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

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



Reference number
ISO 9696:2007(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 9696 was prepared by Technical Committee ISO/TC 147, *Water quality*.

This second edition cancels and replaces the first edition (ISO 9696:1992), which has been technically revised.

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

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This 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 determination of gross alpha activity in non-saline waters for alpha-emitting radionuclides which are not volatile at 350 °C. It is possible to determine supported volatile radionuclides measured to an extent determined by half-life, matrix retention (of the volatile species) and the duration of measurement (counting time).

The method is applicable to raw and potable waters.

The range of application depends on the amount of suspended matter in the water and on the performance characteristics (background count rate and counting efficiency) of the counter.

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 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/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

3 Symbols, definitions and units

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

V_t	Volume of the water sample, in litres
V	Volume of test sample, in litres, equivalent to the mass of solid on the planchet
m	Mass, in milligrams, of ignited residue from volume V
m_r	Mass of the residue deposited on the planchet sample, in milligrams
A	Alpha activity of the calibration source, in becquerels
c_A	Alpha activity concentration, in becquerels per litre
t_0	Background counting time, in seconds
t_g	Sample counting time, in seconds
r_0	Background count rate, per second
r_g	Sample gross count rate, per second
r_s	Calibration count rate, per second
ε	Counting efficiency of the specified calibration source
S	Area of the planchet, in square millimetres
$u(c_A)$	Standard uncertainty associated with the measurement result, in becquerels per litre
U	Expanded uncertainty calculated by $U = k \cdot u(a)$ with $k = 1, 2, \dots$, in becquerels per litre
c_A^*	Decision threshold, in becquerels per litre
$c_A^\#$	Detection limit, in becquerels per litre
$c_A^<, c_A^>$	Lower and upper limits of the confidence interval, in becquerels per litre

4 Principle

Gross alpha radioactivity is determined by using proportional counting or solid scintillation counting on water residue deposited on a planchet. Due to the strong absorption of the residue deposit, it is considered that the alpha emission from the surface is proportional to the alpha activity of the deposit.

Gross alpha determination is not an absolute determination of the sample alpha radioactive content, but a relative determination referring to a specific alpha emitter which constitutes the standard calibration source. This type of determination is also known as alpha index.

The sample is acidified to stabilize it, 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 alpha activity measured by counting in an alpha-particle detector or counting system previously calibrated against an alpha-emitting standard and the alpha activity concentration calculated.

5 Reagents and equipment

5.1 Reagents

Except for the certified reference solution, all reagents shall be of recognized analytical grade and shall not contain any measurable alpha activity.

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

5.1.1 Water, distilled or deionized, for all applications.

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 realizations 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 alpha standard will depend on knowledge of the type of radioactive contaminant likely to be present in the waters being tested. In general, this amounts to a choice between naturally occurring and man-made alpha emitters.

Among standard solutions of artificial alpha-emitting radionuclides, ^{241}Am and ^{239}Pu are commonly used. When ^{239}Pu is used, the presence of ^{241}Pu impurity shall be taken into account as it leads to growth of ^{241}Am in prepared standard solutions of sources. When ^{241}Am is used, the potential interferences of its gamma emission, or alpha-beta crosstalk shall be taken into account (see Reference [1]).

NOTE 1 A uranium compound of certified natural or known isotopic composition has one arguable advantage, in that its specific activity can be calculated from established physical constants and isotopic abundance data which are independent of the calibration procedures of a particular organization.

NOTE 2 Furthermore, since the energies of the alpha emissions from uranium isotopes are less than those from the artificial transuranic nuclides, the use of a uranium standard tends to give a high result for transuranics. Some authorities prefer to err on the high side in situations where the true composition is unknown.

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 Vinyl acetate.

5.1.7 Calcium sulfate, CaSO_4 .

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

5.2.1 Usual laboratory equipment.

5.2.2 Alpha counter.

Alpha activity shall be counted using either a silver-activated zinc sulfide scintillation screen, a silicon charged-particle detector [ion-implanted silicon or surface barrier detector (SSB)] or a gas-flow proportional counter (windowless or with a window of $\leq 100 \mu\text{g} \cdot \text{cm}^{-2}$).

