is filtered, acidified, and oxidized as described in Clause 5. The tracer is added. ^{99}Tc is purified from potential interferents by passing the solution through a TEVA resin, which contains a quaternary amine that selectively extract Tc. Then ^{99}Tc is measured by ICP-MS.

^{95m}Tc, ^{97m}Tc, ^{97}Tc, ^{98}Tc or ^{99m}Tc can be used as a recovery tracer for this method. An approximate detection limit of 2×10^{-10} g·kg^{-1} (0,1 Bq·kg^{-1}) is usually obtained using this method.

A.2 Reagents and apparatus

A.2.1 Reagents

Use only reagents of recognized analytical grade. It is recommended to use acids and bases of trace metal grade or equivalent (a better purity grade can also be employed). Reagent grade acids/bases usually do not have enough purity for ICP-MS measurement.

A.2.1.1 Nitric acid solution, $c(\text{HNO}_3) = 0{,}01$ mol·l^{-1}.

A.2.1.2 Nitric acid solution, $c(\text{HNO}_3) = 0{,}1$ mol·l^{-1}.

A.2.1.3 Nitric acid solution, $c(\text{HNO}_3) = 2$ mol·l^{-1}.

A.2.1.4 Nitric acid solution, $c(\text{HNO}_3) = 12$ mol·l^{-1}.

A.2.1.5 ^{97}Tc standard solution, $c(^{97}\text{Tc}) = 1{,}5 \times 10^{-10}$ mol·l^{-1}, or ^{98}Tc standard solution, $c(^{98}\text{Tc}) = 1{,}5 \times 10^{-10}$ mol·l^{-1}, or ^{95m}Tc standard solution, or ^{97m}Tc standard solution, or ^{99m}Tc standard solution.

A.2.1.6 In standard solution, $c(\text{In(NO}_3)_3) = 1{,}3 \times 10^{-6}$ mol·l^{-1}.

NOTE Other internal standard can be used, but In is considered optimal.

A.2.1.7 Chromatographic extraction resin TEVA, 2 ml cartridge.

A.2.1.8 Laboratory water, distilled or deionized, complying with ISO 3696, grade 3.

A.2.1.9 ^{99}Tc standard solution.

A.2.1.10 Argon gas, at least 99,995 % pure.

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[1] TEVA resin is the trademark of a product supplied by Eichrom Technologies and TrisKem international. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

A.2.2 Apparatus

Usual laboratory equipment including the following:

A.2.2.1 Vacuum filtration system.

A.2.2.2 Filters, of pore size 0,45 µm.

A.2.2.3 Multi-holes vacuum box, e.g. 12 positions.

A.2.2.4 Analytical balance, accuracy 0,1 mg.

A.2.2.5 Disposable polypropylene centrifuge containers, minimum volume of 50 ml.

A.2.2.6 Pipettes.

A.2.2.7 Hot plate.

A.2.2.8 Inductively coupled plasma mass spectrometer.

A.3 Procedure

A.3.1 Sample preparation

Weigh approximately 40 ml of the filtered sample (m) in a 50 ml disposable plastic centrifuge container. Record the sample volume (V) if it is desired to express the final concentration in volume unit.

Add the tracer to the sample and record the mass (m_{TS}). If ^{97}Tc or ^{98}Tc tracer is used: add 1,5 × 10^{-10} mol of ^{97}Tc tracer (m_T) (0,1 ml of ^{97}Tc standard solution, c(^{97}Tc) = 1,5 × 10^{-6} mol·l^{-1}) or 1,5 × 10^{-10} mol of ^{98}Tc tracer (m_T) (0,1 ml of ^{98}Tc standard solution, c(^{98}Tc) = 1,5 × 10^{-6} mol·l^{-1}). If ^{95m}Tc or ^{97m}Tc or ^{99m}Tc is used, add enough ^{95m}Tc or ^{97m}Tc or ^{99m}Tc tracer to give 10 000 counts by gamma spectrometry when counting the sample.

A.3.2 Sample purification by extraction chromatography

Place a 2 ml TEVA resin on top of a multi-holes vacuum box using the appropriate connectors and reservoirs to pass the reagents through the resin.

