ISO/TC 147/SC 3

Secretariat: AFNOR

Date: 2024-06-04

Water quality — Strontium 90 — Test method using ICP-MS

First edition

Date: 2024-04-27

iTeh Standards (https://standards.iteh.ai) Document Preview

ISO/FDIS 472

https://standards.iteh.ai/catalog/standards/iso/87d5cf26-69de-4f25-9a37-961b13bccc3f/iso-fdis-4721

Edited DIS - MUST BE USED FOR FINAL DRAFT

Qualité de l'eau — Strontium 90 — Méthode d'essai par ICP-MS

FDIS stage

iTeh Standards (https://standards.iteh.ai) Document Preview

ISO/FDIS 472

© ISO 20232024

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office CP 401 • Ch. de Blandonnet 8 CH-1214 Vernier, Geneva Phone: + 41 22 749 01 11 <u>EmailE-mail</u>: copyright@iso.org Website: <u>www.iso.org</u>

Published in Switzerland

iTeh Standards (https://standards.iteh.ai) Document Preview

ISO/FDIS 4721

Contents

Forew	ord	vi
Introd	uction	vii
1	Scope	1
2	Normative references	1
3	Terms and definitions	2
4	Symbols	
5	Principle	
5 6	Sampling and sample storage	
-		
7	Chemical reagents and equipment	
7.1 7.2	General	
7.2 7.3	Chemical reagents	
/.3	Apparatus	
8	Chemical separation	6
9	Quality control	7
9.1	General	
9.2	Variables that can influence the measurement	
9.3	Instrument verification	
9.4	Method verification	
10	Expression of results	Q
10.1	General	
10.1	Data analysis	
10.2	Background	
10.3	Mass bias evaluation	
10.4	Use of naturally occurring stable Sr as chemical yield tracer	
- 177710	Internal standard	
	Determination of stable strontium concentration	
	Mass bias evaluation	-
	Sample mass concentration	
	Limit of detection	
	Limit of quantification	
	Isotope dilution method using enriched isotope spike	
	General	
	Isotope spike solution	
	Mass bias evaluation	
	Sample mass concentration	
	Limit of detection	
10.6.6	Limit of quantification	13
10.7	Conversion of mass concentration to activity concentration	14
10.8	Conversion from mass to volume units	14
11	Test report	14
Anney	A (informative) Chemical separation of strontium by crown ether-based extraction	
. mitta	chromatographic resin — Sample strontium solution in 4 mol·l ⁻¹ HNO ₃	16
_		10
Annex	B (informative) Chemical separation of strontium by strontium-specific extraction chromatographic resin — Sample strontium solution in 0,01 mol·l ⁻¹ HNO ₃	18

Annex C (informative) Chemical separation of strontium for large volume water sample by	
cation exchange resin and strontium-specific extraction chromatographic resin	20
Riblingraphy	22

iTeh Standards (https://standards.iteh.ai) Document Preview

ISO/FDIS 472

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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at <u>www.iso.org/patents</u>. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see <u>www.iso.org/iso/foreword.html</u>.

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

SO/FDIS 4721

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 <u>www.iso.org/members.html</u>.

Introduction

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

- — Naturally-occurring radionuclides, including ³H, ¹⁴C, ⁴⁰K and those originating from the thorium and uranium decay series, in particular ²¹⁰Pb, ²¹⁰Po, ²²²Rn, ²²⁶Ra, ²²⁸Ra, ²²⁷Ac, ²³²Th, ²³¹Pa, ²³⁴U₇ and ²³⁸U, 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, gas₇ and coal production, water treatment, and the-production and use of phosphate fertilisers);
- Anthropogenic radionuclides, such as ⁵⁵Fe, ⁵⁹Ni, ⁶³Ni, ⁹⁰Sr, and ⁹⁹Tc, transuranic elements (e.g. Np, Pu, Am, and Cm); and some gamma emitting radionuclides, such as ⁶⁰Co and ¹³⁷Cs, 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 to the environment^[14]] and water bodies. Anthropogenic radionuclides used for 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^{[21,[3,[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,[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 ⁹⁰Sr in drinking water is 10 Bq·l^{-1[4[4]}, 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[5]}, J.

