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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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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/SC3, *Water quality*, SC 3, *Radioactivity measurements* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 230, *Water analysis*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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, ^{3}H, ^{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), ^{3}H, ^{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 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 Bq·l^{-1} for ^{55}Fe activity concentration.

NOTE 1 The guidance level is the activity concentration with an intake of 2 l·d^{-1} of drinking water for one year that results in an effective dose of 0,1 mSv·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 Bq·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 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.

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
$r_{\mathrm{b(A)}}$	s^{-1}	Count rate of reagent blank for energy region (2 to 20) keV
$r_{\mathrm{b(B)}}$	s^{-1}	Count rate of reagent blank for energy region (20 to 3 000) keV
$r_{0\mathrm{(A)}}$	s^{-1}	Detector background for energy region (2 to 20) keV
$r_{0\mathrm{(B)}}$	s^{-1}	Detector background for energy region (20 to 3 000) keV
t_{b}	s	Background count time
V_{s}	l	Sample volume
$u(V_{\mathrm{s}})$	l	Uncertainty on sample volume
ε	$\mathrm{s}^{-1}\ \mathrm{Bq}^{-1}$	General term for detection efficiency
Q_{p}	None	Liquid scintillation quench parameter
$f(Q_{\mathrm{p}})$	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
v_{m}	None	Liquid scintillation counting free parameter

Table 2 — Symbols from ISO 11929-1

Terms from ISO 11929-1	
c_A	Activity concentration of the measurand
$u(c_A)$	Standard uncertainty of the activity concentration
w	Multiplier, comprised of detection efficiency, decay, sample quantity that concerts $r_{\mathrm{net,A(Fe)}}$ to $c_{\mathrm{Fe-55}}$, such that $w=\frac{c_{\mathrm{Fe-55}}}{r_{\mathrm{net,A(Fe)}}}$
$u(w)$	Uncertainty on multiplier *w*, where: $u(w)=w\cdot\sqrt{\left(\frac{u(r_{\mathrm{net,A(Fe)}})}{r_{\mathrm{net,A(Fe)}}}\right)^2+\left(\frac{u(c_{\mathrm{Fe-55}})}{c_{\mathrm{Fe-55}}}\right)^2}$
α	Probability of the error of the false positive decision

Table 2 *(continued)*

Terms from ISO 11929-1	
$k_{1-\alpha}$	Quantiles of the standardised normal distribution for the probabilities $1-\alpha$
β	Probability of the error of the false negative decision
$k_{1-\beta}$	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)
c_A^*	Decision threshold, in Bq l^{-1}
$c_A^\#$	Detection limit, in Bq l^{-1}
Φ	Distribution function of the standardised normal distribution
ω	Distribution function of c_A, where the standard measurement uncertainty is $u(c_A)$
$c_A^{\triangleleft}$	Lower limit of the probabilistically symmetric coverage interval, in Bq l^{-1}
$c_A^{\triangleright}$	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
$C_{\mathrm{Fe(n)}}$	g g^{-1}	Natural iron mass concentration of sample
$u\left(C_{\mathrm{Fe(n)}}\right)$	g g^{-1}	Uncertainty on natural iron mass concentration of sample
$A_{\mathrm{Fe\text{-}55}}$	Bq g^{-1}	Activity concentration of the ^{55}Fe calibration standard solution
$u\left(C_{\mathrm{Fe\text{-}55}}\right)$	Bq g^{-1}	Uncertainty on activity concentration of the ^{55}Fe calibration standard solution
$m_{\mathrm{s(Fe–55)}}$	g	Mass of ^{55}Fe calibration standard solution used
$u\left(m_{\mathrm{s(Fe–55)}}\right)$	g	Uncertainty on mass of ^{55}Fe calibration standard solution used
$r_{\mathrm{s(Fe–55,A)}}$	s^{-1}	Gross count rate of the ^{55}Fe standard source in region A
$u\left(r_{\mathrm{s(Fe–55,A)}}\right)$	s^{-1}	Uncertainty on gross count rate of the ^{55}Fe standard source in region A
$t_{\mathrm{s(Fe–55,A)}}$	s	Count time of ^{55}Fe standard source
$\varepsilon_{\mathrm{Fe–55(A)}}$	s^{-1} Bq^{-1}	Detection efficiency of ^{55}Fe in energy region (2 to 20) keV
$u\left(\varepsilon_{\mathrm{Fe–55(A)}}\right)$	s^{-1} Bq^{-1}	Uncertainty on detection efficiency of ^{55}Fe in energy region (2 to 20) keV
$A_{\mathrm{Fe–59}}$	Bq g^{-1}	Activity concentration of the ^{59}Fe calibration standard solution
$u\left(A_{\mathrm{Fe–59}}\right)$	Bq g^{-1}	Uncertainty on activity concentration of the ^{59}Fe calibration standard solution
$m_{\mathrm{s(Fe–59)}}$	g	Mass of ^{59}Fe calibration standard solution used
$u\left(m_{\mathrm{s(Fe–59)}}\right)$	g	Uncertainty on mass of ^{59}Fe calibration standard solution used
$r_{\mathrm{s(Fe–59,A)}}$	s^{-1}	Gross count rate of the ^{59}Fe standard source in region A
$u\left(r_{\mathrm{s(Fe–59,A)}}\right)$	s^{-1}	Uncertainty on gross count rate of the ^{59}Fe standard source in region A
$t_{\mathrm{s(Fe–59,A)}}$	s	Count time of ^{59}Fe standard source

