



BSI Standards Publication

Water quality - Gross alpha and gross beta activity - Test method using thin source deposit

National foreword

This British Standard is the UK implementation of EN ISO 10704:2019. It is identical to [ISO 10704:2019](#). It supersedes [BS EN ISO 10704:2015](#), which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/3/8, Radioactivity measurements methods.

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.

© The British Standards Institution 2019
Published by BSI Standards Limited 2019

ISBN 978 0 580 96044 4

ICS 13.280; 13.060.60

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 April 2019.

Amendments/corrigenda issued since publication

Date	Text affected
------	---------------

EUROPEAN STANDARD

EN ISO 10704

NORME EUROPÉENNE

EUROPÄISCHE NORM

March 2019

ICS 13.060.60; 13.280

Supersedes EN ISO 10704:2015

English Version

Water quality - Gross alpha and gross beta activity - Test method using thin source deposit (ISO 10704:2019)

Qualité de l'eau - Activités alpha globale et bêta globale - Méthode d'essai par dépôt d'une source fine (ISO 10704:2019)

Wasserbeschaffenheit - Bestimmung der Gesamt-Alpha- und der Gesamt-Beta-Aktivität in nicht-salzhaltigem Wasser - Dünnschichtverfahren (ISO 10704:2019)

This European Standard was approved by CEN on 16 February 2019.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Page

Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms, definitions and symbols	2
4 Principle	3
5 Chemical reagents and equipment	3
5.1 Reagents.....	3
5.1.1 General.....	3
5.1.2 Standard solutions.....	3
5.1.3 Wetting or surfactant agents.....	4
5.1.4 Volatile organic solvents.....	4
5.1.5 Water.....	4
5.1.6 Specific reagents for alpha-emitting radionuclides co-precipitation.....	4
5.2 Equipment.....	4
5.2.1 Laboratory equipment for direct evaporation.....	4
5.2.2 General equipment.....	4
5.2.3 Special equipment for alpha-emitting radionuclide co-precipitation.....	4
5.2.4 Measurement equipment.....	5
6 Sampling	5
7 Procedure	5
7.1 Preliminary.....	5
7.2 Source preparation.....	5
7.2.1 Preparation of planchet.....	5
7.2.2 Evaporation.....	6
7.2.3 Co-precipitation.....	6
7.3 Counting stage.....	7
7.4 Background and blank determination.....	7
7.5 Preparation of counting standard for calibration.....	7
7.6 Preparation of calibration source for self-absorption determination.....	8
7.6.1 General.....	8
7.6.2 Spiking one of two test portions.....	8
7.6.3 Self-absorption curve.....	8
8 Expression of results	9
8.1 General.....	9
8.2 Alpha activity concentration.....	9
8.3 Beta activity concentration.....	9
8.4 Standard uncertainty of the alpha activity concentration.....	10
8.5 Standard uncertainty of the beta activity concentration.....	10
8.6 Decision threshold.....	12
8.6.1 Decision threshold of the alpha activity concentration.....	12
8.6.2 Decision threshold of the beta activity concentration.....	12
8.7 Detection limit.....	12
8.7.1 Detection limit of the alpha activity concentration.....	12
8.7.2 Detection limit of the beta activity concentration.....	13
8.8 Confidence limits.....	13
9 Control of interferences	13
9.1 General.....	13
9.2 Relative humidity.....	14
9.3 Geometry of the deposit.....	14
9.4 Crosstalk.....	14

9.5	Gamma emitters.....	15
9.6	Low beta energy.....	15
9.7	Chlorides	15
9.8	Organic matter	15
9.9	Contamination.....	15
9.10	Losses of activity	15
9.11	Contribution of the natural radionuclides	15
9.12	Losses of activity	16
10	Test report.....	16
	Annex A (informative) Numerical applications.....	18
	Bibliography.....	19

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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: 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.

This second edition cancels and replaces the first edition ([ISO 10704:2009](http://www.iso.org/iso/10704:2009)), which has been technically revised. The main changes compared to the previous edition are as follows:

- Introduction: an introduction has been added;
- [Clause 1](#): the scope has been modified to specify applicability to emergency situations and applicability of waste water as a test sample; information about the exclusion of low energy beta emitters has also been added;
- [Clause 4](#): the filtration has been specified to be carried out at 0,45 µ;
- [5.1.2.2](#): ¹³⁷Cs has been introduced as a standard that can be used;
- [5.2.4](#): the recommended thickness has been increased to up to 400 µg/cm²;
- [7.6.3.1](#): in order to evaluate self-absorption phenomena, spiking method has been recommended to mimic the nature of the salt;
- [Clause 8](#):
 - a new [Formula \(9\)](#) has been introduced to obtain the beta activity concentration when systematic correction is not required;
 - the subsequent Formulae have been renumbered;
- [Clause 9](#): several limitations and interferences have been given;
- [9.1](#): the natural radionuclides contributions have been given.

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 ^{40}K , ^3H , ^{14}C , and those originating from the thorium and uranium decay series, in particular ^{226}Ra , ^{228}Ra , ^{234}U , ^{238}U , ^{210}Po and ^{210}Pb 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 , ^{14}C , ^{90}Sr , 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 can 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 in drinking water is 0,5 Bq/l for gross alpha activity and 1 Bq/l for gross beta activity.

NOTE 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].

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 can 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) can 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 of gross alpha and gross beta activity concentrations in water samples is justified for test laboratories carrying out these measurements, required sometimes by national authorities, as laboratories might 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 - Gross alpha and gross beta activity - Test method using thin source deposit

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 according to this document be carried out by suitably trained staff.