Precondition the extraction resin with 5 ml of the HNO_3 solution of $c(HNO_3)$ = 0,1 mol·l^{-1}.

Pass the sample through the resin at a flow rate of 1 ml·min^{-1} to 2 ml·min^{-1}.

Rinse the resin with 2 × 25 ml of the HNO_3 solution of $c(HNO_3)$ = 0,01 mol·l^{-1}.

Rinse the resin again with 2 × 20 ml of the HNO_3 solution of $c(HNO_3)$ = 2 mol·l^{-1} if the sample contains a significant amount of Mo or Ru.

Elute Tc from the resin into a glass beaker using 25 ml HNO_3 solution of $c(HNO_3)$ = 12 mol·l^{-1}.

A.3.3 Sample preparation for ICP-MS measurement

Evaporate the sample solution on a hot plate until 1 ml is left. It is suggested not to heat at a temperature above 80 °C to optimize the chemical recovery.

Complete to 10 ml with HNO_3 solution of $c(HNO_3)$ = 0,1 mol·l^{-1}.

A.3.4 Measurement

A.3.4.1 Using ^{97}Tc or ^{98}Tc tracers

Measure ^{99}Tc and the tracer in the samples by ICP-MS.

A.3.4.2 Using ^{95m}Tc, ^{97m}Tc or ^{99m}Tc tracers

Measure the tracer in the samples by gamma spectrometry and calculate the chemical recovery (R_c);

Add 1,3 × 10^{-10} mol of ^{115}In (0,1 ml of $c(\mathrm{In(NO_3)_3}) = 1{,}3 \times 10^{-6}$ mol·l^{-1}) internal standard (or equivalent internal standard) to the sample and record the mass of solution added (m_{ISS}).

Measure ^{99}Tc and the internal standard in the samples by ICP-MS.

Annex B
(informative)

Method 2 — TRU[2] resin

B.1 Principle

The sample is filtered, acidified, and oxidized as described in Clause 5. The tracer is added. ^{99}Tc is purified from potential interferents by proceeding to a calcium phosphate co-precipitation followed by a solid phase extraction using an extraction chromatography resin (TRU resin) containing tributyl phosphate (TBP). Then ^{99}Tc is measured by ICP-MS.

Re, ^{95m}Tc, ^{97m}Tc, ^{97}Tc, ^{98}Tc or ^{99m}Tc can be used as a recovery tracer for this method. An approximate detection limit of 2×10^{-10} g·kg^{-1} (0,1 Bq·kg^{-1}) is usually obtained using this method.

B.2 Reagents and apparatus

B.2.1 Reagents

Use only reagents of recognized analytical grade. It is recommended to use acids and bases of trace metal grade or equivalent (a better purity grade can also be employed). Reagent grade acids/bases usually do not have enough purity for ICP-MS measurement.

B.2.1.1 Phosphoric acid solution, $c(H_3PO_4) = 14{,}8$ mol·l^{-1}.

B.2.1.2 Hydrochloric acid solution, $c(HCl) = 12{,}1$ mol·l^{-1}.

B.2.1.3 Sulfuric acid solution, $c(H_2SO_4) = 18$ mol·l^{-1}.

B.2.1.4 Sulfuric acid solution, $c(H_2SO_4) = 2$ mol·l^{-1}.

B.2.1.5 Calcium chloride solution, $c(CaCl_2) = 1$ mol·l^{-1}.

B.2.1.6 Ammonium hydroxide solution, $c(NH_4OH) = 14{,}5$ mol·l^{-1}.

B.2.1.7 Hydrogen peroxide solution, $c(H_2O_2) = 8{,}82$ mol·l^{-1}.

B.2.1.8 Re standard solution, $c(NH_4ReO_4) = 8{,}06 \times 10^{-6}$ mol·l^{-1}, or ^{97}Tc standard solution, $c(^{97}Tc) = 1{,}5 \times 10^{-10}$ mol·l^{-1}, or ^{98}Tc standard solution, $c(^{98}Tc) = 1{,}5 \times 10^{-10}$ mol·l^{-1}, or ^{95m}Tc standard solution, or ^{97m}Tc standard solution, or ^{99m}Tc standard solution.

