
**Water quality — Measurement of gross
beta activity in non-saline water — Thick
source method**

*Qualité de l'eau — Mesurage de l'activité bêta globale des eaux non
salines — Méthode par source concentrée*



Reference number
ISO 9697:2008(E)

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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 9697 was prepared by Technical Committee ISO/TC 147, *Water quality*.

This second edition cancels and replaces the first edition (ISO 9697:1992), which has been technically revised.

Water quality — Measurement of gross beta activity in non-saline water — Thick source method

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

1 Scope

This International Standard specifies a method for the determination of gross beta activity in non-saline waters. The method covers non-volatile radionuclides with maximum beta energies $> 0,3$ MeV. Measurement of very low energy beta emitters, such as ^3H , ^{14}C , ^{35}S and ^{241}Pu , is not included in this International Standard.

The method is applicable to the analysis of raw and potable waters.

2 Normative references

The following referenced documents are indispensable for the application of this documents. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696:1987, *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: 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 9696, *Water quality — Measurement of gross alpha activity in non-saline water — Thick source method*

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* ¹⁾

1) To be published. (Revision of ISO 31-9:1992)

3 Symbols, definitions, and units

For the purposes of this document, the symbols, definitions, and units given in ISO 80000-10, 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 residue deposited on the planchet sample
r_0	background count rate, in reciprocal seconds
$r_{0\alpha}$	background count rate, in reciprocal seconds, from the alpha window
r_g	sample gross count rate, in reciprocal seconds
$r_{g\alpha}$	gross count rate, in reciprocal seconds, from the alpha calibration source
r_s	calibration count rate, in reciprocal seconds
$r_{s\alpha}$	calibration count rate, in reciprocal seconds, from the alpha calibration source
S	area, in square millimetres, of the planchet
t_0	background counting time, in seconds
t_g	sample counting time, 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 $k = 1, 2 \dots$
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
χ	alpha-beta cross-talk, percentage of alpha count going into the beta window from the alpha calibration source

4 Principle

Gross beta activity determination is not an absolute determination of the radioactivity of all beta-emitting radionuclides in a sample, but a relative determination referred to a specific beta emitter that constitutes the standard calibration source. This type of determination is also known as beta index.

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

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, and sample sources can be measured up to 10 mg/cm².

5 Reagents and equipment

5.1 Reagents

Except for the certified reference solution, all reagents shall be of recognised 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 Certified reference solution. A calibration laboratory establishes traceability of its own measurement standards and measuring instruments to the International System of Units (SI) by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. The link to SI units may be achieved by reference to national measurement standards. National measurement standards may be primary standards, which are primary realisations of the SI units or agreed representations of SI units based on fundamental physical constants, or they may be secondary standards which are standards calibrated by another national metrology institute. When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification.

The choice of beta standards will depend on the knowledge of the type of radioactive contaminant likely to be present in the waters being tested. Among standard solutions of beta-emitting radionuclides, ^{90}Sr and ^{40}K are commonly used.

NOTE The beta activity of ^{40}K in natural potassium is 27,4 Bq/g and hence in potassium chloride, it is 14,4/1 000 Bq/mg.

5.1.3 Nitric acid, $c(\text{HNO}_3) = 8 \text{ mol/l}$.

5.1.4 Sulfuric acid, $c(\text{H}_2\text{SO}_4) = 18 \text{ mol/l}$, $\rho = 1,84 \text{ g/ml}$, mass fraction $w(\text{H}_2\text{SO}_4) = 95 \%$.

5.1.5 Volatile organic solvents: methanol and acetone.

5.1.6 Calcium sulfate, CaSO_4 .

CAUTION — As calcium salts may contain trace amounts of ^{226}Ra and/or ^{210}Pb , checks for the presence of these nuclides 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 [passivated implanted planar silicon (PIPS)].

When using a gas-flow proportional counter, it is advisable to choose the beta window to have the beta-alpha cross-talk close to zero and take into account the alpha-beta cross-talk. If equipment other than gas-flow proportional counters is used, then cross-talk may be 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 a PIPS) or gas-flow systems (as used in a proportional counter).

5.2.2 Planchet, counting tray, of surface density at least 2,5 mg/mm² (250 mg/cm²), 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 workers 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

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

Carry out filtration immediately on collection and before acidification, if any, if the measurement of the activity must be done on the filtered sample of water.