1 Scope

This document specifies a method for the determination of gross alpha and gross beta activity concentration for alpha- and beta-emitting radionuclides. Gross alpha and gross beta activity measurement is not intended to give an absolute determination of the activity concentration of all alpha and beta emitting radionuclides in a test sample, but is a screening analysis to ensure particular reference levels of specific alpha and beta emitters have not been exceeded. This type of determination is also known as gross alpha and gross beta index. Gross alpha and gross beta analysis is not expected to be as accurate nor as precise as specific radionuclide analysis after radiochemical separations.

Maximum beta energies of approximately 0,1 MeV or higher are well measured. It is possible that low energy beta emitters can not be detected (e.g. ^3H , ^{55}Fe , ^{241}Pu) or can only be partially detected (e.g. ^{14}C , ^{35}S , ^{63}Ni , ^{210}Pb , ^{228}Ra).

The method covers non-volatile radionuclides, since some gaseous or volatile radionuclides (e.g. radon and radioiodine) can be lost during the source preparation.

The method is applicable to test samples of drinking water, rainwater, surface and ground water as well as cooling water, industrial water, domestic and industrial wastewater after proper sampling, sample handling, and test sample preparation (filtration when necessary and taking into account the amount of dissolved material in the water).

The method described in this document is applicable in the event of an emergency situation, because the results can be obtained in less than 1 h. Detection limits reached for gross alpha and gross beta are less than 10 Bq/l and 20 Bq/l respectively. The evaporation of 10 ml sample is carried out in 20 min followed by 10 min counting with window-proportional counters.

It is the laboratory's responsibility to ensure the suitability of this test method for the water samples tested.

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: Preservation and handling of water samples*

[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 80000-10](#), *Quantities and units — Part 10: Atomic and nuclear physics*

[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 and symbols

For the purposes of this document, the terms, definitions and symbols given in [ISO 80000-10](#) and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

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

V	volume of test sample	l
$\varepsilon_{\alpha}, \varepsilon_{\beta}$	counting efficiency for α and β , respectively	—
$\varepsilon_{a\alpha}, \varepsilon_{a\beta}$	counting efficiency of sample a for α and β , respectively	—
χ	alpha-beta crosstalk	—

4 Principle

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. In order to obtain a thin and homogeneous deposit directly on a planchet, the sample can be progressively evaporated to dryness at a temperature below about 85 °C. Alternatively, for the gross alpha determination, radionuclides can be concentrated via a co-precipitation, the filtered co-precipitate deposited on the planchet being measured[8].

When suspended matter is present, filtration through 0,45 µm filter media is required and the gross alpha and gross beta activity can also be determined for the material retained on the filter.

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

5 Chemical reagents and equipment

5.1 Reagents

5.1.1 General

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

5.1.2 Standard solutions

5.1.2.1 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 interferences of its x and γ emission.

NOTE A uranium compound of certified natural or known isotopic composition has one arguable advantage, in that its specific activity can be calculated from established physical constants and isotopic abundance data which are independent of the calibration procedures of a particular organization. However, a 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.

5.1.2.2 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 $^{90}\text{Sr}/\text{Y}$ in equilibrium or ^{137}Cs are commonly used.

5.1.3 Wetting or surfactant agents

5.1.3.1 Vinyl acetate.

5.1.4 Volatile organic solvents

5.1.4.1 Ethyl alcohol.

5.1.5 Water

5.1.5.1 Water, complying with the requirements of [ISO 3696](#), grade 3.

5.1.6 Specific reagents for alpha-emitting radionuclides co-precipitation

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

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

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

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

5.1.6.5 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 General equipment

5.2.2.1 Filters, of pore size 0,45 μm .

5.2.2.2 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.

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 used.

5.2.3 Special equipment for alpha-emitting radionuclide co-precipitation

5.2.3.1 Hot plate with stirring equipment.

5.2.3.2 Infrared lamp.

5.2.3.3 Vacuum filtration system.

5.2.3.4 Filters, of pore size 0,45 µm.

5.2.4 Measurement equipment

5.2.4.1 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 window-proportional counters (between 80 µg cm⁻² to 400 µg cm⁻²) 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](#). Additional information on sampling of different types of waters can be found in the relative parts of the ISO 5667 series[9][10][11][12][13][14][15][16].

The laboratory sample is not usually acidified as the test portion is directly evaporated on the planchet. Acidification minimizes 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 and, also, 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. For deposits of surface density below 5 mg cm⁻², self-absorption phenomena can be neglected for gross beta measurement except when using low energy beta emitter such as ¹³⁷Cs for calibration[17].

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

IMPORTANT — Due to the ingrowth of radon decay products 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.

7.2.1 Preparation of planchet

Degrease planchets ([5.2.2.2](#)) 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 desiccator 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 (5.2.2.1) with the planchet before use, and record the mass, m_{pf} .

Avoid reuse of planchets to minimize 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 [Formula \(1\)](#):

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

If hygroscopic salt deposit is expected the planchet can also be weighed at the end of the measurement.

To minimize 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 (5.1.3).

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 $7,0 \pm 0,5$.

Add 20 ml of sulfuric acid (5.1.6.3) and boil for 5 min on a hot plate while stirring.

At a temperature of approximately 50 °C, add 1 ml of the barium carrier solution (5.1.6.5) and stir for 30 min.

The barium sulfate precipitates.

