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Water quality — Iron-55 — Test method using liquid scintillation counting

*Qualité de l'eau — Fer-55 — Méthode d'essai par comptage des
scintillations en milieu liquide*

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Contents

Page

Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms and definitions, symbols, and units	2
3.1 General use.....	2
3.2 ⁵⁵ Fe and ⁵⁹ Fe specific terms.....	3
4 Principle	5
5 Reagents	6
5.1 Standard solutions.....	6
5.1.1 Iron-55 and ⁵⁹ Fe standards.....	6
5.1.2 Stable iron standards.....	6
5.1.3 Chemical Quenching agent.....	6
5.1.4 Colour Quenching agent.....	6
5.2 Holdback carrier.....	6
5.3 Water.....	6
5.4 Specific reagents for chemical separation.....	6
5.4.1 Stable iron carrier solution.....	6
5.4.2 Ammonium hydroxide solution, c(NH ₄ OH) = 4 mol l ⁻¹	6
5.4.3 Nitric acid solution, c(HNO ₃) = 7,2 mol l ⁻¹	6
5.4.4 Ammonium hydroxide solution, c(NH ₄ OH) = 6 mol l ⁻¹	6
5.4.5 Hydrochloric acid, c(HCl) = 9 mol l ⁻¹	6
5.4.6 Hydrochloric acid, c(HCl) = 6 mol l ⁻¹	7
5.4.7 Hydrochloric acid, c(HCl) = 4 mol l ⁻¹	7
5.4.8 Hydrochloric acid, c(HCl) = 0,01 mol l ⁻¹	7
5.4.9 Nitric acid solution, c(HNO ₃) = 8 mol l ⁻¹	7
5.4.10 Nitric acid solution, c(HNO ₃) = 2 mol l ⁻¹	7
5.4.11 Sodium hydrogen phosphate, c(Na ₂ HPO ₄) = 0,5 mol l ⁻¹	7
5.4.12 Hydrochloric acid, c(HCl) = 1 mol l ⁻¹	7
6 Equipment	7
6.1 Laboratory equipment for direct evaporation.....	7
6.2 Liquid scintillation vials.....	7
6.3 Measurement equipment: Liquid scintillation counter.....	7
7 Sampling and sample preparation	7
8 Liquid scintillation set up and calibration	8
8.1 Window setting.....	8
8.2 Background.....	8
8.3 Calibration.....	8
9 Procedure	10
9.1 Preliminary.....	10
9.1.1 Stable iron content [11].....	10
9.1.2 Iron separation.....	10
9.2 Iron-55 source preparation.....	11
10 Quality control	11
11 Expression of results	11
11.1 Iron recovery.....	11
11.2 Detection efficiencies.....	12
11.2.1 ⁵⁵ Fe detection efficiency.....	12
11.2.2 ⁵⁹ Fe detection efficiency.....	13
11.3 Iron-55 activity.....	14

11.4	⁵⁵ Fe decision threshold and detection limit.....	15
11.5	Limits of the coverage intervals	15
11.5.1	Limits of the probabilistically symmetric coverage interval.....	15
11.5.2	The limits of the shortest coverage interval.....	15
12	Test report	16
Annex A	(informative) Isolation and purification of iron	18
Annex B	(informative) Analysis flow chart	19
Bibliography	20

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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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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 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 a planned or existing situation, the WHO guidelines for guidance level in drinking water is $1\ 000\ \text{Bq}\cdot\text{l}^{-1}$ for ^{55}Fe activity concentration.

NOTE 1 The guidance level 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 Guideline Levels^[5] mentioned that the activity concentration might not be greater than $10\ 000\ \text{Bq}\cdot\text{l}^{-1}$ for ^{55}Fe .

