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Water quality — Uranium — Test method using alpha liquid scintillation counting

Qualité de l'eau — Uranium — Méthode d'essai par comptage des scintillations alpha en milieu liquide

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Radioactivity from several naturally-occurring and anthropogenic sources is present throughout the environment. Thus, water bodies (e.g. surface waters, ground waters, sea waters) can contain radionuclides of natural, human-made, or both origins.

- Natural radionuclides, including 40K, 3H, 14C, and those originating from the thorium and uranium decay series, in particular 226Ra, 228Ra, 234U, 238U, 210Po and 210Pb can be found in water for natural reasons (e.g. desorption from the soil and washoff by rain water) or can be released from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or phosphate fertilizers production and use).
- Human-made radionuclides such as transuranium elements (americium, plutonium, neptunium, curium), 3H, 14C, 90Sr and gamma emitting radionuclides can also be found in natural waters. Small quantities of these radionuclides are discharged from nuclear fuel cycle facilities into the environment as a result of authorized routine releases. Some of these radionuclides used for medical and industrial applications are also released into the environment after use. Anthropogenic radionuclides are also found in waters as a result of past fallout contaminations resulting from the explosion in the atmosphere of nuclear devices and accidents such as those that occurred in Chernobyl and Fukushima.

Radionuclide activity concentration in water bodies can vary according to local geological characteristics and climatic conditions and can be locally and temporally enhanced by releases from nuclear installation during planned, existing and emergency exposure situations[1]. Drinking-water may thus contain radionuclides at activity concentrations which could present a risk to human health.

The radionuclides present in liquid effluents are usually controlled before being discharged into the environment[2] and water bodies. Drinking waters are monitored for their radioactivity as recommended by the World Health Organization (WHO)[3], so that proper actions can be taken to ensure that there is no adverse health effect to the public. Following these international recommendations, national regulations usually specify radionuclide authorized concentration limits for liquid effluent discharged to the environment and radionuclide guidance levels for waterbodies and drinking waters for planned, existing, and emergency exposure situations. Compliance with these limits can be assessed using measurement results with their associated uncertainties as specified by ISO/IEC Guide 98-3 and ISO 5667-20[4].

Depending on the exposure situation, there are different limits and guidance levels that would result in an action to reduce health risk. As an example, during a planned or existing situation, the WHO guidelines for guidance level for uranium 238 and uranium 234 in drinking water is 10 Bq/l and 1 Bq/l, respectively. The provisional guideline value for total content of uranium in drinking water is 30 µg/l based on its chemical toxicity, which is predominant compared with its radiological toxicity.

NOTE 1 The guidance level is the activity concentration with an intake of 2 l/d of drinking water for one year that results in an effective dose of 0,1 mSv/a for members of the public. This is an effective dose that represents a very low level of risk and which is not expected to give rise to any detectable adverse health effects[3].

In the event of a nuclear emergency, the WHO Codex Guideline Levels[6] state that the activity concentration might not be greater than 100 Bq/l for uranium 235.

NOTE 2 The Codex guidelines levels (GLs) apply to radionuclides contained in foods destined for human consumption and traded internationally, which have been contaminated following a nuclear or radiological emergency. These GLs apply to food after reconstitution or as prepared for consumption, i.e. not to dried or concentrated foods, and are based on an intervention exemption level of 1 mSv in a year for members of the public (infant and adult)[6].

Thus, the test method can be adapted so that the characteristic limits, decision threshold, detection limit and uncertainties ensure that the radionuclide activity concentrations test results can be verified to be below the guidance levels required by a national authority for either planned/existing situations or for an emergency situation[5][6][7].

Usually, the test methods can be adjusted to measure the activity concentration of the radionuclide(s) in either wastewaters before storage or in liquid effluents before being discharged to the environment. The test results will enable the plant/installation operator to verify that, before their discharge, wastewaters/liquid effluent radioactive activity concentrations do not exceed authorized limits.

The test method(s) described in this document may be used during planned, existing and emergency exposure situations as well as for wastewaters and liquid effluents with specific modifications that could increase the overall uncertainty, detection limit and threshold.

The test method(s) may be used for water samples after proper sampling, sample handling and test sample preparation (see the relevant part of the ISO 5667 series).

An International Standard on a test method for total activity concentration of uranium isotopes in water samples is justified for test laboratories carrying out these measurements, required sometimes by national authorities, as laboratories may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

This document is one of a set of International Standards on test methods dealing with the measurement of the activity concentration of radionuclides in water samples.

