BRITISH STANDARD

BS ISO 10704:2009

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

ICS 13.060.60; 13.280

British Standards

National foreword

This British Standard is the UK implementation of ISO 10704:2009.

The UK participation in its preparation was entrusted to Technical Committee EH/3, Water quality.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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This British Standard
was published under
the authority of the
Standards Policy and
Strategy Committee on 31
January 2010

ISBN 978 0 580 59176 1

Amendments/corrigenda issued since publication

Date	Comments

INTERNATIONAL STANDARD

ISO 10704

First edition
2009-11-15

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

Reference number
ISO 10704:2009(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 10704 was prepared by Technical Committee ISO/TC 147, *Water quality*.

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).

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	$\mathrm{Bq\ l^{-1}}$
c_A^*	decision threshold	$\mathrm{Bq\ l^{-1}}$
$c_A^\#$	detection limit	$\mathrm{Bq\ l^{-1}}$
$c_A^\triangleleft$, $c_A^\triangleright$	lower and upper limits of the confidence interval	$\mathrm{Bq\ l^{-1}}$
$f_{\mathrm{a}\alpha}$, $f_{\mathrm{a}\beta}$	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	$\mathrm{s^{-1}}$
$r_{\mathrm{a}\alpha}$, $r_{\mathrm{a}\beta}$	self-absorption sample a count rate from the α and β windows, respectively	$\mathrm{s^{-1}}$
$r_{\mathrm{g}\alpha}$, $r_{\mathrm{g}\beta}$	sample gross count rate from the α and β windows, respectively	$\mathrm{s^{-1}}$
$r_{\mathrm{s}\alpha}$, $r_{\mathrm{s}\beta}$	calibration count rate from the α and β windows, respectively	$\mathrm{s^{-1}}$
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 $k = 1, 2, \ldots$	$\mathrm{Bq\ l^{-1}}$
$u(c_A)$	standard uncertainty associated with the measurement result	$\mathrm{Bq\ l^{-1}}$
V	volume of test sample	l
ε_α, ε_β	counting efficiency for α and β, respectively	—
$\varepsilon_{\mathrm{a}\alpha}$, $\varepsilon_{\mathrm{a}\beta}$	counting efficiency of sample a for α and β, respectively	—
χ	alpha-beta crosstalk	—

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

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 date 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, $c(NH_4OH)$ = 6 mol/l.

5.1.5.2 Nitric acid, concentrated, $c(HNO_3)$ = 15,8 mol/l.

5.1.5.3 Sulfuric acid solution, $c(H_2SO_4)$ = 1 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

5.2.2.2 Infrared lamp

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 ($\leqslant$ 100 µg 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):

$$m_\mathrm{d} = m_\mathrm{pd} - m_\mathrm{p} \qquad (1)$$

To minimise any loss by spitting, maintain the temperature below about 85 °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).

A homogeneous deposit is best achieved on etched or sandblasted planchets. If the deposit is not homogeneously spread, add a wetting agent or surfactant (5.1.2).

7.2.3 Co-precipitation

The recommended working volume is 500 ml.

If a test portion of lower volume is to be analysed, make up to 500 ml with water.

If a test portion of higher volume is to be analysed, concentrate it by evaporation (5.2.1) to 500 ml.

Adjust the pH of the working volume to 7,0 ± 0,5.

Add 20 ml of sulfuric acid (5.1.5.3) and boil for 5 min on a hot plate while stirring (5.2.2.1).

At a temperature of approximately 50 °C, add 1 ml of the barium carrier solution (5.1.5.5) and stir for 30 min.

The barium sulfate precipitates.

Then add 1 ml of the iron carrier solution (5.1.5.4).

Adjust the pH with ammonium hydroxide, (5.1.5.1) drop by drop until iron(III) hydroxide precipitates.

Continue stirring for 30 min.

Filter (5.2.2.4) the co-precipitates.

Place the filter on to the identified planchet and fix it by an annular support to avoid deformation while drying.

Dry at moderate temperature.