Then add 1 ml of the iron carrier solution (5.1.6.4).

Adjust the pH with ammonium hydroxide, (5.1.6.1) drop by drop until iron(III) hydroxide precipitates.

Continue stirring for 30 min.

Filter (5.2.3.4) 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 [Formula \(2\)](#):

$$m_{\text{d}} = m_{\text{pfd}} - m_{\text{pf}} \quad (2)$$

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

7.3 Counting stage

Following evaporation ([7.2.2](#)) or co-precipitation ([7.2.3](#)), if the counting is not performed immediately the planchet with the deposit can be stored in a desiccator.

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.

The counting strategy depends on the objectives of the measurements and the regulator requirements. To monitor natural radionuclides ingrowth or decay (see [9.11](#)), counting procedures should be repeated periodically over a period of one month. If specific counting condition is applied, it is recommended to mention it in the report.

When the counting strategy is defined, the laboratory shall apply it systematically for comparison purposes.

7.4 Background and blank determination

Measure the background activity using a clean planchet ([5.2.2.2](#)) 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 ([5.2.2.2](#)) or filter ([5.2.3.4](#)) 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 ([5.1.2.1](#)) 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, as shown by [Formula \(3\)](#):

$$\varepsilon_{\alpha} = \frac{r_{\text{sa}} - r_{0\alpha}}{A} \quad (3)$$

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 [Formula \(4\)](#):

$$f_{a\alpha} = \frac{\varepsilon_{a\alpha}}{\varepsilon_{\alpha}} \quad (4)$$

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 $\varepsilon_{\beta} = \varepsilon_{a\beta}$ with ($f_{a\beta} = 1$).

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 (spiked test portion minus unspiked test portion) by the activity of sample a [Formula \(5\)](#):

$$\varepsilon_{a\alpha} = \frac{r_{a\alpha} - r_{g\alpha}}{A_a} \quad (5)$$

7.6.3 Self-absorption curve

7.6.3.1 Alpha self-absorption correction

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, $f_{a\alpha}$. 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 [\[18\]](#) to [\[24\]](#). Commonly used curve-fitting techniques can be employed to derive the function and associated uncertainty.

The use of gross alpha self-absorption curves prepared with salts that mimic as closely as possible the salt content and salt nature of the test sample, improves the accuracy of results [\[25\]](#)[\[26\]](#). That consideration apply typically for unknown sample, the evaluation of the self-absorption by the spiking method is recommended.

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.

7.6.3.2 Beta self-absorption correction

Beta self-absorption phenomena can be neglected for gross beta measurement when deposits of surface density is below 5 mg cm^{-2} except when using low energy beta emitter such as ^{137}Cs for calibration.

8 Expression of results

8.1 General

An example of parameter values obtained for a specific test that can be used to check the computation results obtained with the different formulae of this document is given in [Table A.1](#) of [Annex A](#).

8.2 Alpha activity concentration

The alpha activity concentration, c_A , can be obtained using [Formula \(6\)](#):

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

with

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

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

8.3 Beta activity concentration

The beta activity concentration, c_A , can be obtained using [Formula \(7\)](#):

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

with

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

and

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

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 [Formula \(8\)](#):

$$\chi = \frac{r_{s\alpha \rightarrow \beta}}{r_{s\alpha}} \quad (8)$$

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

Because the alpha-beta crosstalk is specific to the alpha standard used for the calibration, the systematic correction is not required, the beta activity concentration, c_A , can be obtained using [Formula \(9\)](#):

$$c_A = \frac{r_{g\beta} - r_{0\beta}}{V \varepsilon_\beta} = [r_{g\beta} - r_{0\beta}]w \quad (9)$$

If the laboratory neglect alpha-beta crosstalk, it has nevertheless to be aware of the orders of magnitude.

8.4 Standard uncertainty of the alpha activity concentration

As specified in ISO/IEC Guide 98-3 (see also [ISO 11929](#)), the standard uncertainty of c_A is calculated using [Formula \(10\)](#):

$$u(c_A) = \sqrt{w^2 \left[u^2(r_{g\alpha}) + u^2(r_{0\alpha}) \right] + c_A^2 u_{\text{rel}}^2(w)} = \sqrt{w^2 \left(\frac{r_{g\alpha}}{t_g} + \frac{r_{0\alpha}}{t_0} \right) + c_A^2 u_{\text{rel}}^2(w)} \quad (10)$$

As the uncertainty of the counting time and the co-precipitation stage are negligible in comparison with other sources of uncertainty^[7], it can be neglected and the relative standard uncertainty of w can be calculated using [Formula \(11\)](#):

$$u_{\text{rel}}^2(w) = u_{\text{rel}}^2(\varepsilon_\alpha) + u_{\text{rel}}^2(V) + u_{\text{rel}}^2(f_{a\alpha}) \quad (11)$$

and the relative standard uncertainty of ε_α is calculated using [Formula \(12\)](#):

$$u_{\text{rel}}^2(\varepsilon_\alpha) = u_{\text{rel}}^2(r_{s\alpha} - r_{0\alpha}) + u_{\text{rel}}^2(A_\alpha) = \frac{(r_{s\alpha}/t_s) + (r_{0\alpha}/t_0)}{(r_{s\alpha} - r_{0\alpha})^2} + u_{\text{rel}}^2(A_\alpha) \quad (12)$$

