INTERNATIONAL STANDARD

ISO 10703

Third edition
2021-06

Water quality — Gamma-ray emitting radionuclides — Test method using high resolution gamma-ray spectrometry

Qualité de l'eau — Radionucléides émetteurs gamma — Méthode d'essai par spectrométrie gamma à haute résolution

Reference number
ISO 10703:2021(E)

COPYRIGHT PROTECTED DOCUMENT

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

Contents

Page

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*, 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).

This third edition cancels and replaces the second edition (ISO 10703:2007), which has been technically revised.

The main changes compared to the previous edition are as follows:

- new common Introduction;
- Scope enlarged to emergency situation and to wastewater, upper dead time increase to 10 %;
- the sample storage conditions are in compliance with ISO 5667-3 (see Clause 10);
- modification of the reference source for calibration (see 6.2);
- calibration efficiency determination by Monte Carlo method (see 11.2.3);
- complete revision of the pulse pile up and dead time;
- complete revision of the true coincidence summing subclause (see 12.1.4);
- addition of the correction factor for dead time and pile up (see 12.1.2);
- introduction of the shortest coverage interval in accordance with the new ISO 11929 series (see 12.5.2);
- modification of the test report (see Clause 13).

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, 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 fertilizer 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 the 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[7]. 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[8]. Water bodies and drinking waters are monitored for their radioactivity content as recommended by the World Health Organization (WHO)[9] 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 water bodies 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.

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 10 Bq·l^{-1} for $^{134/137}$Cs and ^{131}I activity concentration, 1 Bq·l^{-1} for ^{241}Am and 0,1 Bq·l^{-1} for ^{210}Pb.

NOTE 1 The guidance level is the activity concentration with an intake of 2 l/d of drinking water for one year that results in an effective dose of 0,1 mSv/a 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[9].

In the event of a nuclear emergency, the WHO Codex guideline levels[10] mentioned that the activity concentration for infant food might not be greater than 1 000 Bq·kg^{-1} for $^{134/137}$Cs, 100 Bq·kg^{-1} for ^{131}I and 1 Bq·kg^{-1} for ^{241}Am. For food other than infant food, the activity concentration might not be greater than 1 000 Bq·kg^{-1} for $^{134/137}$Cs, 100 Bq·kg^{-1} for ^{131}I and 10 Bq·kg^{-1} for ^{241}Am.

NOTE 2 The Codex guidelines levels (GLs) apply to radionuclides contained in food 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 food, and are based on an intervention exemption level of 1 mSv in a year for members of the public (infant and adult)[10].

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[11][12].

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 discharge 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 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 may 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 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 set of International Standards on test methods dealing with the measurement of the activity concentration of radionuclides in water samples.

Water quality — Gamma-ray emitting radionuclides — Test method using high resolution gamma-ray spectrometry

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 method for the physical pre-treatment and conditioning of water samples and the determination of the activity concentration of various radionuclides emitting gamma-rays with energies between 40 keV and 2 MeV, by gamma-ray spectrometry according to the generic test method described in ISO 20042.

The method is applicable to test samples of drinking water, rainwater, surface and ground water as well as cooling water, industrial water, domestic and industrial wastewater after proper sampling, sample handling, and test sample preparation (filtration when necessary and taking into account the amount of dissolved material in the water). This method is only applicable to homogeneous samples or samples which are homogeneous via timely filtration.

The lowest limit that can be measured without concentration of the sample or by using only passive shield of the detection system is about $5{\cdot}10^{-2}$ Bq/l for e.g. ^{137}Cs[1]. The upper limit of the activity corresponds to a dead time of 10 %. Higher dead times may be used but evidence of the accuracy of the dead-time correction is required.

Depending on different factors, such as the energy of the gamma-rays, the emission probability per nuclear disintegration, the size and geometry of the sample and the detector, the shielding, the counting time and other experimental parameters, the sample may require to be concentrated by evaporation if activities below $5{\cdot}10^{-2}$ Bq/l need to be measured. However, volatile radionuclides (e.g. radon and radioiodine) can be lost during the source preparation.

This method is suitable for application in emergency situations.

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 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste water*

ISO 5667-14, *Water quality — Sampling — Part 14: Guidance on quality assurance and quality control of environmental water sampling and handling*

---

[1] The sample geometry: 3l Marinelli beaker; detector: GE HP N relative efficiency 55 % ; counting time: 18h.

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 and the following 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
blank sample
container of an identical composition to the one used for the water test sample, filled with demineralized water

3.2
dead time
time during spectrum acquisition (real time) during which pulses are not recorded or processed

Note 1 to entry: Dead time is expressed in percent, calculated as follows: real time minus live time, all divided by real time then multiplied by 100

3.3
dead time correction
correction to be applied to the observed number of pulses to take into account the number of pulses lost during the *dead time* (3.2)

3.4
decay constant
λ
quotient of the rate at which a population of radioactive atoms decreases because of radioactive decay by the size of that population of radioactive atoms

Note 1 to entry: It can also be expressed as the quotient of dP by dt,

$$\lambda = \frac{\mathrm{d}P}{\mathrm{d}t} = -\frac{1}{N}\frac{\mathrm{d}N}{\mathrm{d}t}$$

where

dP is the probability of a given nucleus undergoing a spontaneous nuclear transition from that energy state in the time interval dt;

N is the number of nuclei of concern existing at time t.

Note 2 to entry: The time required for half of the original population of radioactive atoms to decay is called the half-life. The relationship between the half-life, $T1/2$, and the decay constant is given by $T1/2 = \ln(2)/\lambda$.

3.5
efficiency
ratio of the number of gamma photons detected in the full energy peak to the number of the same type emitted by the radiation source in the same time interval, under stated conditions of detection

3.6
energy resolution

measure, at a given energy, of the smallest difference between the energy of two gamma-rays which can be distinguished by the apparatus used for *gamma-ray spectrometry* (3.10)

Note 1 to entry: The energy resolution is measured as the full width at half maximum height of the energy peak (FWHM).

3.7
full energy peak

peak of spectral response corresponding to the total absorption of the photon energy in the detector by the photoelectric effect or by consecutive photon interactions of pair production (only for photon energy >1 022 keV), Compton scattering and photoelectric absorption

3.8
gamma cascade

two or more different gamma-photons emitted successively from one nucleus when it de-excites through one or more intermediate energy levels

3.9
gamma radiation

electromagnetic radiation emitted in the process of nuclear transition or particle annihilation

3.10
gamma-ray spectrometry

method of measuring gamma-rays yielding the energy spectrum of the *gamma radiation* (3.9)

3.11
pile-up

processing by a radiation spectrometer of pulses resulting from the simultaneous absorption of particles, or photons, originating from different decaying nuclei, in the radiation detector

Note 1 to entry: As a result, pulses are counted as one single particle or photon with an energy between the individual energies and the sum of these energies.