Table 3 *(continued)*

Symbol	Units	Comments
$\varepsilon_{\text{Fe–59(A)}}$	$\text{s}^{-1}\ \text{Bq}^{-1}$	Detection efficiency of ^{59}Fe in energy region (2 to 20) keV
$u\left(\varepsilon_{\text{Fe–59(A)}}\right)$	$\text{s}^{-1}\ \text{Bq}^{-1}$	Uncertainty on detection efficiency of ^{59}Fe in energy region (2 to 20) keV
$\varepsilon_{\text{Fe–59(B)}}$	$\text{s}^{-1}\ \text{Bq}^{-1}$	Detection efficiency of ^{59}Fe in energy region (20 to 2 000) keV
$u\left(\varepsilon_{\text{Fe–59(B)}}\right)$	$\text{s}^{-1}\ \text{Bq}^{-1}$	Uncertainty on detection efficiency of ^{59}Fe in energy region (20 to 2 000) keV
$r_{\text{s(Fe–59,B)}}$	s^{-1}	Gross count rate of the ^{59}Fe standard source in region B
$u\left(r_{\text{s(Fe–59,B)}}\right)$	s^{-1}	Uncertainty on gross count rate of the ^{59}Fe standard source in region B
$C_{\text{c(Fe)}}$	g g^{-1}	Mass concentration of stable iron carrier
$u\left(C_{\text{c(Fe)}}\right)$	g g^{-1}	Uncertainty on mass concentration of stable iron carrier
$m_{\text{c(Fe)}}$	g	Mass of stable iron carrier used
$u\left(m_{\text{c(Fe)}}\right)$	g	Uncertainty on mass of stable iron carrier used
$C_{\text{s(Fe)}}$	g g^{-1}	Mass concentration of stable iron in the yield solution
$u\left(C_{\text{s(Fe)}}\right)$	g g^{-1}	Uncertainty on mass concentration of stable iron in the yield solution
$R_{\text{(Fe)}}$	None	Recovery of iron
$u\left(R_{\text{(Fe)}}\right)$	None	Uncertainty on recovery of iron
$r_{\text{A(Fe)}}$	s^{-1}	Gross count rate of ^{55}Fe source in region A
$u\left(r_{\text{A(Fe)}}\right)$	s^{-1}	Uncertainty on gross count rate of ^{55}Fe source in region A
$r_{\text{net,A(Fe)}}$	s^{-1}	Net count rate of ^{55}Fe source in region A
$u\left(r_{\text{net,A(Fe)}}\right)$	s^{-1}	Uncertainty on net count rate of ^{55}Fe source in region A
$r_{\text{B(Fe)}}$	s^{-1}	Gross count rate of ^{55}Fe source in region B
$u\left(r_{\text{B(Fe)}}\right)$	s^{-1}	Uncertainty on gross count rate of ^{55}Fe source in region B
$r_{\text{net,B(Fe)}}$	s^{-1}	Net count rate of ^{55}Fe source in region B
$u\left(r_{\text{net,B(Fe)}}\right)$	s^{-1}	Uncertainty on net count rate of ^{55}Fe source in region B
$t_{\text{(Fe)}}$	s	Count time of ^{55}Fe source
$c_{\text{Fe–55}}$	Bq l^{-1}	Activity concentration of ^{55}Fe in sample
$u\left(c_{\text{Fe–55}}\right)$	Bq·l^{-1}	Uncertainty on activity concentration of ^{55}Fe in sample
$m_{\text{Fe(1)}}$	g	Mass of solution recovered for source preparation
$u\left(m_{\text{Fe(1)}}\right)$	g	Uncertainty on mass of solution recovered for source preparation
$m_{\text{Fe(2)}}$	g	Mass of solution used to determine iron content in source solution
$u\left(m_{\text{Fe(2)}}\right)$	g	Uncertainty on mass of solution used to determine iron content in source solution
$m_{\text{Fe(3)}}$	g	Mass of diluted solution for iron content determination
$u\left(m_{\text{Fe(3)}}\right)$	g	Uncertainty on mass of diluted solution for iron content determination
$m_{\text{Fe(4)}}$	g	Mass of solution counted

Table 3 *(continued)*

Symbol	Units	Comments
$u\left(m_{\mathrm{Fe}(4)}\right)$	g	Uncertainty on mass of solution counted
t_d	s	Time elapsed between sampling and measurement
$T_{\mathrm{Fe-55}}$	s	Radioactive half-life of ^{55}Fe
$u\left(T_{\mathrm{Fe-55}}\right)$	s	Uncertainty on radioactive half-life of ^{55}Fe
$D_{\mathrm{Fe-55}}$	None	Decay of ^{55}Fe between sampling and measurement: $D_{\mathrm{Fe-55}} = e^{\left(\frac{\ln 0{,}5 \cdot t_{\mathrm{d}}}{T_{\mathrm{Fe-55}}}\right)}$
$u\left(D_{\mathrm{Fe-55}}\right)$	None	Uncertainty on decay of ^{55}Fe between sampling and measurement

4 Principle

Iron-55 is an anthropogenic radionuclide that decays to ^{55}Mn by electron capture, with the associated emission of manganese K- and L- X-rays, with energies of 5,9 keV and 6,5 keV and a half-life of 2,747 years[8]. ^{55}Fe is generated by neutron capture of ^{54}Fe.

