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

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 11704:2010

<https://standards.iteh.ai/catalog/standards/sist/a82f7532-0f30-4c37-a221-99a698bddb9d/iso-11704-2010>



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 11704:2010

<https://standards.iteh.ai/catalog/standards/sist/a82f7532-0f30-4c37-a221-99a698bddb9d/iso-11704-2010>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2010

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Symbols, definitions and units	2
4 Principle.....	2
5 Reagents and equipment.....	3
6 Sampling.....	4
7 Procedure	4
7.1 Direct counting	4
7.2 Thermal preconcentration	5
7.3 Sample preparation	5
7.4 Liquid scintillation measurement	5
8 Expression of results	7
8.1 Calculation of activity per unit of mass	7
8.2 Standard uncertainty.....	8
8.3 Decision threshold	8
8.4 Detection limit.....	9
8.5 Confidence limits.....	9
8.6 Quality control	10
9 Interference control.....	10
9.1 Contamination	10
9.2 Ingrowth of radon.....	10
9.3 Loss of polonium.....	10
10 Test report.....	10
Bibliography.....	12

Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO 11704:2010](https://standards.iteh.ai/catalog/standards/sist/a82f7532-0f30-4c37-a221-99a698bdbb9d/iso-11704-2010)

<https://standards.iteh.ai/catalog/standards/sist/a82f7532-0f30-4c37-a221-99a698bdbb9d/iso-11704-2010>

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

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

IMPORTANT — It is absolutely essential that tests conducted according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method for the determination of gross alpha and gross beta activity in waters for radionuclides which are not volatile at 80 °C. Radon isotopes and their decay products of short half life are not included in the determination.

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

[ISO 11704:2010](https://standards.iteh.ai/catalog/standards/sist/a82f7532-0f30-4c37-a221-99a698bddb9d/iso-11704-2010)

<https://standards.iteh.ai/catalog/standards/sist/a82f7532-0f30-4c37-a221-99a698bddb9d/iso-11704-2010>

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

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

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

3 Symbols, definitions and units

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

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

4 Principle

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

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

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

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

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

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

5 Reagents and equipment

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

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

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

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

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

5.4 Volatile organic solvents. Methanol or ethanol.

5.5 Certified reference solutions. A calibration laboratory establishes traceability of its own calibration sources and measuring instruments to the International System of Units (SI) by means of an unbroken chain of calibrations or comparisons linking them to relevant certified reference solutions of the SI units of measurement. The link to the SI units may be achieved with respect to national certified reference materials. These may be primary realizations of the SI units, or agreed representations of SI units based on fundamental physical constants, or they may be secondary materials which are materials certified by another national metrology institute. When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability, and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or statement of compliance with an identified metrological specification.

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

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

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

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

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

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

NOTE A ^{90}Sr and ^{90}Y mixture or ^{40}K are commonly used. A potassium solution for atomic absorption spectrometry has one arguable advantage, in that its specific activity can be calculated from established physical constants and isotopic abundance data which are independent of the calibration procedures of a particular organization.

5.6 Equipment.

5.6.1 Analytical balance.

5.6.2 Hot plate with magnetic stirrer and stirring bar.

5.6.3 pH meter.

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

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

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

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

6 Sampling

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

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

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

7 Procedure

7.1 Direct counting

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

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

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

7.2 Thermal preconcentration

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

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

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

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

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

7.3 Sample preparation

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

$$m = \frac{m_1 m_3}{m_2} \quad (1)$$

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

7.4 Liquid scintillation measurement

7.4.1 Preparation of alpha and beta calibration sources

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

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

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