Water quality — Zirconium ‑93 — Test method using ICP-MS

Qualité de l'eau — Zirconium 93 — Méthode d'essai par ICP-MS

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Foreword

INTRODUCTION

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This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements.*

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

Radionuclides are present throughout the environment; thus, water bodies (e.g. surface waters, ground waters, and sea waters) contain radionuclides, which can be of either natural or anthropogenic origin.

— Naturally-occurring radionuclides, including ^{3}H, ^{14}C, ^{40}K and those originating from the thorium and uranium decay series, in particular ^{210}Pb, ^{210}Po, ^{222}Rn, ^{226}Ra, ^{228}Ra, ^{227}Ac, ^{93}Zr, ^{234}U, and ^{238}U, can be found in water bodies due to either natural processes (e.g. desorption from the soil, and runoff by rain water) or released from technological processes involving naturally occurring radioactive materials (e.g. mining, mineral processing, oil, gas, and coal production, water treatment, and production and use of phosphate fertilisers).

— Anthropogenic radionuclides, such as ^{55}Fe, ^{59}Ni, ^{63}Ni, ^{90}Sr, and ^{99}Tc, transuranic elements (e.g. Np, Pu, Am, and Cm) and some gamma emitting radionuclides, such as ^{60}Co and ^{137}Cs, can also be found in natural waters. Small quantities of anthropogenic radionuclides can be discharged from nuclear facilities to the environment as a result of authorized routine releases. The radionuclides present in liquid effluents are usually controlled before being discharged into the environment[1] and water bodies. Anthropogenic radionuclides used in medical and industrial applications can be released to the environment after use. Anthropogenic radionuclides are also found in waters due to contamination from fallout resulting from above-ground nuclear detonations and accidents such as those that have occurred at the Chernobyl and Fukushima nuclear facilities.

Radionuclide activity concentrations in water bodies can vary according to local geological characteristics and climatic conditions and can be locally and temporally enhanced by releases from nuclear facilities during planned, existing, and emergency exposure situations.[2][3] Some drinking water sources can thus contain radionuclides at activity concentrations that can present a human health risk. The World Health Organization (WHO) recommends to routinely monitor radioactivity in drinking waters[4] and to take proper actions when needed to minimize the health risk.

National regulations usually specify the activity concentration limits that are authorized in drinking waters, water bodies, and liquid effluents to be discharged to the environment. These limits can vary for planned, existing, and emergency exposure situations. As an example, during either a planned or existing situation, the WHO guidance level for ^{93}Zr in drinking water is 100 Bq·l^{-1}, see NOTES 1 and 2. Compliance with these limits is assessed by measuring radioactivity in water samples and by comparing the results obtained, with their associated uncertainties, as specified by ISO/IEC Guide 98-3 and ISO 5667-20[5].

NOTE 1   If the value is not specified in Annex 6 of Reference [4], the value has been calculated using the formula provided in Reference [4] and the dose coefficient data from References [6] and [7].

NOTE 2   The guidance level calculated in Reference [4] is the activity concentration that, with an intake of 2 l·d^{-1} of drinking water for one year, results in an effective dose of 0,1 mSv·a^{-1} to members of the public. This is an effective dose that represents a very low level of risk to human health and which is not expected to give rise to any detectable adverse health effects[4].

This document contains method(s) to support laboratories, which need to determine ^{93}Zr in water samples.

The method described in this document can be used for various types of waters (see Clause 1). For radiometric methods, minor modifications such as sample volume and counting time can be made if needed to ensure that the characteristic limit, decision threshold, limit of detection, and uncertainties are below the required limits. For ICP-MS methods, minor modifications to, for example, the sample pre-concentration volume and the interference separation can be made if needed to ensure that limit of detection, limit of quantification and uncertainties are below the required limits. This can be done for several reasons such as emergency situations, lower national guidance limits, and operational requirements.

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WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use.

IMPORTANT — It is essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies a method to determine ^{93}Zr by inductively coupled plasma mass spectrometry (ICP-MS). The mass concentrations obtained can be converted into an activity concentration. The method is applicable to test samples of drinking water, rainwater, surface and ground water, marine water, as well as cooling water, industrial water, domestic, and industrial wastewater after proper sampling and handling, and test sample preparation.