Freshly irradiated natural iron can contain significant quantities of ^{59}Fe and the activity of this nuclide can be estimated by gamma spectrometry.

Iron-55 is separated from an aliquot of the sample matrix by extraction chromatography or ion exchange chromatography and the activity is measured separately by liquid scintillation counting against suitable calibration standards of ^{55}Fe.

To account for the minor impurity ^{59}Fe, twin channel counting, along with gamma spectrometry measurements are recommended.

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.

NOTE A suitable mineralization step is specified in ISO 18589-2[9].

It is necessary to know the concentration of the stable iron in the sample in order to determine the mass of the iron carrier to add and to calculate the chemical yield for the separation of ^{55}Fe.

An analysis flow chart is given in Annex B.

5 Reagents

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

5.1 Standard solutions

5.1.1 Iron-55 and ^{59}Fe standards

These should be traceable to national or international measurement standards, and can be obtained from a number of commercial suppliers and national measurement institutes.

5.1.2 Stable iron standards

These may be obtained from a number of commercial suppliers at a range of concentrations.

5.1.3 Chemical Quenching agent

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

5.1.4 Colour Quenching agent

A suitable chemical should be used, such as tartrazine, $C_{16}H_9N_4Na_3O_9S_2$.

5.2 Holdback carrier

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

5.3 Water

Water conforming with grade 3 of ISO 3696.

5.4 Specific reagents for chemical separation

5.4.1 Stable iron carrier solution

Procure a calibrated solution of 10 g·l $^{-1}$ from a suitable supplier.

5.4.2 Ammonium hydroxide solution, $c(NH_4OH)$ = 4 mol l^{-1}

Dilute 250 ml of concentrated ammonium hydroxide solution to 1 l with water.

WARNING — — Eye protection shall be worn.

5.4.3 Nitric acid solution, $c(HNO_3)$ = 7,2 mol l^{-1}

Dilute 450 ml of concentrated nitric acid to 1 l with water.

5.4.4 Ammonium hydroxide solution, $c(NH_4OH)$ = 6 mol l^{-1}

Dilute 500 ml of concentrated ammonium hydroxide solution to 1 l with water.

5.4.5 Hydrochloric acid, $c(HCl)$ = 9 mol l^{-1}

Dilute 750 ml of concentrated hydrochloric acid to 1 l with water.

5.4.6 Hydrochloric acid, $c(HCl)$ = 6 mol l^{-1}

Dilute 500 ml of concentrated hydrochloric acid to 1 l with water.

5.4.7 Hydrochloric acid, $c(HCl)$ = 4 mol l^{-1}

Dilute 330 ml of concentrated hydrochloric acid to 1 l with water.

5.4.8 Hydrochloric acid, $c(HCl)$ = 0,01 mol l^{-1}

Dilute 1 ml of concentrated hydrochloric acid to 1 l with water.

5.4.9 Nitric acid solution, $c(HNO_3)$ = 8 mol l^{-1}

Dilute 500 ml of concentrated nitric acid to 1 l with water.

5.4.10 Nitric acid solution, $c(\mathrm{HNO_3}) = 2\ \mathrm{mol\ l^{-1}}$

Dilute 125 ml of concentrated nitric acid to 1 l with water.

5.4.11 Sodium hydrogen phosphate, $c(\mathrm{Na_2HPO_4}) = 0{,}5\ \mathrm{mol\ l^{-1}}$

Dissolve 138 g sodium hydrogen phosphate heptahydrate ($\mathrm{Na_2HPO_4 \bullet 7H_2O}$) in 1 l water.

5.4.12 Hydrochloric acid, $c(\mathrm{HCl}) = 1\ \mathrm{mol\ l^{-1}}$

Dilute 83 ml of concentrated hydrochloric acid to 1 l with water.

6 Equipment

6.1 Laboratory equipment for direct evaporation

Usual laboratory apparatus to store and prepare the sample as specified in ISO 5667-3.

A hot plate, an automatic evaporator or any other appropriate apparatus.

6.2 Liquid scintillation vials

Both plastic and glass vials are required.

6.3 Measurement equipment: Liquid scintillation counter

Iron-55 may be counted with a commercial liquid scintillation counter. This may optionally include:

— “conventional” twin photomultiplier tube systems with CIEMAT-NIST[10] efficiency tracing routines (CIEMAT/NIST measurements require additional software for data analysis), and

— triple-to-double coincidence ratio systems (TDCR measurements require additional software for data analysis).

7 Sampling and sample preparation

Sampling, handling and storage of the water samples shall be done as specified in ISO 5667-1 and ISO 5667-3.

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.

The water sample should be acidified with concentrated nitric acid. Hydrochloric acid shall be avoided.

8 Liquid scintillation set up and calibration

8.1 Region setting

Iron-55 measurements require measurements of low energy photon emitters ($^{55}\mathrm{Fe}$) and higher energy beta emitters ($^{59}\mathrm{Fe}$) and so two measurement regions need to be set.

This can be effectively carried out using a tritium source and a $^{63}\mathrm{Ni}$ source, neither of which need to be calibrated. The counting regions are shown in Table 4.

Table 4 — Counting conditions

Region	Energy range	Comments
A	2 keV to 20 keV	This region captures most of the ^{55}Fe data, with electronic noise being largely excluded by setting the lower energy limit to 2 keV
B	20 keV to 2 000 keV	This region captures most of the ^{59}Fe and other high energy contaminants data

8.2 Background

Measure the background activity using an identical clean liquid scintillation vial containing iron carrier as appropriate. Repeated counts confirm the stability of the background level.

