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BSI Standards Publication

Water quality — Measurement of gross alpha and beta activity concentration in non-saline water — Liquid scintillation counting method

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National foreword

This British Standard is the UK implementation of ISO 11704:2010.

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.

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Qualité de l'eau — Mesurage des activités alpha globale et bêta globale des eaux non salines — Méthode de comptage par scintillation liquide

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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 11704 was prepared by Technical Committee ISO/TC 147, *Water quality*.

Water quality — Measurement of gross alpha and beta activity concentration in non-saline water — Liquid scintillation counting 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 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 waters for radionuclides which are not volatile at 80 °C. Radon isotopes and their decay products of short half life are not included in the determination.

The method is applicable to raw and potable waters with a dry residue less than 5 g/l and when no correction for colour quenching is necessary.

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 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*

3 Symbols, definitions and units

For the purposes of this document, the definitions, symbols and abbreviations defined in ISO 80000-10, as well as the following symbols, definitions and units, apply.

a_α, a_β	Alpha and beta activity per mass	Bq g^{-1}
a^*	Decision threshold	Bq g^{-1}
$a^\#$	Detection limit	Bq g^{-1}
$a^\triangleleft, a^\triangleright$	Lower and upper limits of the confidence interval	Bq g^{-1}
A_α, A_β	Activity of the alpha and beta emitter certified reference solution used for the α and β calibration sources	Bq
m	Mass of the test sample	g
m_1	Mass of initial sample subject to heating or possibly concentration	g
m_2	Mass of heated or concentrated sample	g
m_3	Mass of heated or concentrated sample transferred in the vial	g
$m_{\mathrm{S}\alpha}$, $m_{\mathrm{S}\beta}$	Mass of alpha and beta emitters certified reference solutions, respectively	g
$r_{\mathrm{g}\alpha}$, $r_{\mathrm{g}\beta}$	Sample gross count rate, from the alpha and beta windows, respectively	s^{-1}
$r_{0\alpha}$, $r_{0\beta}$, $r_{0\mathrm{T}}$	Blank count rate, from the alpha, beta and total windows, respectively	s^{-1}
$r_{\mathrm{S}\alpha,\alpha}$, $r_{\mathrm{S}\alpha,\beta}$, $r_{\mathrm{S}\alpha,\mathrm{T}}$	Count rate of the alpha calibration source in the alpha, beta and total window	s^{-1}
$r_{\mathrm{S}\beta,\alpha}$, $r_{\mathrm{S}\beta,\beta}$, $r_{\mathrm{S}\beta,\mathrm{T}}$	Count rate of the beta calibration source in the alpha, beta and total window	s^{-1}
t_g	Sample counting time	s
t_0	Blank counting time	s
$t_{\mathrm{s}\alpha}$, $t_{\mathrm{s}\beta}$	Counting time of α and β calibration sources	s
$u(a)$	Standard uncertainty associated with the measurement result	Bq g^{-1}
U	Expanded uncertainty, calculated from $U = ku(a)$, where $k = 1, 2 \ldots$	Bq g^{-1}
$\tilde{u}(\tilde{a}_\alpha)$	Standard uncertainty of a_α as a function of its true value	Bq g^{-1}
ε_α, ε_β	Counting efficiency for alpha and beta, respectively	—
$\tau_\alpha(\chi_{\alpha\to\beta})$	Alpha interference — Fraction of counts observed in the beta window with respect to the total number of counts measured by the counter when an alpha emitter is measured	—
$\tau_\beta(\chi_{\beta\to\alpha})$	Beta interference — Fraction of counts observed in the alpha window with respect to the total number of counts measured by the counter when a beta emitter is measured	—

4 Principle

Gross alpha and beta activity concentrations are determined by using liquid scintillation counting of a water sample mixed with a scintillation cocktail.

Gross alpha and beta determinations are not absolute determinations of the sample radioactive contents, but relative determinations referred to a specific alpha or beta emitter which constitutes the standard calibration sources. These types of determinations are also known as the alpha and beta index.

The aqueous sample is acidified using nitric acid and heated. Subsequently, water with low salt content can be thermally concentrated by slow evaporation to improve the method sensitivity. An aliquot of sample is transferred into a liquid scintillation vial with scintillation cocktail; scintillations from the vial are then counted by equipment with an alpha and beta discrimination device.

