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Water quality — Radium-226 —

Part 3:
Test method using coprecipitation and gamma-spectrometry

Qualité de l'eau — Radium 226 —

Partie 3: Méthode d'essai par coprécipitation et spectrométrie gamma

Reference number
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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

ISO 13165 consists of the following parts, under the general title *Water quality — Radium-226*:

- *Part 1: Test method using liquid scintillation counting*
- *Part 2: Test method using emanometry*
- *Part 3: Test method using coprecipitation and gamma-spectrometry*

Introduction

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

Natural radionuclides, including potassium-40, and those of the thorium and uranium decay series, in particular radium-226, radium-228, uranium-234, uranium-238, and lead-210, can be found in water for natural reasons (e.g. desorption from the soil and wash-off by rain water) or releases from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or phosphate fertilizer production and use).

Human-made radionuclides such as transuranium elements (americium, plutonium, neptunium, curium), tritium, carbon-14, strontium-90, and gamma emitters radionuclides can also be found in natural waters as they can be authorized to be routinely released into the environment in small quantities in the effluent discharge from nuclear fuel cycle facilities and following their use in unsealed form in medicine or industry. They are also found in the water due to the past fallout of the explosion in the atmosphere of nuclear devices and those following the Chernobyl and Fukushima accident.

Human-made radionuclides, such as transuranium elements (americium, plutonium, neptunium, curium), tritium, carbon-14, strontium-90, and some gamma emitting radionuclides, can also be found in natural waters as a result of authorized routine releases into the environment in small quantities in the effluent discharged from nuclear fuel cycle facilities. They are also released into the environment following their use in unsealed form for medical and industrial applications. They are also found in the water as a result of past fallout contamination resulting from the explosion in the atmosphere of nuclear devices and accidents such as those that occurred in Chernobyl and Fukushima.

Drinking water can thus contain radionuclides at activity concentration which could present a risk to human health. In order to assess the quality of drinking water (including mineral waters and spring waters) with respect to its radionuclide content and to provide guidance on reducing health risks by taking measures to decrease radionuclide activity concentrations, water resources (groundwater, river, lake, sea, etc.) and drinking water are monitored for their radioactivity content as recommended by the World Health Organization (WHO) and can be required by some national authorities.

The need of a standard on a test method of radium-226 activity concentrations in water samples is justified for test laboratories carrying out these measurements, required sometimes by national authorities, as they may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

Radium-226 activity concentration can vary widely according to local geological and climatic characteristics and ranges from 0,001 $\text{Bq}\cdot\text{l}^{-1}$ in surface waters up to 50 $\text{Bq}\cdot\text{l}^{-1}$ in natural groundwaters; the guidance level for radium 226 in drinking water as recommended by WHO is 1 $\text{Bq}\cdot\text{l}^{-1}$ (see Reference [13]).

NOTE The guidance level is the activity concentration (rounded to the nearest order of magnitude) with an intake of 2 $\text{l}\cdot\text{d}^{-1}$ of drinking water for 1 year that results in an effective dose of 0,1 $\text{mSv}\cdot\text{y}^{-1}$ for members of the public, an effective dose that represents a very low level of risk that is not expected to give rise to any detectable adverse health effect.

This International Standard is one of a series on determination of the activity concentration of radionuclides in water samples.

Water quality — Radium-226 —

Part 3:
Test method using coprecipitation and gamma-spectrometry

WARNING — Persons using this part of ISO 13165 should be familiar with normal laboratory practice. This part of ISO 13165 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this part of ISO 13165 be carried out by suitably qualified staff.

1 Scope

This part of ISO 13165 specifies the determination of radium-226 (^{226}Ra) activity concentration in all types of water by coprecipitation followed by gamma-spectrometry (see ISO 18589-3).

The method described is suitable for determination of soluble ^{226}Ra activity concentrations greater than 0,02 Bq l^{-1} using a sample volume of 1 l to 100 l of any water type.

For water samples smaller than a volume of 1 l, direct gamma-spectrometry can be performed following ISO 10703 with a higher detection limit.

