BS ISO 9697:2015

BSI Standards Publication

Water quality — Gross beta activity in non-saline water — Test method using thick source

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National foreword

This British Standard is the UK implementation of ISO 9697:2015. It supersedes BS ISO 9697:2008 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/3/8, Radioactivity measurements methods.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Water quality — Gross beta activity in non-saline water — Test method using thick source

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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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

This third edition cancels and replaces the second edition (ISO 9697:2008), which has been technically revised.

Introduction

Radioactivity from several naturally occurring and anthropogenic sources is present throughout the environment. Thus, water bodies (e.g. surface water, groundwater, seawater) can contain the following radionuclides of natural or human-made origins:

- natural radionuclides, including potassium-40 and those originating from the thorium and uranium decay series, particularly radium-226, radium-228, uranium-234, uranium-238, and lead-210, can be found in water for natural reasons (e.g. desorption from the soil and wash-off 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 the production and use of phosphate fertilizer);
- human-made radionuclides, such as transuranium elements (americium, plutonium, neptunium, curium), tritium, carbon-14, strontium-90, and some gamma-emitting radionuclides, can also be found in natural waters as a result of authorized routine releases into the environment in small quantities of the effluent discharged from nuclear fuel cycle facilities. They are also released into the environment following their use in unsealed form for medical and industrial applications. They are also found in the water as a result of past fallout contamination resulting from the explosion in the atmosphere of nuclear devices and accidents such as those that occurred in Chernobyl and Fukushima.

Drinking water can thus contain radionuclides at activity concentrations which could present a risk to human health. In order to assess the quality of drinking water (including mineral waters and spring waters), with respect to its radionuclide content and to provide guidance on reducing health risks by taking measures to decrease radionuclide activity concentrations, water resources (groundwater, river, lake, sea, etc.) and drinking water are monitored for their radioactivity content as recommended by the World Health Organization (WHO) and can be required by some national authorities.

An International Standard on a test method for gross beta activity in water samples is justified for test laboratories carrying out these measurements, required sometimes by national authorities, as laboratories might have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

The screening level for gross beta activity in drinking water, as recommended by WHO, is 1 Bq l^{-1}.[1] If this value is not exceeded, an effective dose of 0,1 mSv $year^{-1}$ should not be exceeded. In case that gross beta screening level is exceeded, it is recommended that the specific radionuclides should be identified and their individual activity concentrations measured. Gross beta measurements based on the evaporation method include a contribution from potassium-40, a naturally occurring beta emitter in a fixed ratio to stable potassium. If the screening level of 1 Bq l^{-1} for gross beta is exceeded, a separate determination of total potassium in water should be performed to subtract the contribution of potassium-40 to beta activity. The factor of 27,6 Bq g^{-1} of beta activity to total potassium should be used to calculate the potassium-40 contribution.[1]

NOTE The screening level is determined based on the activity concentration with an intake of 2 l day^{-1} of drinking water for 1 year that results in an effective dose of less than 0,1 mSv $year^{-1}$ for members of the public, an effective dose that represents a very low level of risk that is not expected to give rise to any detectable adverse health effect.

This International Standard is one of a set of International Standards on test methods dealing with the measurement of gross activity of radionuclides in water samples. Other related standards include

- ISO 9696,
- ISO 10704, and
- ISO 11704.

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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 issues, 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 according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a test method for the determination of gross beta activity concentration in non-saline waters. The method covers non-volatile radionuclides with maximum beta energies of approximately 0,3 MeV or higher. Measurement of low energy beta emitters (e.g. ^{3}H, ^{228}Ra, ^{210}Pb, ^{14}C, ^{35}S, and ^{241}Pu) and some gaseous or volatile radionuclides (e.g. radon and radioiodine) might not be included in the gross beta quantification using the test method described in this International Standard.

This test method is applicable to the analysis of raw and drinking waters. The range of application depends on the amount of total soluble salts in the water and on the performance characteristics (background count rate and counting efficiency) of the counter used.