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

6.2 Pretreatment

The determination of the total solids content of the water may be performed to determine 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 given by:

$$\rho_S = \frac{m_r}{S} \geq 0,1 \quad (1)$$

where

m_r is the mass, in milligrams, of the residue deposited on the planchet sample;

S is the area, in square millimetres, of the planchet.

Use this as a guide to the volume of sample required for the concentration stage which follows.

6.3 Concentration stage

Transfer to a beaker a measured volume, V , in litres, to within ± 1 %, of the sample chosen such that after ignition the value for ρ_S will be at least 0,1.

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

Evaporate carefully on a hotplate until the volume is reduced to about 50 ml and allow to cool.

Transfer the concentrated solution to a weighed silica (or glazed porcelain) dish that has been previously ignited at 350 °C. Wash the beaker carefully with a minimum quantity of water (5.1.1) and transfer the washings to the dish.

NOTE If the beaker is large, it can be convenient to transfer the washings to a smaller beaker. The washings can then be reduced by evaporation to a lower bulk to facilitate transfer to the silica dish.

6.4 Sulfation stage

Some water may, on drying and ignition, produce a residue which is unsuitable for the measurement of radioactivity, because it is hygroscopic or difficult to disperse. A sulfation process is then a suitable treatment for such water samples.

Ensure that the washings in the dish are cool and add $(1 \pm 0,2)$ ml of sulfuric acid (5.1.4).

The volume of sulfuric acid chosen is capable of sulfating about 1,8 g of calcium carbonate. To allow for 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 may show this step to be unnecessary).

Carefully evaporate the contents of the dish to dryness.

To avoid spitting, expose the dish to heat from above (from an infrared lamp) and continue heating until fumes of sulfuric acid are evolved. Then transfer the dishes to a hotplate and continue heating 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 residue and obtain by difference the mass, m , in milligrams, of the ignited residue.

6.6 Source preparation

Grind the residue, transfer the required mass of the residue on to a planchet. If the residue is coarse, grind it in a pestle and mortar. Let this mass be m_r .

If the volume, V , used in 6.3 has led to a value of ρ_S less than 0,1, transfer as much as possible of the residue to the planchet.

Disperse the residue evenly over the planchet by slurring with a few drops of a volatile organic solvent (5.1.5) and allow to dry to a constant mass. Make a note of the time and date of source preparation, and check that no residue has been lost.

Though beta-particle counting efficiency is not seriously affected by the source thickness (see Reference [4]), it is recommended that, for the calibration of the detection system, either standard sources of the same mass loading, m_r , be used, or the procedure repeated with the addition of calcium salts to the water sample to achieve the same mass loading as the calibration standard.

It is also recommended to produce evenly spread counting sources. Vinyl acetate can be added to the solvent to aid in binding the source during evaporation.

6.7 Measurement

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

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

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 ingrowth of daughter radionuclides from naturally occurring radioactive isotopes. The interpretation of the counting data can then be complicated, although, at normal environmental levels, it is rare for these phenomena to be apparent, particularly with long counting times.

6.8 Background determination

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

6.9 Preparation of calibration sources

Accurately weigh approximately 2,5 g of calcium sulfate into a 150 ml beaker. Carefully add $(10 \pm 0,1)$ ml of hot nitric acid (5.1.3), stir and add up to 100 ml of hot water (5.1.1).

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

Transfer the solution to 200 ml silica or porcelain dish (that was previously ignited at $350\text{ }^\circ\text{C}$), weighed to $\pm 0,001$ g, and heat the solution to dryness using an infrared lamp.

Ignite the dry residue in the muffle furnace (5.2.3) at $(350 \pm 10)\text{ }^\circ\text{C}$ for 1 h.

Calculate the activity per unit of mass, in becquerels per gram, of the CaSO_4 (5.1.6) using the mass of the ignited residue and the amount of activity added of the chosen standard.

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.

If a gas-flow proportional counter is used, then an alpha calibration source 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.

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 may 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.

Prepare planchet calibration sources in the manner specified for samples.

Measure these calibration sources in the beta detector.

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 will be expressed as a quantity equivalent to the selected calibration standard, the choice of the standard shall be carefully made in order to minimise the bias.