Water quality — Uranium — Test method using alpha liquid scintillation counting

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document specifies the measurement method for the determination of total activity concentration of uranium isotopes in non-saline waters by extraction and liquid scintillation counting.

This method covers the measurement of soluble uranium isotopes in water in activity concentrations between approximately $2 \cdot 10^{-3}$ Bq/kg and 10 Bq/kg when analysing a 1 l test sample volume with a 60 000 s counting time with a typical alpha LSC instrument.

The ratio ^{234}U/^{238}U can also be determined. This method has not been tested for the measurement of other uranium isotopes.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 80000-10, *Quantities and units — Part 10: Atomic and nuclear physics*

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*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

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

3 Terms, definitions, symbols and units

For the purposes of this document, the terms, definitions, symbols and units given in ISO 80000-10, ISO/IEC Guide 98-3 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

m	mass of the test sample, in grams
$m_{\mathrm{S(U)}}$	mass of ^{236}U standard solution used for the preparation of the calibration sample, in grams
$m_{\mathrm{S(Sr)}}$	mass of ^{90}Sr standard solution used for the preparation of the calibration sample, in grams
A_{S}	activity per unit of mass of the ^{236}U standard solution at the measuring time, in becquerel per gram
r_{S}	count rate of the calibration sample in the alpha window, per second
r_0	blank sample count rate in the alpha window, per second
r_{g}	sample gross count rate in the alpha window, per second
ε	alpha efficiency
a	activity per unit of mass, in becquerel per gram
c_{A}	activity concentration, in becquerel per litre
t_0	blank sample counting time, in seconds
t_{g}	sample counting time, in seconds
t_{S}	calibration sample counting time, in seconds
$u(a)$	standard uncertainty associated with the measurement result; in becquerel per gram
$U(a)$	expanded uncertainty, calculated by $U = k\,u\,(c_{\mathrm{A}})$ with k= 1, 2,..., in becquerel per gram
a^*	decision threshold for the alpha activity, in becquerel per gram
$a^\#$	detection limit for the alpha activity, in becquerel per gram
$a^<, a^>$	lower and upper limits of the confidence interval, in becquerel per gram
$r_{\mathrm{s}\alpha,\beta}$	alpha emitter count rate in beta window
$r_{\mathrm{s}0,\beta}$	blank count rate in beta window
$r_{\mathrm{s}\alpha,\mathrm{T}}$	alpha emitter count rate in total (alpha+beta) window
$r_{\mathrm{s}\beta,\alpha}$	beta emitter count rate in alpha window
$r_{\mathrm{s}0,\alpha}$	blank count rate in alpha window
$r_{\mathrm{s}\beta,\mathrm{T}}$	beta emitter count rate in total (alpha+beta) window
τ_α	alpha spillover parameter
τ_β	beta spillover parameter

4 Principle

The water sample is first acidified with a fixed amount of nitric acid and then, if lower detection limits are required, concentrated by evaporation. Uranium isotopes are then extracted (liquid-liquid extraction) by a scintillation cocktail containing a complexing agent [bis(2-ethylhexyl) hydrogen phosphate, HDEHP] and counted by liquid scintillation. The total activity of uranium isotopes can be thus determined. ^{236}U standard solution is used for calibration (external standard).

Spectral deconvolution can be performed using a suitable software to assess the ^{234}U/^{238}U ratio.

5 Reagents and equipment

5.1 Reagents

All reagents shall be of recognised analytical grade and shall not contain any detectable alpha and beta activity.

5.1.1 Nitric acid, $c(HNO_3)$ = 15,8 mol/l, ρ = 1,42 g/ml, mass fraction $w(HNO_3)$ = 70 %.

5.1.2 Nitric acid, $c(HNO_3)$ = 0,7 mol/l, obtained by dilution of concentrated nitric acid.

5.1.3 Laboratory water, distilled or deionized (complying with grade 3 of ISO 3696).

Deionized water can contain detectable amounts of ^{222}Rn and short lived daughters. It is therefore strongly recommended to boil the water under vigorous stirring and let it stand for one day before its use. An alternative option is to purge it with nitrogen for about 1 h for 2 l volume.

5.1.4 Scintillation cocktail, to be prepared (see 7.1).

5.1.5 Ethanol, 95 %.

5.1.6 p-Xylene, 99 %, for spectroscopy or scintillation grade.