It is the laboratory's responsibility to ensure the suitability of this method for the water samples tested.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 5667-14, *Water quality — Sampling — Part 14: Guidance on quality assurance and quality control of environmental water sampling and handling*

ISO 11929, *Determination of the characteristic limits (decision threshold, detection limit and limits of the confidence interval) for measurements of ionizing radiation — Fundamentals and application*

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

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

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

3 Symbols, definitions, and units

For the purposes of this document, the symbols and designations given in ISO 80000-10, ISO 11929, ISO/IEC Guide 98-3, and ISO/IEC Guide 99, and the following apply.

A	beta activity, in becquerels, of the calibration source
c_A	beta activity concentration, in becquerels per litre
c_A^*	decision threshold, in becquerels per litre
$c_A^\#$	detection limit, in becquerels per litre
$c_A^{\triangleleft}, c_A^{\triangleright}$	lower and upper limits of the confidence interval, in becquerels per litre
m	mass, in milligrams, of ignited residue from volume, V
m_r	mass, in milligrams, of the sample residue deposited on the planchet
r_0	background count rate, per second
$r_{0\alpha}$	background count rate, per second, from the alpha window
r_g	sample gross count rate, per second
$r_{\mathrm{g}\alpha}$	sample gross count rate, per second, from the alpha window
r_s	calibration count rate of the beta source, per second
$r_{\mathrm{s}\alpha}$	calibration count rate of the alpha source, per second, from the alpha window
S	surface area, in square millimetres, of the planchet
t_0	background counting time, in seconds
t_g	sample counting time, in seconds
t_s	calibration count time of the beta source, in seconds
$t_{\mathrm{s}\alpha}$	calibration count time of the alpha source, in seconds
$u(c_A)$	standard uncertainty, in becquerels per litre, associated with the measurement result
U	expanded uncertainty, in becquerels per litre, calculated from $U = ku(c_A)$, with $k = 1, 2 \ldots$
V	volume, in litres, of test sample equivalent to the mass of solid on the planchet
V_t	volume, in litres, of the water sample
ε	counting efficiency for the specified radioactive standard
ρ_S	source thickness, in milligrams per square millimetre, of the sample residue deposited on the planchet
χ	alpha-beta cross-talk, percentage of alpha count going into the beta window from the alpha calibration source

4 Principle

Gross beta measurement is not intended to give an absolute determination of the activity concentration of all beta-emitting radionuclides in a test sample, but rather a screening analysis to ensure particular reference levels of specific beta emitters have not been exceeded. This type of determination is also known as gross beta index. Gross beta analysis is not expected to be as accurate nor as precise as specific radionuclide analysis after radiochemical separations.

The sample, taken, handled, and preserved as specified in ISO 5667-1, ISO 5667-3, and ISO 5667-14, is evaporated to almost dryness, converted to the sulfate form, and ignited at 350 °C. A portion of the residue is transferred onto a planchet and the beta activity measured by counting in an appropriate counting assembly, which is calibrated against a suitable beta calibration source, such as potassium-40 (^{40}K) or strontium-90/yttrium-90 ($^{90}Sr + ^{90}Y$) in equilibrium.

If simultaneous gross alpha and beta measurements are required on the same water sample, the procedure specified in this International Standard is common to that of ISO 9696.[2] However, to simultaneously measure gross alpha activity, the counting source thickness should be at least 0,1 mg mm^{-2}.[3],[4]

A performance criteria example is given in Annex A .

5 Reagents and equipment

5.1 Reagents

All reagents shall be of recognized analytical grade and shall not contain any detectable beta activity.

NOTE A method for preparing reagent blanks to check for the absence of any endemic radioactivity or contamination is given in Clause 7.

5.1.1 Water, complying with ISO 3696:1987, grade 3.

5.1.2 Calibration source, the choice of beta calibration source depends on the knowledge of the type of radioactive contaminant likely to be present in the waters being tested. Among calibration source of beta-emitting radionuclides, ^{90}Sr and ^{40}K are commonly used.