The counter is previously optimized with respect to an alpha and beta discriminator setting and then calibrated against alpha and beta emitter certified reference solutions. In data evaluation, no correction for chemical quenching is applied, since the procedure is designed to provide samples with a constant quenching level.

The method does not account for ^{222}Rn and its daughters of short half life and it is not suitable for ^{3}H and ^{14}C measurement.

When suspended matter is present in significant quantities, a filtration step is required before acidification.

5 Reagents and equipment

All reagents shall be of recognized analytical grade, except for the scintillation cocktail, and shall not contain any detectable alpha and beta activity, except for the radioactive certified reference solutions.

5.1 Nitric acid, $c(\text{HNO}_3) = 15{,}8$ mol/l, $\rho = 1{,}42$ g/ml, mass fraction $w(\text{HNO}_3) = 70$ %.

5.2 Water, ISO 3696[1], grade 3.

Deionized water can contain detectable amounts of ^{222}Rn and short half-life decay products. It is therefore strongly recommended to boil water under vigorous stirring and let it stand for one day before use. Alternatively, use nitrogen flushing for about 1 h for a 2 l sample.

5.3 Scintillation cocktail. Commercially available scintillation cocktails suitable for alpha and beta discrimination (e.g. diisopropylnaphthalene-based cocktails).

5.4 Volatile organic solvents. Methanol or ethanol.

5.5 Certified reference solutions. A calibration laboratory establishes traceability of its own calibration sources and measuring instruments to the International System of Units (SI) by means of an unbroken chain of calibrations or comparisons linking them to relevant certified reference solutions of the SI units of measurement. The link to the SI units may be achieved with respect to national certified reference materials. These may be primary realizations of the SI units, or agreed representations of SI units based on fundamental physical constants, or they may be secondary materials which are materials certified 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 statement of compliance with an identified metrological specification.

NOTE Calibration laboratories fulfilling the requirements of this International Standard are considered to be competent. A calibration certificate bearing an accreditation body logo from a calibration laboratory accredited to this International Standard, for the calibration concerned, is sufficient evidence of traceability of the calibration data reported.

In general, the experimental parameters (efficiency, alpha and beta optimum discrimination) depend on alpha and beta energies, thus the choice of alpha and beta emitter certified reference solutions will depend on knowledge of the type of radioactive contaminant likely to be present in the waters being tested (see ISO 9696[4] and Reference [11]).

5.5.1 Alpha emitter certified reference solution. The alpha emitter certified reference solution shall not contain any unexpected detectable alpha and beta activity.

NOTE ^{236}U is a convenient choice when waters are checked for their natural radioactivity content, as its energy is close to the most widespread natural radionuclides (e.g. uranium and thorium isotopes, ^{226}Ra) and it is commercially available without decay products of short half life. The supplier can supply details of the absence of any decay product.

^{241}Am is commonly used when artificial radionuclide contamination is suspected. ^{239}Pu can be used as well in such circumstances.

5.5.2 Beta emitter certified reference solution. The beta emitter certified reference solution shall not contain alpha-emitting radioisotopes.

NOTE A ^{90}Sr and ^{90}Y mixture or ^{40}K are commonly used. A potassium solution for atomic absorption spectrometry 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.

5.6 Equipment.

5.6.1 Analytical balance.

5.6.2 Hot plate with magnetic stirrer and stirring bar.

5.6.3 pH meter.

5.6.4 Wide-mouth HDPE sample bottles, capacities between 100 ml and 500 ml.

5.6.5 Liquid scintillation counter, with α and β discrimination option, preferably an ultra low level counter to achieve better detection limits.

5.6.6 Polyethylene scintillation vials, capacity 20 ml, such as PET vials, low diffusion PET vials or PTFE-coated polyethylene vials.

PTFE-coated polyethylene vials are the best choice since they prevent both the diffusion of the cocktail into the wall of the vial and the absorption of radon from the outer environment. Glass vials generally worsen α and β discrimination.

6 Sampling

Collect 0,1 l to 1 l of water in a plastic bottle (5.6.4) in accordance with ISO 5667-3. If necessary, filter immediately on collection and before acidification. If possible, acidify immediately with nitric acid (5.1) to a value not lower than pH 1,7 ± 0,2 (7.1) or pH 2,7 ± 0,2 if preconcentration is desired (7.2). Verify by pH meter.

