# INTERNATIONAL STANDARD

Second edition 2019-02

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

*Qualité de l'eau — Activités alpha globale et bêta globale — Méthode d'essai par dépôt d'une source fine* 

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<u>ISO 10704:2019</u> https://standards.iteh.ai/catalog/standards/sist/747d8da6-6d17-487f-b33adea2e71e7871/iso-10704-2019



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#### Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

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 <a href="https://www.iso.org/patents">www.iso.org/patents</a>).

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: <a href="https://www.iso.org/iso/foreword.ltml">www.iso.org/iso/foreword.ltml</a>.

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

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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 <u>www.iso.org/members.html</u>.

This second edition cancels and replaces the first edition (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;
- <u>Clause 1</u>: 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;
- <u>Clause 4</u>: the filtration has been specified to be carried out at  $0,45 \mu$ ;
- <u>5.1.2.2</u>: <sup>137</sup>Cs has been introduced as a standard that can be used;
- <u>5.2.4</u>: the recommended thickness has been increased to up to  $400 \,\mu\text{g/cm}^2$ ;
- <u>7.6.3.1</u>: in order to evaluate self-absorption phenomena, spiking method has been recommended to mimic the nature of the salt;
- <u>Clause 8</u>:
  - 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;
- <u>Clause 9</u>: several limitations and interferences have been given;
- <u>9.1</u>: 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 <sup>40</sup>K, <sup>3</sup>H, <sup>14</sup>C, and those originating from the thorium and uranium decay series, in particular <sup>226</sup>Ra, <sup>228</sup>Ra, <sup>234</sup>U, <sup>238</sup>U, <sup>210</sup>Po and <sup>210</sup>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), <sup>3</sup>H, <sup>14</sup>C, <sup>90</sup>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<sup>[1]</sup>. Drinking-water can thus contain radionuclides at activity concentrations which could present a risk to human health.

The radionuclides present in liquid effuents are usually controlled before being discharged into the environment<sup>[2]</sup> and water bodies. Drinking waters are monitored for their radioactivity as recommended by the World Health Organization (WHO)<sup>[3]</sup> 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<sup>[4]</sup>.

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<sup>[3]</sup>.

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<sup>[5][6][7]</sup>.

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.

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# 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 detected (e.g. <sup>3</sup>H, <sup>55</sup>Fe, <sup>241</sup>Pu) or can only be partially detected (e.g. <sup>14</sup>C, <sup>35</sup>S, <sup>63</sup>Ni, <sup>210</sup>Pb, <sup>228</sup>Ra). (standards.iteh.a)

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 <u>http://www.electropedia.org/</u>

Α	activity of the calibration source	Bq
Aa	activity spiked in sample a, prepared for self-absorption estimation purposes	Bq
$c_A$	activity concentration	Bq l-1
$c_A^*$	decision threshold Teh STANDARD PREVIEW	Bq l-1
$c_A^{\#}$	detection limit (standards.iteh.ai)	Bq l-1
$c_A^{\triangleleft}$ , $c_A^{\triangleright}$	ISO 10704:2019 lower and upper limits of the confidence interval 747d8da6-6d17-487f-b33a- dea2e71e7871/iso-10704-2019	Bq l-1
f <sub>aα</sub> , f <sub>aβ</sub>	self-absorption factor of sample a for $\alpha$ and $\beta$ , respectively	—
m <sub>d</sub>	mass of the deposit	mg
mp	mass of the planchet	mg
m <sub>pd</sub>	mass of the planchet and the deposit	mg
$m_{ m pf}$	mass of the planchet and the filter	mg
$m_{\rm pfd}$	mass of the planchet, the filter and the deposit	mg
r <sub>0α</sub> , r <sub>0β</sub>	background count rate from the $lpha$ and $eta$ windows, respectively	s <sup>-1</sup>
r <sub>aα</sub> , r <sub>aβ</sub>	self-absorption sample a count rate from the $lpha$ and $eta$ windows, respectively	s-1
r <sub>gα</sub> , r <sub>gβ</sub>	sample gross count rate from the $lpha$ and $eta$ windows, respectively	s <sup>-1</sup>
$r_{ m slpha}, r_{ m seta}$	calibration count rate from the $\alpha$ and $\beta$ windows, respectively	s-1
$t_0$	background counting time	S
tg	sample counting time	S
ts	calibration counting time	S
U	expanded uncertainty calculated by $U = k \cdot u(c_A)$ with $k = 1, 2,$	Bq l-1

$u(c_A)$	standard uncertainty associated with the measurement result	Bq l-1
V	volume of test sample	l
ε <sub>α</sub> , ε <sub>β</sub>	counting efficiency for $\alpha$ and $\beta$ , respectively	—
ε <sub>aα</sub> , ε <sub>aβ</sub>	counting efficiency of sample a for $\alpha$ and $\beta$ , 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<sup>[8]</sup>.

When suspended matter is present, filtration through 0,45  $\mu$ m 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 <u>ISO 10704:2019</u> https://standards.iteh.ai/catalog/standards/sist/747d8da6-6d17-487f-b33adea2e71e7871/iso-10704-2019

#### 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 <sup>241</sup>Am solutions and <sup>239</sup>Pu solutions. When <sup>239</sup>Pu is used, the presence of <sup>241</sup>Pu as an impurity shall be taken into account as it leads to growth of <sup>241</sup>Am in prepared standard solutions of sources. When <sup>241</sup>Am is used, take into account the interferences of its x and  $\gamma$  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 date 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, <sup>40</sup>K as potassium chloride, dried to constant mass at 105 °C, can be used. Standard solutions of artificial beta-emitting radionuclides <sup>90</sup>Sr/Y in equilibrium or <sup>137</sup>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,  $c(NH_4OH) = 6 \text{ mol}/2. REVIEW$
- 5.1.6.2 Nitric acid, concentrated, c(HN039=158mol/s.iteh.ai)
- - https://standards.iteh.ai/catalog/standards/sist/747d8da6-6d17-487f-b33a-
- **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.