
**Water quality — Measurement of gross
alpha and gross beta activity in non-
saline water — Thin source deposit
method**

*Qualité de l'eau — Mesurage des activités alpha globale et bêta globale
des eaux non salines — Méthode par dépôt d'une source fine*

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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 10704 was prepared by Technical Committee ISO/TC 147, *Water quality*.

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Water quality — Measurement of gross alpha and gross beta activity in non-saline water — Thin source deposit 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 alpha and gross beta activity in non-saline waters for alpha- and beta-emitting radionuclides.

The method is applicable to raw and potable waters containing a small quantity of dissolved matter. It can, after adaptation, apply to other kind of waters.

The range of application depends upon the amount of dissolved material in the water and on the performance characteristics of the measurement equipment (background count rate and counting efficiency).

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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 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: Guidance on the preservation and handling of water samples*

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*

ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Symbols, definitions and units

For the purposes of this document, the symbols, definitions, and abbreviations given in ISO 80000-10, and the following, apply.

A	activity of the calibration source	Bq
A_a	activity spiked in sample a, prepared for self-absorption estimation purposes	Bq
c_A	activity concentration	Bq l ⁻¹
c_A^*	decision threshold	Bq l ⁻¹
$c_A^\#$	detection limit	Bq l ⁻¹
$c_A^<, c_A^>$	lower and upper limits of the confidence interval	Bq l ⁻¹
$f_{a\alpha}, f_{a\beta}$	self-absorption factor of sample a for α and β , respectively	—
m_d	mass of the deposit	mg
m_p	mass of the planchet	mg
m_{pd}	mass of the planchet and the deposit	mg
m_{pf}	mass of the planchet and the filter	mg
m_{pfd}	mass of the planchet, the filter and the deposit	mg
$r_{0\alpha}, r_{0\beta}$	background count rate from the α and β windows, respectively	s ⁻¹
$r_{a\alpha}, r_{a\beta}$	self-absorption sample a count rate from the α and β windows, respectively	s ⁻¹
$r_{g\alpha}, r_{g\beta}$	sample gross count rate from the α and β windows, respectively	s ⁻¹
$r_{s\alpha}, r_{s\beta}$	calibration count rate from the α and β windows, respectively	s ⁻¹
t_0	background counting time	s
t_g	sample counting time	s
t_s	calibration counting time	s
U	expanded uncertainty calculated by $U = k \cdot u(c_A)$ with $k = 1, 2, \dots$	Bq l ⁻¹
$u(c_A)$	standard uncertainty associated with the measurement result	Bq l ⁻¹
V	volume of test sample	l
$\varepsilon_\alpha, \varepsilon_\beta$	counting efficiency for α and β , respectively	—
$\varepsilon_{a\alpha}, \varepsilon_{a\beta}$	counting efficiency of sample a for α and β , respectively	—
χ	alpha-beta crosstalk	—

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4 Principle

IMPORTANT — Gross alpha and gross beta determinations are not absolute determinations of the sample alpha and beta radioactive contents, but relative determinations referred to specific alpha and beta emitters that constitute the standard calibration sources.

In order to obtain a thin deposit directly on a planchet, the sample can be progressively evaporated to dryness at a temperature below about 85 °C. Alternatively, for alpha determination, it can be concentrated via a coprecipitation, the filtered coprecipitate being measured on to the planchet (Reference [3]). The gross alpha and gross beta activity of the deposit is measured by counting in an alpha- and beta-particle detector or counting system previously calibrated against alpha- and beta-emitting standards.

When suspended matter is present in a significant quantity, a filtration step is required and the gross alpha and gross beta activity can also be determined for the material retained on the filter.

IMPORTANT — Due to the ingrowth of radon daughters over time, the results are dependent on the time elapsed between sample preparation and measurement. For comparison purposes, it is recommended that the measurement be performed at the same time after the preparation of the sample.

5 Chemical reagents and equipment

5.1 Reagents

All reagents shall be of recognised analytical grade and shall not contain any detectable alpha and beta activity, except for radioactive standards solutions.

5.1.1 Standard solutions

5.1.1.1 Alpha standard standards.iteh.ai/catalog/standards/sist/3430d054-7728-4163-90e7-d5acd398bf3f/iso-10704-2009

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

Commonly used standards of artificial alpha-emitting radionuclides employed for this purpose are ^{241}Am solutions and ^{239}Pu solutions. When ^{239}Pu is used, the presence of ^{241}Pu as an 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, take into account the potential interferences of its gamma emission.

NOTE An 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 organisation. However, an uranium compound of known isotopic composition is difficult to obtain. 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 transuranic elements.

5.1.1.2 Beta standard

The choice of beta standard depends on knowledge of the type of radioactive contaminant likely to be present in the waters being tested.

As a natural material, ^{40}K as potassium chloride, dried to constant mass at 105 °C, can be used. Standard solutions of artificial beta-emitting radionuclides ^{90}Sr / ^{90}Y are commonly used.