All the uncertainties related to the calibration source are included in $u_{\text{rel}}^2(A_\alpha)$, 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 (as specified in [ISO 11929](#)); the standard uncertainty of c_A as a function of its true value, calculated using [Formula \(13\)](#):

$$u(\tilde{c}_A) = \sqrt{w^2 \left[\frac{\tilde{c}_A/w + r_{0\alpha}}{t_g} + \frac{r_{0\alpha}}{t_0} \right] + \tilde{c}_A^2 u_{\text{rel}}^2(w)} \quad (13)$$

8.5 Standard uncertainty of the beta activity concentration

As specified in the ISO/IEC Guide 98-3 (see also [ISO 11929](#)), the standard uncertainty of c_A is calculated using [Formula \(14\)](#) to [\(16\)](#):

$$u(c_A) = \sqrt{w^2 \left[u^2(r_{g\beta}) + u^2(r_{0\beta}) + T(\chi) \right] + c_A^2 u_{\text{rel}}^2(w)} = \sqrt{w^2 \left[\frac{r_{g\beta}}{t_g} + \frac{r_{0\beta}}{t_0} + T(\chi) \right] + c_A^2 u_{\text{rel}}^2(w)} \quad (14)$$

with

$$T(\chi) = (r_{g\alpha} - r_{0\alpha})^2 u^2(\chi) + \chi^2 \left(\frac{r_{g\alpha}}{t_g} + \frac{r_{0\alpha}}{t_0} \right) \quad (15)$$

and

$$u(\chi) = \sqrt{\frac{\chi(\chi+1)}{r_{s\alpha} t_{s\alpha}}} \quad (16)$$

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 [Formula \(17\)](#):

$$u_{\text{rel}}^2(w) = u_{\text{rel}}^2(\varepsilon_{\beta}) + u_{\text{rel}}^2(V) \quad (17)$$

and the relative standard uncertainty of ε_{β} is calculated using [Formula \(18\)](#):

$$u_{\text{rel}}^2(\varepsilon_{\beta}) = u_{\text{rel}}^2(r_{s\beta} - r_{0\beta}) + u_{\text{rel}}^2(A_{\beta}) = \frac{(r_{s\beta}/t_s) + (r_{0\beta}/t_0)}{(r_{s\beta} - r_{0\beta})^2} + u_{\text{rel}}^2(A_{\beta}) \quad (18)$$

All the uncertainties related to the calibration source are included in $u_{\text{rel}}^2(A_{\beta})$, 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 (as specified in [ISO 11929](#)); the standard uncertainty of c_A as a function of its true value, calculated by [Formula \(19\)](#):

$$\tilde{u}(\tilde{c}_A) = \sqrt{w^2 \left[\frac{\tilde{c}_A/w + \chi(r_{g\alpha} - r_{0\alpha}) + r_{0\beta}}{t_g} + \frac{r_{0\beta}}{t_0} \right] + \tilde{c}_A^2 u_{\text{rel}}^2(w)} \quad (19)$$

If the alpha-beta crosstalk is neglected, as specified in ISO/IEC Guide 98-3 (see also [ISO 11929](#)), the standard uncertainty of c_A is calculated using [Formula \(20\)](#):

$$u(c_A) = \sqrt{w^2 \left[u^2(r_{g\beta}) + u^2(r_{0\beta}) \right] + c_A^2 u_{\text{rel}}^2(w)} = \sqrt{w^2 \left[\frac{r_{g\beta}}{t_g} + \frac{r_{0\beta}}{t_0} \right] + c_A^2 u_{\text{rel}}^2(w)} \quad (20)$$

As the uncertainty of the counting time and the co-precipitation stage are negligible in comparison with other sources of uncertainty^[Z] it can be neglected and the relative standard uncertainty of w can be calculated using [Formula \(21\)](#):

$$u_{\text{rel}}^2(w) = u_{\text{rel}}^2(w\varepsilon_{\beta}) + u_{\text{rel}}^2(V) \quad (21)$$

and the relative standard uncertainty of ε_{α} is calculated using [Formula \(22\)](#):

$$u_{\text{rel}}^2(\varepsilon_{\beta}) = u_{\text{rel}}^2(r_{s\beta} - r_{0\beta}) + u_{\text{rel}}^2(A_{\beta}) = \frac{(r_{s\beta}/t_s) + (r_{0\beta}/t_0)}{(r_{s\beta} - r_{0\beta})^2} + u_{\text{rel}}^2(A_{\beta}) \quad (22)$$

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

For the calculation of the detection limits, the term $u(\tilde{c}_A)$ is required (as specified in [ISO 11929](#)); the standard uncertainty of c_A as a function of its true value, calculated using [Formula \(23\)](#):

$$\tilde{u}(\tilde{c}_A) = \sqrt{w^2 \left[\frac{\tilde{c}_A/w + r_{0\beta}}{t_g} + \frac{r_{0\beta}}{t_0} \right] + \tilde{c}_A^2 u_{\text{rel}}^2(w)} \quad (23)$$

8.6 Decision threshold

8.6.1 Decision threshold of the alpha activity concentration

The decision threshold, c_A^* , is obtained from [Formula \(13\)](#) for $\tilde{c}_A = 0$ (see [ISO 11929](#)):

$$c_A^* = k_{1-\alpha} \tilde{u}(0) = k_{1-\alpha} w \sqrt{\frac{r_{0\alpha}}{t_g} + \frac{r_{0\alpha}}{t_0}} \quad (24)$$