3.12
transition probability

probability of de-excitation of the nucleus occurring by one specific transition at a given energy state to a less energetic state or to the ground state

4 Symbols

For the purposes of this document, the following symbols apply.

V	Volume of the water sample for test	l
A	Activity of each radionuclide in calibration source, at the calibration time	Bq
c_A, $c_{A,c}$	Activity concentration of each radionuclide, without and with corrections	$\text{Bq}\cdot\text{l}^{-1}$
t_g	Test sample spectrum counting time	s
t_0	Background spectrum counting time	s
t_i	Time between the reference time and the start of the measuring time	s
t_S	Calibration spectrum counting time	s
$n_{N,E}$, $n_{N0,E}$, $n_{Ns,E}$	Number of counts in the net area of the peak, at energy E, in the test sample spectrum, in the background spectrum and in the calibration spectrum, respectively	

$n_{g,E}, n_{g0,E}, n_{gs,E}$	Number of counts in the gross area of the peak, at energy E, in the test sample spectrum, in the background spectrum and in the calibration spectrum, respectively	
$n_{b,E}, n_{b0,E}, n_{bs,E}$	Number of counts in the background of the peak, at energy E, in the test sample spectrum in the background spectrum and in the calibration spectrum, respectively	
ε_E	Detection efficiency at energy E at actual measurement geometry	
f_E	Correction factor considering all necessary corrections	
f_d	Correction factor for decay for a reference date	
$f_{cl,E}$	Correction factor for coincidence losses (summing-out)	
$f_{su,E}$	Correction factor for summing-in effects by coincidences	
$f_{dt\ pu,E}$	Correction factor for dead time and pile up	
P_E	Probability of the emission of a gamma-ray with energy E of each radionuclide, per decay	
λ	Decay constant of each radionuclide	s^{-1}
$u(c_A), u(c_{A,c}), u(fit)$	Standard uncertainty associated with the measurement result (without and with corrections) and with fitting efficiency curve respectively	$Bq \cdot l^{-1}$
U	Expanded uncertainty calculated with $k = 2$.	$Bq \cdot l^{-1}$
$c_A^*, c_{A,c}^*$	Decision threshold, without and with corrections	$Bq \cdot l^{-1}$
$c_A^\#, c_{A,c}^\#$	Detection limit, without and with corrections	$Bq \cdot l^{-1}$
$c_A^\triangleleft, c_A^\triangleright$	Lower and upper limits of the probabilistically symmetric coverage interval	$Bq \cdot l^{-1}$
$c_A^<, c_A^>$	Lower and upper limits of the shortest coverage interval	$Bq \cdot l^{-1}$

5 Principle

Gamma-rays produce electron-hole pairs when interacting with matter. When a voltage is applied across a semiconductor detector, these electron hole-pairs are, after proper amplification, detected as current pulses. The pulse height is related to the energy absorbed from the gamma-photon or photons in the resolving time of the detector and electronics. By discriminating between the height of the pulses, a gamma-ray pulse height spectrum is obtained. After analysis of the spectrum, the various peaks are assigned to the radionuclides which emitted the corresponding gamma-rays using an established detector energy calibration response curve. The concentration of the radionuclides present in the sample is calculated using the established energy-dependent detector efficiency curve.

The determination of the activity concentration of radionuclides emitting gamma-rays with energy below 40 keV and above 2 MeV is also possible within the scope of this document, provided both the calibration of the measuring system and the shielding are adapted to this purpose.

6 Reference sources

6.1 Source(s) for energy calibration

The energy calibration of the spectrometer shall be established using one or more sources containing radionuclides that emit gamma-rays that cover the energy range of interest. Sources can be of any form but the dead time of the spectrometer for the measurements shall be such that the full energy peak shape is not distorted and pulse pile-up is avoided.

The number of peaks (full energy peaks) required depends on the order of polynomial needed for the energy vs. channel calibration curve; normally 5 to 10 peaks should be sufficient. Sources containing long-lived radionuclides (for example ^{152}Eu, ^{241}Am, ^{60}Co or ^{137}Cs) are recommended for this purpose. For periodical checks of the energy calibration, a smaller number of energy peaks may be used.

6.2 Reference source(s) for efficiency calibration

6.2.1 General

The general method to calibrate the spectrometer is to establish the detection efficiency as a function of energy for a defined geometry and energy range. One or more reference sources containing single or multiple radionuclides may be used for this purpose. The activity or emission rates of the radionuclide(s) in the reference source(s) shall be traceable to national or international standards.

The energies of the emitted gamma-rays shall be distributed over the entire energy range of interest, in such a way that the energy-dependent efficiency of the spectrometer can be determined in a sufficiently accurate way. For most purposes, the accuracy is sufficient for an energy range of 60 keV to 1 836 keV if a multi-radionuclide source is used containing all or most of the following radionuclides: ^{241}Am, ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg, ^{51}Cr, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{54}Mn, ^{59}Fe, ^{60}Co, ^{65}Zn or ^{88}Y.

For determining the activity of radionuclides emitting gamma-ray or X–rays in the energy region less than 60 keV, the spectrometry system can be calibrated using a reference source containing the radionuclides of interest.

It may be necessary to take into account true coincidence summing corrections for the calibration radionuclides (for example ^{60}Co and ^{88}Y).

6.2.2 Reference sources for laboratory systems

Reference sources for laboratory-based spectrometry systems shall match, as closely as possible, the geometry, density and matrix of the samples to be measured. Reference sources may be prepared from standardised solutions or purchased as sealed sources. Only standardised solutions or reference sources that are traceable to national or international primary standards of radioactivity shall be used.

If no reference materials are available to match the samples, correction factors shall be calculated, documented and be applied to results from the measurements to take into account differences in detection efficiency due to geometry, density and matrix effects.

If a reference source is prepared by dilution from a standardised solution, the supplier's recommendation on the chemical form of the diluent shall be followed. It is also recommended that the dispensing process includes checks for possible losses of active material and on the accuracy of dispensing (for example gravimetric, volumetric and radiometric techniques should be used and cross-checked).

6.2.3 Reference sources used with numerical methods

Reference sources for gamma-ray spectrometry systems based on numerical models shall be used following the manufacturer's recommendations. The activity or the emission rates of the reference sources shall be traceable to national or international standards.

7 Reagents

The following reagents shall be used when the sample is concentrated by evaporation with iodine retention. Use only reagents of recognized analytical grade and only water complying with grade 3 of ISO 3696.

7.1 Nitric acid, concentrated, $c(\text{HNO}_3)$ = 15,8 mol/l, 69 % volume fraction or mass fraction, [$\rho(\text{HNO}_3)$ = 1,42 g/ml].