5.1.2 Wetting or surfactant agents

5.1.2.1 Vinyl acetate

5.1.3 Volatile organic solvents

5.1.3.1 Ethyl alcohol

5.1.4 Water, complying with the requirements of ISO 3696, grade 3.

5.1.5 Specific reagents for alpha-emitting radionuclides co-precipitation

5.1.5.1 Ammonium hydroxide solution, $c(\text{NH}_4\text{OH}) = 6 \text{ mol/l}$.

5.1.5.2 Nitric acid, concentrated, $c(\text{HNO}_3) = 15,8 \text{ mol/l}$.

5.1.5.3 Sulfuric acid solution, $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/l}$.

5.1.5.4 Iron carrier, solution of 5 mg of iron per millilitre.

5.1.5.5 Barium carrier, solution of 5 mg of barium per millilitre.

5.2 Equipment

5.2.1 Laboratory equipment for direct evaporation

Usual laboratory apparatus to store and prepare the sample as specified in ISO 5667-3.

A hot plate, an automatic evaporator or any other appropriate apparatus.

5.2.2 Special equipment for alpha-emitting radionuclide co-precipitation

5.2.2.1 Hot plate with stirring equipment

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5.2.2.2 Infrared lamp

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5.2.2.3 Vacuum filtration system

5.2.2.4 Filters, of pore size 0,45 μm .

5.2.3 Planchet (counting trays)

The planchet shall be lipped and of stainless steel. The diameter of the planchet is determined taking account of the detector diameter and source holder dimensions of the counter used. In the specific case of co-precipitation, an annular support is used to fix the filter on to a filter holder or on to the planchet.

NOTE As the source, test portion and standard, is spread directly on to the planchet for evaporation, it is easier to produce an even deposit on a roughened metal surface; sand blasting or chemical etching can be applied for this purpose, alternatively, a rippled planchet can be employed.

5.2.4 Measurement equipment: alpha-beta counter

Gross alpha and gross beta activity can be measured using either a silicon surface barrier (SSB) detector or a proportional counter (windowless). Ion-implanted Si detectors and thin ($\leq 100 \mu\text{g cm}^{-2}$) window-proportional counters may also be used. Gross alpha and gross beta activity can also be counted using a silver-activated zinc sulfide scintillation screen and plastic scintillation detector, respectively.

6 Sampling

Sample, handle, and store water samples in accordance with ISO 5667-1 and ISO 5667-3.

The laboratory sample is not usually acidified as the test portion is directly evaporated on the planchet. Acidification minimises the loss of radioactive material from solution by adsorption on the wall of the vial, but is done after filtration, as otherwise it desorbs radioactive material already adsorbed on the particulate material, increases the salt content of the test sample, and thus the thickness of the deposit. If necessary, concentrated nitric acid can be used (it is recommended to avoid hydrochloric acid).

7 Procedure

7.1 Preliminary

Calculate the volume of laboratory sample for gross alpha measurement, i.e. the volume of the test portion, in order to produce a deposit with a surface density lower than 5 mg cm^{-2} on the planchet (References [6] and [7]). For deposits of surface density below 5 mg cm^{-2} , self-absorption phenomena can be neglected for gross beta measurement.

When using the same deposit for the simultaneous gross alpha and gross beta measurement, the planchet surface density limit for alpha activity determinations applies.

7.2 Source preparation

7.2.1 Preparation of planchet

Degrease planchets (5.2.3) using a solvent or a surfactant to ensure that the test portion is well distributed over the entire surface and consequently that there is a deposit of uniform surface density bonded to the planchets. Some suppliers degrease planchets at the end of a cycle of fabrication and deliver, on demand, a certificate of attestation.

Keep planchets that are not to be used immediately in a drier to prevent any modification by ambient atmosphere in the laboratory.

Weigh the planchets before use, and record the mass, m_p . If a co-precipitation method is used, weigh the filter (5.2.2.4) with the planchet before use, and record the mass, m_{pf} .

Avoid reuse of planchets to minimise cross-contamination. If the planchets are reused, their freedom from contamination shall be demonstrated.

7.2.2 Evaporation

Transfer the test portion on to the planchet using automatic or non-automatic equipment with a known uncertainty (pipette, water distribution system) and carefully evaporate to dryness.

The residue deposited should form a thin layer of uniform surface density to limit self-absorption phenomena and to ensure similarity with the calibration source geometry.

After cooling the planchets to ambient temperature, weigh them and record the mass, m_{pd} . The mass deposited, m_d , is given by Equation (1):

$$m_d = m_{pd} - m_p \quad (1)$$

To minimise any loss by spitting, maintain the temperature below about $85 \text{ }^\circ\text{C}$ over the entire planchet surface to avoid any overheated areas.

Before evaporating the test portion to dryness on the planchet, pre-evaporation can be performed with appropriate equipment (5.2.1).