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

8.6.2 Decision threshold of the beta activity concentration

The decision threshold, c_A^* , is obtained from [Formula \(19\)](#) for $\tilde{c}_A = 0$ (see [ISO 11929](#)). This yields:

$$c_A^* = k_{1-\alpha} \tilde{u}(0) = k_{1-\alpha} w \sqrt{\frac{\chi(r_{g\alpha} - r_{0\alpha}) + r_{0\beta}}{t_g} + \frac{r_{0\beta}}{t_0} + T(\chi)} \quad (25)$$

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

If the alpha-beta crosstalk is neglected, the decision threshold, c_A^* , is obtained from [Formula \(23\)](#) for $\tilde{c}_A = 0$ (see [ISO 11929](#)):

$$c_A^* = k_{1-\alpha} \tilde{u}(0) = k_{1-\alpha} w \sqrt{\frac{r_{0\beta}}{t_g} + \frac{r_{0\beta}}{t_0}} \quad (26)$$

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

8.7 Detection limit

8.7.1 Detection limit of the alpha activity concentration

The detection limit, $c_A^\#$, is calculated using [Formula \(27\)](#) (see [ISO 11929](#)):

$$c_A^\# = c_A^* + k_{1-\beta} \tilde{u}(c_A^\#) = c_A^* + k_{1-\beta} \sqrt{w^2 \left[\frac{c_A^\# / w + r_{0\alpha}}{t_g} + \frac{r_{0\alpha}}{t_0} \right] + c_A^{\#2} u_{\text{rel}}^2(w)} \quad (27)$$

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

The detection limit can be calculated by solving [Formula \(27\)](#) for $c_A^\#$ or, more simply, by iteration with a starting approximation $c_A^\# = 2c_A^*$.

When taking $\alpha = \beta$ then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of [Formula \(27\)](#) is given by [Formula \(28\)](#):

$$c_A^\# = \frac{2c_A^* + (k^2 w / t_g)}{1 - k^2 u_{\text{rel}}^2(w)} \quad (28)$$

8.7.2 Detection limit of the beta activity concentration

The detection limit, $c_A^\#$, is calculated using [Formula \(29\)](#) (see [ISO 11929](#)):

$$c_A^\# = c_A^* + k_{1-\beta} \tilde{u}(c_A^\#) = c_A^* + k_{1-\beta} \sqrt{w^2 \left[\frac{c_A^\# / w + \chi(r_{g\alpha} - r_{0\alpha}) + r_{0\beta}}{t_g} + \frac{r_{0\beta}}{t_0} + T(\chi) \right] + c_A^{\#2} u_{\text{rel}}^2(w)} \quad (29)$$

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

If the alpha-beta crosstalk is neglected, the detection limit, $c_A^\#$, is calculated using [Formula \(30\)](#) (see [ISO 11929](#)):

$$c_A^\# = c_A^* + k_{1-\beta} \tilde{u}(c_A^\#) = c_A^* + k_{1-\beta} \sqrt{w^2 \left[\frac{c_A^\# / w + r_{0\beta}}{t_g} + \frac{r_{0\beta}}{t_0} \right] + c_A^{\#2} u_{\text{rel}}^2(w)} \quad (30)$$

The detection limit can be calculated by solving [Formula \(29\)](#) or [\(30\)](#) for $c_A^\#$ or, more simply, by iteration with a starting approximation $c_A^\# = 2c_A^*$.

When taking $\alpha = \beta$ then $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of [Formula \(29\)](#) and [\(30\)](#) is given by [Formula \(31\)](#):

$$c_A^\# = \frac{2c_A^* + (k^2 w / t_g)}{1 - k^2 u_{\text{rel}}^2(w)} \quad (31)$$

8.8 Confidence limits

The lower, c_A^\triangleleft , and upper, c_A^\triangleright , confidence limits are calculated using [Formulae \(32\)](#) and [\(33\)](#) (as specified in [ISO 11929](#)):

$$c_A^\triangleleft = c_A - k_p u(c_A) p = \omega \left(1 - \frac{\gamma}{2} \right) \quad (32)$$

$$c_A^\triangleright = c_A + k_q u(c_A) q = 1 + \frac{\omega \gamma}{2} \quad (33)$$

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

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

$$c_A^{\triangleleft\triangleright} = c_A \pm k_{1-(\gamma/2)} u(c_A) \quad (34)$$

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

9 Control of interferences

9.1 General

The problems discussed below should be recognized as inherent interferences and limitations of the method.

9.2 Relative humidity

If the evaporated sample contains significant levels of hygroscopic material, then this may absorb moisture from the atmosphere, which increases the counting source mass and this, in turn, leads to inconsistent detection efficiencies for the source. Although the use of a desiccator may reduce this effect, the sample may still absorb significant amounts of moisture when it is removed from the desiccator and placed in the sample measurement system. The sample mass before and after the counting can be recorded.

To deal with this issue, several strategies are available. One is to spike the sample with internal standard and to count spiked and non-spiked planchets successively with the same detector; this method is reliant on the relative humidity remaining stable. Alternatively, both planchets may be counted using two detectors in parallel.

Additionally, the sample deposit may be stabilized by treating it with a polymer solution such as collodion or poly(vinyl acetate).

If the techniques described in [ISO 9696](#) and [ISO 9697](#) are used, then the hygroscopic deposit is transformed into a hydrophobic deposit by the sulfation stage. [ISO 11704](#) which is based on liquid scintillation counting, may also be used.

Whatever technique is used, the efficiency and crosstalk determined should be based on the sample configuration.