7.2 Sulfuric acid, concentrated, $c(\text{H}_2\text{SO}_4)$ = 17,9 mol/l, 95 % volume fraction or mass fraction, [$\rho(\text{H}_2\text{SO}_4)$ = 1,84 g/ml].

7.3 Silver nitrate solution, $c(AgNO_3)$ = 3,2 g/l.

Dissolve 3,2 g of silver nitrate in water acidified with 0,1 ml of nitric acid and dilute to a total volume of 1 l with water.

7.4 Potassium iodide solution, $c(KI)$ = 1,3 g/l.

Dissolve 1,3 g of potassium iodide in 1 l of water.

7.5 Sodium sulfite, Na_2SO_3.

7.6 Hydrogen peroxide solution, $c(H_2O_2)$ = 0,3 g/l.

7.7 Sodium carbonate solution, Na_2CO_3, saturated at 20 °C.

8 Gamma-ray spectrometry equipment

8.1 General description

The operation of the measurement system is as follows: in semi-conductor detectors, freed charge is generated by the interaction of ionising radiation with the detector material (through the photoelectric effect, the Compton effect or pair production). A high-voltage supply applies a bias voltage to the detector crystal resulting in an electric field. The freed charge is accelerated by the electric field towards the detector electrodes. The collected charge is converted into an output voltage pulse by a preamplifier and the output pulse is shaped and amplified by the main amplifier.

Two types of electronic systems can be used to process the signal from the detector preamplifier; an analogue amplifier combined with an analogue-to-digital converter (ADC), or a digital DSP (Digital Signal Processor) system. Both systems convert the pulse amplitude and the pulse-height histogram (spectrum) is stored using a multichannel analyzer (MCA). The height of the pulse is proportional to the amount of freed charge and hence to the energy of the ionising radiation striking the detector.

The spectrum stored by the MCA shows a set of peaks (full energy peaks) superimposed on a background continuum from scattered radiation. The full energy peaks are approximately Gaussian in shape. The channel number of the peak centroid depends on the energy of the photon detected. The net full energy peak area is proportional to the number of photons of that energy that have interacted with the detector during the counting period (corrected for dead time). The net full energy peak area is normally determined in the analysis software package by one of two different techniques – summation or fitting

For laboratory use, the spectrometer should be located in a facility with stable temperature following the manufacturer recommendations. It should be noted that changes in temperature can affect the amplifier gain, changing the energy calibration substantially.

The apparatus shall consist of the following necessary parts from 8.2 to 8.8.

8.2 Detector types

The three main geometries of germanium detectors available are planar, coaxial and well-type. Each has specific advantages depending on the circumstances. Coaxial detectors are generally used with large volume samples, whereas the well-type detectors are most efficient for small volume samples. Planar detectors can be useful for detecting photons with energies below 200 keV as they can have better energy resolution than coaxial detectors at these energies. More detailed information on the detectors is given in ISO 20042:2019, Table D.1.

Microphonics phenomena can result in an increase in the Full Width at Half Maximum (FWHM) of the full energy peak. It may be necessary to place the detector on an anti-vibration mat.

8.3 High voltage power supply

When in operation, the power supply should be free from spikes and dropouts, or provided with a power conditioner and/or uninterruptible power supply. Instrument earth connections should be isolated and connected to local ground at one point only to avoid ground loops that may affect the detector resolution.

WARNING — Take necessary safety precautions according to the manufacturer's instructions.

8.4 Preamplifier

The preamplifier strongly influences the quality of the entire measuring system, as both noise and energy resolution depend on the characteristics of the preamplifier.

NOTE Usually the preamplifier is located very close to germanium crystal. Cooling the input stage (FET) of the preamplifier decreases the noise level and improves the energy resolution.

8.5 Cryostat or electric cooler

The cryostat or the electric cooler should be capable of keeping the detector close to the temperature of liquid nitrogen. Operation at a low temperature is required to reduce the leakage current and electronic noise level of the detector and preamplifier; it is recommended that an automatic switch off and an alarm signal be installed which are activated in the case of an increase in the temperature of the detector (e.g. caused by malfunctioning of the cryostat or loss of liquid nitrogen).

A high purity germanium detector may be stored at room temperature, however, it shall be cooled before the bias voltage is applied.

8.6 Shielding

The detector shall be shielded from all sides (including the bottom) with lead, to reduce background signals originating mainly from naturally occurring radionuclides. If measurements in the energy region 40 keV < E < 100 keV are to be made, the internal casing should consist of three successive layers of cadmium/tin, copper and polymethylmethacrylate to achieve a low and constant background by attenuating the X-ray produced in the shielding. Too large a thickness of Cu can increase the background from bremsstrahlung between 100 keV and 200 keV.

Shielding is important to reduce background levels, especially if low activity levels shall be measured. The following measures can be taken to improve the performance of the shield:

- use low activity lead;
- limit the back scattering effect by not having shielding close to the detector if at all possible;
- carefully choose ventilation conditions, air filtration and the construction and other materials of the system, in order to reduce ambient radioactivity and achieve low levels of background radiation. Reduction of the radon inside the shield is desirable for measuring low-activity samples. This may be achieved by displacing the air inside the shield with nitrogen boiled off from the cooling Dewar.

Active shielding can also be used (such as cosmic veto or anti-Compton systems).

8.7 Analogue or digital acquisition electronics

8.7.1 General

Regardless of the ADC or DSP system used, it shall have a good linearity to obtain a linear pulse conversion for the 40 keV to 2 MeV energy range regardless of the gain applied to the signal. Similarly, the conversion system shall provide a minimum of 4096 channels to obtain a good energy resolution which assists the discrimination of peaks and the deconvolution of multiplets.

8.7.2 Analogue electronic (ADC)

Analogue amplifier-based systems should have an adjustable Gaussian pulse shaping system. An adjustment between 4 µs and 8 µs is recommended to obtain the best energy resolution (FWHM). It shall also have a pole-zero setting and an automatic mechanism for restoring the continuous component. A stacking rejection device is recommended for high count rates (>5 000s^{-1}).

An analogue amplifier is usually associated with an analogue-to-digital converter, which converts the pulses coming from the amplifier according to their amplitude.

The data thus converted is stored in a MCA (Multi Channels Analyzer), itself connected to a computer. The computer software to view and save the spectrum reads the MCA memory. These modules can be separated in a NIM-type bin system or integrated into a single unit.

8.7.3 Digital electronic (DSP)

Digital systems are generally integrated into a single unit (referred to as an MCA) comprising signal formatting electronics, scanning and storage electronics. The shaping circuit transforms the input pulses to obtain trapezoidal pulses whose characteristics can be adjusted using "Rise time" and "Flat top" parameters. These parameters should be adjusted to obtain the best energy resolution (FWHM) depending on the type of detector used and the expected count rate. Manufacturers generally indicate the best settings to use. The system shall also have a pole-zero setting.