NOTE This test method also allows other isotopes of radium, ^{223}Ra, ^{224}Ra, and ^{228}Ra, to be determined.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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: Preservation and handling of water samples*

ISO 10703, *Water quality — Determination of the activity concentration of radionuclides — Method by high resolution gamma-ray spectrometry*

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 80000-10, *Quantities and units — Part 10: Atomic and nuclear physics*

3 Terms and definitions

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

3.1
activity
number of spontaneous nuclear disintegrations occurring in a given quantity of material during a suitably small interval of time divided by that interval of time

[SOURCE: ISO 921:1997, 23]

3.2
reference standard
standard, generally having the highest metrological quality available at a given location or in a given organisation, from which measurements made there are derived

3.3
working standard
standard which, usually calibrated against a *reference standard* (3.2), is used routinely to calibrate or check material measures, measuring instruments or reference materials. It can be used as a solution of known *activity* (3.1) concentration obtained by precise dilution or dissolution of a reference standard

4 Symbols

For the purposes of this part of ISO 13165, the symbols defined in ISO 11929 and ISO 80000-10, and the following apply.

Table 1 — List of symbols

V	Volume of the test sample in litres
A	Activity of each radionuclide in calibration source, at the calibration time, in becquerels
c_A, c_{Ac}	Activity concentration of ^{226}Ra, without and with corrections, expressed in becquerels per litre
M_{sulfate}	Mass of barium sulfate, in grams
M_{nitrate}	Mass of barium nitrate, in grams
$M_{\text{carbonate}}$	Mass of barium carbonate, in grams
t_g	Sample spectrum counting time, in seconds
t_0	Background spectrum counting time, in seconds
t_s	Calibration spectrum counting time, in seconds
n_N, n_{N0}, n_{Ns}	Number of counts in the net area of the peak considered, at energy E, in the sample, background and calibration spectra, respectively
n_g, n_{g0}, n_{gs}	Number of counts in the gross area of the peak considered, at energy E, in the sample, background and calibration spectra, respectively
n_b, n_{b0}, n_{bs}	Number of counts in the background of the peak considered, at energy E, in the sample, background and calibration spectra, respectively
ε	Efficiency of the detector at energy E, at actual measurement geometry
R_C	Chemical yield
P	Probability of the emission of a gamma ray with energy, E, of each radionuclide, per decay, i.e. the branching ratio or the number of γ-quanta per decay
$u(c_A)$	Standard uncertainty of activity concentration, in becquerels per litre
$u(x)$	Standard uncertainty of measurand x
$u_{\text{rel}}(x)$	Relative uncertainty: $u(x)/x$
U	Expanded uncertainty calculated by $U = k\,u(c_A)$ with k usually equals 2, in becquerels per litre
c_A^*	Decision threshold, in becquerels per litre

Table 1 *(continued)*

$c_A^{\#}$	Detection limit, in becquerels per litre
$c_A^{\triangleleft}$, $c_A^{\triangleright}$	Lower and upper confidence limits, of each radionuclide, in becquerels per litre

5 Principle of the measurement

Measurement of ^{226}Ra activity concentration in water is carried out in two separate steps: a sample preparation step, followed by a measurement by gamma-spectrometry.[13][14][15]

The sample preparation consists of a filtration of the water sample (see ISO 5667-3) to ensure the measurement of the activity concentration of the ^{226}Ra soluble fraction. The filtration is followed by a pre-concentration, coprecipitation, and accumulation of decay products of ^{226}Ra.

After filtration and acidification of the water sample, the ^{226}Ra is coprecipitated into sulfate using a carrier such as barium nitrate or carbonate. The radium and barium sulfates are then washed, dried, and weighed. The chemical yield is obtained gravimetrically.

For water samples that may have an existing concentration of soluble barium, another tracer should be used to avoid a chemical yield above 100 %.

When a radiometric tracer, such as ^{133}Ba is used as an internal standard to assess the recovery yield, then this recovery yield is used instead of R_c in the Formulae (2) and (4).

The ^{226}Ra activity concentration of the precipitate is measured by gamma-spectrometry using a high purity germanium detector with low system background.

The presence of other gamma emitters, such as ^{235}U, in the precipitate can interfere with the quantification of ^{226}Ra activity.

The assessment of the soluble ^{226}Ra activity concentration is carried out by the measurement of its decay products, ^{214}Pb and ^{214}Bi, at equilibrium reached four weeks after the last coprecipitation step.

6 Reagents and equipment

6.1 Reagents

Unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity and no undesirable radioactivity.