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 $1\ \text{mSv}$ 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^[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 parts 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 series 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 — Iron-55 — 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 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 a test method for the determination of iron-55 (^{55}Fe) 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 the ^{55}Fe activity concentrations in the range from the limit of detection up to 120 mBq l⁻¹. These values can be achieved with a counting time between 7 200 s and 10 800 s for a sample volume from 0,5 l to 1,5 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 limits are influenced by amount of stable iron 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 11929-1, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 1: Elementary applications*

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 and definitions, symbols, and units

For the purposes of this document, the terms, definitions, symbols and abbreviated terms given in ISO 80000-10 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/>

3.1 General use

Table 1 — General symbols and units

Symbol	Units	Comments
$r_{b(A)}$	s ⁻¹	Count rate of reagent blank for energy region (2 to 20) keV
$r_{b(B)}$	s ⁻¹	Count rate of reagent blank for energy region (20 to 3 000) keV
$r_{0(A)}$	s ⁻¹	Detector background for energy region (2 to 20) keV
$r_{0(B)}$	s ⁻¹	Detector background for energy region (20 to 3 000) keV
t_b	s	Background count time
V_s	l	Sample volume
$u(V_s)$	l	Uncertainty on sample volume
ε	s ⁻¹ Bq ⁻¹	General term for detection efficiency
Q_p	None	Liquid scintillation quench parameter
$f(Q_p)$	None	Empirical function linking a quench parameter, Q_p , to a detection efficiency, ε .
E	keV	Beta particle energy
$S(E)$	None	Energy distribution for beta particles emitted by a particular radionuclide
$\frac{1}{m}$	None	Liquid scintillation counting free parameter

Table 2 — Symbols from ISO 11929-1

Terms from ISO 11929-1	
y	Determined value of the estimate of the measurand
$u(y)$	Standard uncertainty of the estimate of the measurand
w	Multiplier, comprised of detection efficiency, decay, sample quantity that converts $r_{\text{net},A(\text{Fe})}$ to $c_{\text{Fe-55}}$, such that $w = \frac{c_{\text{Fe-55}}}{r_{\text{net},A(\text{Fe})}}$
$u(w)$	Uncertainty on multiplier w , where: $u(w) = w \cdot \sqrt{\left(\frac{u(r_{\text{net},A(\text{Fe})})}{r_{\text{net},A(\text{Fe})}\right)^2 + \left(\frac{u(c_{\text{Fe-55}})}{c_{\text{Fe-55}}}\right)^2}$
α	Probability of the error of the first kind
$k_{1-\alpha}$	Quantiles of the standardised normal distribution for the probabilities $1-\alpha$
β	Probability of the error of the second kind
$k_{1-\beta}$	Quantiles of the standardised normal distribution for the probabilities $1-\beta$
k	Coverage factor ($k = 2$ is associated with a level of confidence of approximately 95,5 % for a Gaussian distribution)
y^*	Decision threshold
$y^\#$	Detection limit
$y^<$	Lower confidence limit of y
$y^>$	Upper confidence limit of y
Φ	Distribution function of the standardised normal distribution
ω	Distribution function of y , where the standard measurement uncertainty is $u(y)$
c_A	Concentration of the measurand
$u(c_A)$	Uncertainty on the concentration of the measurand
$c_A^<$	Lower limit of the probabilistically symmetric coverage interval, in Bq l^{-1}
$c_A^>$	Upper limit of the probabilistically symmetric coverage interval, in Bq l^{-1}
$c_A^<$	Lower limit of the shortest coverage interval, in Bq l^{-1}
$c_A^>$	Upper limit of the shortest coverage interval, in Bq l^{-1}

3.2 ^{55}Fe and ^{59}Fe specific terms

Table 3 — Symbols and units specific to ^{55}Fe analysis

Symbol	Units	Comments
$C_{\text{Fe}(n)}$	g g^{-1}	Natural iron mass concentration of sample
$u(C_{\text{Fe}(n)})$	g g^{-1}	Uncertainty on natural iron mass concentration of sample
$A_{\text{Fe-55}}$	Bq g^{-1}	Activity concentration of the ^{55}Fe calibration standard solution
$u(C_{\text{Fe-55}})$	Bq g^{-1}	Uncertainty on activity concentration of the ^{55}Fe calibration standard solution
$m_{\text{s}(\text{Fe-55})}$	g	Mass of ^{55}Fe calibration standard solution used
$u(m_{\text{s}(\text{Fe-55})})$	g	Uncertainty on mass of ^{55}Fe calibration standard solution used

Table 3 (continued)