The limit of detection depends on the sample volume, the instrument used, the background count rate, the detection efficiency and the chemical yield. In this document, the limit of detection of the method described in this document, using currently available apparatus, is approximately 90 Bq·l^{-1} (or Bq·kg^{-1}), which is lower than the WHO criteria for safe consumption of drinking water (100 Bq·l^{-1})[4][4].

This The method described in this document covers the measurement of ^{93}Zr in water at activity concentrations between 0,09 Bq·l^{-1} and 100 Bq·l^{-1}. Samples with higher activity concentrations than 100 Bq·l^{-1} can be measured if a dilution is performed.

The method described in this document is applicable in the event of an emergency situation.

In this method, Filtration of the test sample is necessary for the method described in this document. The analysis of ^{93}Zr adsorbed to suspended matter is not covered by this method. The analysis of the insoluble fraction requires a mineralization step that is not covered by this document. In this case, the measurement is made on the different phases obtained.

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 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 water*

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

ISO 17294-1:2004 2024, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines*

ISO 17294-2:20162023, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of selected elements including uranium isotopes*

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

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

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Symbols

α	Measurement bias constant which allows a correction for signal intensity bias between the tracer and the analyte	
C	Activity concentration	$Bq \cdot g^{-1}$
C_s	Specific activity corresponding to one gram of the radionuclide	$Bq \cdot g^{-1}$
C_T	Activity concentration of the tracer	Bq
C_{TS}	Activity of the tracer added to a sample	$Bq \cdot g^{-1}$
k	Coverage factor for uncertainties	
L_D	Detection limit in mass concentration, the lowest mass concentration that can be considered statistically different from a blank sample	$g \cdot kg^{-1}$
L_Q	Limit of quantification, the lowest mass concentration that can be quantified with statistical uncertainty	$g \cdot kg^{-1}$
m	Mass of sample	kg
m/z	Mass-to-charge ratio measured by ICP-MS	
m_A	Analyte mass added to a spiked solution	g
m_{AS}	Mass of the analyte solution added to a control sample or for measurement calculation	g
m_C	Mass of the calibration standard tracer added to sample	g
m_{CS}	Mass of calibration standard solution added to sample	g
m_{IS}	Mass of the internal standard added to blank and sample	g
m_{ISS}	Mass of the internal standard solution added to a blank or a sample	g
m_T	Mass of tracer added to blank and sample	g
m_{TB}	Mass of tracer added to the reagent blank	g
m_{TS}	Mass of tracer solution added to blank or sample	g
N	Counts per second measured when performing ICP-MS measurement for a sample at a given mass-to-charge ratio	Counts s^{-1}

N_0	Counts per second measured when performing ICP-MS measurement of a blank sample at a given mass-to-charge ratio	Counts·s⁻¹
$\bar{N}_0$	Average number of counts per second for several blank samples measured by ICP-MS at a given mass-to-charge ratio	Counts·s⁻¹
N_{net}	Net number of counts per second, $N - N_0$	Counts·s⁻¹
N_{netIS}	Net number of counts per second at the internal standard mass-to-charge ratio	Counts·s⁻¹
N_{netT}	Net number of counts per second in samples where a tracer has been added to assess chemical recovery	Counts·s⁻¹
N_{SP}	Net number of counts per second in the spiked reagent blank	Counts·s⁻¹
N_T	Counts per second at analyte mass-to-charge ratio present as impurities	Counts·s⁻¹
N_{US}	Net number of counts per second in the unspiked reagent blank sample	Counts·s⁻¹
R_c	Chemical recovery following purification measured by ICP-MS	
S_{N_0}	Standard deviation obtained by measurement of 10 test portions of the blank sample	Counts·s⁻¹
U	Expanded uncertainty and the coverage factor k with $k = 1, 2, ..., U = k \cdot u$	Bq
$u(C)$	Standard uncertainty of the activity concentration result	Bq
$u(\rho)$	Standard uncertainty associated with the measurement result	g
ρ	Analyte mass concentration	g·kg⁻¹
ρ_A	Mass concentration of the analyte standard solution	g·g⁻¹
ρ_c	Mass concentration of the calibration standard solution	g·g⁻¹
ρ_{IS}	Mass of internal standard element or isotope per unit volume of the internal standard solution	g·l⁻¹
ρ_T	Mass concentration of tracer solution	g·g⁻¹
ρ_V	Mass of analyte per sample unit volume	g·l⁻¹
V	Volume of sample	l
α	Measurement bias constant which allows a correction for signal intensity bias between the tracer and the analyte	

5 Principle

The principle of measurement of analysis using ICP-MS is described in ISO 17294-1 and ISO 17294-2.