If a gas-flow proportional counter is used, it is advisable to choose the alpha window so that the beta-alpha cross-talk is close to zero.

If windowless gas-flow proportional counters are 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 problems if operated in a vacuum (as in the case of an SSB) or gas flow systems (as used in a proportional counter).

5.2.3 Planchets, of thickness at least $2,5 \text{ mg} \cdot \text{mm}^{-2}$ ($250 \text{ mg} \cdot \text{cm}^{-2}$), lipped and of stainless steel; counting tray.

NOTE 1 The diameter of the planchet to be used is determined by the counter requirements, i.e. the detector diameter and source holder dimensions.

NOTE 2 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).

5.2.4 Muffle furnace, capable of maintaining a temperature of $(350 \pm 10) ^\circ\text{C}$.

6 Procedure

6.1 Sampling

Sampling, handling and storage of the water samples shall be done 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 it is desired to measure the activity of the filtered sample of water.

NOTE Acidification of the water sample will minimize the loss of radioactivity material from solution by adsorption. If carried out before filtration, it will desorb radioactive material already 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 $350 ^\circ\text{C}$ and sulfation of the residue, calculate the volume of sample required to produce a mass of solid residue slightly in excess of R such as:

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

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

The analysis should be carried out in an area where radio-tracers are not used.

6.3 Concentration stage

Transfer to a beaker a measured volume, V , in litres, ($\pm 1\%$) of the sample chosen such that after ignition the value for R will be above 0,1.

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

Evaporate carefully on a hot-plate 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 distilled water 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 concentrated to a low bulk to facilitate transfer to the silica dish.

6.4 Sulfation stage

Ensure that the washings in the dish are cool and add 1 ml ($\pm 20\%$) of sulfuric acid (5.1.4).

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. This sulfation process is a suitable treatment for many such samples. The volume of sulfuric acid (5.1.4) chosen is capable of sulfating about 1,8 mg 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 hot-plate and continue heating until no further fumes are evolved.

6.5 Ignition stage

Transfer the dish and contents to the muffle furnace (5.2.4), ignite for 1 h at a temperature of (350 ± 10) °C and allow to cool in a desiccator. Note the time and date of removal from the furnace.

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

6.6 Source preparation

Weigh the ash on a planchet in order to get the value for R above 0,1. If the ash is not fine, grind it in a pestle and mortar.

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

As alpha-particle counting efficiency is seriously affected by the source thickness (see Reference [2]), it follows that either standard sources of the same mass loading, m_r , are required for calibration of the detection system or the procedure is repeated with the addition of calcium salts to the water sample to achieve the same mass loading as the calibration standard.

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.

As alpha-particle counting efficiency is seriously affected by the source thickness, it is essential to produce evenly spread counting sources. Polyvinyl acetate (PVA) (5.1.6) can be added to the solvent [for example, 1 g of PVA dissolved in 100 ml (+5 %) solvent] to aid in binding the source during evaporation.

6.7 Measurement

Set up the counter (5.2.2) according to the manufacturer's instructions.

Immediately after drying the source, start measuring the activity on the planchet (5.2.3) 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 the detection limit and decision threshold required.

NOTE Recounting over a period of one month can reveal ingrowth of daughter radionuclides from radium isotopes. The interpretation of such data can be complicated by the presence of uranium and/or thorium series radionuclides. At normal environmental levels, it is rare for these phenomena to be apparent, particularly when the counting time is long. However, over a period of one month, sources from raw waters which are naturally high in ^{226}Ra can show an ingrowth of up to four times the initial count.

6.8 Background determination

Measure the background activity using a clean planchet (5.2.3). Repeated counts will confirm the stability of background.

6.9 Preparation of the calibration source

Accurately weigh approximately 2,5 g of calcium sulfate (5.1.7) into a 150 ml beaker. Carefully add 10 ml ($\pm 1\%$) 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 °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 (5.2.4) at (350 ± 10) °C for 1 h.