Symbol	Units	Comments
$r_{s(\text{Fe-55,A})}$	s ⁻¹	Gross count rate of the ⁵⁵ Fe standard source in region A
$u(r_{s(\text{Fe-55,A})})$	s ⁻¹	Uncertainty on gross count rate of the ⁵⁵ Fe standard source in region A
$t_{s(\text{Fe-55,A})}$	s	Count time of ⁵⁵ Fe standard source
$\varepsilon_{\text{Fe-55(A)}}$	s ⁻¹ Bq ⁻¹	Detection efficiency of ⁵⁵ Fe in energy region (2 to 20) keV
$u(\varepsilon_{\text{Fe-55(A)}})$	s ⁻¹ Bq ⁻¹	Uncertainty on detection efficiency of ⁵⁵ Fe in energy region (2 to 20) keV
$A_{\text{Fe-59}}$	Bq g ⁻¹	Activity concentration of the ⁵⁹ Fe calibration standard solution
$u(A_{\text{Fe-59}})$	Bq g ⁻¹	Uncertainty on activity concentration of the ⁵⁹ Fe calibration standard solution
$m_{s(\text{Fe-59})}$	g	Mass of ⁵⁹ Fe calibration standard solution used
$u(m_{s(\text{Fe-59})})$	g	Uncertainty on mass of ⁵⁹ Fe calibration standard solution used
$r_{s(\text{Fe-59,A})}$	s ⁻¹	Gross count rate of the ⁵⁹ Fe standard source in region A
$u(r_{s(\text{Fe-59,A})})$	s ⁻¹	Uncertainty on gross count rate of the ⁵⁹ Fe standard source in region A
$t_{s(\text{Fe-59,A})}$	s	Count time of ⁵⁹ Fe standard source
$\varepsilon_{\text{Fe-59(A)}}$	s ⁻¹ Bq ⁻¹	Detection efficiency of ⁵⁹ Fe in energy region (2 to 20) keV
$u(\varepsilon_{\text{Fe-59(A)}})$	s ⁻¹ Bq ⁻¹	Uncertainty on detection efficiency of ⁵⁹ Fe in energy region (2 to 20) keV
$\varepsilon_{\text{Fe-59(B)}}$	s ⁻¹ Bq ⁻¹	Detection efficiency of ⁵⁹ Fe in energy region (20 to 2 000) keV
$u(\varepsilon_{\text{Fe-59(B)}})$	s ⁻¹ Bq ⁻¹	Uncertainty on detection efficiency of ⁵⁹ Fe in energy region (20 to 2 000) keV
$r_{s(\text{Fe-59,B})}$	s ⁻¹	Gross count rate of the ⁵⁹ Fe standard source in region B
$u(r_{s(\text{Fe-59,B})})$	s ⁻¹	Uncertainty on gross count rate of the ⁵⁹ Fe standard source in region B
$C_{c(\text{Fe})}$	g g ⁻¹	Mass concentration of stable iron carrier
$u(C_{c(\text{Fe})})$	g g ⁻¹	Uncertainty on mass concentration of stable iron carrier
$m_{c(\text{Fe})}$	g	Mass of stable iron carrier used
$u(m_{c(\text{Fe})})$	g	Uncertainty on mass of stable iron carrier used
$C_{s(\text{Fe})}$	g g ⁻¹	Mass concentration of stable iron in the yield solution
$u(C_{s(\text{Fe})})$	g g ⁻¹	Uncertainty on mass concentration of stable iron in the yield solution
$R_{(\text{Fe})}$	None	Recovery of iron
$u(R_{(\text{Fe})})$	None	Uncertainty on recovery of iron
$r_{A(\text{Fe})}$	s ⁻¹	Gross count rate of ⁵⁵ Fe source in region A
$u(r_{A(\text{Fe})})$	s ⁻¹	Uncertainty on gross count rate of ⁵⁵ Fe source in region A
$r_{\text{net,A}(\text{Fe})}$	s ⁻¹	Net count rate of ⁵⁵ Fe source in region A
$u(r_{\text{net,A}(\text{Fe})})$	s ⁻¹	Uncertainty on net count rate of ⁵⁵ Fe source in region A
$r_{B(\text{Fe})}$	s ⁻¹	Gross count rate of ⁵⁵ Fe source in region B
$u(r_{B(\text{Fe})})$	s ⁻¹	Uncertainty on gross count rate of ⁵⁵ Fe source in region B