5.1.7 Bis(2-ethylhexyl) hydrogen phosphate (HDEHP), 95 %.

5.1.8 Naphthalene, 99 %, scintillation grade.

5.1.9 2-(4-Biphenylyl)-6-phenyl benzoxazole (PBBO), scintillation grade.

5.1.10 Radioactive standard solution.

Alpha and beta emitter standard solutions (respectively ^{236}U and ^{90}Sr/^{90}Y or other suitable beta emitter radionuclide) shall be provided with calibration certificates containing at least the activity concentration, measurement uncertainty and/or statement of compliance with an identified metrological specification.

5.2 Equipment

5.2.1 Balance.

5.2.2 Hot plate with magnetic stirrer and stirring bar.

5.2.3 pH meter.

5.2.4 Wide-mouth HDPE sample bottles, volumes between 100 ml and 500 ml.

5.2.5 Liquid scintillation counter, with α/β discrimination option, with thermostated counting chamber and preferably a commercially available low background counter to achieve better detection limits.

5.2.6 Polyethylene scintillation vials, PTFE coated, 20 ml.

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 exhibit a considerably higher background and generally degrade both α/β discrimination and α peaks resolution.

6 Sampling

Collect the sample in accordance with ISO 5667-1. Store the water sample (from 0,1 l to 1 l) in a plastic bottle in accordance with ISO 5667-3. Acidify the sample with HNO_3: if the sample is not supposed to be concentrated by evaporation, add 5 ml of HNO_3 to 95 g of sample; if pre-concentration has to be carried out, add 5 ml of HNO_3 to 1 000 g of sample (see 8.1 and 8.2). When necessary, carry out filtration immediately on collection and before acidification.

Acidification of the water sample minimizes the loss of radioactive material from solution by adsorption. If the filtration of the sample is required, the acidification is performed afterward, otherwise radioactive material already adsorbed on the particulate material can be desorbed. Acidified samples may be stored for two months before being analysed (ISO 5667-3).

7 Instrument set up and calibration

7.1 Preparation of extractant scintillation cocktail

To prepare 500 ml of cocktail, weigh in a beaker:

— Naphthalene	90,0 g;
— PBBO	2,0 g;
— HDEHP	25,0 g.

Add approximately 450 ml of xylene and stir for about 1 h with a magnetic stirrer. Transfer to a 500 ml tared flask, bring to volume with xylene, shake and filter, if necessary, with glass wool.

Transfer the cocktail to a dark bottle preferably equipped with a 10 ml dispenser.

7.2 Preparation of alpha emitter calibration source

Transfer an accurately known amount $m_{S(U)}$ of the ^{236}U standard solution into a beaker. Add 100 ml of 0,7 M HNO_3 and extract twice, in a separatory funnel, by two 10 ml portions of scintillation cocktail. Transfer both cocktail portions into a scintillation vial and purge gently with argon or nitrogen to remove traces of ^{222}Rn.

7.3 Preparation of beta emitter calibration source

Transfer an accurately known amount $m_{S(Sr)}$ of the ^{90}Sr standard solution at isotopic equilibrium with ^{90}Y into a beaker. Add 100 ml of 0,7 M HNO_3 and extract twice, in a separatory funnel, by two 10 ml portions of scintillation cocktail. Transfer both cocktail portions into a scintillation vial and purge gently with argon or nitrogen to remove traces of ^{222}Rn.

7.4 Optimization of counting conditions

Set the alpha and beta counting window so that the energies of all the alpha and beta emitters of interest are covered.

Count for an appropriate period the alpha and beta counting standards in alpha/beta-discrimination, under different discriminator settings.

Determine the alpha (τ_α) and beta (τ_β) spillover parameter for each discriminator setting, as shown by Formulae (1) and (2):

$$\tau_\alpha = \frac{r_{S\alpha,\beta} - r_{0,\beta}}{r_{S\alpha,T} - r_{0,T}} \qquad (1)$$

$$\tau_\beta = \frac{r_{S\beta,\alpha} - r_{0,\alpha}}{r_{S\beta,T} - r_{0,T}} \qquad (2)$$

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 standards).

An example of discriminator setting determination is given in Annex A.

NOTE Good alpha-beta separation ($\tau < 0{,}10$) can be achieved by using a scintillation cocktail which is non-miscible with water and which contains naphthalene.