For a method blank, measure the blank activity using clean water and reagents under conditions representative of the measurement method. Repeated counts confirm the stability of the blank level.

8.3 Calibration

Measure the background for a period of at least equal to that of the counting time used for the test sample and standards.

The count rate of the reagent blank is denoted, $r_{\mathrm{b(A)}}$ for the region A (^{55}Fe) and $r_{\mathrm{b(B)}}$ for region B (^{59}Fe) which can be replaced by the appropriate background count rate value, $r_{\mathrm{0(A)}}$ and $r_{\mathrm{0(B)}}$ if these values are equivalent.

The counting efficiency of ^{55}Fe is determined in region A, $\varepsilon_{\mathrm{Fe-55(A)}}$ with a standard ^{55}Fe solution of known activity and purity; the detection efficiency in region B is zero for ^{55}Fe. The detection efficiency is determined by dividing the net count rate in region A by the activity of the source: $A_{\mathrm{Fe-55}} m_{\mathrm{s(Fe-55)}}$ in Formula (1):

$$\varepsilon_{\mathrm{Fe-55(A)}} = \frac{\left[r_{\mathrm{s(Fe-55,A)}} - r_{\mathrm{0(A)}} \right]}{A_{\mathrm{Fe-55}} \cdot m_{\mathrm{s(Fe-55)}}} \tag{1}$$

The counting efficiency of ^{59}Fe is determined in region A, $\varepsilon_{\mathrm{Fe-59(A)}}$ and region B, $\varepsilon_{\mathrm{Fe-59(B)}}$ with a standard ^{59}Fe solution of known activity concentration and purity. The detection efficiency is determined by dividing the net count rate in regions A and B by the activity of the source in Formulae (2) and (3):

$$\varepsilon_{\mathrm{Fe-59(A)}} = \frac{\left[r_{\mathrm{s(Fe-59,A)}} - r_{\mathrm{0(A)}} \right]}{A_{\mathrm{Fe-59}} \cdot m_{\mathrm{s(Fe-59)}}} \tag{2}$$

and

$$\varepsilon_{\mathrm{Fe-59(B)}} = \frac{\left[r_{\mathrm{s(Fe-59,}B\mathrm{)}} - r_{\mathrm{0(B)}} \right]}{A_{\mathrm{Fe-59}} \cdot m_{\mathrm{s(Fe-59)}}} \tag{3}$$

In each case a chemical quench correction curve shall be prepared by adding a suitable chemical quenching agent (for example nitromethane) to a series of sources of known activity. Also, a colour quench correction curve shall also be prepared by adding a suitable chemical quenching agent (for example tartrazine) to a series of sources of known activity. Each source should be consistent with respect to:

- approximate activity;
- carrier mass;

- solution volume;
- scintillation fluid volume.

The shape of the quench correction curve cannot be predicted, but takes the general form in Formula (4):

$$\varepsilon = f\left(Q_\mathrm{p}\right) \tag{4}$$

It is advisable to use efficiency tracing routines, if appropriate [10]. Efficiency tracing is a relative or tracing activity measurement method suitable for activity determination of pure beta, beta-gamma, pure electron capture and electron capture-gamma decaying radionuclide using a commercial liquid scintillation counter.

The efficiency tracing requires one to know the experimental counting efficiency of one tracer radionuclide (tritium is usually employed for beta emitters) for different degrees of chemical quench and to compute the counting efficiency at the photocathode output for different values of the free parameter, v_m, associated with the efficiency model in Formula (5):

$$\varepsilon = \int_0^E S(E) \cdot \left(1 - e^{-v_\mathrm{m}}\right) dE \tag{5}$$

NOTE The derivation of the free parameter, v_m, is outside the scope of this document. See, for example, L'Annunziata – Handbook of Radioactivity for a detailed explanation.

To obtain a universal curve, which allows to determine the counting efficiency for any radionuclide, a model for the counting efficiency as a function of the free parameter shall be developed for each radionuclide.

Acceptance limits for efficiency should be defined. It is advisable to check the method linearity. The efficiency should be assessed using calibration samples whose activities should cover the whole working range.

Efficiencies should be verified with a periodicity established by the laboratory and whenever changes in materials (e.g. scintillation cocktail) or when maintenance operations are performed on the scintillation counter.

A verification or a recalibration is necessary when requisites of instrument quality control are not met.

9 Procedure

9.1 Preliminary

9.1.1 Stable iron content [11]

Measure the iron $\left(C_{\mathrm{Fe(n)}}\right)$ concentration in the sample according to various protocols already described in other International Standards. These protocols include the following:

- ICP-OES according to ISO 11885;
- ICP-MS according to ISO 17294-2;
- AAS according to ISO 15586.

9.1.2 Iron separation

Take a suitable aliquot of sample $\left(V_\mathrm{S}\right)$ and add a known mass calibrated iron $\left(C_{\mathrm{c(Fe)}} \text{ and } m_{\mathrm{c(Fe)}}\right)$ carrier (5.4.1). Uncalibrated holdback carriers of antimony, caesium, calcium, cerium, chromium, cobalt, manganese, nickel, ruthenium, silver, strontium and zinc may be added depending on sample composition.

Add 4 mol l^{-1} ammonium hydroxide solution (5.4.2) to the sample, until alkaline, with pH>10. Warm the solution to coagulate the precipitate.