NOTE The beta activity of ^{40}K in natural potassium is 27,6 Bq g^{-1}, i.e. 14,4 Bq g^{-1} in potassium chloride.[1]

5.1.3 Nitric acid, $c(HNO_3) = 8$ mol/l^{-1}.

5.1.4 Sulfuric acid, $c(H_2SO_4) = 18$ mol/l^{-1}, $\rho = 1{,}84$ g/ml^{-1}, mass fraction $w(H_2SO_4) = 95$ %.

5.1.5 Volatile organic solvents, methanol or acetone.

5.1.6 Calcium sulfate, $CaSO_4$.

5.1.7 Vinyl acetate, $((C_4H_6O_2)n)$.

CAUTION — As calcium salts can contain trace amounts of ^{226}Ra and/or ^{210}Pb, checks for the presence of these radionuclides shall be made.

5.2 Equipment

Usual laboratory equipment and, in particular, the following:

5.2.1 Beta counter, preferably of the gas-flow proportional type, incorporating a plastic scintillation detector or a silicon-charged particle detector.

When using a gas-flow proportional counter, it is advisable to choose the electronic beta window with minimal beta-alpha cross-talk and correct for the alpha-beta cross-talk using a ^{239}Pu alpha source. If equipment other than gas-flow proportional counters is used, then cross-talk can be insignificant and ignored.

If a windowless gas-flow proportional counter is used, carry out regular checks for possible contamination of the counting system by counting blank samples.

NOTE The particulate nature of the source to be counted can give rise to contamination if operated in a vacuum (as in the case of silicon-charged particle detector) or gas-flow systems (as used in a proportional counter).

5.2.2 Planchet with counting tray, of surface density at least 2,5 mg/mm^{-2} (250 mg/cm^{-2}), having a lipped edge and made of stainless steel.

The diameter of the planchet to be used is determined by the counter requirements, i.e. the detector diameter and source holder dimensions.

NOTE An evenly spread source is required and some analysts find it easier to produce this on a polished metal surface, whereas others prefer to use an etched or roughened planchet (sand blasting and chemical etching has been applied for this purpose).

5.2.3 Muffle furnace, capable of being maintained at (350 ± 10) °C.

6 Procedure

6.1 Sampling

Collection, handling, and storage of water samples shall be performed as specified in ISO 5667-1, ISO 5667-3, and ISO 5667-14.

If the measurement of the activity in the filtered water sample is required, carry out filtration immediately on collection and before acidification.

NOTE Acidification of the water sample minimises the loss of radioactive material from solution by adsorption. If carried out before filtration, acidification desorbs radioactive material initially adsorbed on the particulate material.

6.2 Pretreatment

The determination of the total solids content of the water can be performed to estimate the smallest volume of water needed for the measurement. Making due allowance for changes in composition due to ignition at 350 °C and sulfation of the residue, calculate the volume of sample required to produce a mass per unit area of solid residue slightly in excess of ρ_S (mg/mm^{-2}) given by:

$$\rho_s = \frac{m_r}{S} \geq 0{,}1 \tag{1}$$

Use this as a guide to determine the volume of sample required for the concentration stage below.

6.3 Concentration stage

Transfer to a beaker a measured volume, *V*, in litres, of the sample chosen such that after ignition the value for ρ_S is at least 0,1 mg/mm^{-2}.

With very soft waters, it is possible that the volume required to produce $\rho_S \geq 0{,}1$ mg/mm^{-2} is impractically large. In these circumstances, the largest practicable volume should be used or calcium salts should be added.

Evaporate the sample carefully on a hotplate until the volume is reduced to about 50 ml.

After cooling, transfer the concentrated solution to a weighed-silica or glazed porcelain dish that has been previously ignited at 350 °C. Rinse the beaker carefully with a minimum quantity of water (5.1.1) and transfer the rinses to the dish.

NOTE If the beaker is large, it might be more convenient to transfer the rinses to a smaller beaker. The rinses can then be evaporated to a lower volume to facilitate sample transfer to the silica dish.

6.4 Sulfation stage

After drying and ignition, some water residue can be hygroscopic or difficult to disperse, and thus, unsuitable for the activity measurement. A sulfation process is then a suitable treatment for such water samples.

Ensure that the rinses in the dish are cool and add (1 ± 0,2) ml of sulfuric acid (5.1.4).