NOTE Acidification of the water sample minimizes the loss of radioactive material from solution by adsorption. If carried out before filtration, it desorbs radioactive material already adsorbed on to the particulate material.

The relatively low acidification of the sample does not ensure long-term preservation. Prepare the sample within 48 h of collection. Underground waters are usually more stable than raw waters (see ISO 5667-3).

7 Procedure

7.1 Direct counting

Transfer a weighed (5.6.1) aliquot of the water sample of approximately 50 g, m_1, into a beaker. If the laboratory sample has not yet been acidified, acidify the aliquot using nitric acid (5.1) to pH 1,7 ± 0,2 (verify by pH meter, 5.6.3).

Cover the beaker and heat to approximately 80 °C while stirring for 30 min (5.6.2) to remove the dissolved ^{222}Rn. Allow the aliquot to cool and weigh it again to account for the losses due to evaporation. Record the mass as m_2.

NOTE The necessary amount of acid is small (normally about 0,15 g for a 50 g sample) and its mass can be neglected.

7.2 Thermal preconcentration

It is possible to apply a thermal preconcentration when soft waters are considered (e.g. dry residue less than 500 mg/l, as in most drinking waters) in order to increase the sensitivity of the method. Hard waters (dry residue more than 500 mg/l) may give rise to salt precipitations or to a difficult homogenization with the scintillation cocktail.

Transfer a weighed (5.6.1) aliquot of the water sample of approximately 200 g, m_1, into a beaker. If the laboratory sample has not yet been acidified, acidify the aliquot using nitric acid (5.1) to pH 2,7 ± 0,2 (verify by pH meter).

Slowly evaporate the aliquot on a hot plate (5.6.2) to a final quantity of approximately 20 g. Allow the aliquot to cool to room temperature and weigh the concentrated aliquot. Record the mass as m_2. The pH of the concentrated aliquot shall be 1,7 ± 0,2.

No precipitation should be observed, otherwise direct counting (7.1) or smaller preconcentration factors shall be applied.

NOTE If unknown, only a rough evaluation of the dry residue is needed. Any commonly used technique can be adopted.

7.3 Sample preparation

Clean the scintillation vial (5.6.6) with ethanol or methanol (5.4). Transfer a weighed (5.6.1) test portion (7.1 or 7.2), m_3, of the aliquot into the vial. Add the scintillation cocktail (5.3) and shake vigorously. Calculate the exact mass, m, of sample analysed:

$$m = \frac{m_1 \, m_3}{m_2} \tag{1}$$

The relative amounts of test portion and scintillation cocktail depend on the characteristics of the latter. Follow the manufacturer's instructions. With many commercially available cocktails, 8:12 volume ratios can be used. The sample:cocktail ratio has to be chosen once and for all and used for every measurement (e.g. 8 ml test portion + 12 ml scintillation cocktail). It is important to ensure homogeneity of the test portion and cocktail mix before measurement.

7.4 Liquid scintillation measurement

7.4.1 Preparation of alpha and beta calibration sources

Transfer an accurately known amount, $m_{S\alpha}$ (e.g. corresponding to an activity of about 10 Bq), of the chosen alpha emitter certified reference solution (5.5.1) into a scintillation vial (5.6.6). Let the activity at the time of measurement be A_α. Dilute with water (5.2) to the previously chosen mass (e.g. 8 g). Add the scintillation cocktail (5.3), e.g. 12 ml, and mix thoroughly.

In the same way, prepare the chosen beta emitter certified reference solution (5.5.2). Let the amount of the beta solution transferred into the vial be $m_{S\beta}$, and the activity at the time of measurement be A_β.

The pH of the diluted certified reference solutions shall be 1,7 ± 0,2.

7.4.2 Optimization of counting conditions

Set the alpha- and beta-counting windows of the scintillation counter (5.6.5) so that the energies of all the alpha and beta emitters of interest are covered (see the manufacturer's instructions).

Usually all alpha emitters are included in the determination. In principle, all beta emitter energies can be covered, as the liquid scintillation counting sensitivity is good also for very low (e.g. < 0,2 MeV) beta energies. Nevertheless a common choice is to set the beta-counting window so that all beta emitter energies lower than 0,2 MeV are excluded in order to get results comparable to those obtained with other standard methods, e.g. ISO 9697[5] (see Reference [11]).