Calculate the activity per unit of mass of the CaSO_4 , in $\text{Bq}\cdot\text{g}^{-1}$, using the mass of the ignited residue and the amount of activity added of the chosen standard (^{241}Am , ^{239}Pu or U of natural isotopic composition).

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

WARNING — This procedure requires the preparation of a dry powder spiked with alpha-emitting radionuclides. Accidental intake of radioactive particulates shall be avoided and the use of a sample closed cabinet glove box 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 carry fine powder particles into the air.

Prepare planchet (5.2.3) sources in the manner specified for samples.

Measure these calibration sources in the alpha counter (5.2.2).

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 will be expressed as an equivalent quantity to the selected calibration standard, the choice of the standard shall be carefully made in order to minimize the bias. Counting efficiency data obtained with a silver-activated zinc sulfide scintillation screen detector for several radionuclides are reported as an example in Table 1.

Table 1 – Example of counting efficiency data

Radionuclide		Maximum energy per disintegration MeV	Counting efficiency ^{a, b} %
²⁵² Cf		6,1	13,0
²⁴¹ Am		5,5	11,0
²³⁹ Pu		5,1	7,1
U (natural isotopic composition)	²³⁸ U	4,2	6,4
	²³⁴ U	4,8	
	²³⁵ U	4,4	
^a Counting efficiency varies between radionuclides present in the residue, sample preparations and the detectors used.			
^b The sensitivity and bias obtained when using different radioactive standards is indicated in this table with counting efficiency for a constant size source increasing with alpha-particle energy.			

6.11 Optimization of the determination

As alpha particles are absorbed by matter, it is necessary to optimize the thickness of the source; in this method, the thickness should be $> 0,1 \text{ mg} \cdot \text{mm}^{-2}$ ($10 \text{ mg} \cdot \text{cm}^{-2}$).

Even 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 Contamination check

7.1 General

Check the contamination of reagents by evaporating the volumes of the reagents used in the procedures onto separate planchets (5.2.3). Check the contamination of the whole system by processing $1 \text{ l} \pm 10 \text{ ml}$ of distilled water (5.1.1) acidified with 20 ml of nitric acid (5.1.3), to which $0,1 \text{ RS} \pm 1 \text{ mg}$ 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 $0,1 \text{ RS} \pm 1 \text{ mg}$ of the silica gel on a planchet (5.2.3). 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 using the procedure for checking the contamination of the whole system given in the first paragraph of this clause.

7.2 Radon isotopes losses

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

7.3 Polonium losses

Some alpha-emitting polonium isotopes which occur naturally as members of the uranium and thorium decay series may comprise a significant proportion of the gross alpha activity of some waters. The element and some of its compounds, particularly the halides which sublime at relatively low temperatures, may be volatilized (for example, see References [3] and [4]). Nitrates and sulfates are stable up to temperatures of at least 400 °C to 500 °C, and losses of polonium are therefore not expected for samples which have been acidified with nitric acid and subjected to sulfation.

8 Expression of results

8.1 Calculation of activity concentration

The sample activity concentration c_A , in becquerels per litre of the water sample can be obtained using Equation (2):

$$c_A = (r_g - r_0) / V \cdot \varepsilon = (r_g - r_0) \cdot w \quad \text{with} \quad w = 1 / V \cdot \varepsilon \quad (2)$$

The counting efficiency is calculated using Equation (3):

$$\varepsilon = \frac{r_s - r_0}{A} \cdot 1000 \quad (3)$$

and Equation (4):

$$V = \frac{V_t}{m} \cdot m_r \quad (4)$$

8.2 Standard uncertainty

As specified in the GUM (see also ISO 11929), the combined uncertainty of c_A is calculated, from Equation (1), by:

$$u(c_A) = \sqrt{w^2 \cdot [u^2(r_g) + u^2(r_0)] + c_A^2 \cdot u_{\text{rel}}^2(w)} = \sqrt{w^2 \cdot (r_g/t_g + r_0/t_0) + c_A^2 \cdot u_{\text{rel}}^2(w)} \quad (5)$$