6.11 Optimisation of the determination

When using constant 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 on to separate planchets. Check the contamination of the whole measurement system by processing $(1\,000 \pm 10)$ ml of water (5.1.1) acidified with 20 ml of nitric acid (5.1.3), to which $(0,1\rho_S S \pm 1)$ 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 radioactivity, or include a blank determination in the main procedure following the steps required for the sample preparation.

7.2 Potential losses of radon isotopes

Some radionuclides are lost by volatilisation when this method is used. Of the radionuclides in the uranium series, ^{222}Rn is substantially lost during processing. When ^{226}Ra is present, ^{222}Rn and its alpha- and beta-emitting daughters will subsequently grow in the source. A similar effect occurs with radionuclides from the ^{232}Th series.

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 Equation (2):

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

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 Equation (3):

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

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

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

where $r_{s\alpha \rightarrow \beta}$ 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, then the alpha-beta cross-talk can be neglected and $\chi = 0$.

8.2 Standard uncertainty

As specified in ISO/IEC NP Guide 98-3^[2] (see also ISO 11929^[1]), the standard uncertainty of c_A is calculated from Equation (2) by:

$$u(c_A) = \sqrt{w^2 \left[\left(\frac{r_g}{t_g} + \frac{r_0}{t_0} \right) + T(\chi) \right] + c_A^2 u_{\text{rel}}^2(w)} \quad (5)$$

with

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

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

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

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

$$u_{\text{rel}}^2(\varepsilon) = u_{\text{rel}}^2(r_s - r_0) + u_{\text{rel}}^2(A) = \frac{(r_s/t_s + r_0/t_0)}{(r_s - r_0)^2} + u_{\text{rel}}^2(A) \quad (8)$$

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

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

For the calculation of the characteristic limits according to ISO 11929^[1], one needs $\tilde{u}(\tilde{c}_A)$, i.e the standard uncertainty of c_A as a function of its true value, calculated using Equation (10):

$$\tilde{u}(\tilde{c}_A) = \sqrt{w^2 \left\{ \frac{[(\tilde{c}_A/w) + \chi(r_{g\alpha} - r_{0\alpha}) + r_0]}{t_g} + \frac{r_0}{t_0} + T(\chi) \right\} + \tilde{c}_A^2 u_{\text{rel}}^2(w)} \quad (10)$$

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

8.3 Decision threshold

The decision threshold, c_A^* , is obtained from Equation (10) for $\tilde{c}_A = 0$ (see also ISO 11929^[1]) giving

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

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

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

8.4 Detection limit

The detection limit, $c_A^\#$, is calculated using Equation (12) (see ISO 11929^[1]):

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

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

The detection limit can be calculated by solving Equation (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 Equation (12) is given by Equation (13):

$$c_A^\# = \frac{2 c_A^* + (k^2 w) / t_g}{1 - k^2 u_{\text{rel}}^2(w)} \quad (13)$$

8.5 Confidence limits

The lower, c_A^\triangleleft , and upper, c_A^\triangleright , confidence limits are calculated using Equations (14) and (15) (see ISO 11929^[1]):

$$c_A^\triangleleft = c_A - k_p u_c(c_A) \quad p = \omega \frac{(1-\gamma)}{2} \quad (14)$$

$$c_A^\triangleright = c_A + k_p u_c(c_A) \quad q = (1-\omega) \frac{\gamma}{2} \quad (15)$$

where ω is given by

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

in which F is the distribution function of the standardised normal distribution;

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

If $c_A \geq 4 u_c(c_A)$, ω may be set to unity. In this case:

$$c_A^\triangleleft, c_A^\triangleright = c_A \pm k_{(1-\gamma)/2} u_c(c_A) \quad (16)$$

with $\gamma = 0,05$, $k_{(1-\gamma)/2} = 1,96$ 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:

- reference to this International Standard (ISO 9697);
- identification of the sample;

- c) units in which the results are expressed;
- d) test result, $c_A \pm u(c_A)$ or $c_A \pm U$, with the associated k value.

Complementary information can be provided such as:

- e) probabilities α , β and $(1 - \gamma)$;
- f) decision threshold and detection limit;
- g) 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^[1]), 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;
- h) 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.

Bibliography

- [1] 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* ²⁾
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2) To be published. [Revision of ISO 11929 (all parts)]

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