6.1.1 Concentrated nitric acid solution (HNO_3), 65 % or 69 %.

6.1.2 Nitric acid solution (HNO_3), diluted to 2,28 %.

6.1.3 Nitric acid solution (HNO_3), diluted to 18 %.

6.1.4 Carrier solution, 10,0 $g \cdot l^{-1} \cdot Ba^{2+}$.

Slowly dissolve barium nitrate $Ba(NO_3)_2$ (19,0 g) or barium carbonate $BaCO_3$ (14,3 g) in one litre of nitric acid solution (6.1.2). The salt mass shall be weighed accurately (better than 1 %) and recorded.

It is recommended to use a $Ba(NO_3)_2$ or $BaCO_3$ reagent with a low ^{226}Ra activity concentration. The presence of ^{226}Ra in the reagent shall be controlled using a blank sample prepared with distilled or demineralized water.

6.1.5 Concentrated sulfuric acid solution (H_2SO_4), 98 %.

6.1.6 Cellulose ester filter of porosity, 0,45 µm.

6.2 Equipment

Usual laboratory equipment and, in particular, the following:

6.2.1 Peristaltic or membrane pump.

6.2.2 Magnetic bar stirrer.

6.2.3 Centrifuge and tubes.

6.2.4 Precision balance.

6.2.5 Drying oven.

6.2.6 Gas-tight container.

6.2.7 Gamma-spectrometry system, with low background capability.

7 Sampling

The method can be used on water samples up to 100 l.

7.1 Sample collection

The sampling conditions shall comply with ISO 5667-3.

If required, the filtration is carried out during or immediately after collection and before acidification, otherwise, radioactive material already adsorbed onto the suspended particulate material can be desorbed.

It is recommended that plastic single-use type containers are used for sample collection.

The volume of the water sample to collect for measurement depends on the detection limits required by the customer.

7.2 Sample transport and storage

The water sample shall be transported and stored according to ISO 5667-3.

When pre-concentration is desired, acidify the sample to between pH 1 and pH 3 with HNO_3. Acidification of the water sample minimizes the loss of radioactive material from solution by adsorption.

The test shall be performed as soon as possible.

8 Procedures

8.1 Blank sample preparation

Prepare a blank with distilled or demineralized water by performing the operations described in step a) to i) in 8.2.

8.2 Sample preparation

a) To prevent adsorption effects and proliferation of algae, the water sample is acidified to pH 1 with nitric acid (see 6.1.1). The water sample volume can be 1 l to 100 l. Acidification of the sample shall not be performed with hydrochloric acid as barium sulfate is partially soluble in it.

b) Pour dropwise, precisely, 50 ml of carrier solution (see 6.1.4) into the solution to be analysed, with vigorous stirring. In the case of natural water rich in sulfate ions, such as seawater, the barium and radium have to be mixed before the sulfate is precipitated.

c) Add 20 ml of concentrated H_2SO_4 (see 6.1.5) to precipitate the sulfate.

d) Stop mixing.

e) Leave the barium radium sulfate precipitate $Ba(Ra)SO_4$ to settle for about 24 h.

f) Separate most of the supernatant, e.g. using the pump, and centrifuge the remainder at about 2 000 rpm for about 5 min or filter to recover the precipitate.

g) Wash the precipitate with nitric acid (see 6.1.3) in the centrifuge tube, shake, and centrifuge again.

   Repeat steps f) and g) two times.

h) Eliminate the supernatant.

i) Rinse the precipitate with water (e.g. shaking and centrifuging each time).

j) Dry the precipitate in an oven at 105 °C for at least 24 h.

k) Weigh a gas-tight container.

l) Place the precipitate in the weighed gas-tight container and tightly seal to prevent any loss of radon (see ISO 18589-3).

m) Weigh the container.

8.3 Counting procedure

^{226}Ra activity is assessed by gamma-spectrometry using the detection of emissions of ^{214}Pb and/or ^{214}Bi following an ingrowth period of approximately 4 weeks, during which, ^{222}Rn reaches secular equilibrium with ^{226}Ra (see ISO 18589-3).

Calibration shall be performed using the same geometry as the one used to count the sample precipitate.

In the case of counting before the equilibrium is reached, a correction for the subsequent accumulation of the decay products has to be applied.