Where the standard uncertainty of the counting time is neglected and the relative standard uncertainty of w is calculated using Equation (6):

$$u_{\text{rel}}^2(w) = u_{\text{rel}}^2(\varepsilon) + u_{\text{rel}}^2(V) \quad (6)$$

the relative standard uncertainty of ε is calculated using Equation (7):

$$u_{\text{rel}}^2(\varepsilon) = u_{\text{rel}}^2(r_s - r_0) + u_{\text{rel}}^2(A) = (r_s/t_s + r_0/t_0) / (A \cdot \varepsilon)^2 + u_{\text{rel}}^2(A) \quad (7)$$

where $u_{\text{rel}}^2(A)$ includes all the uncertainties related to the calibration source activity: certificate of calibration, preparation of the standard solution and addition of the standard to the calibration source.

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

$$u_{\text{rel}}^2(V) = u_{\text{rel}}^2(V_t) + u_{\text{rel}}^2(A) + u_{\text{rel}}^2(m) \quad (8)$$

For the calculation of the characteristic limits (see ISO 11929), one needs $\tilde{u}(\tilde{c}_A)$, i.e. the combined standard uncertainty of c_A as a function of its true value, \tilde{c}_A , calculated using Equation (9):

$$\tilde{u}(\tilde{c}_A) = \sqrt{w^2 \cdot [(\tilde{c}_A / w + r_0) / t_g + r_0 / t_0] + \tilde{c}_A^2 \cdot u_{\text{rel}}^2(w)} \quad (9)$$

8.3 Decision threshold

The decision threshold, c_A^* , is obtained from Equation (9) for $\tilde{c}_A = 0$ (see ISO 11929). This yields:

$$c_A^* = k_{1-\alpha} \cdot \tilde{u}(0) = k_{1-\alpha} \cdot w \cdot \sqrt{r_0 / t_g + r_0 / t_0} \quad (10)$$

c_A^* is expressed in becquerels per litre.

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

8.4 Detection limit

The detection limit, $c_A^\#$, is calculated using Equation (11) (see ISO 11929):

$$c_A^\# = c_A^* + k_{1-\beta} \cdot \tilde{u}_c(c_A^*) = c_A^* + k_{1-\beta} \cdot \sqrt{w^2 \cdot [(c_A^* / w + r_0) / t_g + r_0 / t_0] + c_A^{*\#2} \cdot u_{\text{rel}}^2(w)} \quad (11)$$

$c_A^\#$ is expressed in becquerels per litre.

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

The detection limit can be calculated by solving Equation (11) 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 Equation (11) is given by Equation (12):

$$c_A^\# = \frac{2 \cdot c_A^* + (k^2 \cdot w) / t_g}{1 - k^2 \cdot u_{\text{rel}}^2(w)} \quad (12)$$

8.5 Confidence-interval limits

The lower, c_A^\triangleleft , and upper, c_A^\triangleright , confidence-interval limits are calculated using Equations (13) and (14) (see ISO 11929):

$$c_A^\triangleleft = c_A - k_p \cdot u(c_A) ; p = \omega \cdot (1 - \gamma / 2) \quad (13)$$

$$c_A^\triangleright = c_A + k_q \cdot u(c_A) ; q = 1 - \omega \cdot \gamma / 2 \quad (14)$$

where

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

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

$\omega = 1$ may be set if $c_A \geq 4 \cdot u(c_A)$. In this case:

$$c_A^{\triangleleft}, c_A^{\triangleleft} = c_A \pm k_{1-\gamma/2} \cdot u(c_A) \quad (15)$$

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

9 Test report

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

- a) reference to this International Standard (ISO 9696:2007);
- b) 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 the detection limit;
- g) depending on the customer request, 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;
- h) mention of any relevant information likely to affect the results.

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1) In preparation. [Revision of ISO 11929:2005 (all parts)]

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