It is possible to choose a working point such that τ_α is slightly higher than τ_β; this reduces both the detection efficiency and the background and minimizes the interference by any beta emitter. Since the same discriminator setting is used for both calibration and sample measurement, no correction for misclassification is necessary in the calculations.

7.5 Detection efficiency

The extraction efficiency and the counting efficiency are calculated together by extracting and counting a ^{236}U standard solution (7.2).

Determine the alpha efficiency, as shown by Formula (3):

$$\varepsilon = \frac{r_S - r_0}{A_S\, m_S} \qquad (3)$$

Acceptance limits for efficiency should be defined.

The term ε includes both counting and extraction efficiencies and usually ranges from 90 % to 100 %.

A more accurate estimate of efficiency may be obtained by preparing and measuring five or more calibration samples.

Efficiencies should be verified with a periodicity established by the laboratory and whenever changes in materials (e.g. scintillation cocktail), analyst or when maintenance operations are performed on the scintillation counter. A verification or a recalibration is necessary when requisites of instrument quality control (see Clause 11) are not met.

7.6 Blank sample preparation and measurement

Prepare the blank sample by the same procedure used for test samples (see Clause 8). Acidify 95 ml of laboratory water with approximately 5 ml of concentrated HNO_3 (final concentration 0,7 M) and extract it twice, in a separatory funnel, by two 10 ml portion of scintillation cocktail. Transfer both cocktail portions into a scintillation vial and purge gently with argon or nitrogen to remove traces of ^{222}Rn.

Acceptance limits for blank samples should be defined, also on the basis of the desired sensitivity. For this purpose the use of control charts is advisable[8].

It is recommended to count blank samples for the same time length as the usual samples.

Blank measurements should be performed with a periodicity established by the laboratory (e.g. monthly) and whenever changes in materials (e.g. scintillation cocktail batch) or when maintenance operations are performed on the scintillation counter. Verification or a recalibration is necessary when requisites of instrument quality control (see Clause 11) are not met.

8 Procedure

8.1 Without pre-concentration

Transfer a weighed aliquot (m) of the initial water sample (approximately 95 g) into a beaker. Acidify it with approximately 5 ml of concentrated HNO_3, a 0,7 M final concentration in HNO_3 is obtained.

Heat to approximately 80 °C under stirring for 30 min in a covered beaker to allow the dissolved ^{222}Rn to desorb. Let the sample cool down and weigh it again and add, if necessary, laboratory water to restore the initial weight.

8.2 Pre-concentration by evaporation

It is possible to apply a pre-concentration step by evaporation when soft waters are considered (e.g. dry residue < 500 mg/l, as most drinking waters) in order to decrease the detection limit of the method. Hard waters may give rise to salt precipitations which can be hardly completely dissolved.

Transfer a weighed aliquot (m) of the initial water sample (1 000 g approximately) into a beaker. Add approximately 5 ml of concentrated HNO_3 to achieve a concentration of 0,07 M.

Slowly evaporate the sample on a hot plate until a quantity of approximately 100 g. Let the sample cool down to room temperature. The resulting nitric acid concentration in the evaporated fraction should be 0,7 M.

No precipitation should be observable, otherwise direct counting (8.1) or smaller pre-concentration factors are to be applied.

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

8.3 Sample preparation

Transfer the heated or concentrated sample (approximately 100 ml) to a separatory funnel and extract it twice, in a separatory funnel, by two 10 ml portions of scintillation cocktail. Transfer both cocktail portions into a scintillation vial and purge gently with argon or nitrogen to remove traces of ^{222}Rn. Clean the exterior of the vial with ethanol and transfer it into the counting chamber.

8.4 Sample measurement

Count the sample using the chosen optimum counting conditions (see 7.4). The counting time depends on the sample count rate (r_g.) and also on precision and detection limit required.

9 Expression of results

9.1 Calculation of activity per unit of mass

Calculate the uranium activity per unit of mass, a, of the test water sample by Formula (4):

$$a = \frac{r_g - r_0}{\varepsilon\, m} = (r_g - r_0)\, w \quad \text{with} \quad w = \frac{1}{\varepsilon\, m} \tag{4}$$

If the result has to be expressed in activity concentration c_A (Bq per unit volume) then the initial result expressed in becquerel per unit of mass shall be multiplied by the density of the water sample.