The ^{226}Ra activity concentration can be determined directly from its own energy emission at 186 keV, without the need for decay products ingrowth. The disadvantages of this technique relate primarily in that the emission probability of this gamma photopeak is weak (3,28 %), and furthermore by the presence of the interfering primary gamma emission of ^{235}U at 185,7 eV that has a higher emission probability of 57,24 %. Assuming the equilibrium between ^{226}Ra and ^{214}Bi, the theoretical detection limit of ^{226}Ra can be improved using the energy line of ^{214}Bi at 609 keV to measure the ^{226}Ra activity.

9 Quality assurance and quality control programme

9.1 General

Quality control operations shall meet the requirements of ISO/IEC 17025 and ISO 10703.

9.2 Influence quantities

Special precautions shall be taken to minimize the influence of quantities that can affect the measurement results:

- loss of ^{222}Rn from the container to be measured;
- ^{226}Ra present in a $Ba(NO_3)_2$ or $BaCO_3$ reagent. The presence of ^{226}Ra in reagent shall be assessed by measuring a blank sample;
- wide temporal variations of the radon activity concentration can be observed in the atmosphere of the laboratory. The continuous monitoring of the ^{222}Rn activity concentration in the laboratory using a radon measuring device is recommended, since even in well-ventilated rooms, considerable variations are possible in special weather conditions (see ISO 11665-1 and ISO 11665-5);
- to maintain a constant temperature and relative humidity, the air should be continuously renewed through an air-conditioning system. In order to limit the release of radon from material within the walls, the walls of the laboratory should be painted with a suitable material such as epoxy paint.[12]

A case may be added around the detector and filled with a radon-free gas (e.g. N_2, air passed through a granulated activated charcoal filter) in order to prevent any accumulation of radon in the vicinity of the detector.

9.3 Instrument verification

Major instrument parameters (energy calibration, efficiency calibration as a function of energy, and background spectrum) shall be periodically checked within a quality assurance programme established by the laboratory and following the manufacturer's instructions (see ISO 10703).

9.4 Method verification

Periodical verifications of the accuracy of the method shall be run. These may be accomplished by the following:

- participating in intercomparison exercises;
- analysing reference materials.

Method repeatability shall also be checked, e.g. by replicate measurements.

The acceptance limits of the tests mentioned in the preceding shall be defined.

9.5 Demonstration of analyst capability

If an analyst has not used this procedure before, a precision and bias test shall be performed by running a duplicate measurement of a reference or spiked material. Acceptance limits shall be defined by the laboratory.

A similar test shall be performed by analysts routinely using this procedure with a periodicity defined by the laboratory. Acceptance limits shall be defined.

10 Expression of results

10.1 General

Except for the chemical yield, R_C, computation for the activity concentration is developed in ISO 10703, and in this clause, only final formulae are presented.

10.2 Water-soluble ^{226}Ra activity concentration

The activity concentration measured by gamma-spectrometry, expressed in Bq·l^{-1}, has to be corrected for the barium sulfate or other precipitation yield (R_C) determination.

This yield is the ratio of the mass of separated barium sulfate to the mass of barium nitrate or barium carbonate introduced:

$$R_C = \frac{M_{\text{sulfate}}}{M_{\text{nitrate}}} \times 0{,}893 \quad \text{or } R_C = \frac{M_{\text{sulfate}}}{M_{\text{carbonate}}} \times 1{,}18 \tag{1}$$

where 0,893 and 1,18 are the molar mass ratios.

Thus, for an undisturbed peak at an energy, E, the activity concentration measured by gamma-spectroscopy is expressed in Bq·l^{-1} as shown in Formula (2):

$$c_A = \frac{n_g - n_b}{P \cdot \varepsilon \cdot V \cdot R_C \cdot t_g} = (n_g - n_b) \cdot w / t_g \quad \text{with } w = \frac{1}{P \cdot \varepsilon \cdot V \cdot R_C} \tag{2}$$

where ε is given by the calibration curve in the measurement set-up (see ISO 10703).

It should be considered that for the purpose of this part of ISO 13165, the correction factor, f_E, that appears in ISO 10703, is taken equal to 1 and the radiochemical yield, R_C, is taken into account.

