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

**Part 1:
Test method using liquid scintillation
counting**

*Qualité de l'eau — Nickel 59 et Nickel 63 —
Partie 1: Méthode d'essai par comptage des scintillations en milieu
liquide*

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

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 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 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 washoff 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 can 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].

Depending on the exposure situation, there are different limits and guideline levels (GLs) that would result in an action to reduce health risk. As an example, during a planned or existing situation, the WHO GL for drinking water is $1\,000\text{ Bq}\cdot\text{l}^{-1}$ for Ni activity concentration.

NOTE 1 The 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].

In the event of a nuclear emergency, the WHO Codex GLs^[6] mentioned that the activity concentration might not be greater than $10\,000\text{ Bq}\cdot\text{l}^{-1}$ for Ni.

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^{[7],[8]}.

Usually, the test methods can be adjusted to measure the activity concentration of the radionuclide(s) in either wastewaters before storage or in liquid effluents before being discharged to the environment.

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The test results will enable the plant/installation operator to verify that, before their discharge, wastewaters/liquid effluent radioactive activity concentrations do not exceed authorized limits.

The test method(s) described in this document 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 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 need to obtain a specific accreditation for radionuclide measurement in drinking water samples.

This document is one of a set of International Standards on test methods dealing with the measurement of the activity concentration of radionuclides in water samples.

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Water quality — Nickel-59 and nickel-63 —

Part 1:

Test method using liquid scintillation counting

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.

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 liquid scintillation counting (LSC). Using currently available liquid scintillation counters, this test method can measure ^{59}Ni activity concentrations of $50 \text{ mBq}\cdot\text{l}^{-1}$ and ^{63}Ni activity concentrations of $20 \text{ mBq}\cdot\text{l}^{-1}$ with a counting time of 200 min and a sample volume of 1,5 l.

NOTE These performance indicators are wholly dependent on the measurement regimes in individual laboratories; in particular, the detection limits for ^{59}Ni are entirely dependent on the levels of ^{63}Ni that can be 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 detection 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/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

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

3 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/>

4 General symbols and nickel-59 and nickel-63 specific symbols

Table 1 provides the general symbols used in this document and Table 2 provides the ⁵⁹Ni and ⁶³Ni specific symbols used in this document.

Table 1 — General symbols and units

Symbol	Description	Unit
c_A	Activity concentration of the measurand	Bq · l ⁻¹
c_A^*	Decision threshold, expressed as an activity concentration	Bq · l ⁻¹
$c_A^\#$	Detection limit, expressed as an activity concentration	Bq · l ⁻¹
$c_A^<$	Lower limit of the probabilistically symmetric coverage interval	Bq · l ⁻¹
$c_A^>$	Upper limit of the probabilistically symmetric coverage interval	Bq · l ⁻¹
$c_A^{<}$	Lower limit of the shortest coverage interval	Bq · l ⁻¹
$c_A^{>}$	Upper limit of the shortest coverage interval	Bq · l ⁻¹
E	Beta particle energy	keV
$f(Q_p)$	Empirical function linking a quench parameter, Q_p , to a detection efficiency, ϵ	—
$k_{1-\alpha}$	Quantiles of the standardised normal distribution for the probabilities $1 - \alpha$	—
$k_{1-\beta}$	Quantiles of the standardised normal distribution for the probabilities $1 - \beta$	—
$r_{g,A}$	Count rate of sample for energy region (2–20) keV	s ⁻¹
$r_{g,B}$	Count rate of sample for energy region (20–100) keV	s ⁻¹
$r_{b,A}$	Count rate of reagent blank for energy region (2–20) keV	s ⁻¹
$r_{b,B}$	Count rate of reagent blank for energy region (20–100) keV	s ⁻¹
$r_{0,A}$	Detector background for energy region (2–20) keV	s ⁻¹
$r_{0,B}$	Detector background for energy region (20–100) keV	s ⁻¹
$r_{net,A}$	Net count rate for energy region (2–20) keV	s ⁻¹
$r_{net,B}$	Net count rate for energy region (20–100) keV	s ⁻¹
R_{Ni}	Nickel recovery	—
S_E	Energy distribution for beta particles emitted by a particular radionuclide	—
t_s	Standard count time	s
t_g	Sample count time	s
t_b	Blank count time	s
t_0	Background count time	s
Q_p	Liquid scintillation quench parameter	—
$u_{R_{Ni}}$	nickel recovery uncertainty	—
u_x	Uncertainty of the parameter “x”	unit of x
u_y	Standard uncertainty of the estimate of the measurand	Bq · l ⁻¹
v_m	Liquid scintillation counting free parameter	—
V_g	Sample volume	l

Table 1 (continued)

Symbol	Description	Unit
x	Generic input quantity	—
y	Estimate of the measurand, primary measurement result of the measurand	$\text{Bq} \cdot \text{l}^{-1}$
α	Probability of reporting a false-positive result	—
β	Probability of reporting a false-negative result	—
ε	General term for detection efficiency	$\text{s}^{-1} \cdot \text{Bq}^{-1}$
γ	Proportion of data outside the coverage interval of the measurand	—
Φ	Distribution function of the standardised normal distribution	—
ω	Distribution function of y , where the standard measurement uncertainty is $u(y)$	—