The counterpoint, dry samples that have obtained a static charge, may ionize the carrier gas in the gas proportional counting system yielding increased counts above the level of radioactivity that is actually present. Accumulation of static charge may occur when conditions of low humidity (<60 % relative humidity) exist in the laboratory. If such conditions exist, the static charge may be successfully removed by connecting a small wire to a grounded metal structure and touching the planchet with the wire before counting.

A usual method^[27] suggested to convert salts to oxides is by heating the sample intensely until it glows with a characteristic dull-red colour. This process is not recommended due to the potential radionuclides losses such as ¹³⁷Cs.

9.3 Geometry of the deposit

Non-uniformity of the sample residue in counting planchet interferes with the accuracy and precision of the method. The requirements also apply for calibration using self-absorption mass curves. For instance, sources made with calcium carbonate or sodium sulfate can induce homogeneous or non-homogenous deposit, respectively^{[28][29]}.

9.4 Crosstalk

In general, the determination of alpha activity in the presence of beta activity is not subject to significant interference. Although crosstalk cannot be eliminated when counting on the beta plateau, beta to alpha crosstalk can be minimized during set-up of the instrument by adjusting the alpha and beta discriminators. While minimizing crosstalk also minimizes corrections needed for most samples, it should be kept in mind that if there is a significant difference in alpha and beta activities significant crosstalk may still be present. In cases where unusually high ratios of beta activity to alpha activity are present in samples, determining the alpha activity of samples in a separate measurement from the beta by counting on the alpha plateau provides the most effective discrimination against beta-to-alpha crosstalk and the most accurate measurement of alpha activity in samples.

The issue of alpha to beta crosstalk is different. Decay of alpha emitting radionuclides is often accompanied by the emission of conversion electrons, auger electrons and X-rays, which produce pulses indistinguishable from beta pulses, thus causing significant alpha to beta crosstalk^[30]. Consequently, the crosstalk correction is only appropriate if the radionuclides assayed are exactly the same as used for calibration, otherwise an unpredictable bias can be expected for gross beta determination.

When the correction strategy is defined the laboratory shall apply it systematically for comparison purposes.

9.5 Gamma emitters

Moderate to high levels of gamma radiation can be misinterpreted as alpha or beta activity by gas-flow proportional counters. This interference can be minimized by using a guard detector and employing anti-coincident counting techniques.

9.6 Low beta energy

Proportional counters operated in the “windowed” mode configuration are limited to detecting (beta) particles with an energy of 0,1 MeV or greater. To detect “soft” betas, the counter needs to be operated in the “windowless” mode configuration.

9.7 Chlorides

If the sample is known or suspected to contain significant chloride (acid or salts), the chloride should be converted to nitrate before transferring to a stainless steel planchet. (Chlorides attack the stainless steel, depositing heavy metal ions on top of the sample, thereby increasing inelastic scattering. Subsequently, no correction can be made for this effect[27]).

9.8 Organic matter

If the sample contains organic matter, the evaporation residue can be inappropriate for counting. The sample can be treated with acids to oxidize organic matter[27]. The additional step may induce losses of ^{14}C . Another solution is to use liquid scintillation counter (see [ISO 11704](#)[31]).

9.9 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 the activity of the test sample.

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

9.10 Losses of activity

Volatile radioisotopes of C, ^3H , I, Po, and adsorbed Rn may be lost when samples are heated.

9.11 Contribution of the natural radionuclides

The most common natural radiological contaminants found in samples are the uranium isotopes (^{234}U , ^{235}U , and ^{238}U), the radium isotopes (^{224}Ra , ^{226}Ra , ^{228}Ra), lead isotope ^{210}Pb and polonium isotope ^{210}Po .

Isotopes of radon can be lost by volatilization during the evaporation step. Once the residue forms, ^{222}Rn and ^{220}Rn produced by ^{226}Ra and ^{224}Ra respectively are trapped in the residue[32][33].

The gross alpha and gross beta activity are differently affected.

- The contribution of ^{224}Ra decay products to the gross alpha activity decreases with the time between collection and analysis and becomes negligible (less than 2,5 %) at 21 days.
- The contribution of the ^{228}Ra decay products to the gross alpha activity increases with the time between collection and analysis and can become significant (9 %) after three months.

- The contribution of the ^{226}Ra decay products to the gross alpha activity increases with the time between preparation and analysis, becomes more than 16 % after one day and reaches 95 % after 16 days. It can show an ingrowth of four times the initial count.
- The contribution of the ^{210}Pb decay product ^{210}Po to the gross alpha activity increases with the time between collection and analysis, becomes more than 11 % after one month, and reaches 50 % after 5 months.
- The contribution of the ^{238}U decay products to the gross beta activity increases with the time between preparation and analysis becomes more than 2 % after one day and reaches 95 % after 3 months days.
- The contribution of the ^{226}Ra decay products to the gross beta activity increases with the time between preparation and analysis, becomes more than 16 % after one day and reaches 95 % after 16 days.
- The contribution of ^{224}Ra decay products to the gross beta activity decreases with the time between collection and analysis and becomes negligible (less than 2,5 %) at 21 days.
- The contribution of the ^{228}Ra decay product ^{228}Ac to the gross beta activity increases rapidly with the time between collection and analysis and reaches 93 % after one day.
- The contribution of the ^{210}Pb decay product ^{210}Bi to the gross beta activity increases with the time between collection and analysis, becomes more than 13 % after one day, and reaches 95 % after 22 days.

The problem is complicated when seasonal variations of the concentration of natural radionuclides are observed in the water sampled^[34].

When the correction strategy is defined, the laboratory shall keep it for comparison purposes.