After cooling the planchet and the filter to ambient temperature, weigh them and record their mass, m_{pfd}. Determine the mass deposited, m_{d}, using Equation (2):

$$m_{\mathrm{d}} = m_{\mathrm{pfd}} - m_{\mathrm{pf}} \qquad (2)$$

NOTE Radium, polonium and actinides co-precipitate quantitatively with barium sulfate or iron(III) hydroxide (Reference [2]).

7.3 Counting stage

Following evaporation (7.2.2) or co-precipitation (7.2.3), keep the planchet with the deposit in a drier before measurement.

The measurement of the residue activity on the planchet is performed by counting for an appropriate duration to reach the required characteristic limits and depending on the test portion and background count rates.

Due to the ingrowth of radon daughters over time, perform the measurement as soon as possible. For comparison purposes, it is recommended to perform the measurement at the same time after the preparation of the sample. In order to check for ingrowth of daughters of radium, the counting procedures should be repeated periodically over a period of one month.

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 times are long. However, over a period of one month, sources from raw waters which are naturally high in ^{226}Ra can show an ingrowth of four times the initial count.

7.4 Background and blank determination

Measure the background activity using a clean planchet (5.2.3) under conditions representative of the measurement method. Repeated counts confirm the stability of the background level.

If reagents are used, measure the blank activity using a clean planchet and reagents under conditions representative of the measurement method. Repeated counts confirm the stability of the blank level.

7.5 Preparation of counting standard for calibration

Prepare a geometry- and matrix-matched calibration source [planchet (5.2.3) or filter (5.2.2.4) with precipitates and annular attachment to the planchet] to closely mimic the procedure applied to test portions in order to obtain the same retro-diffusion effect.

Add an accurately known amount (about 5 Bq to 10 Bq) of a standard solution (5.1.1.1) to the starting volume of water and use the same source preparation procedure or directly add the standard solution to the planchet.

These standard sources are measured in the detector with a counting duration that leads to a counting uncertainty of 1 % (more than 10 000 counts should be recorded).

The counting efficiency is calculated by dividing the net count rate (test portion minus background) by the activity of the calibration source.

$$\varepsilon_{\alpha} = \frac{r_{\mathrm{s}\alpha} - r_{0\alpha}}{A} \tag{3}$$

A commercially available certified calibration source may be used.

7.6 Preparation of calibration source for self-absorption determination

7.6.1 General

As alpha-particle counting efficiency is directly dependent on the source thickness, estimate the self-absorption factor, defined by Equation (4):

$$f_{\mathrm{a}\alpha} = \frac{\varepsilon_{\mathrm{a}\alpha}}{\varepsilon_{\alpha}} \tag{4}$$

Two possibilities are given:

- spiking a single sample (7.6.2);
- spiking a set of samples of increasing mass to plot a self-absorption curve.

For beta particle counting efficiency, self-absorption phenomena are negligible $\varepsilon_{\beta} = \varepsilon_{\mathrm{a}\beta}$ ($f_{\mathrm{a}\beta} = 1$).

7.6.2 Spiking one of two test portions

Two test portions from the same laboratory sample are prepared under the same conditions with one test portion spiked with a known activity of the standard solution.

The specific efficiency is calculated by dividing the net count rate (unspiked test portion minus spiked test portion) by the activity of sample a [Equation (5)]:

$$\varepsilon_{\mathrm{a}\alpha} = \frac{r_{\mathrm{a}\alpha} - r_{\mathrm{g}\alpha}}{A_{\mathrm{a}}} \tag{5}$$

7.6.3 Self-absorption curve

A set of calibration sources with the same standard activity and increasing mass is prepared with the same sample preparation procedure to allow the determination of a series of absorption factors, $f_{\mathrm{a}\alpha}$. A function relating the absorption factor to the mass can be derived from the measurement data.

Several mathematical models are proposed to describe self-absorption phenomena such as linear, hyperbolic, exponential, polynomial, power laws (see References [3] to [9]). Commonly used curve-fitting techniques can be employed to derive the function and associated uncertainty.