NOTE 1 The iron hydroxide ($Fe(OH)_3$) precipitate is visible as a brown precipitate.

Centrifuge the solution at a suitable rate to give effective separation of supernate and precipitate, and reject the supernate. Wash the precipitate with water (5.3) and recentrifuge; repeat the wash cycle twice.

It is essential to wash any residual NaOH solution from the precipitate.

Redissolve the solid in 7,2 mol l^{-1} nitric acid (5.4.3), warming if necessary.

Pass the solution through a suitable anion exchange resin[1] or extraction chromatography resin[2] to remove any actinide elements. Wash the column with 3 bed volumes of 7,2 mol l^{-1} nitric acid (5.4.3).

NOTE 2 This step removes any uranium and thorium from the sample.

NOTE 3 Other methods of separation of ^{55}Fe are available, such as (i) separation using anion exchange resin and then purification by liquid-liquid extraction with N-nitroso N-phenythydroxylamine (cupferron) in chloroform, and (ii) by liquid-liquid extraction with methyl-isobutyl ketone – (see Reference [12]). Solvent extraction methods rely on the use of volatile and semi volatile organic compounds.

Precipitate iron by adding 6 mol l^{-1} ammonium hydroxide solution (5.4.4) to the solution, and warm to coagulate the precipitate.

Centrifuge the solution at 2 000 s^{-1}, retain the precipitate for ^{55}Fe analysis.

Purify the iron fraction according to Annex A.

9.2 Iron-55 source preparation

Take the solution from step A1.6 or A.2.5 as appropriate.

Add 10 ml of 0,5 mol l^{-1} Na_2HPO_4 (5.4.11) to the solution and add 6 mol l^{-1} NH_4OH (5.4.4) until the pH is 3,0, then gently warm the solution on a hot plate to complete the precipitation of iron as $Fe(PO_4)$.

Centrifuge the solution at 2 000 s^{-1} and reject the supernate. Wash the precipitate with warm water and recentrifuge. Repeat the wash cycle twice.

Dissolve the precipitate in the minimum amount of 6 mol l^{-1} hydrochloric acid (5.4.6). Transfer the solution with water to a clean, pre-weighed glass or plastic scintillation vial, obtaining a solution mass of approximately 5 g and record the mass of solution, $m_{Fe(1)}$.

Remove approximately 0,5 g of the solution, $m_{Fe(2)}$ and dilute to approximately 50 g with 1 mol l^{-1} hydrochloric acid (5.4.12), recording the mass, $m_{Fe(3)}$ of this ‘Iron yield solution’.

Weigh approximately 2,5 g of the $m_{Fe(1)}$ solution into a clean plastic scintillation vial $m_{Fe(4)}$. Add a suitable amount of an appropriate scintillant and count, using the regions set in Clause 8.

NOTE The intense colour of $FeCl_6^{3-}$ affects counting efficiency.

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[1] Dowex AG1-X8 is an example of a suitable anion exchange resin, available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

[2] Eichrom or TrisKem TEVA, UTEVA and DGA resins are examples of suitable extraction chromatography media. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

10 Quality control

Periodically, check the measurement performances of the instruments using sources of constant activity, covering the energy range to be measured. This can usually be done with the background, ^{3}H and ^{14}C sealed check sources supplied with the counter. The results of such checks should be plotted on a Shewhart control chart and analysed using ISO 7870-2.

11 Expression of results

11.1 Iron recovery

Using the $m_{\mathrm{Fe}(3)}$ solution from 9.2 measure the iron concentration in the solution used to prepare the counting sources, as in 9.1.1, $C_{\mathrm{s(Fe)}}$.

The concentration of stable iron in the counting solution is given in Formula (6):

$$\frac{C_{\mathrm{s(Fe)}} . m_{\mathrm{Fe}(3)}}{m_{\mathrm{Fe}(2)}} \tag{6}$$

and so the total amount of iron recovered is given in Formula (7):

$$\frac{C_{\mathrm{s(Fe)}} \cdot m_{\mathrm{Fe}(3)}}{m_{\mathrm{Fe}(2)}} \cdot m_{\mathrm{Fe}(1)} \tag{7}$$

The total amount of iron in the sample is given in Formula (8):

$$C_{\mathrm{Fe(n)}} \cdot V_{\mathrm{s}} + C_{\mathrm{c(Fe)}} \cdot m_{\mathrm{c(Fe)}} \tag{8}$$

Thus, the recovery of iron, $R_{\mathrm{(Fe)}}$, is given in Formula (9):

$$R_{\mathrm{(Fe)}} = \frac{\left[C_{\mathrm{s(Fe)}} . m_{\mathrm{Fe}(3)} . m_{\mathrm{Fe}(1)} \right]}{\left[C_{\mathrm{Fe(n)}} \cdot V_{\mathrm{s}} + C_{\mathrm{c(Fe)}} \cdot m_{\mathrm{c(Fe)}} \right] \cdot m_{\mathrm{Fe}(2)}} \tag{9}$$