The volume of sulfuric acid chosen is sufficient for sulfating about 1,8 g of calcium carbonate. To ensure an excess of acid, the initial volume of sample should be chosen such that the total solids content does not exceed 1 g (experience with some waters can show this step to be unnecessary).

Carefully evaporate the contents of the dish to dryness.

To avoid spitting, heat the dish from above using an infrared lamp until fumes of sulfuric acid are evolved. Then, transfer the dishes to a hotplate until no further fumes are evolved.

6.5 Ignition stage

Transfer the dish and contents to the muffle furnace (5.2.3), ignite for 1 h at a temperature of (350 ± 10) °C and allow to cool to room temperature in a desiccator.

Weigh the dish and the residue and obtain by difference, m, in milligrams, the mass of the ignited residue.

6.6 Source preparation

If the residue is coarse, grind it in a pestle and mortar. Transfer the required mass of the residue onto a planchet (5.2.2). Let this mass be m_r.

If the volume of the sample used (V, in 6.3) has led to a value of ρ_S less than 0,1 mg/mm^{-2}, transfer as much as possible of the residue to the planchet.

Disperse the residue evenly over the planchet by slurrying with a few drops of a volatile organic solvent (5.1.5, methanol or acetone) and allow it to dry to a constant mass. It is also recommended that, to produce evenly spread counting sources, vinyl acetate (5.1.7) can be added to the solvent to aid in binding the source. Make a note of the time and date of source preparation, and check that no residue has been lost.

Although beta-particle counting efficiency is not seriously affected by the source thickness up to 0,2 mg mm^{-2} (see References[4] and [5]), it is recommended that, for the calibration of the detection system, either use standard sources of the same mass loading (m_r) or repeat the procedure with the addition of calcium salts to the water sample to achieve the same mass loading as the calibration standard.

6.7 Measurement

Set up the counter (5.2.1) according to the manufacturer's instructions.

Immediately after drying the source, measure the activity on the planchet by counting for an appropriate duration. Note the time, date, and counting duration.

The counting time depends on the sample and background count rates, and also on the detection limit and decision threshold required.

NOTE Re-counting over a period of one month can reveal in-growth of daughter radionuclides from naturally occurring radioactive isotopes. The interpretation of the counting data can then be complicated. However, at normal environmental levels, it is rare for these phenomena to be apparent, particularly with a long counting time.

6.8 Determination of counting background

Measure the background activity using a clean planchet. Let the measured counting rate be r_0. Repeated counts confirm the stability of the counting background.

6.9 Preparation of calibration sources

Accurately weigh approximately 2,5 g of calcium sulfate into a 150 ml beaker. Carefully add (10 ± 0,1) ml of hot 8 mol l^{-1} nitric acid (5.1.3), stir and add up to 100 ml of hot water (5.1.1).

Add an exactly known amount (about 5 Bq to 10 Bq) of the appropriate standard (5.1.2).

Transfer the solution to 200 ml silica or porcelain dish (that was previously ignited at 350 °C), weighed to ± 0,001 g, and evaporate the solution to dryness using an infrared lamp.

Ignite the dry residue in the muffle furnace (5.2.3) at (350 ± 10) °C for 1 h.

Calculate the activity per unit of mass of the $CaSO_4$ (5.1.6) in becquerels per gram, by dividing the added activity of the chosen standard over the mass of the ignited residue.

Crush (and grind with a pestle and mortar, if necessary) the residue to produce a fine powder in a manner similar to that used for the preparation of the sample.

Prepare planchet calibration sources in the manner as specified for samples.

Measure these calibration sources in the beta counter.

If a gas-flow proportional counter is used, then an alpha calibration source (^{239}Pu) shall be prepared in the same way as for the beta calibration source with a known amount (about 5 Bq to 10 Bq) of the appropriate standard.

If potassium chloride is used as a calibration source, dry the KCl powder to constant mass at 105 °C. The beta activity of the KCl calibration source is given in 5.1.2.