WARNING — This procedure requires the preparation of a dry deposit spiked with alpha-emitting radionuclides. Avoid accidental ingestion of radioactive particulates by use of a sample closed cabinet glove box 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.

8 Expression of results

8.1 Alpha activity concentration

The alpha activity concentration, c_A, can be obtained using Equation (6):

$$c_A = \frac{r_{\mathrm{g}\alpha} - r_{0\alpha}}{V\,\varepsilon_\alpha\, f_{\mathrm{a}\alpha}} = (r_{\mathrm{g}\alpha} - r_{0\alpha})w \tag{6}$$

with

$$w = \frac{1}{V\,\varepsilon_\alpha\, f_{\mathrm{a}\alpha}}$$

NOTE It is advisable to choose the alpha and beta windows to reduce the alpha-beta crosstalk to a minimum.

8.2 Beta activity concentration

The beta activity concentration, c_A, can be obtained using Equation (7):

$$c_A = \frac{r_{\mathrm{g}\beta} - r_{0\beta} - \chi(r_{\mathrm{g}\alpha} - r_{0\alpha})}{V\,\varepsilon_\beta\, f_{\mathrm{a}\beta}} = \left[r_{\mathrm{g}\beta} - r_{0\beta} - \chi(r_{\mathrm{g}\alpha} - r_{0\alpha})\right]w \tag{7}$$

with

$$w = \frac{1}{V\,\varepsilon_\beta}$$

and

$$f_{\mathrm{a}\beta} = 1$$

Take any alpha-beta crosstalk into account, in order to derive the correct beta count rate. If needed, the alpha-beta crosstalk correction factor, χ, can be calculated using Equation (8):

$$\chi = \frac{r_{\mathrm{s}\alpha\to\beta}}{r_{\mathrm{s}\alpha}} \tag{8}$$

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

8.3 Standard uncertainty of the alpha activity concentration

As specified in ISO/IEC Guide 98-3 (see also ISO 11929[1]), the standard uncertainty of c_A is calculated using Equation (9):

$$u(c_A) = \sqrt{w^2 \left[u^2(r_{\mathrm{g}\alpha}) + u^2(r_{0\alpha}) \right] + c_A{}^2\, u_{\mathrm{rel}}^2(w)} = \sqrt{w^2 \left(\frac{r_{\mathrm{g}\alpha}}{t_{\mathrm{g}}} + \frac{r_{0\alpha}}{t_0} \right) + c_A{}^2\, u_{\mathrm{rel}}^2(w)} \tag{9}$$

As the uncertainty of the counting time and the coprecipitation stage are negligible in comparison with other sources of uncertainty (Reference [2]), it can be neglected and the relative standard uncertainty of w can be calculated using Equation (10):

$$u_{\mathrm{rel}}^2(w) = u_{\mathrm{rel}}^2(\varepsilon_\alpha) + u_{\mathrm{rel}}^2(V) + u_{\mathrm{rel}}^2(f_{\mathrm{a}\alpha}) \tag{10}$$

and the relative standard uncertainty of ε_α is calculated using Equation (11):

$$u_{\mathrm{rel}}^2(\varepsilon_\alpha) = u_{\mathrm{rel}}^2(r_{\mathrm{s}\alpha} - r_{0\alpha}) + u_{\mathrm{rel}}^2(A_\alpha) = \frac{(r_{\mathrm{s}\alpha}/t_{\mathrm{s}}) + (r_{0\alpha}/t_0)}{(r_{\mathrm{s}\alpha} - r_{0\alpha})^2} + u_{\mathrm{rel}}^2(A_\alpha) \tag{11}$$

All the uncertainties related to the calibration source are included in $u_{\mathrm{rel}}^2(A_\alpha)$, i.e. those of the standard solution and the preparation of the calibration source.