And thus the uncertainty on $R_{\mathrm{(Fe)}}$, $u\left(R_{\mathrm{(Fe)}}\right)$, is given in Formula (10):

$$u\left(R_{\mathrm{(Fe)}}\right) = R_{\mathrm{(Fe)}} \cdot \sqrt{\begin{array}{c} \left[\frac{u\left(C_{\mathrm{s(Fe)}}\right)}{C_{\mathrm{s(Fe)}}}\right]^2 + \left[\frac{u\left(m_{\mathrm{Fe}(1)}\right)}{m_{\mathrm{Fe}(1)}}\right]^2 + \left[\frac{u\left(m_{\mathrm{Fe}(2)}\right)}{m_{\mathrm{Fe}(2)}}\right]^2 + \left[\frac{u\left(m_{\mathrm{Fe}(3)}\right)}{m_{\mathrm{Fe}(3)}}\right]^2 + \\ \frac{\left\{\left[\frac{u\left(C_{\mathrm{Fe(n)}}\right)}{C_{\mathrm{Fe(n)}}}\right]^2 + \left[\frac{u\left(V_{\mathrm{s}}\right)}{V_{\mathrm{s}}}\right]^2\right\} \cdot \left[C_{\mathrm{Fe(n)}} \cdot V_{\mathrm{s}}\right]^2 + \left\{\left[\frac{u\left(C_{\mathrm{c(Fe)}}\right)}{C_{\mathrm{c(Fe)}}}\right]^2 + \left[\frac{u\left(m_{\mathrm{c(Fe)}}\right)}{m_{\mathrm{c(Fe)}}}\right]^2\right\} \cdot \left[C_{\mathrm{c}(Fe)} \cdot m_{\mathrm{c(Fe)}}\right]^2}{\left[C_{\mathrm{Fe(n)}} \cdot V_{\mathrm{s}} + C_{\mathrm{c(Fe)}} \cdot m_{\mathrm{c(Fe)}}\right]^2} \end{array}}$$

(10)

11.2 Detection efficiencies

11.2.1 ^{55}Fe detection efficiency

The calculation of ^{55}Fe detection efficiency in energy region A is given in Formula (1) in 8.3:

$$\varepsilon_{\text{Fe-55}(A)} = \frac{\left[r_{s(\text{Fe-55,A})} - r_{0(\text{A})}\right]}{A_{\text{Fe-55}} \cdot m_{\text{s(Fe-55)}}}$$

And thus the uncertainty on $\varepsilon_{\text{Fe-55(A)}}$, $u\left(\varepsilon_{\text{Fe-55(A)}}\right)$, is given in Formula (11):

$$u_{\left(\varepsilon_{\text{Fe-55(A)}}\right)} = \varepsilon_{\text{Fe-55(A)}} \cdot \sqrt{\left[\frac{u_{\left(A_{\text{Fe-55}}\right)}}{A_{\text{Fe-55}}}\right]^2 + \left[\frac{u\left(m_{\text{s(Fe-55)}}\right)}{m_{\text{s(Fe-55)}}}\right]^2 + \frac{\left[\frac{r_{\text{s}(\text{Fe-55},A)}}{t_{\text{s}(\text{Fe-55},A)}} + \frac{r_{0(\text{A})}}{t_{\text{b}}}\right]}{\left[r_{\text{s}(\text{Fe-55},A)} - r_{0(\text{A})}\right]^2}} \tag{11}$$

11.2.2 ^{59}Fe detection efficiency

The calculation of ^{59}Fe detection efficiency in energy region A is given in Formula (2) in 8.3:

$$\varepsilon_{\text{Fe-59(A)}} = \frac{\left[r_{\text{s(Fe-59,A)}} - r_{0(\text{A})}\right]}{A_{\text{Fe-59}} \cdot m_{\text{s(Fe-59)}}}$$

And thus, the uncertainty on $\varepsilon_{\text{Fe-59(A)}}$, $u\left(\varepsilon_{\text{Fe-59(A)}}\right)$, is given in Formula (12):

$$u_{\left(\varepsilon_{\text{Fe-59(A)}}\right)} = \varepsilon_{\text{Fe-59(A)}} \cdot \sqrt{\left[\frac{u_{\left(A_{\text{Fe-59}}\right)}}{A_{\text{Fe-59}}}\right]^2 + \left[\frac{u\left(m_{\text{s(Fe-59)}}\right)}{m_{\text{s(Fe-59)}}}\right]^2 + \frac{\left[\frac{r_{\text{s}(\text{Fe-59},A)}}{t_{\text{s}(\text{Fe-59},A)}} + \frac{r_{0(A)}}{t_{\text{b}}}\right]}{\left[r_{\text{s}(\text{Fe-59},A)} - r_{0(A)}\right]^2}} \tag{12}$$

Also, the calculation of ^{59}Fe detection efficiency in energy region B is given in Formula (3) in 8.3:

$$\varepsilon_{\text{Fe-59}(B)} = \frac{\left[r_{s(\text{Fe-59},B)} - r_{0(B)}\right]}{A_{\text{Fe-59}} \cdot m_{s(\text{Fe-59})}}$$

And thus, the uncertainty on $\varepsilon_{\text{Fe-59}(A)}$, $u\left(\varepsilon_{\text{Fe-59}(A)}\right)$, is given in Formula (13):

$$u_{\left(\varepsilon_{\text{Fe-59}(B)}\right)} = \varepsilon_{\text{Fe-59}(B)} \cdot \sqrt{\left[\frac{u_{\left(A_{\text{Fe-59}}\right)}}{A_{\text{Fe-59}}}\right]^2 + \left[\frac{u\left(m_{\text{s(Fe-59)}}\right)}{m_{\text{s(Fe-59)}}}\right]^2 + \frac{\left[\frac{r_{\text{s}(\text{Fe-59},B)}}{t_{\text{s}(\text{Fe-59},B)}} + \frac{r_{0(B)}}{t_{\text{b}}}\right]}{\left[r_{\text{s}(\text{Fe-59},B)} - r_{0(B)}\right]^2}} \tag{13}$$