WARNING — In order to determine the alpha rejection rate in the beta window, this procedure requires the preparation of a dry powder spiked with beta- and alpha-emitting radionuclides. Accidental intake of radioactive particulates shall be avoided and the use of a closed cabinet glove box for the sample is recommended for the preparation of the spiked sources. Alternatively, a laboratory fume cupboard can be used, provided that the extract draught is not excessive and liable to create disturbance or cause the suspension of fine powder particles in the air.

6.10 Sensitivity and bias

These are dependent on the amount of inorganic material in the water, the characteristics of the detector and the radionuclides present.

As the result of the measurement is expressed as an equivalent concentration to the selected calibration standard, the standard shall be carefully chosen in order to minimize the bias.

6.11 Optimization of the determination

When using constant source thickness, it is necessary to make the sample and standard source as nearly alike as possible, i.e. in terms of density of material and distribution of radioactivity.

7 Source control

7.1 Contamination check

Check the contamination of reagents by evaporating the volumes of the reagents used in the procedure onto separate planchets. Check the contamination of the whole measurement system by processing (1 000 ± 10) ml of water (5.1.1) acidified with 20 ml of 8 mol l^{-1} nitric acid (5.1.3), to which ($0{,}1\ \rho_S\ S$ ± 1) mg of chromatographic reagent grade silica gel has been added, and measure the activity. This can be compared with the activity of a direct slurry of $0{,}1\ \rho_S\ S$ mg of the silica gel on a planchet. Ensure that the activity is not significantly different from that of the processed silica gel sample.

If the activity is significantly different, select reagents with lower activity, or include a blank determination and correction in the main procedure following the steps required for the sample preparation.

7.2 Potential disequilibrium of radionuclides

Some radionuclides are lost by volatilisation when this method is used. In the uranium decay series, ^{222}Rn is substantially lost during sample processing. When ^{226}Ra is present, ^{222}Rn and its alpha- and beta-emitting daughters subsequently grow in the source. Re-counting over a period of 3 weeks can reveal in-growth of the progenies of ^{226}Ra. Some short-lived radionuclides (e.g. ^{224}Ra), if unsupported by the parent ^{228}Th, could also be missed due to the time delay between the sampling and counting.

8 Expression of results

8.1 Calculation of activity concentration

When a gas-flow proportional counter is used, the sample gross beta activity concentration, c_A, in becquerels per litre, of the water sample is given by Formula (2):

$$c_A = \frac{r_g - r_0 - \chi\left(r_{g\alpha} - r_{0\alpha}\right)}{V\varepsilon} = \left[r_g - r_0 - \chi\left(r_{g\alpha} - r_{0\alpha}\right)\right]w \qquad (2)$$

where

V is the volume, in litres, of test sample equivalent to the mass of solid on the planchet, given by

$$V = \frac{V_t}{m} m_r$$

$$w = 1/(V\varepsilon)$$

The counting efficiency, ε, is given by Formula (3) equivalent to the specific beta calibration source used. Note that different calibration source used for beta efficiency determination will lead to different test results.

$$\varepsilon = \frac{r_s - r_0}{A} \qquad (3)$$

If needed, the alpha-beta cross-talk correction factor, χ, can be calculated using Formula (4):

$$\chi = \frac{r_{\mathrm{s}\alpha \rightarrow \beta}}{r_{\mathrm{s}\alpha}} \qquad (4)$$

where

$r_{\mathrm{s}\alpha \rightarrow \beta}$ is the count rate in the beta window when the alpha calibration source is measured.

If equipment other than a gas-flow proportional counter is used and the alpha-beta cross-talk can be neglected, then $\chi = 0$.