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

NOTE 2 The guidance level calculated in Reference [4[4]] is the activity concentration that results in an effective dose of 0,1 mSv·a⁻¹ to members of the public for an intake of 2 l·d⁻¹ of drinking water for one year. 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[4],].

This document contains method(s) to support laboratories, which need to determine ⁹⁰Sr in water samples. The method(s) described in this document can be used for various types of waters (see <u>Clause 1Clause 1).)</u>. 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, detection limit, and uncertainties are below the required limits. For ICP-MS methods, minor modifications <u>such asto</u>, for example, the sample pre-concentration volume and <u>the</u> interference separation, can be made if needed to ensure that the 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.

iTeh Standards (https://standards.iteh.ai) Document Preview

ISO/FDIS 472

Water quality — Strontium 90 – Test method using ICP-MS

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 methods to determine strontium-90 (⁹⁰Sr) by inductively coupled plasma mass spectrometry (ICP-MS). The mass concentrations obtained can be converted into an activity concentration.

The method <u>described in this document</u> is applicable to test samples of supply<u>/ water</u>, drinking water, rainwater, surface and ground water, as well as cooling water, industrial water, domestic₇ and industrial wastewater after proper sampling and handling₇ 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 and chemical pre-concentration, is approximately 5 Bq·l⁻¹, which is lower than the WHO criteria for safe consumption of drinking water (10 Bq·l⁻¹)[4][4].

<u>ThisThe</u> method <u>described in this document</u> covers the measurement of 90 Sr in water at activity concentrations up to 1 000 Bq·l⁻¹. Samples with higher activity concentrations than 1 000 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 ⁹⁰Sr 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/IEC Guide 99:2007, International vocabulary of metrology — Basic and general concepts and associated terms (VIM)

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 $17294-1:\frac{20042024}{2024}$, Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines

ISO 17294-<u>-</u>2:<u>20162023</u>, 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, ISO/IEC Guide 99 and ISO 80000-10 apply.

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

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

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

4 Symbols

iTeh Standards

а	Regression line slope to s://standards.iteh.ai)	s-¹∙kg-1
b	Coordinate at the origin of the regression line	S^{-1}
С	Activity concentration corresponding to the mass concentration, ρ_{\star} measured for a given radionuclide	Bq∙kg ⁻¹
C _{int} https: €₅CS	Internal standard correction factor //standards/tenal/catalog/standards/iso/87d5ct26-69de-4t25-9a37-961b13bccc3f/iso-fdi Specific activity corresponding to one gram of the radionuclide	s-4721 Bq∙g-1
I_{84}	Isotopic abundance of ⁸⁴ Sr: 0, 0056<u>005</u> 6 (natural isotopic abundance is assumed)	
I_{86}	Isotopic abundance of ⁸⁶ Sr: 0, 0986<u>098</u> 6 (natural isotopic abundance is assumed)	
<u>k</u>	<u>Coverage facto used as a multiplier of the combined standard uncertainty in order to</u> <u>obtain an expanded uncertainty</u>	=
$L_{\rm D}$	Limit of detection, the lowest mass concentration that can be detected with statistical uncertainty	g∙kg⁻¹
L_{Q}	Limit of quantification, the lowest mass concentration that can be quantified with statistical uncertainty	g∙kg⁻¹
М	Isotope mass number	
т	Mass of sample	kg
Δm	Mass difference	
$m_{ m c}$	Mass of calibration standard tracer added to standard	g
m_{cs}	Mass of calibration standard added to standard	g
$m_{ m IS}$	Mass of the internal standard added to blank and sample	g
$m_{\rm ISS}$	Mass of the internal standard solution added to a blank or a sample	g