Table 2 — Symbols and units specific to ^{59}Ni and ^{63}Ni analysis

Symbol	Description	Unit
$A_{59\text{Ni}}$	Activity concentration of the ^{59}Ni calibration standard solution	$\text{Bq} \cdot \text{g}^{-1}$
$A_{63\text{Ni}}$	Activity concentration of the ^{63}Ni calibration standard solution	$\text{Bq} \cdot \text{g}^{-1}$
$c_{A,59\text{Ni}}$	Activity concentration of ^{59}Ni in sample	$\text{Bq} \cdot \text{l}^{-1}$
$\tilde{c}_{A,59\text{Ni}}$	True value of the activity concentration of ^{59}Ni in sample	—
$c_{A,63\text{Ni}}$	Activity concentration of ^{63}Ni in sample	$\text{Bq} \cdot \text{l}^{-1}$
$C_{c,\text{Ni}}$	Mass concentration of stable nickel carrier	$\text{g} \cdot \text{g}^{-1}$
$C_{g,\text{Ni}}$	Mass concentration of stable nickel of sample	$\text{g} \cdot \text{g}^{-1}$
$C_{y,\text{Ni}}$	Mass concentration of stable nickel in the yield solution	$\text{g} \cdot \text{g}^{-1}$
$D_{63\text{Ni}}$	Decay of ^{63}Ni between sampling and measurement; $D_{63\text{Ni}} = e^{-\left(\frac{\ln 0,5 \cdot t_d}{T_{63\text{Ni}}}\right)}$	—
$m_{c,\text{Ni}}$	Mass of stable nickel carrier	g
$m_{\text{Ni},1}$	Mass of solution recovered for source preparation	g
$m_{\text{Ni},2}$	Mass of solution used to determine stable nickel content in source solution	g
$m_{\text{Ni},3}$	Mass of diluted solution for stable nickel content determination	g
$m_{\text{Ni},4}$	Mass of solution counted	g
$m_{s,59\text{Ni}}$	Mass of ^{59}Ni calibration standard solution used	g
$m_{s,63\text{Ni}}$	Mass of ^{63}Ni calibration standard solution used	g
R_{Ni}	Recovery of nickel	—
$r_{s,59\text{Ni},A}$	Gross count rate of the ^{59}Ni source in region A	s^{-1}
$r_{s,63\text{Ni},A}$	Gross count rate of the ^{63}Ni source in region A	s^{-1}
$r_{s,63\text{Ni},B}$	Gross count rate of the ^{63}Ni source in region B	s^{-1}
t_d	Time elapsed between sampling and measurement	s
t_{Ni}	Count time of source	s
$T_{63\text{Ni}}$	Radioactive half-life of ^{63}Ni	s
$\tilde{u}_{\tilde{c}_{A,59\text{Ni}}}$	Combined standard uncertainty of $\tilde{c}_{A,59\text{Ni}}$	—
$w_{59\text{Ni}}$	Multiplier linking net count rate of ^{59}Ni to the measured activity concentration of ^{59}Ni , where: $w_{59\text{Ni}} = \frac{m_{\text{Ni},1}}{\varepsilon_{59\text{Ni},A} \cdot R_{\text{Ni}} \cdot m_{\text{Ni},4} \cdot V_g}$	$\text{Bq} \cdot \text{l}^{-1} \cdot \text{s}$

Table 2 (continued)

Symbol	Description	Unit
$w_{63\text{Ni}}$	Multiplier linking net count rate of ^{63}Ni to the measured activity concentration of ^{63}Ni , where: $w_{63\text{Ni}} = \frac{m_{\text{Ni},1}}{\epsilon_{63\text{Ni},A} \cdot R_{\text{Ni}} \cdot m_{\text{Ni},4} \cdot V_g}$	$\text{Bq} \cdot \text{l}^{-1} \cdot \text{s}$
$\epsilon_{59\text{Ni},A}$	Detection efficiency of ^{59}Ni in region A	$\text{s}^{-1} \cdot \text{Bq}^{-1}$
$\epsilon_{63\text{Ni},A}$	Detection efficiency of ^{63}Ni in region A	$\text{s}^{-1} \cdot \text{Bq}^{-1}$
$\epsilon_{63\text{Ni},B}$	Detection efficiency of ^{63}Ni in region B	$\text{s}^{-1} \cdot \text{Bq}^{-1}$
χ	Ratio between the detection efficiency of ^{63}Ni in channel A and the detection efficiency if ^{63}Ni in channel B	—
$T(\chi)$	Uncertainty term in channel A, arising from counts in channel B arising from ^{63}Ni	s^{-2}

5 Principle

Nickel-59 is an anthropogenic radionuclide with a half-life of 76 000 years^[9] that decays to ^{59}Co by electron capture, with the associated emission of cobalt K- and L- X-rays, with energies of 6,9 keV and 7,7 keV. ^{59}Ni is generated by neutron capture of the stable isotope ^{58}Ni .

