
**Water quality — Lead-210 — Test
method using liquid scintillation
counting**

*Qualité de l'eau — Plomb 210 — Méthode d'essai par comptage des
scintillations en milieu liquide*

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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. 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. www.iso.org/patents

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

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Introduction

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

- natural radionuclides, including potassium-40, and those originating from the thorium and uranium decay series, particularly radium-226, radium-228, uranium-234, uranium-238, and lead-210, can be found in water for natural reasons (e.g. desorption from the soil and wash-off by rain water) or can be released from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or the production and use of phosphate fertilizer);
- human-made radionuclides, such as transuranium elements (americium, plutonium, neptunium, curium), tritium, carbon-14, strontium-90, and gamma-emitting radionuclides, can also be found in natural waters as a result of authorized routine releases into the environment in small quantities of the effluent discharged from nuclear fuel cycle facilities. They are also released into the environment following their use in unsealed form for medical and industrial applications. They are also found in the water as a result of past fallout contamination resulting from the explosion in the atmosphere of nuclear devices and accidents such as those that occurred in Chernobyl and Fukushima.

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

An International Standard on a test method for lead-210 activity concentrations in water samples is justified for test laboratories carrying out these measurements, required sometimes by national authorities, as laboratories may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

Lead-210 activity concentration can vary according to local geological and climatic characteristics and usually ranges from 2 mBq·l⁻¹ to 300 mBq·l⁻¹ (References [12][13]). The guidance level for lead-210 in drinking water, as recommended by WHO, is 100 mBq·l⁻¹ (Reference [14]).

NOTE The guidance level is the activity concentration with an intake of 2 l·day⁻¹ of drinking water for 1 year that results in an effective dose of 0,1 mSv·year⁻¹ for members of the public, an effective dose that represents a very low level of risk that is not expected to give rise to any detectable adverse health effect.

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Water quality — Lead-210 — Test method using liquid scintillation counting

WARNING — Persons using ISO 13163 should be familiar with normal laboratory practice. ISO 13163 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 ISO 13163 be carried out by suitably trained staff.

1 Scope

ISO 13163 specifies the determination of lead-210 (^{210}Pb) activity concentration in samples of all types of water using liquid scintillation counting (LSC). For raw and drinking water, the sample should be degassed in order to minimize the ingrowth of ^{210}Pb from radon-222 (^{222}Rn).

Using currently available liquid scintillation counters, this test method can measure the ^{210}Pb activity concentrations in the range of less than $20 \text{ mBq}\cdot\text{l}^{-1}$ to $50 \text{ mBq}\cdot\text{l}^{-1}$. These values can be achieved with a counting time between 180 min and 720 min for a sample volume from 0,5 l to 1,5 l.

Higher ^{210}Pb activity concentrations can be measured by either diluting the sample or using smaller sample aliquots or both.

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, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

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

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*

3 Symbols

For the purposes of this document, the symbols and designations given in ISO 80000-10, ISO 11929, ISO/IEC Guide 98-3, and ISO/IEC Guide 99 and the following apply.

c_{coeff}	coefficient of ^{210}Bi ingrowth to equilibrium in the sample between the end of bismuth elution and time of counting
c_A	activity concentration in the sample, in becquerel per litre
c_{A0}	activity concentration of the standard, in becquerel per litre
c_A^*	decision threshold, in becquerel per litre
$c_A^\#$	detection limit, in becquerel per litre
$c_A^{\triangleleft}, c_A^{\triangleright}$	lower and upper limits of the confidence interval, in becquerel per litre
R_c	chemical yield
r_b	count rate of the reagent blank, in reciprocal second
r_g	sample count rate, in reciprocal second
r_s	calibration count rate, in reciprocal second
r_0	background count rate, in reciprocal second
S1	eluted solution containing lead
t_g	sample counting time, in second
t_s	calibration counting time, in second
t_0	background counting time, in second
U	expanded uncertainty, calculated by $U = ku(c_A)$ with $k = 1, 2, \dots$, in becquerel per litre
$u(c_A)$	standard uncertainty associated with the measurement result, in becquerel per litre
V	volume of the eluted phase, in litre
V_e	total volume of the test sample plus the carrier, in litre
V_s	volume of the standard test sample, in litre
V_{sample}	volume of the sample, in litre
V_1	volume of the aliquot from S1 for ^{210}Pb counting, in litre
V_2	volume of the aliquot from S1 for the determination of the chemical yield of lead, in litre
ε	detection efficiency related to ^{210}Pb
ρ	concentration of lead of the eluate, in milligram per litre
ρ_e	concentration of lead in the sample after the addition of the carrier, in milligram per litre

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4 Principle

^{210}Pb is a natural beta-emitting radionuclide with a maximum beta-energy of 63,9 keV and a half-life of 22,23 years (References [15][16]). It appears in the ^{238}U decay series ($4n+2$) as a long-lived decay product of ^{222}Rn (see Figure 1).