$m_{ m SP}$	Mass of the enriched spike solution	g
m <mark>≁_</mark> _z	Mass_to_charge ratio measured by ICP-MS	g
Ν	Counts per second measured when performing ICP-MS measurement for a sample at a given mass-to-charge ratio	S ⁻¹
N ₀	Counts per second measured when performing ICP-MS measurement of a blank sample at a given mass-to-charge ratio	S ⁻¹
ℕ0	Average number of counts per second for several blank samples measured by ICP-MS at a given mass-to-charge ratio	S ⁻¹
N _{net}	Net number of counts per second, $N - N_0$	S ^{−1}
r	Measured isotopic ratio	—
R	Corrected isotopic ratio	—
S_{N_0}	Standard deviation obtained by measurement of ten test portions of the blank sample	—
U	Expanded uncertainty and the coverage factor k with $k = 1, 2, \dots, U = k \cdot u$	—
u	Uncertainty such as counts per second, mass added etc.	
u(c)	Standard uncertainty of the activity concentration result	Bq∙kg ⁻¹
u(c) <u>u_{cal}</u>	Standard uncertainty of the activity concentration result <u>Uncertainty associated with the calibration</u>	Bq∙kg ⁻¹ —
		Bq·kg ⁻¹
<u>U_{cal}</u>	<u>Uncertainty associated with the calibration</u> Uncertainty on isotopic abundance of ⁸⁴ Sr: 0, 0002 <u>000 2</u> (natural isotopic abundance is	Bq·kg ⁻¹
<u>U_{cal}</u> u(I ₈₄)	Uncertainty associated with the calibration Uncertainty on isotopic abundance of ⁸⁴ Sr: 0, 0002 <u>000</u> (natural isotopic abundance is assumed) Uncertainty on isotopic abundance of ⁸⁶ Sr: 0, 0020 <u>002</u> (natural isotopic abundance is	Bq·kg ⁻¹ — — —
<u>И_{саl}</u> и(I ₈₄) и(I ₈₆)	Uncertainty associated with the calibration Uncertainty on isotopic abundance of ⁸⁴ Sr: 0, 0002 000 <u>2</u> (natural isotopic abundance is assumed) Uncertainty on isotopic abundance of ⁸⁶ Sr: 0, 0020002 0 (natural isotopic abundance is assumed)	Bq⋅kg ⁻¹ — — — 1
<u>U_{cal}</u> u(I ₈₄) u(I ₈₆) u(ρ)	Uncertainty associated with the calibration Uncertainty on isotopic abundance of ⁸⁴ Sr: 0, 0002 000 2 (natural isotopic abundance is assumed) Uncertainty on isotopic abundance of ⁸⁶ Sr: 0, 0020002 0 (natural isotopic abundance is assumed) Standard uncertainty associated with the measurement result	Bq⋅kg ⁻¹ — — 1 8-4721
$\frac{u_{cal}}{u(I_{84})}$ $u(I_{86})$ $u(\rho)$ V	Uncertainty associated with the calibrationUncertainty on isotopic abundance of ⁸⁴ Sr: 0,00020002 (natural isotopic abundance is assumed)Uncertainty on isotopic abundance of ⁸⁶ Sr: 0,0020002 0 (natural isotopic abundance is assumed)Standard uncertainty associated with the measurement resultVolume of sampleMeasurement bias constant which allows a correction for signal intensity bias between	Bq·kg ⁻¹ — — 1 g·kg ⁻¹
$\frac{u_{cal}}{u(I_{84})}$ $u(I_{86})$ $u(\rho)$ V $h\alpha_{ps:}$	Uncertainty associated with the calibration Uncertainty on isotopic abundance of ⁸⁴ Sr: 0, 0002 000 <u>2</u> (natural isotopic abundance is assumed) Uncertainty on isotopic abundance of ⁸⁶ Sr: 0, 0020002 0 (natural isotopic abundance is assumed) Standard uncertainty associated with the measurement result Volume of sample Measurement bias constant which allows a correction for signal intensity bias between the tracer and the analyte	= - - 1 s-4 72 1
$\frac{u_{cal}}{u(I_{84})}$ $u(I_{86})$ $u(\rho)$ V $h\alpha ps:$ ρ	Uncertainty associated with the calibrationUncertainty on isotopic abundance of ⁸⁴ Sr: 0,0002,000 2 (natural isotopic abundance is assumed)Uncertainty on isotopic abundance of ⁸⁶ Sr: 0,002,0002 0 (natural isotopic abundance is assumed)Standard uncertainty associated with the measurement resultVolume of sampleMeasurement bias constant which allows a correction for signal intensity bias between the tracer and the analyteAnalyte mass for a given radionuclide per sample unit mass	 l s-4 72 1 g·kg ⁻¹

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 mass concentration of ^{90}Sr in water samples $[\underline{11}[\underline{12}][\underline{13}][\underline{14}]]$

The results can be converted in activity concentrations using specific activity as a conversion factor<u>, which is</u> given in <u>Table 1</u><u>Table 2</u>.