Nickel-63 is an anthropogenic radionuclide with a half-life of 98,7 years^[10] that decays to ^{63}Cu by beta particle emission with a maximum beta energy of 67,0 keV. ^{63}Ni is generated by neutron capture of the stable isotope ^{62}Ni .

Freshly irradiated natural nickel can have an $^{59}\text{Ni}/^{63}\text{Ni}$ activity ratio of 0,009 and this increases to 0,01 after 20 years, to 0,012 after 50 years and to 0,018 after 100 years, although these values are very dependent on the irradiation history and any subsequent processing of the material being measured.

Both ^{59}Ni and ^{63}Ni are separated from an aliquot of the sample matrix by precipitation, extraction chromatography or ion exchange chromatography and their activities are measured simultaneously by liquid scintillation counting against suitable calibration standards of ^{59}Ni and ^{63}Ni .

For samples with high activity concentration, dilution of the sample is required to avoid resin and detector saturation during the separation and counting steps, respectively.

Suspended material is removed prior to analysis by filtration using 0,45 μm filters. The analysis of the insoluble fraction requires a mineralization step that is not covered by this document.

NOTE General guidance on sample pre-treatment is given in ISO 18589-2^[11].

It is necessary to know the concentration of stable nickel in the sample in order to determine the mass of the iron and nickel carriers to add and to calculate the chemical yield for the separation of ^{59}Ni and ^{63}Ni .

When suspended matters are significant, a filtration step is required and ^{59}Ni and ^{63}Ni activities can also be determined in the filter deposit.

6 Reagents

All reagents shall be of recognized analytical grade and shall not contain any detectable alpha and beta activity, except for radioactive standard solutions.

6.1 Standard solutions

6.1.1 Nickel-59 and ^{63}Ni standards

Nickel-59 and ^{63}Ni standards should be traceable to national or international measurement standards, and can be obtained from a number of commercial suppliers and national metrology institutes.

6.1.2 Stable nickel standards

Stable nickel standards can be obtained from a number of commercial suppliers at a range of concentrations.

6.2 Chemical quenching agent

A suitable chemical should be used, such as nitromethane, CH_3NO_2 .

6.3 Colour quenching agent

A suitable chemical should be used, such as tartrazine, $\text{C}_{16}\text{H}_9\text{N}_4\text{Na}_3\text{O}_9\text{S}_2$.

6.4 Holdback carrier

Prepare a mixed solution of antimony, caesium, calcium, cerium, chromium, cobalt, iron, manganese, ruthenium, silver, strontium and zinc, containing approximately 10 mg g^{-1} of each element. It is not necessary to calibrate this solution.

6.5 Water

Water complying with grade 3 of ISO 3696. [23655-1:2022](https://standards.iteh.ai/catalog/standards/sist/23655-1-2022)

6.6 Specific reagents for chemical separation

6.6.1 Ammonium citrate solution, $c_{\text{C}_6\text{H}_{14}\text{N}_2\text{O}_7} = 1 \text{ mol}\cdot\text{l}^{-1}$

Dissolve 22,62 g of ammonium citrate ($\text{C}_6\text{H}_{14}\text{N}_2\text{O}_7$) in 80 ml of water and dilute to 100 ml with water (6.5).

6.6.2 Ammonium citrate solution, $c_{\text{C}_6\text{H}_{14}\text{N}_2\text{O}_7} = 0,2 \text{ mol}\cdot\text{l}^{-1}$

Dissolve 4,52 g of ammonium citrate ($\text{C}_6\text{H}_{14}\text{N}_2\text{O}_7$) in 80 ml of water and dilute to 100 ml with water (6.5).

6.6.3 Ammonium hydroxide solution, $c_{\text{NH}_4\text{OH}} = 6 \text{ mol}\cdot\text{l}^{-1}$

Dilute 500 ml of concentrated ammonia (NH_4OH) solution to 1 l with water (6.5).

6.6.4 Dimethylglyoxime solution, 1 %, $c_{\text{C}_4\text{H}_8\text{N}_2\text{O}_2} = 0,086 \text{ mol}\cdot\text{l}^{-1}$

Dissolve 1 g dimethylglyoxime ($\text{C}_4\text{H}_8\text{N}_2\text{O}_2$) in 100 ml ethanol.

6.6.5 Hydrochloric acid solution, $c_{\text{HCl}} = 6 \text{ mol}\cdot\text{l}^{-1}$

Dilute 500 ml of concentrated hydrochloric acid (HCl) to 1 l with water (6.5).

6.6.6 Hydrochloric acid solution, $c_{\text{HCl}} = 1 \text{ mol}\cdot\text{l}^{-1}$

Dilute 83 ml of concentrated hydrochloric acid (HCl) to 1 l with water (6.5).