B.2.1.9 In standard solution, $c(In(NO_3)_3) = 1{,}3 \times 10^{-6}$ mol·l^{-1}.

NOTE Other internal standard can be used, but In is considered optimal.

B.2.1.10 Chromatographic extraction resin TRU, 2 ml cartridge.

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[2] TRU resin is the trademark of a product supplied by Eichrom Technologies and TrisKem international. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

B.2.1.11 Laboratory water, distilled or deionised, complying with ISO 3696 grade 3.

B.2.1.12 ^{99}Tc standard solution.

B.2.1.13 Argon gas, at least 99,995 % pure.

B.2.2 Apparatus

Usual laboratory equipment including the following.

B.2.2.1 Vacuum filtration system.

B.2.2.2 Filters, of pore size 0,45 µm.

B.2.2.3 Centrifuge.

B.2.2.4 Multi-holes vacuum box, e.g. 12 positions.

B.2.2.5 Analytical balance, accuracy 0,1 mg.

B.2.2.6 Disposable polypropylene centrifuge container, minimum volume of 50 ml.

B.2.2.7 Pipettes.

B.2.2.8 Hot plate.

B.2.2.9 Inductively coupled plasma mass spectrometer.

B.3 Procedure

B.3.1 Sample preparation

Weigh approximately 40 ml of the filtered sample (m) in a 50 ml disposable plastic centrifuge container. Record the sample volume (V) if it is desired to express the final concentration in volume unit.

Add the tracer to the sample and record the mass (m_{TS}). If ^{97}Tc, ^{98}Tc, or Re tracer is used: add 8,06 × 10^{-10} mol of Re tracer (m_T) (0,1 ml of Re standard solution, $c(NH_4ReO_4) = 8{,}06 \times 10^{-6}$ mol·l^{-1}) or 1,5 × 10^{-10} mol of ^{97}Tc tracer (m_T) (0,1 ml of ^{97}Tc standard solution, $c(^{97}Tc) = 1{,}5 \times 10^{-6}$ mol·l^{-1}) or 1,5 × 10^{-10} mol of ^{98}Tc tracer (m_T) (0,1 ml of ^{98}Tc standard solution, $c(^{98}Tc) = 1{,}5 \times 10^{-6}$ mol·l^{-1}). If ^{95m}Tc or ^{97m}Tc or ^{99m}Tc is used, add enough ^{95m}Tc or ^{97m}Tc or ^{99m}Tc tracer to give 10 000 counts by gamma spectrometry when counting the sample.

Add 5 × 10^{-4} mol of Ca^{2+} (0,5 ml of calcium chloride solution, $c(CaCl_2) = 1$ mol·l^{-1}) (except for sea water, since the amount of calcium is already sufficient), 2,96 × 10^{-3} mol of H_3PO_4 (0,2 ml of H_3PO_4 solution, $c(H_3PO_4) = 14{,}8$ mol·l^{-1}), and 8,82 × 10^{-4} mol of H_2O_2 (0,1 ml of H_2O_2 solution, $c(H_2O_2) = 8{,}82$ mol·l^{-1}). Mix the sample to homogenize and wait 5 min.

B.3.2 Calcium phosphate precipitation

Add 1,45 × 10^{-2} mol of NH_4OH (1 ml of 14,5 mol·l^{-1} NH_4OH solution, $c(NH_4OH) = 14{,}5$ mol l^{-1}) and mix the sample. Wait 5 min for the precipitate to be formed.

Centrifuge the sample.

Transfer the supernatant in a disposable 50 ml plastic container and add 7,92 × 10^{-2} mol of H_2SO_4 (4,4 ml of H_2SO_4 solution, c(H_2SO_4) = 18 mol·l^{-1}) to the supernatant. Mix the sample and wait 30 min to let the sample cool to room temperature.

CAUTION — The dissolution of H_2SO_4 in water is highly exothermic, which could make the plastic softer. It is important to be careful when closing the container cap.