ICP-MS has been successfully used to measure the concentration of ^{93}Zr in water samples[8],[9].

The results can be converted in activity concentrations using specific activity as a conversion factor given in Table 1. The typical measurement time is several minutes per sample, including sample uptake, counting time and washout before the next sample.

Table 1 — Zirconium-93 half-life and specific activity[10]

Isotope	Half-life	Specific activity
	years	Bq·g⁻¹
^{93}Zr	$1{,}61\,(6)\cdot10^6$	$88{,}3\,(33)\cdot10^7$

An example of the limit of detection that can be obtained with ICP-MS is given in Table 2~~Table 2~~.~~.~~

Table 2 — Examples of ~~l~~imit of detection[8],[9][8][9]

Isotope	Limit of detection $\mu g \cdot l^{-1}$	Limit of detection $Bq \cdot l^{-1}$
^{93}Zr	1,0	90

Radionuclide measurement by ICP-MS is affected by several interferences~~,~~ which are outlined in Table 3~~Table 3~~.~~.~~

Table 3 — Interferences affecting ICP-MS measurement

~~Interference name~~Name of interference	Description	^{93}Zr interference
Isobaric interference	~~A~~Stable or radioactive ~~isotope~~isotopes with a similar mass to the analyte	^{93}Nb, ^{93}Mo
Polyatomic interference	Stable or radioactive isotopes combining in ~~the~~plasma to form a polyatomic ion with a similar mass to the analyte	$^{92}Mo^1H$, $^{92}Zr^1H$, $^{77}Se^{16}O$, $^{61}Ni^{16}O_2$, $^{53}Cr^{40}Ar$
Tailing	Stable or radioactive isotopes of one or two mass units on either side of the analyte with a relatively high abundance ($>10^6$) relative to the analyte	^{92}Zr, ^{92}Mo

It is important to ensure that all potential interferences have been removed prior to measurement. The most significant interference affecting ^{93}Zr measurement by ICP-MS is stable isobaric ^{93}Nb. Because ^{93}Nb is monoisotopic, it is more challenging to correct for this interference and separation is critically important. A possible radioactive isobaric interference from ^{93}Mo shall also be considered. The other interferences that shall be considered are polyatomic: $^{92}Mo^1H$, $^{92}Zr^1H$, $^{77}Se^{16}O$, $^{61}Ni^{16}O_2$ and $^{53}Cr^{40}Ar$. Tailing interferences from ^{92}Zr and ^{92}Mo shall also be considered.

It is important to know the interference separation factor achievable by chemical separation and the ICP-MS instrument used. This can initially be assessed by running stable element standards at increasing concentrations to monitor the impact at $m/z = 93$ before and after chemical and/or instrument separation.

Certain ICP-MS designs (i.e. those equipped with a reaction cell) can potentially remove interferences online without the need for offline chemical separation. In such cases, matrix-matched calibration standards should be prepared using an appropriate reference standard or standard addition.

An aliquot of a water sample can be directly measured by ICP-MS to determine the stable element composition. High matrix samples (such as seawater) ~~might~~can need to be diluted to a greater extent before this measurement, depending on the sample introduction system of the instrument used; some designs offer online aerosol dilution capability that can run high matrix samples such as seawater without prior dilution.

If any interference has an impact on the ^{93}Zr result that cannot be corrected for, then the result cannot be considered to be valid. The interference from $^{92}Mo^1H$ can be monitored at the interference-free Mo isotope at $m/z = 95$ ~~and,~~ by correcting for ^{92}Mo using natural isotopic ratios. The same approach can be used to correct for $^{92}Zr^1H$ by monitoring Zr at $m/z = 91$. Selenium can be monitored at $m/z = 78$, nickel can be monitored at $m/z = 60$ and chromium can be monitored at $m/z = 52$. This type of correction should only be used if absolutely necessary, as it increases the measurement uncertainty and affects the limit of detection and measurement precision.