For the calculation of the detection limits, the term $\tilde{u}(\tilde{c}_A)$ is required (see ISO 11929[1]); the standard uncertainty of c_A as a function of its true value, calculated using Equation (12):

$$\tilde{u}(\tilde{c}_A) = \sqrt{w^2 \left[\frac{(\tilde{c}_A / w) + r_{0\alpha}}{t_{\mathrm{g}}} + \frac{r_{0\alpha}}{t_0} \right] + \tilde{c}_A^2\, u_{\mathrm{rel}}^2(w)} \tag{12}$$

8.4 Standard uncertainty of the beta activity concentration

As specified in the ISO/IEC Guide 98-3 (see also ISO 11929[1]), the standard uncertainty of c_A is calculated using Equations (13) to (15):

$$u(c_A) = \sqrt{w^2 \left[u^2(r_{\mathrm{g}\beta}) + u^2(r_{0\beta}) + T(\chi) \right] + c_A{}^2\, u_{\mathrm{rel}}^2(w)} = \sqrt{w^2 \left[\frac{r_{\mathrm{g}\beta}}{t_{\mathrm{g}}} + \frac{r_{0\beta}}{t_0} + T(\chi) \right] + c_A{}^2\, u_{\mathrm{rel}}^2(w)} \tag{13}$$

with

$$T(\chi) = \left(r_{\mathrm{g}\alpha} - r_{0\alpha} \right)^2 u^2(\chi) + \chi^2 \left(\frac{r_{\mathrm{g}\alpha}}{t_{\mathrm{g}}} + \frac{r_{0\alpha}}{t_0} \right) \tag{14}$$

and

$$u(\chi) = \sqrt{\frac{\chi(\chi + 1)}{r_{\mathrm{s}\alpha}\, t_{\mathrm{s}\alpha}}} \tag{15}$$

As the uncertainty of the counting time is small in comparison to other sources of uncertainty, it can be neglected and the relative standard uncertainty of w is calculated using Equation (16):

$$u_{\mathrm{rel}}^2(w) = u_{\mathrm{rel}}^2\left(\varepsilon_\beta\right) + u_{\mathrm{rel}}^2(V) \tag{16}$$

and the relative standard uncertainty of ε_β is calculated using Equation (17):

$$u_{\mathrm{rel}}^2(\varepsilon_\beta) = u_{\mathrm{rel}}^2(r_{\mathrm{s}\beta} - r_{0\beta}) + u_{\mathrm{rel}}^2(A_\beta) = \frac{(r_{\mathrm{s}\beta}/t_\mathrm{s}) + (r_{0\beta}/t_0)}{(r_{\mathrm{s}\beta} - r_{0\beta})^2} + u_{\mathrm{rel}}^2(A_\beta) \tag{17}$$

All the uncertainties related to the calibration source are included in $u_{\mathrm{rel}}^2(A_\beta)$, i.e. those of the standard solution and the preparation of the calibration source.

For the calculation of the detection limits, the term $\tilde{u}(\tilde{c}_A)$ is required (see ISO 11929[1]); the standard uncertainty of c_A as a function of its true value, calculated by Equation (18):

$$\tilde{u}(\tilde{c}_A) = \sqrt{w^2 \left[\frac{(\tilde{c}_A / w) + \chi\left(r_{\mathrm{g}\alpha} - r_{0\alpha}\right) + r_{0\beta}}{t_\mathrm{g}} + \frac{r_{0\beta}}{t_0} + T\left(\chi\right) \right] + \tilde{c}_A^2\, u_{\mathrm{rel}}^2(w)} \tag{18}$$

8.5 Decision threshold

8.5.1 Decision threshold of the alpha activity concentration

The decision threshold, c_A^*, is obtained from Equation (12) for $\tilde{c}_A = 0$ (see ISO 11929[1]):

$$c_A^* = k_{1-\alpha}\, \tilde{u}(0) = k_{1-\alpha}\, w \sqrt{\frac{r_{0\alpha}}{t_\mathrm{g}} + \frac{r_{0\alpha}}{t_0}} \tag{19}$$