B.3.3 Sample purification by extraction chromatography

Place a 2 ml extraction resin (i.e. approximately 0,7 g of dry resin) on top of a multi-holes vacuum box using the appropriate connectors and reservoirs to pass the reagents through the resin.

Precondition the extraction resin with 10 ml of the H_2SO_4 solution of $c(H_2SO_4)$ = 2 mol·l^{-1}.

Pass the sample through the resin at a flow rate of 3 ml·min^{-1} to 4 ml·min^{-1}.

Rinse the resin with 30 ml of the H_2SO_4 solution $c(H_2SO_4)$ = 2 mol·l^{-1}.

Bring to boiling water in a clean beaker on a hot plate (near boiling water).

Elute Tc and Re from the resin using approximately 14 ml of the near boiling water in a 50 ml plastic container. The elution should be done as quickly as possible to maintain the temperature of the water high. The volume of water can be measured by a graduated reservoir on top of the resin such as a syringe. Let the solution cool to room temperature.

CAUTION — Wear appropriate protective gloves when manipulating the hot water beaker to avoid burns.

B.3.4 Measurement

B.3.4.1 Using ^{97}Tc, ^{98}Tc or Re tracers

Measure ^{99}Tc and the tracer in the samples by ICP-MS.

B.3.4.2 Using ^{95m}Tc, ^{97m}Tc or ^{99m}Tc tracers

Measure the tracer in the samples by gamma spectrometry and calculate the chemical recovery (R_c).

Add 1,3 × 10^{-10} mol of ^{115}In (0,1 ml of $c(In(NO_3)_3)$ = 1,3 × 10^{-6} mol·l^{-1}) internal standard (or equivalent internal standard) to the sample and record the mass of solution added (m_{ISS});

Measure ^{99}Tc and the internal standard in the samples by ICP-MS.

Annex C
(informative)

Method 3 — Anion exchange resin

C.1 Principle

The sample is filtered, acidified, and oxidized as described in Clause 5. The tracer is added. ^{99}Tc is purified from potential interferents by passing the solution through an anion exchange resin, which contains a quaternary amine that selectively extract Tc. Then ^{99}Tc is measured by ICP-MS.

^{95m}Tc, ^{97m}Tc, ^{97}Tc, ^{98}Tc or ^{99m}Tc can be used as a recovery tracer for this method. An approximate detection limit of $0,5 \times 10^{-10}$ g·kg^{-1} (0,3 Bq·kg^{-1}) is usually obtained using this method.

C.2 Reagents and apparatus

C.2.1 Reagents

Use only reagents of recognized analytical grade. It is recommended to use acids and bases of trace metal grade or equivalent (a better purity grade can also be employed). Reagent grade acids/bases usually do not have enough purity for ICP-MS measurement.

C.2.1.1 Hydrochloric acid solution, $c(\mathrm{HCl}) = 0,5$ mol·l^{-1}.

C.2.1.2 Nitric acid solution, $c(\mathrm{HNO_3}) = 10$ mol·l^{-1}.

C.2.1.3 Nitric acid solution, $c(\mathrm{HNO_3}) = 0,5$ mol·l^{-1}.

C.2.1.4 ^{97}Tc standard solution, $c(^{97}\mathrm{Tc}) = 1,5 \times 10^{-10}$ mol·l^{-1}, or ^{98}Tc standard solution, $c(^{98}\mathrm{Tc}) = 1,5 \times 10^{-10}$ mol·l^{-1}, or ^{95m}Tc standard solution, or ^{97m}Tc standard solution, or ^{99m}Tc standard solution.

C.2.1.5 In standard solution, $c(\mathrm{In(NO_3)_3}) = 1,3 \times 10^{-6}$ mol·l^{-1}.

NOTE Other internal standard can be used, but In is considered optimal.

C.2.1.6 Strong basic anion exchange resin, 0,8 ml.

C.2.1.7 Laboratory water, distilled or deionised, complying with ISO 3696 grade 3.

C.2.1.8 ^{99}Tc standard solution.

C.2.1.9 Argon gas, at least 99,995 % pure.

C.2.2 Apparatus

Usual laboratory equipment including the following.

C.2.2.1 Vacuum filtration system.