10.3 Standard uncertainty of activity concentration

According to the *Guide to the Expression of Uncertainty in Measurement* [9], the combined uncertainty of c_A is calculated by Formula (3):

$$u(c_A) = \sqrt{(w / t_g)^2 \cdot (u^2(n_g) + u^2(n_b)) + c_A^2 \cdot u_{\text{rel}}^2(w)} \tag{3}$$

where the standard uncertainty of the counting time is neglected, and the relative standard uncertainty of w is calculated by Formula (4):

$$u_{\text{rel}}^2(w) = u_{\text{rel}}^2(P) + u_{\text{rel}}^2(V) + u_{\text{rel}}^2(\varepsilon) + u_{\text{rel}}^2(R_C) \tag{4}$$

Details about the calculation of $u_{\text{rel}}^2(\varepsilon)$ can be found in ISO 10703.

10.4 Decision threshold

In accordance with ISO 11929, the decision threshold, c_A^*, is obtained from the Formula (3) for $c_A = 0$. This yields the following:

$$c_A^* = k_{1-\alpha} \cdot w \cdot \sqrt{2} \cdot u(n_b) / t_g \tag{5}$$

where $\alpha = 0{,}05$ and then, $k_{1-\alpha} = 1{,}65$ is often chosen by default.

10.5 Detection limit

The detection limit, $c_A^\#$, is calculated according to ISO 11929 using the implicit Formula (6):

$$c_A^{\#} = c_A^{*} + k_{1-\beta} \cdot \sqrt{w^2 \left[(c_A^{\#} / w + n_b / t_g) / t_g + u^2(n_b) / t_g^2) + c_A^{\#2} \cdot u_{\mathrm{rel}}^2(w) \right]} \tag{6}$$

where $\beta = 0{,}05$ and then, $k_{1-\beta} = 1{,}65$ is often chosen by default.

The detection limit can be calculated by solving Formula (6) for $c_A^{\#}$ or, more simply, by iteration with a starting approximation, $c_A^{\#} = 2 \cdot c_A^{*}$.

When taking $k_{1-\alpha} = k_{1-\beta} = k$, the solution of Formula (6) is given by Formula (7):

$$c_A^{\#} = \frac{2c_A^{*} + \left(k^2 \,.\, w\right) / t_g}{1 - k^2 u_{\mathrm{rel}}^2 \left(w\right)} \tag{7}$$

10.6 Limits of the confidence interval

The lower, $C_A^{\triangleleft}$, and upper, $C_A^{\triangleright}$, limits of the confidence interval are calculated using the following formulae (see ISO 11929):

$$C_A^{\triangleleft} = C_A - k_p \cdot u\left(C_A\right) \ , \ p = \omega \cdot \left(1 - \gamma/2\right) \tag{8}$$

$$C_A^{\triangleright} = C_A + k_q \cdot u\left(C_A\right) \ , \ q = 1 - \omega \cdot \gamma/2 \tag{9}$$

where

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

$\omega = 1$ may be set if $C_A \geq 4 \times u\left(C_A\right)$. In this case,

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

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

10.7 Corrections for contributions from other radionuclides and background

10.7.1 General

If the determination of soluble ^{226}Ra activity concentration is determined directly from the gamma ray emissions of the ^{226}Ra, other gamma emitters such as ^{235}U can interfere. In this case, the contributing radionuclide has another gamma line from which the contribution to the line in question can be estimated, taking into account the branching ratios of the gamma lines.

If the determination of soluble ^{226}Ra activity concentration is estimated by the measurement of its decay products ^{214}Pb and ^{214}Bi at equilibrium, reached four weeks after the coprecipitation step, it should be taken into account that the gamma line of the radionuclide to be determined occurs also in the background of the spectrometer. By measuring a background spectrum without a sample for a counting time, t_0, this contribution can be corrected for, taking into account the different counting times of the two spectra.

For both cases, the activity concentration can be calculated using a model according to Formula (10):

$$c_{Ac} = (n_N / t_g - x \cdot n_{\mathrm{N0}} / t_0) \cdot w \tag{10}$$

For the both cases of contribution, this model gives the necessary corrections, which are developed in ISO 10703.