Count for an appropriate period the alpha and beta calibration sources in alpha and beta discrimination mode (see the manufacturer's instructions), under different discriminator settings.

Let the counting rate be $r_{\mathrm{S}\alpha,\beta}$ and $r_{\mathrm{S}\alpha,\mathrm{T}}$ respectively for the counts of the alpha calibration source in beta and in total (undiscriminated) window. Let the counting rate be $r_{\mathrm{S}\beta,\alpha}$ and $r_{\mathrm{S}\beta,\mathrm{T}}$ respectively for the counts of the beta calibration source in alpha and in total (undiscriminated) window.

Determine the alpha (τ_α) and beta (τ_β) interference parameter for each discriminator setting (background contribution is often negligible):

$$\tau_\alpha = \frac{r_{\mathrm{S}\alpha,\beta} - r_{0\beta}}{r_{\mathrm{S}\alpha,\mathrm{T}} - r_{0\mathrm{T}}} \tag{2}$$

$$\tau_\beta = \frac{r_{\mathrm{S}\beta,\alpha} - r_{0\alpha}}{r_{\mathrm{S}\beta,\mathrm{T}} - r_{0\mathrm{T}}} \tag{3}$$

The best discriminator setting (working point) is chosen in order to minimize both τ_α and τ_β.

The selected counting conditions are used for all other subsequent counts (sample, blank and calibration sources).

NOTE 1 The term interference means the misclassification of alpha pulses in the beta-counting window, or vice versa.

NOTE 2 Examples of setting determination are given in Reference [11].

It is necessary for the values of both alpha and beta interference in the chosen working conditions to be adequately low (e.g. less than 5 %); in this situation the uncertainty introduced in the activity determination is generally included in the range of values defined by the total uncertainty. Otherwise it is necessary to account for interference in the calculations of sample activity, decision threshold, and detection limit (see ASTM D7283[9] and Reference [11]).

It is known that quenching can affect both interference and detection efficiency (see ISO 9696[4], ISO 9697[5], ASTM D7283[9], UNI 11260[10], and References [11] to [14]). However, the strict control of the pH of samples (as outlined above) allows quenching variability and its effects on results to be minimized. It is therefore advisable to determine an acceptability range for quench values of any measured vial (sample, blank or calibration source), e.g. on the basis of repeated measurements of replicate samples. When measuring samples, blank or calibration source vials, their quenching should be checked (as monitored by the instrument — see the manufacturer's instructions) and compared with the acceptability range.

NOTE 3 Optical quench or colour quench can not be pointed out by the parameter given by the instrument (this is a general problem of LSC counting). The presence of optical quench can be determined by considering the external source spectrum (see the manufacturer's instructions). Optical quench can lead to underestimation of results, whose extent is unpredictable.

7.4.3 Blank sample preparation and measurement

Acidify water (5.2) to pH 1,7 ± 0,2. Transfer the chosen quantity (e.g. 8 ml) into the scintillation vial (5.6.6) and weigh the vial. Add the scintillation cocktail (5.3), e.g. 12 ml, and mix thoroughly.

Count (5.6.5) the blank sample using the chosen optimum counting conditions. Let the measured counting rates be $r_{0\alpha}$ and $r_{0\beta}$, respectively, for the counts in the alpha and beta windows.

Repeated counts allow a confirmation of the stability of the blank count rate, the use of an appropriate control chart is recommended.

7.4.4 Alpha and beta efficiencies

Let the counting rates be $r_{\mathrm{S}\alpha,\alpha}$ and $r_{\mathrm{S}\beta,\beta}$, respectively, for the counts of the alpha calibration source in the alpha window and for the counts of the beta calibration source in the beta window, as measured with the previously defined best discriminator setting.

Determine the alpha- and beta-counting efficiencies:

$$\varepsilon_\alpha = \frac{r_{\mathrm{S}\alpha,\alpha} - r_{0\alpha}}{A_\alpha} \tag{4}$$

$$\varepsilon_\beta = \frac{r_{\mathrm{S}\beta,\beta} - r_{0\beta}}{A_\beta} \tag{5}$$

7.4.5 Sample measurement

Count the sample using the chosen optimum counting conditions. Let the measured counting rates be $r_{\mathrm{g}\alpha}$ and $r_{\mathrm{g}\beta}$, respectively, for the counts in the alpha and beta windows.