9.2 Standard uncertainty

According to ISO/IEC Guide 98-3, the standard uncertainty of a is calculated by Formula (5):

$$u(a)=\sqrt{w^2\left[u^2\left(r_\mathrm{g}\right)+u^2\left(r_0\right)\right]+a^2\ u_\mathrm{rel}^2\left(w\right)}=\sqrt{w^2\ \left(r_\mathrm{g}/t_\mathrm{g}+r_0/t_0\right)+a^2\ u_\mathrm{rel}^2\left(w\right)} \tag{5}$$

where the uncertainty of the counting time is neglected.

The relative standard uncertainty of w is calculated by Formula (6):

$$u_\mathrm{rel}^2\left(w\right)=u_\mathrm{rel}^2\left(\varepsilon\right)+u_\mathrm{rel}^2\left(m\right) \tag{6}$$

The relative standard uncertainty of ε is calculated by Formula (7):

$$u_\mathrm{rel}^2\left(\varepsilon\right)=u_\mathrm{rel}^2\left(r_s-r_0\right)+u_\mathrm{rel}^2\left(A_s\right)+u_\mathrm{rel}^2\left(m_s\right)=\left(r_s/t_s+r_0/t_0\right)/\left(r_s-r_0\right)^2+u_\mathrm{rel}^2\left(A_s\right)+u_\mathrm{rel}^2\left(m_s\right) \tag{7}$$

Repeatability contribution to $u(\varepsilon)$ should also be taken into account. It can be calculated as standard deviation of five or more calibration samples prepared and measured under repeatability conditions (see 7.5).

Calculate the characteristic limits in accordance with ISO 11929 and $\tilde{u}\left(\tilde{a}\right)$, i.e. the standard uncertainty of a as a function of its true value, using Formula (8):

$$\tilde{u}\left(\tilde{a}\right)=\sqrt{w^2\cdot\left[\left(\tilde{a}/w+r_0\right)/t_g+r_0/t_0\right]+\tilde{a}^2\ u_\mathrm{rel}^2\left(w\right)} \tag{8}$$

9.3 Decision threshold

The decision threshold, a^*, is obtained from Formula (9) for $\tilde{a}=0$. This yields:

$$a^*=k_{1-\alpha}\ \tilde{u}\left(0\right)=k_{1-\alpha}w\sqrt{r_0/t_\mathrm{g}+r_0/t_0} \tag{9}$$

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

9.4 Detection limit

Calculate the detection limit, $a^\#$, (is in the definitions) using Formula (10):

$$a^\#=a^*+k_{1-\beta}\ \tilde{u}\left(a^\#\right)=a^*+k_{1-\beta}\sqrt{w^2\left[\left(a^\#/w+r_0\right)/t_\mathrm{g}+r_0/t_0\right]+a^{\#2}u_\mathrm{rel}^2\left(w\right)} \tag{10}$$

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

The detection limit can be calculated by solving Formula (10) for $a^\#$ or, more simply, by iteration with a starting approximation $a^\#$ = 2 a^*.

When taking $\alpha = \beta$ then $k_{1-\alpha} = k_{1-\beta} = k$, the solution of Formula (10) is given by Formula (11):

$$a^\#=\frac{2\,a^*+\left(k^2\ w\right)/t_\mathrm{g}}{1-k^2\ u_\mathrm{rel}^2\left(w\right)} \tag{11}$$

9.5 Confidence limits

Calculate in accordance with ISO 11929, the lower, $a^{<}$, and upper, $a^{>}$, limits of the confidence interval using Formulae (12) and (13):

$$a^{<} = a - k_{\mathrm{p}}\, u(a)\,;\, p = \omega \cdot (1 - \gamma/2) \tag{12}$$

$$a^{>} = a + k_{\mathrm{q}}\, u(a)\,;\, q = 1 - \omega \cdot \gamma/2 \tag{13}$$

where

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

$\omega = 1$ may be set if $a \geq 4\,u(a)$ then Formula (14) applies:

$$a^{<,>} = a \pm k_{1-\gamma/2} \cdot u(a) \tag{14}$$

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

9.6 Calculations using the concentration activity

The activity concentration may be calculated by multiplying the activity per unit of mass by the density ρ in gram per litre, as shown by Formulae (15) and (16):

$$c_A = \frac{r_{\mathrm{g}} - r_0}{\varepsilon\, m}\rho = \left(r_{\mathrm{g}} - r_0\right) w \quad \text{with} \quad w = \frac{\rho}{\varepsilon\ m} \tag{15}$$

$$u_{\mathrm{rel}}^2(w) = u_{\mathrm{rel}}^2(\varepsilon) + u_{\mathrm{rel}}^2(m) + u_{\mathrm{rel}}^2(\rho) \tag{16}$$

The uncertainty, the characteristics limits and the limits of the confidence interval may be calculated using the previous expression [Formulae (5), (9), (10), (12) and (13)] with Formulae (15) and (16).