8.8 Computer, including peripherical devices and software

The computer, in combination with the available hardware and software should be able to:

- read the data from the MCA;
- reproduce these data on a video display or a printer and store them;
- determine the relation between channel number and corresponding energy over the entire energy range to be studied, by making use of the appropriate reference source, energy calibration;
- determine the energy-dependent efficiency over the entire energy range to be studied, by making use of the appropriate reference source;
- detect peaks, determine the characteristics of the detected peaks such as the centroid, the peak FWHM, the number of net counts collected under the peak, and determine the uncertainty of this number;
- identify the radionuclides responsible for the observed full energy peaks by making use of radionuclide references (see Clause 9);
- calculate the activity concentration of the respective radionuclides on the basis of the number of counts, the counting time, the counting efficiency and the data given in radionuclide references (see Clause 12);
- calculate the combined uncertainty of the activity concentration of the identified radionuclides;
- calculate the detection limit and the decision threshold of radionuclides to be measured but not found in the sample.

It is recommended that the results of the computer analysis of the spectrum be visually checked regularly for obvious anomalies or errors. To check the performance of the apparatus, the use of a laboratory standard is recommended. Participation in intercomparison exercises also helps to test the performance of the apparatus and analysis.

Whenever necessary, calculations and identification may be performed manually.

9 Nuclear decay data

Nuclear decay data (half-lives, gamma-ray energy and emission probabilities) are available from the scientific literature, databases and other publications. There are often differences between the values depending on the source data used and the evaluation method.

To ensure consistent results from gamma-ray spectrometry measurements, nuclear decay data used for instrument calibration and for estimating the activity content of samples, should therefore be taken from the Decay Data Evaluation Project (http://www.lnhb.fr/nuclear-data/). If no evaluation of the nuclear decay data of the radionuclide of interest is available in this database, the following databases should be consulted:

- Joint Evaluated Fission and Fusion (JEFF) Library (https://www.oecd-nea.org/dbdata/);
- National Nuclear Data Center database (https://www.nndc.bnl.gov/);
- Evaluated Nuclear Data File (ENDF) (https://www-nds.iaea.org/exfor/endf.htm).

10 Sampling

The sample shall be collected and preserved as specified in ISO 5667-1, ISO 5667-3, ISO 5667-10 and ISO 5667-14. Particular attention shall be given to the following:

- the time elapsed between sampling and the start of the measurement of the sample;
- homogeneity of the sample; if any particulate matter is present which can cause heterogeneity, this shall be removed by filtration, and the residue shall be measured separately if necessary;
- for the sampling, polyethylene bottles should be used, cleaned with 1 mol/l hydrochloric acid, followed by leaching with dilute nitric acid solution and rinsing with distilled or deionized water (any other cleaning treatment may be used if its effectiveness is demonstrated);
- depending on the elapsed time between sampling and measurement (e.g. if storage is more than seven days) as well as the radionuclide to measure, the sample should be acidified to pH < 2 with nitric acid; if particulate matter is removed by filtration or centrifugation, this shall be carried out before acidification;
- between acidification and measurement, the sample should be transported and/or stored in the absence of light and at a temperature of between 1 °C and 5 °C.

If radioiodine is to be determined, hydrochloric acid should be used instead of nitric acid for acidification of the sample.

NOTE In some cases, it can be desirable to add a carrier solution to the sample. For example, when waste water from a nuclear power plant is measured, a carrier solution as described in Annex A can be used.

11 Procedure

11.1 Sample preparation

11.1.1 General

This document describes three different ways for preparing the water sample. The method of sample preparation used shall be mentioned in the test report.

The choice of the method depends on the limit of detection required. However, if radionuclides of iodine have to be determined, the choice can only be made between 11.1.2 or 11.1.4, depending on the limit of detection required.

11.1.2 Direct measurement without preparation

Direct measurement can be used to get results rapidly, for example in situations such as emission monitoring or accident control. The sample is measured directly, preferably in a Marinelli type beaker.

When the water sample is filtered, the residue on the filter shall be measured separately. In the test report, the method shall be referred to as “direct measurement”, giving the results for “liquid” and for “solid” both referring to the volume of the sample. The mass concentration of suspended solids shall also be given.

NOTE 1 Homogenization of the suspended matter in the sample can be made by adding a gelling agent.

NOTE 2 Partial evaporation of the sample can be performed to reduce the volume and concentrate the activity before the direct measurement.

11.1.3 Evaporation without iodine retention

Evaporate the water to dryness or to a volume suitable for the measurement conditions, with the possible addition of a carrier. If necessary, determine the mass of the residue and measure the activity of the quantity corresponding to the mass of the standard geometry. In the test report, the method shall be referred to as "evaporation without iodine retention".

11.1.4 Evaporation with iodine retention

Add to the sample, while stirring, 10 ml of potassium iodide solution (7.4), 0,1 g of sodium sulfite (7.5) and 5 ml of concentrated sulfuric acid (7.2) per litre of unfiltered water sample. After stirring for 5 min, add 10 ml of silver nitrate solution (7.3) and 10 ml of hydrogen peroxide solution (7.6) per litre. Adjust the pH to a value of 9, using saturated sodium carbonate solution (7.7).

Evaporate and dry the sample in accordance with 11.1.3. In the test report, the method shall be referred to as "evaporation with iodine retention".

In the case of partial evaporation, the concentrated solution cannot be measured in this state, it is then necessary to dissolve the precipitate of silver iodide or to proceed with a filtration step followed by distinct filter and filtrate measurements.

11.2 Calibration

11.2.1 General

Energy and efficiency calibration procedures are given below, following procedures described in ISO 20042.

11.2.2 Energy calibration

For reliable radionuclide identification, the energy calibration should be accurate to better than 0,2 keV over the energy range of interest. The energy calibration of the spectrometer shall therefore be established using one or more sources containing radionuclides that emit gamma-rays that cover the energy range of interest. This calibration establishes the relationship between the channel number of the photopeak centroid and the known energy of the photons. This task is normally carried out using commercially-available software to determine the photopeak centroid (channel) and to match this to the energy (keV). The software also determines the full width half maximum of the full energy peaks. This information is used by the photopeak search algorithm and for the calculation of characteristic limits.

The count rate in the detector shall be such that the full energy peak shape is not distorted due to pulse pile-up; a dead time less than 10 % normally suffices. For a germanium spectrometer, the full energy peak position should be determined with an accuracy of better than 0,2 keV.

A calibration curve (energy vs channel) should be calculated by least-squares fitting, so that photopeak energies may be interpolated. A linear or quadratic function is normally sufficient but higher order polynomials may be required. A similar process should be used to determine the FWHM as a function of energy.