Samples should be counted immediately after preparation, to minimize undesirable ingrowth of ^{222}Rn and other decay products from ^{226}Ra (ISO 9696[4], ASTM D7283[9]).

NOTE The duration of counting depends on the sample count rate and also on precision and the detection limit required.

8 Expression of results

8.1 Calculation of activity per mass

When the beta interference is negligible, calculate the alpha activity per mass, a_α, of the water sample by:

$$a_\alpha = \frac{r_{\mathrm{g}\alpha} - r_{0\alpha}}{m\,\varepsilon_\alpha} = (r_{\mathrm{g}\alpha} - r_{0\alpha})\,w_\alpha \qquad \text{where} \qquad w_\alpha = \frac{1}{m\,\varepsilon_\alpha} \tag{6}$$

When the alpha interference is negligible, calculate the beta activity per mass, a_β, of the water sample by:

$$a_\beta = \frac{r_{\mathrm{g}\beta} - r_{0\beta}}{m\,\varepsilon_\beta} = (r_{\mathrm{g}\beta} - r_{0\beta})\,w_\beta \qquad \text{where} \qquad w_\beta = \frac{1}{m\,\varepsilon_\beta} \tag{7}$$

If the result has to be expressed in becquerels per volume then multiply the initial result expressed in becquerels per mass by the density of the water sample.

8.2 Standard uncertainty

As specified in ISO/IEC Guide 98-3:2008[7], the standard uncertainty of a_α is calculated, from Equation (8), by:

$$u\left(a_\alpha\right)=\sqrt{w_\alpha^2\left[u^2(r_{\mathrm{g}\alpha})+u^2(r_{0\alpha})\right]+\left(r_{\mathrm{g}\alpha}-r_{0\alpha}\right)^2 u_{\mathrm{rel}}^2\left(w_\alpha\right)}=\sqrt{w_\alpha^2\left(\frac{r_{\mathrm{g}\alpha}}{t_\mathrm{g}}+\frac{r_{0\alpha}}{t_0}\right)+a_\alpha^2\, u_{\mathrm{rel}}^2\left(w_\alpha\right)} \tag{8}$$

where the uncertainty of the counting time is neglected and the relative standard uncertainty of w is calculated using Equation (9):

$$u_{\mathrm{rel}}^2(w_\alpha)=u_{\mathrm{rel}}^2(\varepsilon_\alpha)+u_{\mathrm{rel}}^2(m) \tag{9}$$

the relative standard uncertainty of m is calculated using Equation (10):

$$u_{\mathrm{rel}}^2(m)=u_{\mathrm{rel}}^2(m_1)+u_{\mathrm{rel}}^2(m_2)+u_{\mathrm{rel}}^2(m_3) \tag{10}$$

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

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

$u_{\mathrm{rel}}^2(A_\alpha)$ includes all the uncertainties related to the calibration source, i.e. in the certified reference solution and the preparation of the calibration source.

For the calculation of the characteristic limits, one needs $\tilde{u}(\tilde{a}_\alpha)$ (see ISO 11929[6]), i.e. the standard uncertainty of a_α as a function of its true value, calculated using Equation (12):

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

In the same way, the standard uncertainty of the beta activity per mass is calculated using Equation (13):

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

and the standard uncertainty of a_β as a function of its true value using Equation (14):

$$\tilde{u}(\tilde{a}_\beta)=\sqrt{w_\beta^2\left[\frac{(\tilde{a}_\beta/w_\beta+r_{0\beta})}{t_\mathrm{g}}+\frac{r_{0\beta}}{t_0}\right]+\tilde{a}_\beta^2\, u_{\mathrm{rel}}^2(w_\beta)} \tag{14}$$

NOTE If an analytical balance is used, the mass uncertainty contribution to the total uncertainty can be neglected.

