
**Water quality — Determination of the
activity concentration of radionuclides —
Method by high resolution gamma-ray
spectrometry**

*Qualité de l'eau — Détermination de l'activité volumique des
radionucléides — Méthode par spectrométrie gamma à haute résolution*

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 10703 was prepared by Technical Committee ISO/TC 147, *Water quality*.

This second edition cancels and replaces the first edition (ISO 10703:1997), which has been technically revised.

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Introduction

This International Standard allows (after proper sampling, sample handling and, when necessary or desirable, sample preparation) the simultaneous determination of the activity concentration of several gamma-ray emitting radionuclides in water samples by gamma-ray spectrometry using high purity germanium [HPGe] detectors. Gamma-ray emitting radionuclides are widespread both as naturally occurring and as man-made radionuclides. Therefore, environmental samples usually contain a multitude of different gamma-ray emitters and high resolution gamma-ray spectrometry provides a useful analytical tool for environmental measurements.

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Water quality — Determination of the activity concentration of radionuclides — Method by high resolution gamma-ray spectrometry

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard 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 ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method for the simultaneous determination of the activity concentration of various radionuclides emitting gamma rays with energies $40 \text{ keV} < E < 2 \text{ MeV}$ in water samples, by gamma-ray spectrometry using germanium detectors with high energy resolution in combination with a multichannel analyser.

NOTE 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 International Standard, provided both the calibration of the measuring system and the shielding are adapted to this purpose.

This International Standard includes the procedures for energy calibration, determination of the energy dependent sensitivity of the measuring system, the analysis of the spectra and the determination of the activity concentration of the various radionuclides in the sample studied. It is only applicable to homogeneous samples. Samples with activities typically between 1 Bq and 10^4 Bq can be measured as such, i.e. without dilution or concentration of the sample or special (electronic) devices.

Depending on different factors, such as the energy of the gamma rays and 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 should be concentrated by evaporation when activities below about 1 Bq have to be measured. Also, when the activity is considerably higher than 10^4 Bq, the sample should be either diluted or an aliquot of the sample should be taken or the source to detector distance should be increased, or a correction for pile-up effects should be applied.

2 Normative references

The following referenced documents are indispensable for the application 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 31-9, *Quantities and units — Part 9: Atomic and nuclear physics*

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 10703:2007(E)

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

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

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

Guide to the expression of uncertainty in measurement (GUM), BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML

IEC 60973, *Test procedures for germanium gamma-ray detectors*

IEC 61151, *Nuclear instrumentation — Amplifiers and preamplifiers used with detectors of ionizing radiation — Test procedures*

IEC 61452, *Nuclear instrumentation — Measurement of gamma-ray emission rates of radionuclides — Calibration and use of germanium spectrometers*

3 Terms and definitions

For the purposes of this document, the definitions, symbols and abbreviations given in ISO 31-9 and the following apply.

3.1 blank sample

container of an identical composition to the one used for the water test sample filled with radon free demineralized water

3.2 dead time

time interval which must elapse between the occurrence of two consecutive pulses or ionising events for them to be recognized by the detection system as separate pulses or events

3.3 dead time correction

correction to be applied to the observed number of pulses in order to take into account the number of pulses lost during the dead time

3.4 decay constant

λ
(radionuclide in a particular energy state) quotient of dP by dt , where dP is the probability of a given nucleus undergoing a spontaneous nuclear transition from that energy state in the time interval dt

$$\lambda = \frac{dP}{dt} = - \frac{1}{N} \frac{dN}{dt}$$

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

3.5 efficiency

under stated conditions of detection, the ratio of the number of detected gamma-photons to the number of gamma-photons of the same type emitted by the radiation source in the same time interval

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

3.7**full energy peak**

peak of spectral response curve corresponding to the total absorption of the photon energy in the sensitive detector volume 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 within the resolution time, 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

3.11**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 As a result, they 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**

fraction of the nuclei which disintegrates in a specific way

4 Symbols and units

V	Volume of the water sample for test, in litres
A	Activity of each radionuclide in calibration source, at the calibration time, in becquerels
$c_A, c_{A,C}$	Activity concentration ¹⁾ of each radionuclide, without and with corrections, expressed in becquerels per litre
t_g	Sample spectrum counting time, in seconds
t_0	Background spectrum counting time, in seconds
t_s	Calibration spectrum counting time, in seconds
$n_{N,E}, n_{N0,E}, n_{Ns,E}$	Number of counts in the net area of the peak, at energy E , in the 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 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 sample spectrum in the background spectrum and in the calibration spectrum, respectively
ε_E	Efficiency of the detector at energy E at actual measurement geometry
P_E	Probability of the emission of a gamma ray with energy E of each radionuclide, per decay
λ	Decay constant of each radionuclide, in reciprocal seconds
$u(c_A), u(c_{A,C})$	Standard uncertainty associated with the measurement result, without and with corrections, in becquerels per litre
U	Expanded uncertainty calculated by $U = k \cdot u(c_A)$ with $k = 1, 2, \dots$, in becquerels per litre
$c_A^*, c_{A,C}^*$	Decision threshold, without and with corrections, in becquerels per litre
$c_A^\#, c_{A,C}^\#$	Detection limit, without and with corrections, in becquerels per litre
$c_A^\triangleleft, c_A^\triangleright$	Lower and upper limits of the confidence interval, in becquerels per litre

1) "Volumic activity" is an alternative name for "Activity concentration".

5 Principle

Gamma rays cause 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 the previously obtained energy detector calibration. The concentration of the radionuclides present in the sample is calculated using the previously obtained energy-dependent detector efficiency.

6 Reference sources

All certified reference sources shall be traceable to a national or international standard.

6.1 Reference source(s) for energy calibration

One or more reference sources emitting gamma rays with accurately known energies covering the entire energy range to be studied shall be used.

It is recommended that photon-emitting sources be used which cover the energy region of interest. Choose the source so that at least nine full energy peaks uniformly divided throughout the energy range of interest are available; sources containing long-lived radionuclides (europium-152, americium-241, cobalt-60, caesium-137) are recommended for this purpose. For a periodical control on the energy calibration, a smaller number of energy peaks can be used.

6.2 Reference source(s) for efficiency calibration

One or more reference sources, traceable to national or international standards, for which the uncertainty of the activity is stated shall be used. Multi-radionuclide sources may also be used. The energies of the emitted gamma rays shall be distributed over the entire energy range to be analysed, in such a way that the energy-dependent efficiency of the measuring apparatus can be determined in a sufficiently accurate way. For most purposes, the accuracy is sufficient if the difference in counting efficiency between two subsequent energies is smaller than 10 % of the counting efficiency at 120 keV, if the required radionuclides are available. For determining the activity of radionuclide-emitting gamma rays in the energy region $40 \text{ keV} < E < 100 \text{ keV}$, the counting efficiency for these gamma rays should be determined by calibration with this particular radionuclide.

NOTE For the energy range $100 \text{ keV} < E < 2\,000 \text{ keV}$, the following radionuclides can be used: manganese-54, cobalt-57, zinc-65, strontium-85, yttrium-88, cadmium-109, tin-113, caesium-137, cerium-139. Radionuclides with cascade transitions (e.g. cobalt-60 and caesium-134) are applied with caution. As mercury is volatile it cannot be incorporated in solid sources prepared by evaporation.

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 shall be used for all applications.

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

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