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

8.5.2 Decision threshold of the beta activity concentration

The decision threshold, c_A^*, is obtained from Equation (18) for $\tilde{c}_A = 0$ (see ISO 11929[1]). This yields:

$$c_A^* = k_{1-\alpha}\, \tilde{u}(0) = k_{1-\alpha}\, w \sqrt{\frac{\chi(r_{\mathrm{g}\alpha} - r_{0\alpha}) + r_{0\beta}}{t_\mathrm{g}} + \frac{r_{0\beta}}{t_0} + T\left(\chi\right)} \tag{20}$$

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

NOTE In the case of a simultaneous alpha and beta activity measurement, the possibility of alpha-beta crosstalk is taken into account, in order to correct the alpha decision threshold.

8.6 Detection limit

8.6.1 Detection limit of the alpha activity concentration

The detection limit, $c_A^\#$, is calculated using Equation (21) (see ISO 11929[1]):

$$c_A^\# = c_A^* + k_{1-\beta}\, \tilde{u}(c_A^\#) = c_A^* + k_{1-\beta} \sqrt{w^2 \left[\frac{(c_A^\# / w) + r_{0\alpha}}{t_\mathrm{g}} + \frac{r_{0\alpha}}{t_0} \right] + c_A^{\#2}\, u_{\mathrm{rel}}^2(w)} \tag{21}$$

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

The detection limit can be calculated by solving Equation (21) for $c_A^{\#}$ or, more simply, by iteration with a starting approximation, $c_A^{\#} = 2\, c_A^{*}$.

When taking $\alpha = \beta$ then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of Equation (21) is given by Equation (22):

$$c_A^{\#} = \frac{2\, c_A^{*} + (k^2 w / t_{\mathrm{g}})}{1 - k^2\, u_{\mathrm{rel}}^2(w)} \tag{22}$$

8.6.2 Detection limit of the beta activity concentration

The detection limit, $c_A^{\#}$, is calculated using Equation (23) (see ISO 11929[1]):

$$c_A^{\#} = c_A^{*} + k_{1-\beta}\, \tilde{u}(c_A^{\#}) = c_A^{*} + k_{1-\beta} \sqrt{w^2 \left\{ \left[\frac{(c_A^{\#}/w) + \chi\left(r_{\mathrm{g}\alpha} - r_{0\alpha}\right) + r_{0\beta}}{t_{\mathrm{g}}} \right] + \frac{r_{0\beta}}{t_0} + T(\chi) \right\} + c_A^{\#2}\, u_{\mathrm{rel}}^2(w)} \tag{23}$$

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

The detection limit can be calculated by solving Equation (23) for $c_A^{\#}$ or, more simply, by iteration with a starting approximation $c_A^{\#} = 2\, c_A^{*}$.

When taking $\alpha = \beta$ then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of Equation (23) is given by Equation (24):

$$c_A^{\#} = \frac{2 c_A^{*} + (k^2\, w / t_{\mathrm{g}})}{1 - k^2\, u_{\mathrm{rel}}^2(w)} \tag{24}$$

8.7 Confidence limits

The lower, $c_A^{\triangleleft}$, and upper, $c_A^{\triangleright}$, confidence limits are calculated using Equations (25) and (26) (see ISO 11929[1]):

$$c_A^{\triangleleft} = c_A - k_p\, u(c_A) \qquad p = \omega\left(1 - \frac{\gamma}{2}\right) \qquad \text{as defined in ISO 11929[1]} \tag{25}$$

$$c_A^{\triangleright} = c_A + k_q\, u(c_A) \qquad q = 1 - \frac{\omega\,\gamma}{2} \qquad \text{as defined in ISO 11929[1]} \tag{26}$$

where $\omega = \Phi[y/u(y)]$ in which Φ is the distribution function of the standardised normal distribution.

Set $\omega = 1$ if $c_A \geqslant 4\, u(c_A)$. In this case:

$$c_A^{\triangleleft\triangleright} = c_A \pm k_{1-(\gamma/2)}\, u\left(c_A\right) \tag{27}$$

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

9 Interference control

9.1 Contamination

Check the contamination of reagents by evaporating the volumes of the reagents used in the procedures on to separate planchets. Ensure that the activities are negligible compared with that of the test sample.