11.3 Iron-55 activity

Calculate the net count rate in regions A and B as given in Formulae (14) and (15).

$$r_{\mathrm{net},A(\mathrm{Fe})} = r_{A(\mathrm{Fe})} - r_{0(A)} \tag{14}$$

and

$$r_{\mathrm{net},B(\mathrm{Fe})} = r_{B(\mathrm{Fe})} - r_{0(B)} \tag{15}$$

If the value $r_{\mathrm{net},B(Fe)}$ is greater than the background, and the uncertainty on that term given in Formula (16):

$$r_{\mathrm{net},B(\mathrm{Fe})} > 2.u\left(r_{\mathrm{net},B(\mathrm{Fe})}\right) \tag{16}$$

Then the net count rate of ^{55}Fe in region A is given in Formula (17):

$$r_{\mathrm{net},A(\mathrm{Fe})} = r_{A(\mathrm{Fe})} - r_{0(A)} - r_{\mathrm{net},B(\mathrm{Fe})} \cdot \frac{\varepsilon_{\mathrm{Fe-59}(A)}}{\varepsilon_{\mathrm{Fe-59}(B)}} \tag{17}$$

The activity may be calculated as given in Formula (18):

$$c_{\mathrm{Fe-55}} = \frac{r_{\mathrm{net},A(\mathrm{Fe})} \cdot m_{\mathrm{Fe}(1)}}{\varepsilon_{\mathrm{Fe-55}(A)} \cdot R_{(\mathrm{Fe})} \cdot m_{\mathrm{Fe}(4)} \cdot D_{\mathrm{Fe-55}} \cdot V_{\mathrm{s}}} \tag{18}$$

The count rate uncertainty is calculated as given in Formula (19):

$$u\left(r_{\mathrm{net},A(\mathrm{Fe})}\right) = \sqrt{\frac{r_{A(\mathrm{Fe})}}{t_{Fe}} + \frac{r_{0(A)}}{t_{\mathrm{b}}}} \tag{19}$$

If ^{59}Fe is present, this modifies to the form given in Formula (20):

$$u\left(r_{\mathrm{net},A(\mathrm{Fe})}\right) = \sqrt{\frac{r_{A(\mathrm{Fe})}}{t_{Fe}} + \frac{r_{0(A)}}{t_{\mathrm{b}}} + \left\{\left[\frac{u\left(r_{\mathrm{net},B(\mathrm{Fe})}\right)}{r_{\mathrm{net},B(\mathrm{Fe})}}\right]^2 + \left[\frac{u\left(\varepsilon_{\mathrm{Fe-59}(A)}\right)}{\varepsilon_{\mathrm{Fe-59}(A)}}\right]^2 + \left[\frac{\varepsilon_{\mathrm{Fe-59}(B)})}{\varepsilon_{\mathrm{Fe-59}(B)}}\right]^2\right\} \cdot \left[r_{\mathrm{net},B(\mathrm{Fe})} \cdot \frac{\varepsilon_{\mathrm{Fe-59}(A)}}{\varepsilon_{\mathrm{Fe-59}(B)}}\right]^2}$$

(20)

Then, the uncertainty on $c_{\mathrm{Fe-55}}$, $u\left(c_{\mathrm{Fe-55}}\right)$, is given in Formula (21):

$$u\left(c_{\mathrm{Fe-55}}\right) = c_{\mathrm{Fe-55}} \cdot \sqrt{\left[\frac{u\left(r_{\mathrm{net},A(\mathrm{Fe})}\right)}{r_{\mathrm{net},A(\mathrm{Fe})}}\right]^2 + \left[\frac{u\left(m_{\mathrm{Fe}(1)}\right)}{m_{\mathrm{Fe}(1)}}\right]^2 + \left[\frac{u\left(\varepsilon_{\mathrm{Fe-55}(A)}\right)}{\varepsilon_{\mathrm{Fe-55}(A)}}\right]^2 + \left[\frac{u\left(R_{(\mathrm{Fe})}\right)}{R_{(\mathrm{Fe})}}\right]^2 + \left[\frac{u\left(m_{\mathrm{Fe}(4)}\right)}{m_{\mathrm{Fe}(4)}}\right]^2 + \left[\frac{u\left(D_{\mathrm{Fe-55}}\right)}{D_{\mathrm{Fe-55}}}\right]^2 + \left[\frac{u\left(V_{\mathrm{s}}\right)}{V_{\mathrm{s}}}\right]^2}$$

(21)

11.4 ^{55}Fe decision threshold and detection limit

The assumption made is that $t_{(\mathrm{Fe})}=t_\mathrm{b}=t$, and $k_{1-\alpha}=k_{1-\beta}=k$, and that the multiplier, *w*, is given in Formula (22):

$$w=\frac{m_{\mathrm{Fe}(1)}}{\varepsilon_{\mathrm{Fe-55}(A)}\cdot R_{(\mathrm{Fe})}\cdot m_{\mathrm{Fe}(4)}\cdot D_{\mathrm{Fe-55}}\cdot V_\mathrm{s}} \tag{22}$$

Using the model described in ISO 11929 series, the decision threshold is given in Formula (23):

$$c_A{}^{*}=k_{1-\alpha}\cdot\tilde{u}(0) \tag{23}$$

NOTE 1 This can be explicitly stated as: $c_A{}^{*}=k_{1-\alpha}\cdot w\cdot u\left(r_{0(A)}\right)\cdot\sqrt{2}$.