10.7.2 Contribution from other radionuclides

The gamma line to be corrected at the energy, E_1, has the net peak area n_{N,E_1}. The contribution of another radionuclide having more than one gamma line at gamma energy, E_1, is calculated through the ratio of the respective emission probability, deducted from the gamma line, E_2. Using Formula (10) with $x = P_{E_1} / P_{E_2}$ and $t_0 = t_g$, this yields to:

$$c_{Ac} = (n_{N,E_1} - x \cdot n_{N,E_2}) \cdot w / t_g \tag{11}$$

Neglecting the standard uncertainty of *x*, the standard uncertainty of c_{Ac} is calculated by Formula (12):

$$u^2(c_{Ac}) = (w / t_g)^2 \left\{ n_{g,E_1} + u^2(n_{b,E_1}) + x^2 \left[n_{g,E_2} + u^2(n_{b,E_2}) \right] \right\} + c_{Ac}^2 \cdot u_{\mathrm{rel}}^2(w) \tag{12}$$

The decision threshold, c_{Ac}^*, is given by Formula (13):

$$c_{Ac}{}^* = k_{1-\alpha} \cdot (w / t_g) \cdot \sqrt{n_{b,E_1} + u^2(n_{b,E_1}) + x(n_{g,E_2} - n_{b,E_2}) + x^2 [n_{g,E_2} + u^2(n_{b,E_2})]} \tag{13}$$

and the detection limit, $c_{Ac}^\#$, by Formula (14):

$$c_{Ac}^\# = c_{Ac}^* + k_{1-\beta} \sqrt{(w / t_g)^2 (c_{Ac}^\# t_g / w + n_{b,E_1} + u^2(n_{b,E_1}) + x(n_{g,E_2} - n_{b,E_2}) + x^2 [n_{g,E_2} + u^2(n_{b,E_2})]) + c_{Ac}^{\#\,2} u_{\mathrm{rel}}^2(w)} \tag{14}$$

The detection limit can be calculated by solving Formula (14) for $c_{Ac}^\#$ or, more simply, by iteration with a starting approximation: $c_{Ac}^\# = 2 \cdot c_{Ac}^*$.

When taking $k_{1-\alpha} = k_{1-\beta} = k$, the solution of Formula (14) is given by Formula (15):

$$c_{Ac}^\# = \frac{2 \cdot c_{Ac}^* + \left(k^2 \cdot w\right) / t_g}{1 - k^2 \cdot u_{\mathrm{rel}}\left(w\right)} \tag{15}$$

10.7.3 Contribution from background

In this case, Formula (10) is used for the correction with $x = 1$ and $u(x) = 0$. n_{N0} is the net peak area of the gamma line in the background spectrum and t_0 the counting time of the background spectrum. This yields:

$$c_{Ac} = (n_N / t_g - n_{N0} / t_0) \cdot w \tag{16}$$

The standard uncertainty of c_{Ac} is calculated by Formula (17):

$$u^2(c_{Ac}) = w^2(n_g / t_g^2 + n_{g0} / t_0^2 + u^2(n_b) / t_g^2 + u^2(n_{b0}) / t_0^2) + c_{Ac}^2 \cdot u_{\text{rel}}^2(w) \tag{17}$$

The decision threshold, c_{Ac}^*, is given by Formula (18):

$$c_{Ac}^* = k_{1-\alpha} \cdot w \cdot \sqrt{[n_b + u^2(n_b)] / t_g^2 + [n_{g0} + u^2(n_{b0})] / t_0^2 + (n_{g0} - n_{b0}) / t_0 t_g} \tag{18}$$

and the detection limit, $c_{Ac}^\#$, by Formula (19):

$$c_{Ac}^\# = c_{Ac}^* + k_{1-\beta} \sqrt{w^2[\frac{c_{Ac}^\#}{t_g w} + (n_b + u^2(n_b)] / t_g^2 + [n_{g0} + u^2(n_{b0})] / t_0^2 + (n_{g0} - n_{b0}) / t_0 t_g) + c_{Ac}^{\#2} u_{\text{rel}}^2(w)} \tag{19}$$

The detection limit can be calculated by solving Formula (19) for $c_{Ac}^\#$ or, more simply, by iteration with a starting approximation: $c_{Ac}^\# = 2 \cdot c_{Ac}^*$.

When taking $k_{1-\alpha} = k_{1-\beta} = k$, the solution of Formula (19) is given by Formula (20):

$$c_{Ac}^\# = \frac{2 \cdot c_{Ac}^* + \left(k^2 \cdot w\right) / t_g}{1 - k^2 \cdot u_{\text{rel}}^2 \left(w\right)} \tag{20}$$

The limits of the confidence interval are calculated according to Formulae (8) and (9).