^{210}Pb is separated from its daughters, bismuth-210 and polonium-210, by extraction chromatography and its activity is measured by liquid scintillation counting, either directly after its separation or indirectly after ingrowth of its progeny bismuth-210. Other separation methods exist (Reference [17]).

To avoid the possible interferences of the isotopes lead-211 and lead-214 and their progenies during the liquid scintillation counting, it is recommended to wait at least 3 h between elution of lead and the sample counting to allow these radionuclides to fully decay.

For radioisotopes with longer half-lives such as lead-212 and its progenies, their interferences are avoided by choosing appropriate counting windows as their energies are much higher than the energy of ^{210}Pb (see 7.4.2).

For samples with high activity concentration, dilution of the sample is required to avoid resin and detector saturation during the separation and counting steps, respectively.

Suspended material is removed prior to analysis by filtration using 0,45 μm filters. The analysis of the insoluble fraction requires a mineralization step that is not covered by ISO 13163.

NOTE A suitable mineralization step is specified in ISO 18589-2.[10]

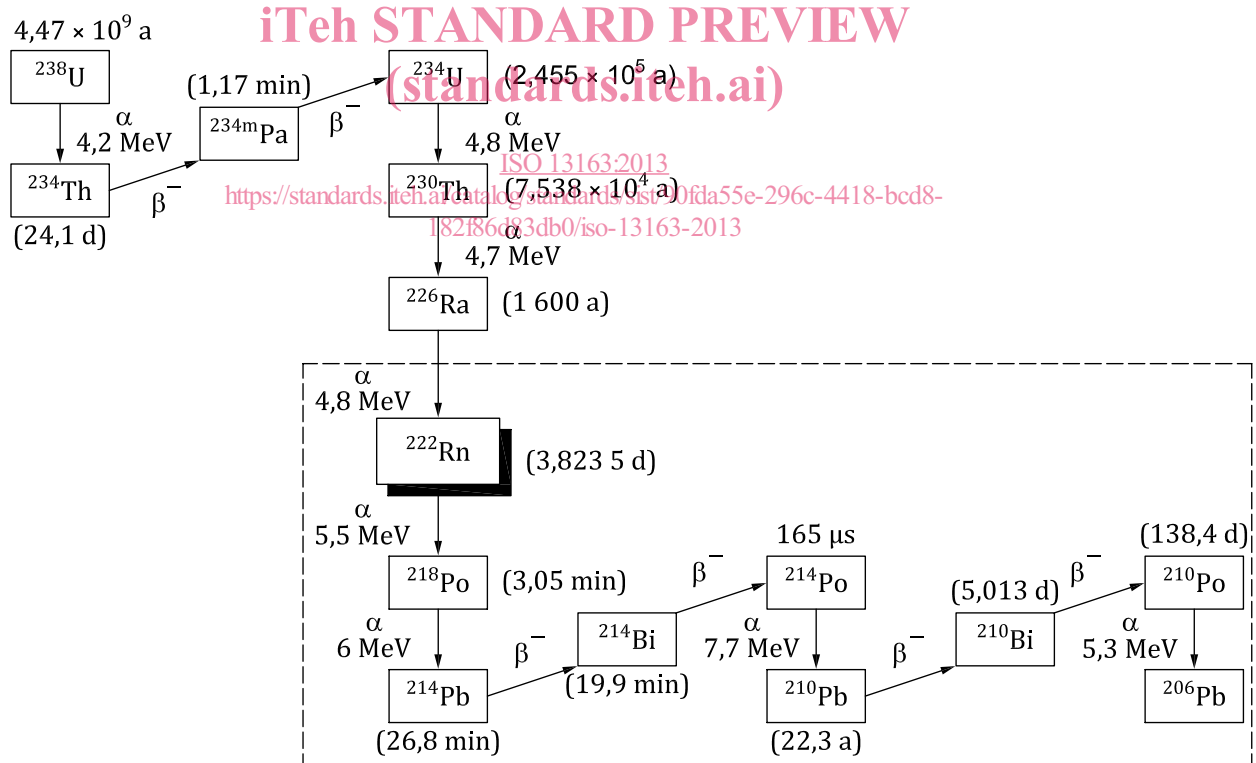


Figure 1 — Uranium-238 and its decay products (see ISO 13164-1)

It is necessary to know the concentration of the stable lead in the sample in order to determine the mass of the lead carrier to add and to calculate the chemical yield for the separation of ^{210}Pb .

It is possible to confirm the radiopurity of the ^{210}Pb fraction by monitoring ^{210}Bi ingrowth activity up to equilibration via repeated counting over an appropriate period of time.

5 Reagents and equipment

Use only reagents of recognized analytical grade.