10 Interference control

Thorium isotopes can be partially co-extracted with uranium. Thorium presence is shown by alpha peaks of ^{228}Th at the energy lines 5,32 MeV (68 %) and 5,26 MeV (32 %). As alpha emissions of ^{232}Th are not distinguishable from those of ^{238}U, the ^{232}Th contribution cannot be removed. When ^{228}Th alpha peaks are clearly visible and therefore thorium presence is demonstrated, an alternative method shall be used for uranium quantification (see e.g. ISO 13166[9]).

11 Quality control

Measurement methods shall be selected and associated procedures performed by suitably skilled staff under a quality assurance programme and control.

Confidence in the measurement results is maintained by regular use of certified reference materials and the participation in interlaboratory comparisons and proficiency testing (see ISO/IEC 17025).

Laboratory procedures shall ensure that laboratory and equipment contamination as well as cross sample contamination is avoided.

12 Test report

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

a) the test method used with reference to this document, i.e. ISO 13169;

b) all information necessary for complete identification of the sample;

c) units in which the results are expressed;

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

Complementary information can be provided such as:

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

f) decision threshold and the detection limit;

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

   1) when the activity per unit of mass, a , is compared with the decision threshold (see ISO 11929) the result of the measurement should be expressed as $\leq a^*$ when the result is less than the decision threshold;

   2) when the activity per unit of mass, a, is compared with the detection limit, the result of the measurement can be expressed as $\leq a^\#$ when the result is below the detection limit. If the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose;

h) mention of any relevant information likely to affect the results.

Annex A
(informative)

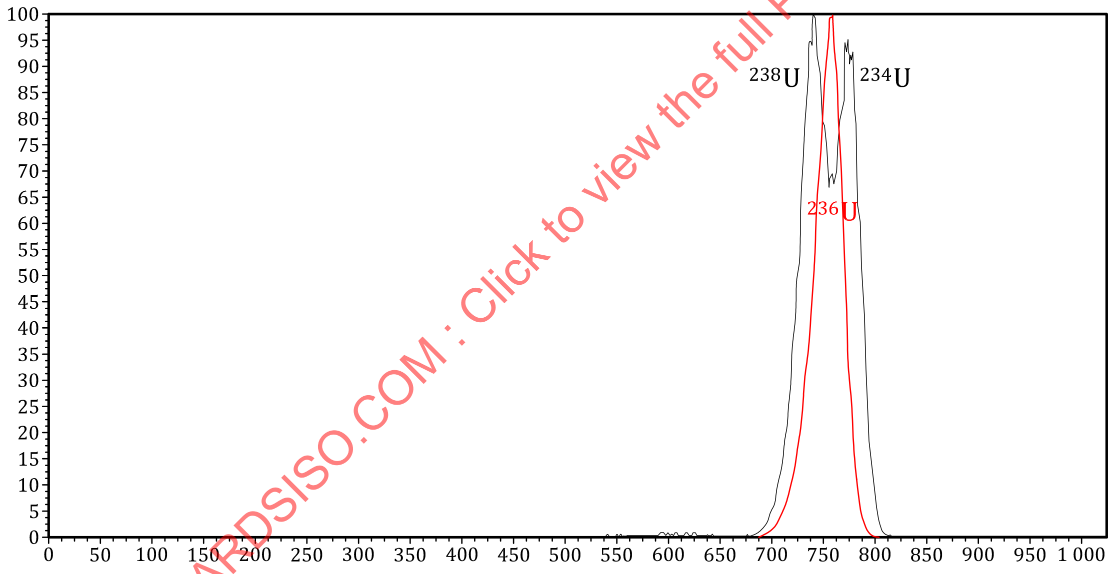
Set-up parameters and validation data

A.1 General

The following measurements have been performed by a Quantulus[1] 1220 (PerkinElmer) liquid scintillation counter. PTFE coated polyethylene vials Polyvials SLD[1] (Zinsser Analytic GmbH) and scintillation cocktail prepared as described in 7.1 have been used[10].