Care shall be taken when extrapolating the energy calibration curve. Photopeaks in the background spectrum or X-rays should be used to check the accuracy of the extrapolated curve, and the extrapolation should be plotted to check for large deviations from a linear function due to quadratic or higher order components.

11.2.3 Efficiency calibration

The following approaches may be considered for determining the detection efficiency:

- direct comparison with a reference source of the same radionuclide in the same matrix and geometry. This approach is appropriate if the aim of the measurement is to determine the activity of a single radionuclide or if the photon energy is in an energy region that is difficult to calibrate (typically 20 keV to 100 keV). It may also be required if true coincidence summing corrections are significant (for example, when measuring a gamma-ray emitting radionuclide with a complex decay scheme using a high efficiency detector such as a well detector);
- measurement of the full energy peak detection efficiency as a function of energy;
- calculation of the full energy peak detection efficiency as a function of energy by Monte Carlo simulation or other modelling technique. Such numerical models are sensitive to input parameters such as the detector dimensions, and therefore shall be checked using at least one reference source containing radionuclides that emit gamma-rays covering the energy range of interest. If a discrepancy is found between efficiency calculated using the model and the reference source, the discrepancy shall be investigated and corrections applied to the numerical model.

The same algorithm for analysis of the spectrum shall be used for both calibration and sample measurement.

The detection efficiency is affected by the following factors:

- the detector;
- the geometry of the sample with respect to the detector (solid angle);
- the density of the sample and the sample container characteristics;
- the sample mass and chemical composition;
- the heterogeneity of the sample matrix with respect to activity, density and chemical composition.

When any of these factors is changed, the detection efficiency shall be re-evaluated for the new conditions.

If no numerical model is used, the test sample measurement shall be performed with comparable measuring conditions as used for calibrating the gamma-ray spectrometry system. In particular, the measurement geometry, the position of the reference source in relation to the detector and the test sample and reference source matrices should be identical. Corrections should be applied to account for differences in the matrix and other factors that can affect the detection efficiency, as appropriate. If the efficiency calibration method uses a numerical model, the parameters used to describe the sample shall match the item being measured.

In most practical measurements, the instrument dead time for the calibration measurements is higher than for the sample measurements. The activity of the efficiency calibration source shall be such that count rate does not distort the full energy peak shape and that an accurate dead-time correction is applied by the analysis software; a dead time of less than 10 % normally suffices. Higher dead times may be used but evidence of the accuracy of the dead-time correction shall be required.

The full energy peaks in the spectrum shall contain sufficient counts such that the uncertainty in the full energy peak area is small in comparison with other components of uncertainty. A full energy peak area of 10 000 counts is normally sufficient. The full energy peak shall be discarded from the efficiency calibration data set if visual inspection shows that it is not Gaussian in shape.

The detection efficiency for each full energy peak in the calibration spectrum shall be calculated using Formula (1) (when reference source is used):

$$\varepsilon_E = \frac{n_{\mathrm{Ns},E}/t_s}{A \cdot P_E} \tag{1}$$

NOTE This formula assumes negligible decay of the calibration source during the acquisition time.

For an undisturbed peak at an energy E, the count $n_{\mathrm{Ns},E}$ in the net-peak area of a γ-spectrum is calculated using Formula (2):

$$n_{\mathrm{Ns},E} = n_{\mathrm{gs},E} - n_{\mathrm{bs},E} \tag{2}$$

The efficiency for each specific radionuclide i, at energy E, is determined using Formula (3):

$$\varepsilon_{i,E} = \frac{n_{\mathrm{Ns},E}/t_s}{A_i \cdot P_E} \tag{3}$$

For an undisturbed peak at an energy E, the count $n_{N,E}$ in the net-peak area of a γ-spectrum is calculated using Formula (4):

$$n_{N,E} = n_{\mathrm{g},E} - n_{\mathrm{b},E} \tag{4}$$

The detection efficiency as a function of energy should be calculated and, if required, a function fitted to enable detection efficiency to be interpolated. The detection efficiency is a varying function of energy and software packages offer a choice of different functions. The selection of the fit function shall be based on an evaluation of the goodness of fit parameter (chi-squared) and visual inspection of the function to confirm that the fitted function does not deviate from a smooth curve due to using too many variable parameters. Extrapolation of the efficiency calibration function to energies below the range of the calibration points shall not be used. Extrapolation of the efficiency calibration function to energies above the range of the calibration points is not recommended unless supported by other evidence such as modelling.

12 Expression of results

12.1 Calculation of the activity concentration

12.1.1 General

The activity concentration, c_A, of each radionuclide present in the sample is obtained from the net count $n_{N,E}$ from the peak of an individual gamma line without any interference using Formula (5):

$$c_A = \frac{n_{N,E}/t_{\mathrm{g}}}{P_E \cdot \varepsilon_E \cdot V \cdot f_E} \tag{5}$$

where

f_E is calculated according to Formula (6):

$$f_E = f_\mathrm{d} \cdot f_{cl,E} \cdot f_{su,E} \cdot f_{\mathrm{dt}\ pu,E} \tag{6}$$

Formula (5) might be expressed as:

$$c_A = \frac{n_{N,E}/t_\mathrm{g}}{P_E \cdot \varepsilon_E \cdot V \cdot f_E} = \frac{n_{\mathrm{g},E} - n_{\mathrm{b},E}}{P_E \cdot \varepsilon_E \cdot V \cdot f_E \cdot t_\mathrm{g}} = (n_{\mathrm{g},E} - n_{\mathrm{b},E}) \cdot w / t_g \text{ with } w = \frac{1}{P_E \cdot \varepsilon_E \cdot V \cdot f_E} \tag{7}$$

When samples are acidified, it could be appropriate to acidify blank water in the same manner. The same consideration apply for salt carriers, if applied.

Demineralized water shall not contain radon or other gamma-emitting radionuclides.

12.1.2 Dead time and pile up corrections (see ISO 20042)

The pulse processing electronics and analysis software normally apply a correction for the dead time introduced by the electronics. However, the analogue pulse may extend beyond this time period; if a second pulse arrives on the tail of the pulse, the pulse height may be distorted and a pulse may be lost from the full energy peak in the spectrum.

The effect may be seen in the spectrum as increased full energy peak widths (FWHM) and low-energy tails. The effect shall not be neglected for low-activity measurements as it may be significant for the reference source used for calibration.

The effect depends on the instrument and electronics used.

An additional correction may be needed if the dead time changes significantly during the measurement time (for example when measuring high-activity short half-life samples). Specialist dead-time correction systems are needed in this case[8].

Low count rates (less than a few thousand pulses per second, equivalent to dead times of a few percent) should be used when possible, by choosing appropriate sample sizes and geometry.

Short shaping times may be selected on the main amplifier to reduce pulse lengths. However, this may have the effect of increasing the FHWM of full energy peaks.

Care shall be taken to ensure that the pole-zero adjustment has been made as an incorrect setting may affect the magnitude of this effect.

For dead times less than 10 %, the correction for pile-up is typically less than 1 %, though the magnitude of the correction depends on the spectrometer type and settings. If higher dead times are used, a correction should be applied as described in ISO 20042.

12.1.3 Decay corrections

The specific activity shall be corrected to take into account the radioactive decay during the counting time and during the time between the reference time (t = 0) and the measuring time ($t = t_\mathrm{i}$). The decay factor,f_d, shall be calculated by:

$$f_\mathrm{d}^{-1} = e^{\lambda \cdot t_\mathrm{i}} \cdot \left[\frac{\lambda \cdot t_\mathrm{g}}{1 - e^{-\lambda \cdot t_\mathrm{g}}} \right] \tag{8}$$

12.1.4 True coincidence summing

12.1.4.1 General

A detailed description of true coincidence summing is given in the Annex B.

12.1.4.2 Probability of true-coincidence summing

Because true-coincidence summing requires the detection of two photons from the same decay it is strongly dependent on the following:

- the detection efficiency for the photons that participate in the summing process. This is a function of the detector type and the counting geometry that is used. For example, the probability of detecting two photons as a summed event increases with increasing detector size and decreasing source-to-detector distance.
- The nuclide that is being measured. More specifically, the decay schema of the nuclide. Earlier we have learned that summing in and out depends on the gamma-ray decay schema (e.g. the presence of a cascade and the mode of decay, such as internal conversion or electron capture). There are some nuclei that are unaffected (such as ^{137}Cs) and others (such as ^{60}Co and ^{88}Y) that demonstrate significant summing effects.

12.1.4.3 Consequences for source-based efficiency calibrations and for sample count

Most commercially available multi-nuclide sources include nuclides that exhibit true-coincidence summing. For example it is common to use ^{60}Co and ^{88}Y as the high-energy photon-emitting nuclides and both of these suffer from summing out. Using nuclides that have summing-out effects in close geometries results in peak count rates that are lower than they would have been if the nuclide didn't have summing-out effects. The result of this is that the efficiency at these energies appears to be lower than the true efficiency. If these efficiency points are used without correction, then the calculated efficiency is biased low compared to the true efficiency. As a result, when the efficiency is used to calculate the activity for a summing-free sample measurement, the result is higher than the true activity.

For sample counts, the summing-out effects cause a reduction in the measured peak count rates; the measured activities is therefore lower than the true activities. Summing in has the effect to generate summation peak that can interfere[2]. It should be noted that summing-out effects are most common. It is also the more significant effect since the underestimating of nuclide activities can have safety implications.

12.1.4.4 Reducing the effect of true-coincidence summing

Since the probability of true-coincidence summing is dependent on the detection efficiency, the effect can be reduced by moving the sample away from the detector. However, the impact of reducing the overall efficiency of the counting system can result in increased counting times (in order to maintain counting statistics) and therefore reduced productivity for sample counting.

If the coincidence summing is mainly due to x-rays (as may be the case when the nucleus decays by electron capture) then the coincidence summing can be reduced by introducing an attenuator that attenuates most low energy photons. The optimum treatment is to use specific software to quantify and correct for true-coincidence-summing effects. This approach is applicable for all measurement geometries and all commonly measured nuclides that are known to be affected.

Most of the theoretical methods to calculate summation are related with the use of transport theory and Monte-Carlo techniques (see References [15] to [20]). Given the difficulties to model the detectors, some experimental procedures can be applied for each specific situation.

Further information is given in References [21] and [22].

---

[2] E.g. a ^{152}Eu summation peak at 167,7 keV interferes with ^{201}Tl at 167,5 keV, ^{110m}Ag summation peak at 1331,5 keV interferes with ^{60}Co at 1332,5 keV, ^{57}Co summation peak at 136 keV interferes with ^{201}Tl at 136 keV,

12.2 Standard uncertainty

According to the ISO/IEC Guide 98-3, the standard uncertainty of c_A is calculated according to Formula (9):

$$u(c_A)=\sqrt{(w/t_\mathrm{g})^2\cdot[u^2(n_{\mathrm{g},E})+u^2(n_{\mathrm{b},E})]+c_A^2\cdot u_\mathrm{rel}^2(w)} \tag{9}$$

Where the standard uncertainty of the counting time is neglected and the relative standard uncertainty of w is calculated by Formula (10):

$$u_\mathrm{rel}^2(w)=u_\mathrm{rel}^2(P_E)+u_\mathrm{rel}^2(V)+u_\mathrm{rel}^2(\varepsilon_E)+u_\mathrm{rel}^2(f_E) \tag{10}$$

Taking into account Formula (3), the relative standard uncertainty of ε_E is calculated by Formula (11):

$$u_\mathrm{rel}^2\left(\varepsilon_E\right)=u_\mathrm{rel}^2\left(n_{Ns,E}\right)+u_\mathrm{rel}^2\left(A\right)+u_\mathrm{rel}^2\left(P_E\right)=u_\mathrm{rel}^2\left(n_{\mathrm{gs},E}-n_{\mathrm{bs},E}\right)+u_\mathrm{rel}^2\left(A\right)+u_\mathrm{rel}^2\left(P_E\right)+u_\mathrm{rel}^2\left(fit\right) \tag{11}$$

where $u_\mathrm{rel}(A)$ includes all the uncertainties related to the calibration source: calibration certificate of the standard solution and preparation of the calibration source and the addition of this calibration source.

For the calculation of the characteristic limits (see ISO 11929-1), $\tilde{u}(\tilde{c}_A)$, is needed, i.e. the combined standard uncertainty of c_A as a function of its true value. For a true value, $\tilde{c}_A$, it is expected $n_{\mathrm{g},E}/t_\mathrm{g}=\tilde{c}_A/w+n_{\mathrm{b},E}/t_\mathrm{g}$ and with $u^2(n_\mathrm{g})=n_\mathrm{g}$, the following is obtained:

$$\tilde{u}(\tilde{c}_A)=\sqrt{w^2\cdot[(\tilde{c}_A/w+n_{\mathrm{b},E}/t_\mathrm{g})/t_\mathrm{g}+u^2(n_{\mathrm{b},E})/t_\mathrm{g}^2]+\tilde{c}_A^2\cdot u_\mathrm{rel}^2(w)} \tag{12}$$

The uncertainties $u\left(n_N\right)$, $u\left(n_\mathrm{g}\right)$ and $u\left(n_\mathrm{b}\right)$ are calculated according to the ISO/IEC Guide 98-3, taking into account that the individual counts n_i in a channel i of a multi-channel spectrum are the result of a Poisson process and hence $u^2\left(n_i\right)=n_i$ holds. The values of n_N, n_g and n_b and their associated standard uncertainties $u\left(n_N\right)$, $u\left(n_\mathrm{g}\right)$ and $u\left(n_\mathrm{b}\right)$ can be calculated with a computer code. Since there are various methods of subtracting the background below a peak in order to derive the number of counts in the net peak area, no generally applicable formulas can be given. An example of the simple case of linear background subtraction is given in Annex C. For calculating the decision threshold and the detection limit at least $n_N, n_\mathrm{b}, u\left(n_N\right)$ and $u\left(n_\mathrm{b}\right)$ shall be known.

12.3 Decision threshold

The decision threshold, c_A^*, is obtained from formula (12) for $\tilde{c}_A=0$ according to ISO 11929-1 and ISO 11929-3. This yields:

$$c_A^*=k_{1-\alpha}\cdot\tilde{u}\left(0\right)=k_{1-\alpha}\cdot\left(w/t_\mathrm{g}\right)\cdot\sqrt{n_{\mathrm{b},E}+u^2\left(n_{\mathrm{b},E}\right)} \tag{13}$$

$\alpha=0{,}05$ and then, $k_{1-\alpha}=1{,}65$ is often chosen by default.

12.4 Detection limit

The detection limit, $c_A^{\#}$, is calculated using the implicit Formula (14) according to ISO 11929-1 and ISO 11929-3:

$$c_A^{\#} = c_A^{*} + k_{1-\beta} \cdot \tilde{u}\left(c_A^{\#}\right) = c_A^{*} + k_{1-\beta}\sqrt{w^2\left[\left(c_A^{\#} / w + n_{\mathrm{b},E} / t_{\mathrm{g}}\right) / t_{\mathrm{g}} + u^2\left(n_{\mathrm{b},E}\right) / t_{\mathrm{g}}^2\right] + c_A^{\#2} \cdot u_{\mathrm{rel}}^2\left(w\right)} \quad (14)$$

$\beta = 0{,}05$ and then, $k_{1-\beta} = 1{,}65$ is often chosen by default.

The detection limit can be calculated by solving Formula (14) for $c_A^{\#}$ or, more simply, by iteration with a starting approximation $c_A^{\#} = 2 \cdot c_A^{*}$.

When taking $k_{1-\alpha} = k_{1-\beta} = k$, the solution of Formula (14) is given by formula (15):

$$c_A^{\#} = \frac{2 \cdot c_A^{*} + \left(k^2 \cdot w\right) / t_{\mathrm{g}}}{1 - k^2 \cdot u_{rel}^2\left(w\right)} \quad (15)$$

12.5 Limits of the coverage intervals

12.5.1 Limits of the probabilistically symmetric coverage interval

The lower, $c_A^{\triangleleft}$, and upper, $c_A^{\triangleright}$, coverage limits are calculated using Formulae (16) and (17) according to ISO 11929-1 and ISO 11929-3:

$$c_A^{\triangleleft} = c_A - k_p \cdot u(c_A) \text{ ; } p = \omega \cdot (1 - \gamma/2) \quad (16)$$

$$c_A^{\triangleright} = c_A + k_q \cdot u(c_A) \text{; } q = 1 - \omega \cdot \gamma/2 \quad (17)$$

where

$\omega = \Phi[y/u(y)]$, 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 $c_A \geq 4 \cdot u(c_A)$. In this case:

$$c_A^{\triangleleft}, c_A^{\triangleright} = c_A \pm k_{1-\gamma/2} \cdot u(c_A) \quad (18)$$

$\gamma = 0{,}05$ and then, $k_{1-\gamma/2} = 1{,}96$ is often chosen by default.

12.5.2 The shortest coverage interval

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

$$ {c_A}^{<}, {c_A}^{>} = c_A \pm k_p \cdot u(c_A); p = [1 + \omega \cdot (1 - \gamma)]/2 \quad (19) $$

or if ${C_A}^{<} < 0$, were the result by:

$$ {c_A}^{<} = 0; {c_A}^{>} = c_A \pm k_q \cdot u(c_A); q = 1 - \omega \cdot \gamma \quad (20) $$

$\omega = \Phi[y/u(y)]$, Φ being the distribution function of the standardized normal distribution;

The relations $0 \le {c_A}^{<} < {c_A}^{>}$ apply and the approximation of Formula (18) is valid.

12.6 Corrections for contributions from other radionuclides and background

12.6.1 General

In gamma-ray spectrometry, two types of contribution frequently have to be corrected for.

a) The gamma line of the radionuclide to be determined contains contributions from gamma radiation of another radionuclide in the sample. The contributing radionuclide has another gamma line from which the contribution to the line in question can be estimated taking into account the emission probabilities of the gamma lines.

b) The gamma line of the radionuclide to be determined occurs also in the background of the spectrometer. By measuring a background spectrum with a blank sample for a counting time t_0, this contribution can be corrected for, taking into account the different counting times of the two spectra.

For both cases, the specific activity can be calculated using a model according to Formula (21):

$$ c_{A,\mathrm{c}} = (n_{N,E}/t_{\mathrm{g}} - x \cdot n_{N0,E}/t_0) \cdot w \quad (21) $$

where x is a factor whose expression depends on the type of correction. For both contribution cases, this model gives the necessary correction.

12.6.2 Contribution from other radionuclides

The gamma line to be corrected at the energy E_1 has the net peak area $n_{N,E1.}$ The contribution of the radionuclide is calculated using the ratio of the contributing radionuclide for gamma energy E_1. Formula (21) gives the necessary correction with $x = P_{E_1} \cdot \varepsilon_1 / P_{E_2} \cdot \varepsilon_2$ and $t_0 = t_\mathrm{g}$. This yields

$$c_{A,\mathrm{c}} = (n_{N,E_1} - x \cdot n_{N,E_2}) \cdot w / t_\mathrm{g} \tag{22}$$