11 Expression of results

This test report shall meet the requirements of ISO/IEC 17025 and shall contain at least the following information:

a) the test method used, together with a reference to this part of ISO 13165, i.e. ISO 13165-3:2016;

b) a reference to the measuring and evaluation procedure implemented;

c) the identification of the sample;

d) the measurement date;

e) the units in which the results are expressed;

f) the test result, $C_A \pm u\left(C_A\right)$ or $C_A \pm U$, with the associated *k* value and reference date.

Complementary information can be provided, such as the following:

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

- the decision threshold and the detection limit; depending on the customer request, the following are the different ways to present the result:
    - when the ^{226}Ra activity concentration is compared with the decision threshold (see ISO 11929), the result of the measurement shall be expressed as $\leq c_A^*$ when the result is below the decision threshold;
    - when the ^{226}Ra activity concentration is compared with the detection limit, the result of the measurement can be expressed as 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;
- mention of any relevant information likely to affect the results.

The test report can be completed by any useful information according to ISO/IEC 17025.

Annex A
(informative)

Uranium and its decay chain

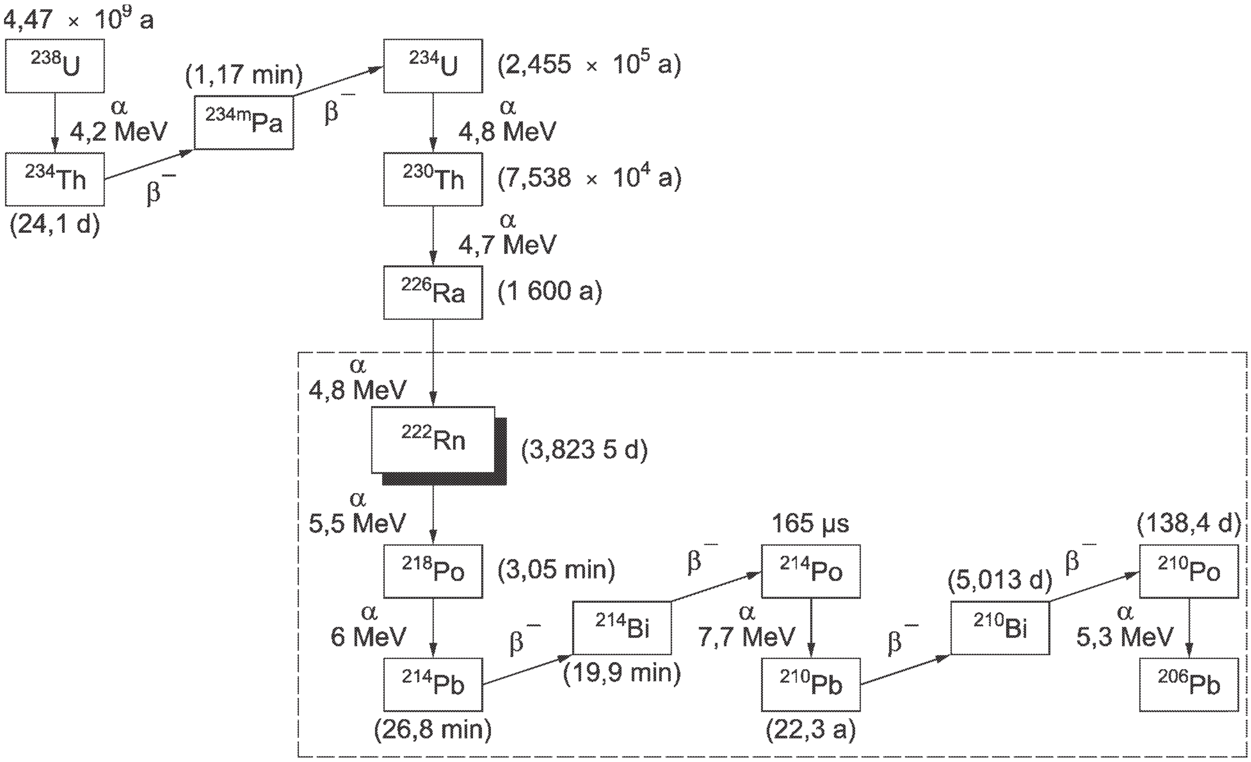

Figure A.1 — Uranium and its decay chain [16]

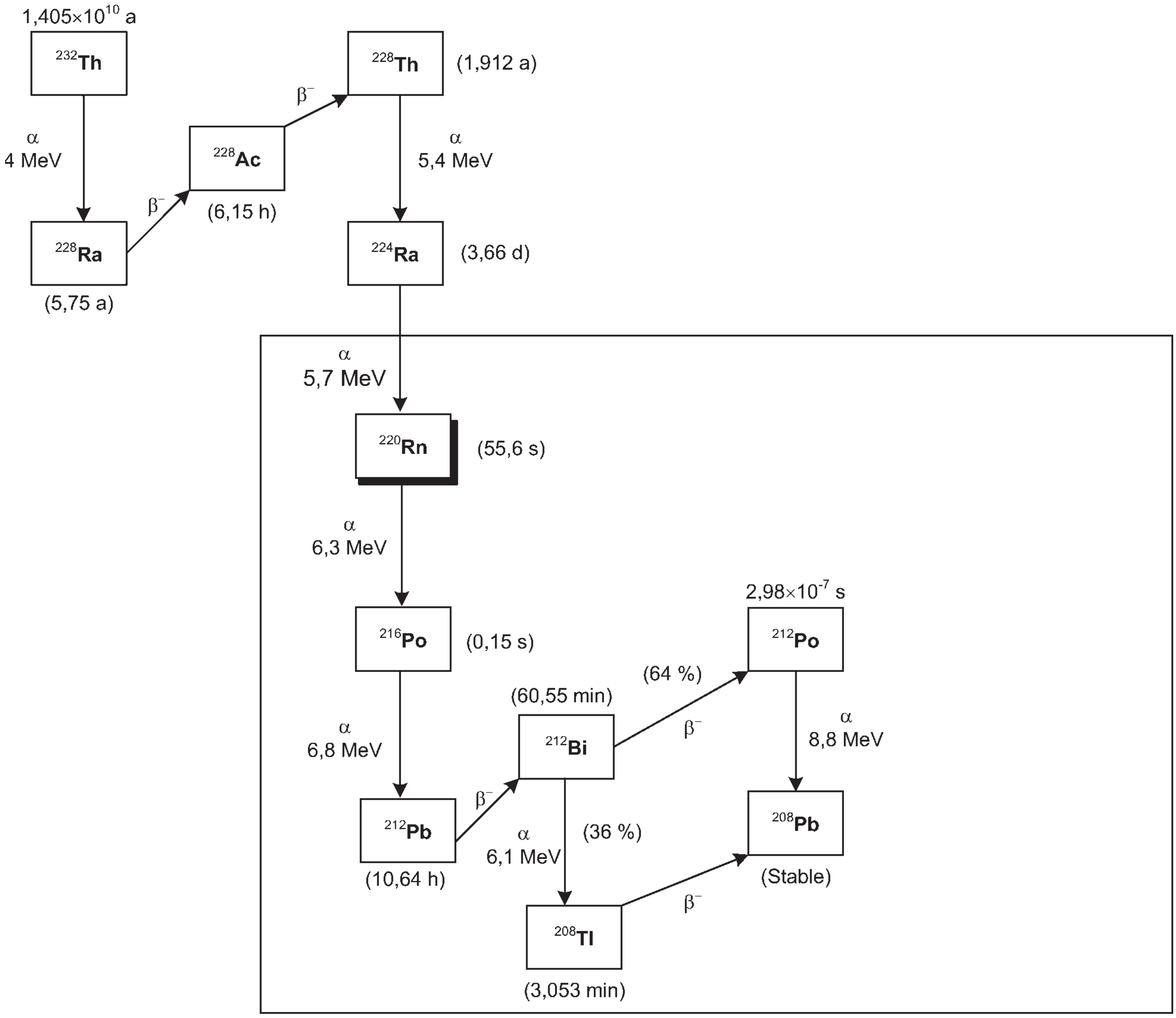

Figure A.2 — Thorium 232 and its decay chain [16]

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1) Withdrawn. Replaced by ISO 80000-10.

2) Withdrawn standard.

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