A.2 Instrument set up and calibration

Superimposed alpha spectra of natural uranium (^{234}U, ^{235}U and ^{238}U – green) and ^{236}U (red) are shown in Figure A.1. ^{235}U peak is not resolved. The scintillation cocktail used is described in 7.1.



Key

X channels

Y counts

Figure A.1 — Superposed LSC spectra of natural uranium and 236U

The spillover diagram obtained as described in 7.4 is reported in Figure A.2. Calibration sources of ^{236}U and ^{90}Sr/^{90}Y have been used. Counting window was (620 to 820) channels.

Alpha (τ_α) and beta (τ_β) spillover parameter are reported on y axis and the discrimination parameter (PSA) on x axis.

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[1] Quantulus and Polyvials SLD are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

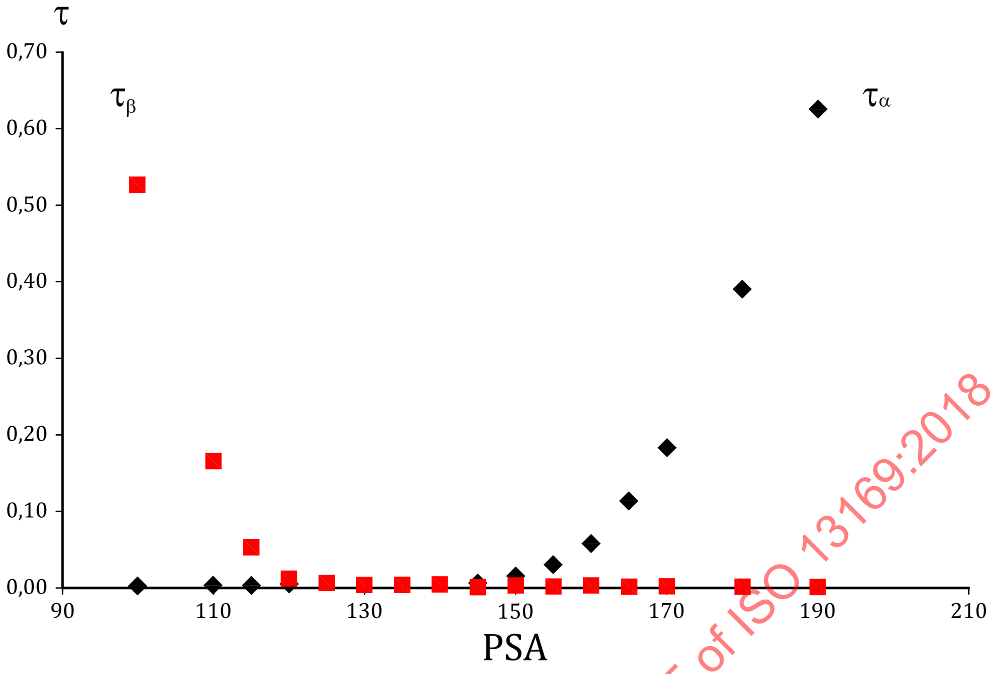

Figure A.2 — Spillover diagram

Overall efficiency was determined as described in 7.5. Results are reported in Table A.1.

Table A.1 — Calibration parameters

Calibration RN	Activity	Replicates	Average value ε	ε average combined uncertainty[a]	s(ε) – stand. dev.
^{236}U	10 Bq	3	0,994	0,024	0,011

[a] ε average combined uncertainty is the average of individual uncertainties of efficiency values. These combined uncertainties take into account uncertainties of counting, background and standard activity.

The variance of replicate measurement is not considered in the calculation of efficiency uncertainty since the method repeatability contribution is evaluated.

A.3 Expression of results

Decision threshold and detection limits calculated as in 9.3 and 9.4 are reported in Table A.2 for the above reported conditions (1,0 kg samples). The same parameters for non-concentrated samples (0,1 kg) are reported too. Above reported efficiency and blank values are used.

Table A.2 — Characteristic limits

Actual mass of test sample kg	Counting time min	Background counts	Background uncert. (k=1) counts	Decision threshold Bq/kg	Detection limit Bq/kg
0,1	1 000	125	21	$8 \cdot 10^{-3}$	$2 \cdot 10^{-2}$
1,0	1 000	125	21	$8 \cdot 10^{-4}$	$2 \cdot 10^{-3}$