5.1 Reagents

5.1.1 Nitric acid, HNO_3 , concentrated, i.e. $700 \text{ g}\cdot\text{l}^{-1}$.

5.1.2 Hydrochloric acid, HCl , concentrated, i.e. $370 \text{ g}\cdot\text{l}^{-1}$.

5.1.3 Hydrochloric acid solution, $2 \text{ mol}\cdot\text{l}^{-1}$ HCl .

5.1.4 Nitric acid solution, $1 \text{ mol}\cdot\text{l}^{-1}$ HNO_3 .

5.1.5 Nitric acid solution, $0,1 \text{ mol}\cdot\text{l}^{-1}$ HNO_3 .

5.1.6 Solution of Fe(III), approximately $1 \text{ g}\cdot\text{l}^{-1}$ in $0,1 \text{ mol}\cdot\text{l}^{-1}$ HNO_3 or $0,5 \text{ mol}\cdot\text{l}^{-1}$ HCl .

5.1.7 Standard solution of Pb(II), approximately $1 \text{ g}\cdot\text{l}^{-1}$ in $0,1 \text{ mol}\cdot\text{l}^{-1}$ HNO_3 or $2 \text{ mol}\cdot\text{l}^{-1}$ HCl .

5.1.8 Ammonia, NH_4OH , concentrated, e.g. $280 \text{ g}\cdot\text{l}^{-1}$.

5.1.9 Ammonium citrate or citric acid solution, $0,01 \text{ mol}\cdot\text{l}^{-1}$ to $0,1 \text{ mol}\cdot\text{l}^{-1}$ or **EDTA solution**, $0,01 \text{ mol}\cdot\text{l}^{-1}$.

5.1.10 Chromatographic extraction resin, e.g. a crown ether 18C6-type resin.

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5.1.11 Liquid scintillation cocktail, chosen according to the characteristics of the sample to be analysed and the properties of the detection equipment. The characteristics of the scintillation cocktail shall allow the mixture to be homogeneous and stable.

5.1.12 Laboratory water, distilled or deionized, complying with ISO 3696,^[1] grade 3.

Deionized water can contain detectable amounts of ^{222}Rn and its short-lived daughters. It is therefore strongly recommended that water be boiled under vigorous stirring and allowed to stand for 1 day before use; otherwise, degassing with nitrogen for about 1 h per 2 l is recommended.

All reagents shall be of high purity (containing no detectable lead) or with certified lead content. This is validated by performing regular reagent blank checks.

5.1.13 Radioactive solution, ^{210}Pb standard solution in equilibrium with ^{210}Bi for the determination of the counting yield in liquid scintillation.

5.1.14 Quenching agent, e.g. nitric acid, acetone, organochlorine compounds (e.g. chloroform), nitromethane. Any one of these quenching agents can be used.

CAUTION — Some quenching agents are dangerous or toxic.

5.2 Equipment

Usual laboratory equipment and in particular the following.

5.2.1 Centrifuge or vacuum filtration system.

5.2.2 Membrane filter, of pore size 0,45 µm.

5.2.3 Analytical balance, accuracy 0,1 mg.

5.2.4 Equipment for the measurement of stable lead, e.g. atomic absorption spectroscopy, ICP-MS, ICP-OES.

5.2.5 Beta-counter, liquid scintillation counter provided with a display system and facility for recording spectra.

5.2.6 Scintillation vials, e.g. of polyethylene, adapted to the liquid scintillation counter.

6 Sampling and storage

6.1 Sampling

It is important that the laboratory receive a representative sample, unmodified during transport or storage and in an undamaged container (see ISO 5667-3).

6.2 Sample storage

Samples shall be stored according to the general requirements of ISO 5667-3.

^{222}Rn in a sample at $100 \text{ Bq}\cdot\text{l}^{-1}$ will generate approximately $40 \text{ mBq}\cdot\text{l}^{-1}$ of ^{210}Pb for a storage time of 10 days. Thus, the storage time for ^{210}Pb shall be taken into consideration when the sample contains radon.

7 Procedure

The measurement is realized in the following three stages:

- stage 1: preconcentration of lead by co-precipitation with $\text{Fe}(\text{OH})_3$ prepared *in situ* (Reference [18]);
- stage 2: separation of lead on the extraction chromatographic resin (References [17][18][19][20][21][22][23]);
- stage 3: determination of the beta-activity of ^{210}Pb or its progeny, ^{210}Bi (Reference [24]).

The chemical yield of the separation is obtained by measuring the yield of the stable lead used as a carrier. It is thus necessary to take the following steps.

- Measure the original lead content in the sample to determine the quantity of the carrier to add.
- Measure the lead content of the aliquot loaded with the carrier before chemical separation.
- Measure the lead content in the final eluate to be used for the counting of ^{210}Pb in order to calculate the chemical yield.

The measurement of the stable lead for the determination of the chemical yield can be carried out according to various protocols already described in other International Standards. These protocols include the following:

- ICP-OES according to ISO 11885;[5]
- ICP-MS according to ISO 17294-2;[9]
- AAS according to ISO 15586.[8]

The beta-activity of ^{210}Pb is measured by liquid scintillation counting.