Neglecting the standard uncertainty of x, the standard uncertainty of $c_{A,\mathrm{c}}$ is calculated by:

$$u^2(c_{A,\mathrm{c}}) = (w/t_\mathrm{g})^2 \left\{ n_{\mathrm{g},E_1} + u^2(n_{\mathrm{b},E_1}) + x^2 \left[n_{\mathrm{g},E_2} + u^2(n_{\mathrm{b},E_2}) \right] \right\} + c_{A,\mathrm{c}}^2 \cdot u_\mathrm{rel}^2(w) \tag{23}$$

and with a true value $\tilde{c}_{A,\mathrm{c}}$ instead of $c_{A,\mathrm{c}}$:

$$\tilde{u}_c{}^2(\tilde{c}_{A,\mathrm{c}}) = (w/t_\mathrm{g})^2 \left\{ \tilde{c}_{A,\mathrm{c}}\, t_\mathrm{g} / w + n_{\mathrm{b},E_1} + u^2(n_{\mathrm{b},E_1}) + x\,(n_{\mathrm{g},E_2} - n_{\mathrm{b},E_2}) + x^2 \left[n_{\mathrm{g},E_2} + u^2(n_{\mathrm{b},E_2}) \right] \right\} + \ldots$$

$$\ldots + \tilde{c}_{A,\mathrm{c}}{}^2 \cdot u_\mathrm{rel}^2(w) \tag{24}$$

Then, the decision threshold $c_{A,\mathrm{c}}^*$ is given by:

$$c_{A,\mathrm{c}}^* = k_{1-\alpha} \cdot (w/t_\mathrm{g}) \cdot \sqrt{n_{\mathrm{b},E_1} + u^2(n_{\mathrm{b},E_1}) + x(n_{\mathrm{g},E_2} - n_{\mathrm{b},E_2}) + x^2 \left[n_{\mathrm{g},E_2} + u^2(n_{\mathrm{b},E_2}) \right]} \tag{25}$$

and the detection limit , $c_{A,\mathrm{c}}^\#$, by:

$$c_{A,\mathrm{c}}^\# = c_{A,\mathrm{c}}^* + k_{1-\beta} \times \ldots$$

$$\ldots \times \sqrt{\left(w / t_\mathrm{g}\right)^2 \left\{ c_{A,\mathrm{c}}^\# t_\mathrm{g} / w + n_{\mathrm{b},E_1} + u^2\left(n_{\mathrm{b},E_1}\right) + x\left(n_{\mathrm{g},E_2} - n_{\mathrm{b},E_2}\right) + x^2 \left[n_{\mathrm{g},E_2} + u^2\left(n_{\mathrm{b},E_2}\right) \right] \right\} + c_{A,\mathrm{c}}^{\#2} u_\mathrm{rel}^2\left(w\right)}$$

(26)

The detection limit can be calculated by solving Formula (24) for $c_{A,\mathrm{c}}^\#$ or, more simply, by iteration with a starting approximation $c_{A,\mathrm{c}}^\# = 2 \cdot c_{A,\mathrm{c}}^*$.

When taking $k_{1-\alpha} = k_{1-\beta} = k$ the solution of Formula (26) is given by Formula (27):

$$c_{A,\mathrm{c}}^\# = \frac{2 \cdot c_{A,\mathrm{c}}^* + \left(k^2 \cdot w\right) / t_\mathrm{g}}{1 - k^2 \cdot u_\mathrm{rel}^2(w)} \tag{27}$$

12.6.3 Contribution from background

In this case, Formula (21) is used for the correction with $x=1$ and $u(x)=0$. $n_{N0,E}$ is the net peak area of the gamma line in the background spectrum and t_0 the counting time of the background spectrum. This yields

$$c_{A,\mathrm{c}}=(n_{N,E}/t_\mathrm{g}-n_{N0,E}/t_0)\cdot w \tag{28}$$

The standard uncertainty of $c_{A,\mathrm{c}}$ is calculated by:

$$u^2(c_{A,\mathrm{c}})=w^2(n_{\mathrm{g},E}/t_\mathrm{g}^2+n_{\mathrm{g}0,E}/t_0^2+u^2(n_{\mathrm{b},E})/t_\mathrm{g}^2+u^2(n_{\mathrm{b}0,E})/t_0^2)+c_{A,\mathrm{c}}^2\cdot u_\mathrm{rel}^2(w) \tag{29}$$

and with a true value $\tilde{c}_{A,c}$ of $c_{A,c}$

$$\begin{aligned}\tilde{u}_c^2\left(\tilde{c}_{A,\mathrm{c}}\right)=w^2\left\{\tilde{c}_{A,\mathrm{c}}/t_\mathrm{g}w+\left[n_{\mathrm{b},E}+u^2\left(n_{\mathrm{b},E}\right)\right]/t_\mathrm{g}^2+\left[n_{\mathrm{g}0,E}+u^2\left(n_{\mathrm{b}0,E}\right)\right]/t_0^2+\left(n_{\mathrm{g}0,E}-n_{\mathrm{b}0,E}\right)/t_0t_\mathrm{g}\right\}+\ldots\\ \ldots+\tilde{c}_{A,\mathrm{c}}^2u_\mathrm{rel}^2(w)\end{aligned} \tag{30}$$

Then, the decision threshold $c_{A,\mathrm{c}}^*$ is given by:

$$c_{A,\mathrm{c}}^*=k_{1-\alpha}\cdot w\cdot\sqrt{[n_{\mathrm{b},E}+u^2(n_{\mathrm{b},E})]/t_\mathrm{g}^2+[n_{\mathrm{g}0,E}+u^2(n_{\mathrm{b}0,E})]/t_0^2+(n_{\mathrm{g}0,E}-n_{\mathrm{b}0,E})/t_0t_\mathrm{g}} \tag{31}$$

and the detection limit $c_{A,\mathrm{c}}^\#$ by:

$$c_{A,\mathrm{c}}^\#=c_{A,\mathrm{c}}^*+k_{1-\beta}\times\ldots$$

$$\ldots\times\sqrt{w^2\left\{\frac{c_{A,\mathrm{c}}^\#}{t_\mathrm{g}w}+\left[n_{\mathrm{b},E}+u^2\left(n_{\mathrm{b},E}\right)\right]/t_\mathrm{g}^2+\left[n_{\mathrm{g}0,E}+u^2\left(n_{\mathrm{b}0,E}\right)\right]/t_0^2+\left(n_{\mathrm{g}0,E}-n_{\mathrm{b}0,E}\right)/t_0t_\mathrm{g}\right\}+c_{A,\mathrm{c}}^{\#2}u_\mathrm{rel}^2(w)}$$

(32)

The detection limit can be calculated by solving Formula (32) for $c_{A,\mathrm{c}}^\#$ or, more simply, by iteration with a starting approximation $c_{A,\mathrm{c}}^\#=2\cdot c_{A,\mathrm{c}}^*$.

When taking $k_{1-\alpha}=k_{1-\beta}=k$, the solution of Formula (32) is given by Formula (33):

$$c_{A,\mathrm{c}}^\#=\frac{2\cdot c_{A,\mathrm{c}}^*+\left(k^2\cdot w\right)/t_\mathrm{g}}{1-k^2\cdot u_\mathrm{rel}^2(w)} \tag{33}$$

The limits of the coverage intervals are calculated according to Formulae (16) and (17) or (19) and (20).

13 Test report

The test report shall conform to ISO/IEC 17025 requirements. It shall contain the following information:

a) reference to this document i.e. ISO 10703:2021;

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 $\leq c_A^*$,

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

Complementary information can be provided such as:

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

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 likely to affect the results;

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 this document. They have the consequence that it is decided too frequently that the physical effect is absent when in fact it is not absent.