8.2 Standard uncertainty

As specified in ISO/IEC Guide 98-3 (see also ISO 11929), the standard uncertainty of c_A is calculated from Formula (2) by:

$$u(c_A) = \sqrt{w^2 \left[\left(\frac{r_{\mathrm{g}}}{t_{\mathrm{g}}} + \frac{r_0}{t_0} \right) + T(\chi) \right] + c_A^2 \, u_{\mathrm{rel}}^2(w)} \qquad (5)$$

with

$$T(\chi) = (r_{\mathrm{g}\alpha} - r_{0\alpha})^2 u^2(\chi) + \chi^2 \left(\frac{r_{\mathrm{g}\alpha}}{t_{\mathrm{g}}} + \frac{r_{0\alpha}}{t_0} \right) \qquad (6)$$

where the uncertainty of the counting time is neglected and the relative standard uncertainty of w is calculated using Formula (7):

$$u_{\mathrm{rel}}^2(w) = u_{\mathrm{rel}}^2(\varepsilon) + u_{\mathrm{rel}}^2(V) \qquad (7)$$

The relative standard uncertainty of ε is calculated using Formula (8):

$$u_{\mathrm{rel}}^2(\varepsilon) = u_{\mathrm{rel}}^2(r_{\mathrm{s}} - r_0) + u_{\mathrm{rel}}^2(A) = \frac{(r_{\mathrm{s}} / t_{\mathrm{s}} + r_0 / t_0)}{(r_{\mathrm{s}} - r_0)^2} + u_{\mathrm{rel}}^2(A) \qquad (8)$$

and the relative standard uncertainty of χ is calculated using Formula (9):

$$u(\chi) = \sqrt{\frac{\chi(\chi + 1)}{(r_{\mathrm{s}\alpha} \, t_{\mathrm{s}\alpha})}} \qquad (9)$$

For the calculation of the characteristic limits according to ISO 11929, one needs $\tilde{u}(\tilde{c}_A)$ i.e the standard uncertainty of c_A as a function of its true value, calculated using Formula (10):

$$\tilde{u}(\tilde{c}_A) = \sqrt{w^2 \left\{ \frac{[(\tilde{c}_A / w) + \chi(r_{\mathrm{g}\pm} - r_{0\pm}) + r_0]}{t_{\mathrm{g}}} + \frac{r_0}{t_0} + T(\chi) \right\} + \tilde{c}_A^2 \, u_{\mathrm{rel}}^2(w)} \qquad (10)$$

If equipment other than a gas-flow proportional counter is used and the alpha-beta cross-talk can be neglected, then $\chi = 0$.

8.3 Decision threshold

The decision threshold, c_A^*, is obtained from Formula (10) for $\tilde{c}_A = 0$ (see also ISO 11929) giving

$$c_A^* = k_{1-\alpha}\,\tilde{u}(0) = k_{1-\alpha}\,w\sqrt{\frac{[\chi(r_{\mathrm{g}\pm} - r_{0\pm}) + r_0]}{t_\mathrm{g}} + \frac{r_0}{t_0} + T(\chi)} \tag{11}$$

α = 0,05 with $k_{1-\alpha}$ = 1,65 are often chosen by default.

If equipment other than a gas-flow proportional counter is used and the alpha-beta cross-talk can be neglected, then χ = 0.

8.4 Detection limit

The detection limit, $c_A^\#$, is calculated using Formula (12) (see ISO 11929):

$$c_A^\# = c_A^* + k_{1-\beta}\,\tilde{u}(c_A^\#) = c_A^* + k_{1-\beta}\sqrt{w^2\left\{\frac{\left[\left(c_A^\#/w\right) + \chi(r_{\mathrm{g}\alpha} - r_{0\alpha}) + r_0\right]}{t_\mathrm{g}} + \frac{r_0}{t_0} + T\left(\chi\right)\right\} + c_A^{\#2}u_\mathrm{rel}^2(w)} \tag{12}$$

β = 0,05 with $k_{1-\beta}$ = 1,65 are often chosen by default.

The detection limit can be calculated by solving Formula (12) for $c_A^\#$ or, more simply, by iteration with a starting approximation $c_A^\# = 2\,c_A^*$.

When taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta}$ and the solution of Formula (12) is given by Formula (13):

$$c_A^\# = \frac{2c_A^* + \left(k^2\,w\right)/t_\mathrm{g}}{1 - k^2 u_\mathrm{rel}^2\left(w\right)} \tag{13}$$

8.5 Confidence limits

The lower, $c_A^\triangleleft$, and upper, $c_A^\triangleright$, confidence limits are calculated using Formulae (14) and (15) (see ISO 11929):

$$c_A^\triangleleft = c_A - k_\mathrm{p}\,u_\mathrm{c}(c_A) \qquad p = \omega\frac{(1-\gamma)}{2} \tag{14}$$

$$c_A^\triangleright = c_A + k_q \cdot u_\mathrm{c}(c_A) \qquad q = 1 - \frac{\omega\gamma}{2} \tag{15}$$

where ω is given by

$$\omega = \Phi\left[\frac{y}{u(y)}\right]$$

where

Φ is the distribution function of the standardised normal distribution;

$1 - \gamma$ is the probability for the confidence interval of the measurand.

If $c_A \geq 4\, u_\mathrm{c}(c_A)$, ω can be set to unity. In this case

$$c_A^{\triangleleft}, c_A^{\triangleright} = c_A \pm k_{(1-\gamma)/2}\, u_\mathrm{c}(c_A) \tag{16}$$

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

9 Test report

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

a) the test method used, together with a reference to ISO 9697;

b) an identification of the sample;

c) the dates of sample preparation and measurement

d) the units in which the results are expressed;

e) the test result, $c_A \pm u(c_A)$ or $c_A \pm U$, with the associated k value;

f) the radionuclide reference used for beta efficiency calibration. In case of a gas proportional counter, specify the alpha reference used for the alpha-to-beta crosstalk correction factor.

Complementary information can be provided such as:

g) the probabilities α, β, and $(1 - \gamma)$;

h) decision threshold and detection limit;

i) dependent on customer requirements, there are different ways to present the result:

   1) when the activity concentration, c_A, is compared with the decision threshold (see ISO 11929), the result of the measurement should be expressed as $\leq c_A^*$ when the result is below the decision threshold;

   2) when the activity concentration, c_A, is compared with the detection limit, the result of the measurement can be expressed as $\leq c_A^\#$ when the result is below the detection limit. If the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose;

j) details of any operating procedures not specified in this International Standard, or regarded as optional, together with details of any incidents likely to have affected the results.

Annex A
(informative)

Example of performance criteria

The method described in this International Standard is identical to the method as described in NEN 5627:2005. NEN 5627:2005 was validated in 2005 in the Netherlands as part of a large project to validate the radioactivity measurement standards.

The project was commissioned by the Dutch department of Ministry of Housing, Spatial Planning and the Environment to NEN, the Netherlands Standardization Institute.

The validation was carried out by the Center for Advanced Radiation Technology (KVI), the Nuclear Research and Consultancy Group (NRG), and the National Institute for Public Health and the Environment (RIVM), all institutes from the Netherlands.

The results of this validation are listed in Table A.1.

Table A.1 — Performance criteria

Parameter	**Sample**[a]		
	1	2	3
Number of participating laboratories	8	8	7
Number of determinations per laboratory	3	3	3
Number of rejected results	0	0	4
Average determination result (a_m), in Bq/g	10,48	37,48	5,26
True value, in Bq/g	11,97	43,65	4,39
Repeatability standard deviation (s_r), in Bq/g	0,21	0,75	0,12
Repeatability variation coefficient (vc_r), in %	2,0	2,0	2,4
Repeatability limit (r), in Bq/g	0,58	2,10	0,35
Reproducibility standard deviation (s_R), in Bq/g	0,30	1,13	0,82
Reproducibility variation coefficient (vc_R), in %	2,9	3,0	15,7
Reproducibility limit (R), in Bq/g	0,85	3,16	2,31
Systematic deviation (δ)in Bq/g	-1,49	-6,17	0,87
Limit of detection (AG_r), in Bq/g	0,046	0,046	0,046

[a] Sample 1: $CaSO_4$-powder with KCl and ^{90}Sr, with low beta-activity [(12,0 ± 0,3) Bq/g].

Sample 2: $CaSO_4$-powder with KCl and ^{90}Sr, with high beta-activity [(43,7 ± 1,0) Bq/g].

Sample 3: $CaSO_4$-powder with ^{90}Sr, with low beta-activity [(4,4 ± 0,1) Bq/g] and high alpha-activity [^{241}Am; (55,7 ± 1,2) Bq/g].

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