And the detection limit is given in Formula (24):

$$c_A{}^{\#}=c_A{}^{*}+k_{1-\beta}\cdot\tilde{u}\left(c_A{}^{\#}\right) \tag{24}$$

NOTE 2 This can be explicitly stated as: $c_A{}^{\#}=\dfrac{k\cdot w\cdot\left[u\left(r_{0(A)}\right)\cdot\sqrt{8}-\dfrac{k}{t}\right]}{\left[1-k^2\cdot u_\mathrm{rel}^2(w)\right]}$.

With $k_{1-\alpha}=k_{1-\beta}=k$

11.5 Limits of the coverage intervals

11.5.1 Limits of the probabilistically symmetric coverage interval

The lower, $c_\mathrm{A}^{\triangleleft}$ and upper, $c_\mathrm{A}^{\triangleright}$ coverage limits are calculated using Formulae (25) and (26) according to ISO 11929-1 :

$$c_\mathrm{A}^{\triangleleft}=c_\mathrm{A}-k_p\cdot u\left(c_\mathrm{A}\right);p=\omega\cdot\left(1-{}^{\gamma}\!/_{2}\right) \tag{25}$$

$$c_\mathrm{A}^{\triangleright}=c_\mathrm{A}+k_q\cdot u\left(c_\mathrm{A}\right);q=1-\frac{\omega\cdot\gamma}{2} \tag{26}$$

where

$w=\Phi\dfrac{c_A}{u(c_A)}$ Φ being the distribution function of the standardized normal distribution;

$1-\gamma$ is the probability for the coverage interval of the measurand;

$\omega=1$ may be set if $c_\mathrm{A}>4\cdot u\left(c_\mathrm{A}\right)$. In this case, Formula (27) is obtained:

$$c_\mathrm{A}^{\triangleleft}=c_\mathrm{A}^{\triangleright}=c_\mathrm{A}\pm k_{1-{}^{\gamma}\!/_{2}}\cdot u\left(c_\mathrm{A}\right) \tag{27}$$

Often, a probability of 0,05 is chosen. In this case, $\gamma=0{,}05$ and then $k_{1-{}^{\gamma}\!/_{2}}=1{,}96$ is often chosen by default.

11.5.2 The limits of the shortest coverage interval

As described in detail in ISO 11929-1, the lower limit of the shortest coverage interval, $c_{\mathrm{A}}^{<}$, and the upper limit of the shortest coverage interval, $c_{\mathrm{A}}^{>}$, are calculated from a primary measurement result, c_{A}, of the measurand and the standard uncertainty, $u(c_{\mathrm{A}})$, associated with c_{A}, either by Formulae (28) or (29):

$$c_{\mathrm{A}}^{<}, c_{\mathrm{A}}^{>} = c_{\mathrm{A}} \pm k_{\mathrm{p}} \cdot u(c_{\mathrm{A}}), \text{where } p = \left[1 + \omega \cdot {}^{(1-\gamma)}\!/_{2}\right] \tag{28}$$

or if $c_A^{<} < 0$, then the result is given in Formula (29):

$$c_{\mathrm{A}}^{<} < 0; c_{\mathrm{A}}^{>} = c_{\mathrm{A}} + k_{\mathrm{q}} \cdot u(c_{\mathrm{A}}), \text{where } q = 1 + \omega \cdot \gamma \tag{29}$$

$w = \Phi \dfrac{c_A}{u(c_A)}$ Φ being the distribution function of the standardized normal distribution.

The relations $0 \leq c_{\mathrm{A}}^{\triangleleft} < c_{\mathrm{A}}^{\triangleright}$ apply and the approximation of Formula (27) is valid.

12 Test report

The test report shall conform to the requirements of ISO/IEC 17025 and shall contain at least the following information:

a) reference to this document (ISO 22515:2021);

b) identification of the sample;

c) units in which the results are expressed;

d) the test result;

   1) when the activity concentration, c_A is compared with the decision threshold (see ISO 11929 series);

      — if the result is less than the decision threshold, the result of the measurement is expressed as $\leq c_{\mathrm{A}}$,

      — if the result is greater than the decision threshold, the result of the measurement is expressed as $c_A \pm u(c_A)$ or $c_A \pm U$ with the associated k value,

   2) when the activity concentration, c_A is compared with the detection limit;

      — if the result is less than the detection limit, the result of the measurement is expressed as $\leq c_A^{\#}$;

      — if the result is greater than the detection limit, the result of the measurement is expressed as $c_A \pm u(c_A)$ or $c_{\mathrm{A}} \pm U$ with the associated k value.

   NOTE Occasionally, it is requested by the customer or regulator to compare the primary measurement result, c_A, with the detection limit, $c_A^{\#}$, in order to decide whether the physical effect is recognized or not. Such stipulations are not in accordance with the ISO 11929 series. They have the consequence that it is decided too frequently that the physical effect is absent when in fact it is not absent.

Complementary information can be provided such as:

e) the uncertainty can also be expressed as the limits of the probabilistically symmetric coverage interval $c_{\mathrm{A}}^{\triangleleft}, c_{\mathrm{A}}^{\triangleright}$ and/or the limits of the shortest coverage interval;

f) probabilities α, β and $1-\gamma$;

g) decision threshold and the detection limit;

h) if the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose;

i) mention of any relevant information to affect the results.