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**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*

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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.

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# 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
$\varepsilon$	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,  $^{241}\text{Am}$  and  $^{239}\text{Pu}$  are commonly used. When  $^{239}\text{Pu}$  is used, the presence of  $^{241}\text{Pu}$  impurity shall be taken into account as it leads to growth of  $^{241}\text{Am}$  in prepared standard solutions of sources. When  $^{241}\text{Am}$  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**,  $\text{CaSO}_4$ .

As calcium salts may contain trace amounts of  $^{226}\text{Ra}$  and/or  $^{210}\text{Pb}$ , 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 $(350 \pm 10) ^\circ\text{C}$ .

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## 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  $(350 \pm 10)$  °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.