8.3 Decision threshold

The decision threshold, a_α^*, is obtained from Equation (12) for $\tilde{a}_\alpha=0$ (see ISO 11929[6]). This yields:

$$a_\alpha^*=k_{1-\alpha}\,\tilde{u}(0)=k_{1-\alpha}\,w_\alpha\sqrt{\frac{r_{0\alpha}}{t_\mathrm{g}}+\frac{r_{0\alpha}}{t_0}} \tag{15}$$

and, in the same way, the decision threshold, a_β^*:

$$a_\beta^* = k_{1-\alpha}\,\tilde{u}(0) = k_{1-\alpha}\,w_\beta \sqrt{\frac{r_{0\beta}}{t_g} + \frac{r_{0\beta}}{t_0}} \qquad (16)$$

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

8.4 Detection limit

The detection limit, $a_\alpha^\#$, is calculated using Equation (17) (see ISO 11929[6]):

$$\begin{aligned} a_\alpha^\# &= a_\alpha^* + k_{1-\beta}\,\tilde{u}(a_\alpha^\#) = \\ &= a_\alpha^* + k_{1-\beta}\sqrt{w_\alpha^2\left[\frac{(a_\alpha^\# / w_\alpha + r_{0\alpha})}{t_g} + \frac{r_{0\alpha}}{t_0}\right] + (a_\alpha^\#)^2\,u_{\text{rel}}^2(w_\alpha)} \end{aligned} \qquad (17)$$

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

The detection limit can be calculated by solving Equation (17) for $a_\alpha^\#$ or, more simply, by iteration with an initial approximation $a_\alpha^\# = 2a_\alpha^*$.

When taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of Equation (17) is given by Equation (18):

$$a_\alpha^\# = \frac{2a_\alpha^* + \left(k^2 w_\alpha\right)/t_g}{1 - k^2\,u_{\text{rel}}^2\left(w_\alpha\right)} \qquad (18)$$

In the same way, the detection limit of the beta activity per mass is given by Equation (19):

$$a_\beta^\# = \frac{2a_\beta^* + \left(k^2 w_\beta\right)/t_g}{1 - k^2\,u_{\text{rel}}^2\left(w_\beta\right)} \qquad (19)$$

8.5 Confidence limits

The lower, $a^\triangleleft$, and upper, $a^\triangleright$, confidence limits are calculated using Equations (20) and (21) (see ISO 11929[6]):

$$a^\triangleleft = a - k_p\,u(a) \quad \text{with} \quad p = \omega(1 - \gamma/2) \qquad (20)$$

$$a^\triangleright = a + k_q\,u(a) \quad \text{with} \quad q = 1 - \omega\,\gamma/2 \qquad (21)$$

where

$$\omega = \Phi\left[y/u(y)\right]$$

in which Φ is the distribution function of the standardized normal distribution.

If $a \geqslant 4\,u(a)$, ω may be set to 1. In this case:

$$a^{\triangleleft,\triangleright} = a \pm k_{1-\gamma/2}\,u(a) \qquad (22)$$

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

8.6 Quality control

Precision, sensitivity and bias are dependent on the characteristics of the detector, the radionuclides present, and the certified reference solution selected both for the discriminator setting and for counting efficiency. The radionuclides used for the calibration shall be carefully chosen in order to minimize the bias.

The repeatability of this method has been determined and is reported in Reference [11].

9 Interference control

9.1 Contamination

Check the contamination of reagents by processing blank measurements using appropriate aliquots of distilled water. If a significant contribution of the reagents to the gross alpha or beta activity is found, select reagents with lower content of radioactivity. Run contamination checks with every new reagent batch.

The contamination of the counting system should be controlled by passing appropriate background samples (e.g. background vials supplied by the manufacturer of the counting system).

9.2 Ingrowth of radon

Radon isotopes should be volatilized during the preconcentration step. If ^{226}Ra and ^{228}Th/^{224}Ra are present, ^{222}Rn and ^{220}Rn and their alpha- and beta-emitting daughters grow in during the measurement.

9.3 Loss of polonium

Loss of polonium isotopes due to volatilization is not expected, since the samples are acidified using nitric acid and heated at temperatures far below 400 °C.

10 Test report

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

a) a reference to this International Standard (ISO 11704:2010);

b) identification of the sample;

c) the radionuclides used for the calibration;

d) the units in which the results are expressed;

e) the test result, $a \pm u(a)$ or $a \pm U$, with the associated k value.

Complementary information can be provided such as:

f) probabilities α, β and $(1 - \gamma)$;

g) decision threshold and the detection limit;

h) depending on the customer request there are different ways to present the result:

    1) when the activity per mass, a, is compared with the decision threshold (see ISO 11929[6]), the result of the measurement should be expressed as $\leqslant a^*$ when the result is below the decision threshold,

    2) when the activity per mass, a, is compared with the detection limit, the result of the measurement can be expressed as $\leqslant a^\#$ 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;

i) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s).

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