If the activity is not negligible, select reagents with lower radioactivity, or include a blank determination in the main procedure.

9.2 Losses of activity

9.2.1 Radon isotopes

Some radionuclides, such as radon, can be lost by volatilisation during the evaporation step. Thus, although ^{222}Rn is lost during processing, ^{226}Ra daughter radionuclides subsequently grow into the counting source. A similar effect occurs with ^{224}Ra daughter radionuclides in the ^{232}Th series.

9.2.2 Polonium

Some alpha-emitting polonium isotopes, which occur naturally as members of the uranium and thorium decay series, may contribute to 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 volatilised (Reference [10], pp. 39, 64 ff. and 77 ff.).

10 Test report

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

a) reference to this International Standard;

b) all information necessary for the complete 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:

- 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 $\leqslant c_A^*$ when the result is below the decision threshold,
- when the activity concentration, c_A, is compared with the detection limit, the result of the measurement can be expressed as $\leqslant c_A^{\#}$ 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) all operating details not specified in this International Standard, or regarded as optional, together with details of any incident that may have influenced 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] LLAURADÓ, M., VALLÉS, I., ABELAIRAS, A., ALONSO, A., DÍAZ, M.F., GARCÍA, R., DE LUCAS, M.J., SUÁREZ, J.A. *Procedimiento para la determinación del índice de actividad alfa total en muestras de agua: Métodos de coprecipitación y evaporación* [Determination of the total alpha activity index in water samples: Coprecipitation and evaporation methods]. Consejo de Seguridad Nuclear, Madrid, 2006. 30 p. (*Colección Informes Técnicos* 11.2006, *Serie Vigilancia Radiológica Ambiental, Procedimiento* 1.9.) Available (2009-06-11) at: http://www.csn.es/publicaciones/pro_09.pdf

[3] RODRÍGUEZ, P.B., SÁNCHEZ, A.M., TOMÉ, F.V. Experimental studies of self-absorption and backscattering in alpha-particle sources. *Appl. Radiat. Isot*. 1997, **48**, pp. 1215-1220

[4] JETER, H.W. An extended range method for gross alpha-/beta-particle measurements in water samples. *Radioact. Radiochem*. 1998, **9**, pp. 16-25

[5] MORÓN, M.C., GARCÍA-TENORIO, R., GARCÍA-MONTAÑO, E., GARCIA-LEON, M., MADURGA, G. An easy method for the determination of Ra isotopes and actinide alpha emitters from the same water sample. *Int. J. Appl. Radiat. Isot.* 1986, **37**, pp. 383-389

[6] SEMKOW, T.M., BARI, A., PAREKH, P.P., HAINES, D.K., GAO, H., BOLDEN, A.N., DAHMS, K.S., SCARPITTA, S.C., THERN, R.E., VELAZQUEZ, S. Experimental investigation of mass efficiency curve for alpha radioactivity counting using a gas-proportional detector. *Appl. Radiat. Isot*. 2004, **60**, pp. 879-886

[7] SEMKOW, T.M., JETER, H.W., PARSA, B., PAREKH, P.P., HAINES, D.K., BARI, A. Modeling of alpha mass-efficiency curve. *Nucl. Instrum. Meth. Phys. Res. A* 2005, **538**, pp. 790-800

[8] SUAREZ-NAVARRO, J.A., PUJOL, L.I., DE PABLO, M.A. Rapid determination of gross alpha-activity in sea water by coprecipitation. *J. Radioanal. Nucl. Chem*. 2002, **253**, pp. 47-52

[9] ZIKOVSKY, L. Uranyl ions, nitrate ions and some major cations as causes of anomalous results when determining gross alpha radioactivity in ground water. *Radioact. Radiochem*. 2000, **11**, pp. 26-28

[10] BAGNALL, K.W. *Chemistry of the rare radio elements: Polonium-actinium*. Butterworth, London. 177 p.

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