9.12 Losses of activity

Volatile radioisotopes of C, ^3H , I, Po, and adsorbed Rn may be lost when samples are heated.

If an oxidation step is required, it may induce losses of ^{14}C .

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 volatilized^[35]. The problem can be solved by converted halide to nitrate^[27].

10 Test report

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

- a) a reference to this document, i.e. [ISO 10704:2019](#);
- 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 the following:

- 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:
- as recommended by [ISO 11929](#), the test result has to be compared with the decision threshold. When the activity concentration c_A is below or equal to the decision threshold, a contribution from the sample has not been observed and the result of the measurement should be expressed as $\leq c_A^*$;
 - if the customer requests that the activity concentration c_A is to be compared with the detection limit, then the result of the measurement can be expressed as $\leq c_A^\#$ when the result is below or equal to 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 document, or regarded as optional, together with details of any incident that may have influenced the results. When the time elapsed between collection of the sample and the sample preparation, and the time between the preparation of the sample and the count, significantly impact the final result, the procedure shall be specified. The crosstalk correction strategy shall also be specified.

Annex A (informative)

Numerical applications

Table A.1 — Example of parameter values obtained for a specific test that can be used to check the computation results obtained with the different formulae of this document

Parameter	Unit	Value	Parameter	Unit	Result
$r_{g\alpha}$	s ⁻¹	0,008 33	$C_{A\alpha}$	Bq.l ⁻¹	0,111
$r_{g\beta}$	s ⁻¹	0,100	$C_{A^*\alpha}$	Bq.l ⁻¹	0,034 9
$r_{0\alpha}$	s ⁻¹	0,001 67	$C_{A^\#\alpha}$	Bq.l ⁻¹	0,080 0
$r_{0\beta}$	s ⁻¹	0,041 7	$u(C_{A\alpha})$	Bq.l ⁻¹	0,024 5
t_g	s	7 200	ω		1,00
t_0	s	7 200	p		0,975
ε_α		0,300	q		0,975
ε_β		0,500	k_p		1,96
χ		0,080 0	k_q		1,96
$f_{a\alpha}$		0,500	$C_{A^<\alpha}$	Bq.l ⁻¹	0,063
$f_{a\beta}$		1,00	$C_{A^>\alpha}$	Bq.l ⁻¹	0,159
V	l	0,040 0	$C_{A\beta}$	Bq.l ⁻¹	0,289
W_α	l ⁻¹	16,7	$C_{A^*\beta}$	Bq.l ⁻¹	0,029 5
W_β	l ⁻¹	5,00	$C_{A^\#\beta}$	Bq.l ⁻¹	0,063 1
$u_{rel}(\varepsilon_\alpha)$		0,036 6	$u(C_{A\beta})$	Bq.l ⁻¹	0,039 1
$u_{rel}(\varepsilon_\beta)$		0,030 4	ω		1,00
$U_{rel}(f_{a\alpha})$		0,100	p		0,975
$U_{rel}(f_{a\beta})$		0,100	q		0,975
$u_{rel}(V)$		0,025	k_p		1,960
$u^2_{rel}(w_\alpha)$		0,012 0	k_q		1,960
$u^2_{rel}(w_\beta)$		0,011 5	$C_{A^<\beta}$	Bq.l ⁻¹	0,212
$r_{s\alpha}$	s ⁻¹	10	$C_{A^>\beta}$	Bq.l ⁻¹	0,366
t_{sa}	s	1 000	α, β, γ	%	5
$u(\chi)$		0,002 94	$k_{1-\alpha} = k_{1-\beta} = k$		1,645
$T(\chi)$	s ⁻²	1,08E-08	$k_{1-\gamma/2}$		1,96

For example, to calculate in Microsoft Excel®¹⁾:

$$\omega = \text{NORMSDIST}[u(c_A)/c_A], k_p = \text{NORMSINV}(p), k_q = \text{NORMSINV}(q)$$

1) Excel® is a trademark of Microsoft. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Bibliography

- [1] ICRP. (2007) Annals of the ICRP - Publication 103: The 2007 Recommendations of the International Commission on Radiological Protection Editor J. VALENTIN Published for The International Commission on Radiological Protection
- [2] IAEA. Environmental and Source Monitoring for Purposes of Radiation Protection. Safety Guide No. RS-G-1.8. International Atomic Energy Agency, Vienna, 2005
- [3] WHO. Guidelines for drinking-water quality. World Health Organization, Geneva, Third Edition, 2010
- [4] [ISO 5667-20](#), *Water quality — Sampling — Part 20: Guidance on the use of sampling data for decision making — Compliance with thresholds and classification systems*
- [5] ALINORM 06/29/12 (2006). Report of the 38th session of Codex Committee on Food Additives and Contaminants
- [6] FAO-WHO Codex Alimentarius. (2011) Fact Sheet on Codex Guideline Levels for Radionuclides in Foods Contaminated Following a Nuclear or Radiological Emergency - prepared by Codex Secretariat (2 May 2011)
- [7] IAEA GSG-2, *Criteria for use in preparedness and response for a nuclear or radiological emergency (Jointly sponsored by FAO, IAEA, ILO, OECD/NEA, PAHO, OCHA, WHO)*. International Atomic Energy Agency, Vienna, 2011
- [8] 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
- [9] [ISO 5667-4](#), *Water quality — Sampling — Part 4: Guidance on sampling from lakes, natural and man-made*
- [10] [ISO 5667-5](#), *Water quality — Sampling — Part 5: Guidance on sampling of drinking water from treatment works and piped distribution systems*
- [11] [ISO 5667-6](#), *Water quality — Sampling — Part 6: Guidance on sampling of rivers and streams*
- [12] [ISO 5667-7](#), *Water quality — Sampling — Part 7: Guidance on sampling of water and steam in boiler plants*
- [13] [ISO 5667-8](#), *Water quality — Sampling — Part 8: Guidance on the sampling of wet deposition*
- [14] [ISO 5667-10](#), *Water quality — Sampling — Part 10: Guidance on sampling of waste waters*
- [15] [ISO 5667-11](#), *Water quality — Sampling — Part 11: Guidance on sampling of groundwaters*
- [16] [ISO 5667-14](#), *Water quality — Sampling — Part 14: Guidance on quality assurance and quality control of environmental water sampling and handling*
- [17] KRIEGER H.L. Interim radiochemical methodology for drinking water. US Environmental Protection Agency 1976 EPA-600 /4-75-008 (Revised), pp 4-75 1-2
- [18] 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

