
**Water quality — Nickel-59 and
nickel-63 —**

**Part 2:
Test method using ICP-MS**

Qualité de l'eau — Nickel-59 et nickel-63 —

Partie 2: Méthode d'essai par 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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.

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 23655 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 (e.g. surface waters, ground waters, sea waters) can contain radionuclides of natural, human-made, or both origins:

- 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 (e.g. desorption from the soil and wash off by rain water) or can be released from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or phosphate fertilizers production and use);
- human-made radionuclides such as transuranium elements (americium, plutonium, neptunium, curium), ^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 can 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^[4] and ISO 5667-20^[5].

NOTE 1 The guideline level (GL) is the activity concentration with an intake of $2\text{ l}\cdot\text{d}^{-1}$ of drinking water for one year that results in an effective dose of $0,1\text{ mSv}\cdot\text{a}^{-1}$ 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].

NOTE 2 The Codex 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)^[6].

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^{[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 can be used during planned, existing and emergency exposure situations as well as for wastewaters and liquid effluents with specific modifications that can increase the overall uncertainty, detection limit and threshold.

The test method(s) can 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 support the need of test laboratories carrying out these measurements, that are sometimes required by national authorities, as they can be required 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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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. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of any other restrictions.

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

1 Scope

This document specifies the determination of nickel-59 and nickel-63 (^{59}Ni and ^{63}Ni) activity concentration in samples of all types of water using inductively coupled plasma mass spectrometry (ICP-MS).

Using currently available ICP-MS, this test method can measure ^{59}Ni activity concentrations of 300 $\text{mBq}\cdot\text{l}^{-1}$ and ^{63}Ni activity concentrations of 200 $\text{Bq}\cdot\text{l}^{-1}$. These values can be achieved with a sample volume of 1,0 l. Higher activity concentrations can be measured by either diluting the sample or using smaller sample aliquots or both.

NOTE These performance indicators are wholly dependent on the measurement regimes in individual laboratories; in particular, the detection limit is influenced by amount of stable nickel present.

The range of application depends on the amount of dissolved material in the water and on the performance characteristics of the measurement equipment (background count rate and counting efficiency).

It is the laboratory's responsibility to ensure the suitability 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 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 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 15586, *Water quality — Determination of trace elements using atomic absorption spectrometry with graphite furnace*

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 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 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/>

3.2 Symbols

The symbols used in this document are explained in [Table 1](#).

Table 1 — Symbols and units

Symbol	Description	Unit
b_{59}	Mass bias for ^{59}Ni (relative to ^{58}Ni)	—
b_{63}	Mass bias for ^{63}Ni (relative to ^{63}Ni)	—
c	Concentration	$\text{mol}\cdot\text{l}^{-1}$
$C_{A,59\text{Ni}}$	^{59}Ni activity concentration	$\text{Bq}\cdot\text{l}^{-1}$
$C_{A,63\text{Ni}}$	^{63}Ni activity concentration	$\text{Bq}\cdot\text{l}^{-1}$
$C_{S,59\text{Ni}}$	^{59}Ni calibration standard solution activity concentration	$\text{Bq}\cdot\text{g}^{-1}$
$C_{S,63\text{Ni}}$	^{63}Ni calibration standard solution activity concentration	$\text{Bq}\cdot\text{g}^{-1}$
$C_{c,\text{Ni}}$	Mass concentration of stable nickel carrier	$\text{g}\cdot\text{g}^{-1}$
$C_{m,\text{Ni}}$	Mass concentration of stable nickel in the measurement solution	$\text{g}\cdot\text{g}^{-1}$
$C_{y,\text{Ni}}$	Mass concentration of stable nickel in the yield solution	$\text{g}\cdot\text{g}^{-1}$
$C_{g,\text{Ni}}$	Mass concentration of natural nickel in the sample	$\text{g}\cdot\text{g}^{-1}$
$\varepsilon_{59\text{Ni}}$	Response of the ICP-MS to ^{59}Ni at $\frac{m}{z} = 59$	$\text{s}^{-1}\cdot\text{g}^{-1}$
$\varepsilon_{63\text{Ni}}$	Response of the ICP-MS to ^{63}Ni at $\frac{m}{z} = 63$	$\text{s}^{-1}\cdot\text{g}^{-1}$
I_{58}	Isotopic abundance of ^{58}Ni : 0,680 77 (natural isotopic abundance is assumed)	—
k	Associated coverage factor	—
L_A	Avogadro constant: $6,022\ 140\ 76 \times 10^{23}$ atoms per mole (defined as an exact quantity, with zero uncertainty)	mol^{-1}
L_D	Detection limit	—
L_Q	Limit of quantification	—
m	Mass of an ion	—
$m_{a,59\text{Ni}}$	Atomic mass of ^{59}Ni 58,934 346 7	$\text{g}\cdot\text{mol}^{-1}$
$m_{a,63\text{Ni}}$	Atomic mass of ^{63}Ni 62,929 669 4	$\text{g}\cdot\text{mol}^{-1}$
$m_{c,\text{Ni}}$	Mass of stable nickel carrier used	g