- [19] JETER H.W. An extended range method for gross alpha-/beta-particle measurements in water samples. *Radioact. Radiochem.* 1998, **9** pp. 16–25
- [20] 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
- [21] 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
- [22] SEMKOW T.M., JETER H.W., PARSA B., PAREKH P.P., HAINES D.K., BARI A. Modeling of alpha mass-efficiency curve. *Nucl. Instrum. Methods Phys. Res. A.* 2005, **538** pp. 790–800
- [23] 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
- [24] 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
- [25] TUCKER B.J., & ARNDT S. Gross alpha analytical modifications that improve wastewater treatment compliance. *Waste Management Symposium* 2007
- [26] TIMON A.F., VARGAS M.J., SANCHEZ A.B.R., PEREZ J.T., SANCHEZ A.M. Influence of source composition and particle energy on the determination of gross alpha activity *Appl. Radiat. Isot.* 2013, 82(Issue 1), pp 376-381 ISSN: 0969-8043 Online ISSN: 1872-9800
- [27] BRUG W.P., & McCROAN K.D. Laboratory Method for Gross Alpha and Beta Activity Determination. RP710(a), DOE Method. 1997, pp. 1-21
- [28] RUANO , SANCHEZ A.B., MARTIN SANCHEZ A., de LA TORRE PÉREZ J. Gross alpha and beta indices in water samples revisited. *Appl. Radiat. Isot.* 2013, **81** pp. 136–139
- [29] VALENTIN C. P., KRATKY J., BRANDL A. Investigation of Alpha and Beta Self-Absorption Factors in the Calibration of Water Sample Measurements *Health Physics* **Vol 103** suppl 2 124-130
- [30] SEMKOW T.M., & PAREKH P.P. Principles of gross alpha and beta radioactivity detection in water. *Health Phys.* 2001, **81** (5) pp. 567–574
- [31] [ISO 11704](#), *Water quality — Measurement of gross alpha and beta activity concentration in non-saline water — Liquid scintillation counting method*
- [32] ARNDT M.F., *Evaluation of gross alpha and uranium measurements for MCL compliance.* Water Research Foundation (Denver, CO) for the US Environmental Protection Agency. 2010, pp. 1-22
- [33] Schumacher B., Griggs J., Askren D., Litman B., Shannon B., Mehrhoff M., Nelson A. Schultz „K , SCHULTZ M.K., *Development of rapid radiochemical method for gross alpha and gross beta activity concentration in flowback and produced waters from hydraulic fracturing operations.* US Environmental Protection Agency. 2014, EPA/600/R-14/107, pp. 1-21
- [34] PHROMMAVANH V. Study of uranium migration in environment: experimental approach and geochemical modelling. *Rapport CEA-R-6219.* 2009 pp. 1-218
- [35] BAGNALL K.W. *Chemistry of the rare radio elements: Polonium-actinium.* Butterworth, London. 1957, pp 177

This page deliberately left blank

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards-based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Copyright in BSI publications

All the content in BSI publications, including British Standards, is the property of and copyrighted by BSI or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use.

Save for the provisions below, you may not transfer, share or disseminate any portion of the standard to any other person. You may not adapt, distribute, commercially exploit, or publicly display the standard or any portion thereof in any manner whatsoever without BSI's prior written consent.

Storing and using standards

Standards purchased in soft copy format:

- A British Standard purchased in soft copy format is licensed to a sole named user for personal or internal company use only.
- The standard may be stored on more than 1 device provided that it is accessible by the sole named user only and that only 1 copy is accessed at any one time.
- A single paper copy may be printed for personal or internal company use only.
- Standards purchased in hard copy format:
- A British Standard purchased in hard copy format is for personal or internal company use only.
- It may not be further reproduced – in any format – to create an additional copy. This includes scanning of the document.

If you need more than 1 copy of the document, or if you wish to share the document on an internal network, you can save money by choosing a subscription product (see 'Subscriptions').

Reproducing extracts

For permission to reproduce content from BSI publications contact the BSI Copyright & Licensing team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email subscriptions@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Useful Contacts

Customer Services

Tel: +44 345 086 9001

Email (orders): orders@bsigroup.com

Email (enquiries): cservices@bsigroup.com

Subscriptions

Tel: +44 345 086 9001

Email: subscriptions@bsigroup.com

Knowledge Centre

Tel: +44 20 8996 7004

Email: knowledgecentre@bsigroup.com

Copyright & Licensing

Tel: +44 20 8996 7070

Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK