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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

Reference number
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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 www.iso.org/directives).

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For an explanation of 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 www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 13163:2013), which has been technically revised.

The main changes compared to the previous edition are as follows:

- addition of the common introduction;
- transfer of separation processes to an annex.

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.

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, ^{3}H, ^{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), ^{3}H, ^{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][2] Drinking-water can thus contain radionuclides at activity concentrations that could present a risk to human health.

The radionuclides present in liquid effluents are usually controlled before being discharged into the environment[3] and water bodies. Drinking waters are monitored for their radioactivity as recommended by the World Health Organization (WHO)[4] 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[5].

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[4] for guidance level in drinking water is 0,1 Bq·l^{-1} for ^{210}Pb activity concentration.

NOTE 1 The guidance level is the activity concentration with an intake of 2 l·d^{-1} of drinking water for one year that results in an effective dose of 0,1 mSv·a^{-1} 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[4].

In the event of a nuclear emergency, the WHO Codex Guideline Levels[6] mention that the activity concentration might not be greater than 0,1 Bq·l^{-1} for ^{210}Pb.

NOTE 2 The Codex guidelines levels (GLs) apply to radionuclides contained in foods destined for human consumption and traded internationally, which have been contaminated following a nuclear or radiological emergency. These GLs apply to food after reconstitution or as prepared for consumption, i.e., not to dried or concentrated foods, and are based on an intervention exemption level of 1 mSv in a year for members of the public (infant and adult)[6].

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[2][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 may be used during planned, existing and emergency exposure situations as well as for wastewaters and liquid effluents with specific modifications that can increase the overall uncertainty, detection limit, and threshold.

The test method(s) may be used for water samples after proper sampling, sample handling, and test sample preparation (see the relevant part of the ISO 5667 series).

This document has been developed to answer the need of test laboratories carrying out these measurements, that are sometimes required by national authorities, as they might need to obtain a specific accreditation for radionuclide measurement in drinking water samples.

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

WARNING — Persons using this document should be familiar with normal laboratory practices. 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 and to determine the applicability of any other restrictions.

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

1 Scope

This document specifies a method for the measurement of ^{210}Pb in all types of waters by liquid scintillation counting (LSC).

The method is applicable to test samples of supply/drinking water, rainwater, surface and ground water, as well as cooling water, industrial water, domestic, and industrial wastewater after proper sampling and handling, and test sample preparation. Filtration of the test sample is necessary. Lead-210 activity concentration in the environment can vary and usually ranges from 2 mBq l^{-1} to 300 mBq l^{-1} [27][28].

Using currently available liquid scintillation counters, the limit of detection of this method for ^{210}Pb is generally of the order of 20 mBq l^{-1} to 50 mBq l^{-1}, which is lower than the WHO criteria for safe consumption of drinking water (100 mBq l^{-1}).[4][6] 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 activity concentrations can be measured by either diluting the sample or using smaller sample aliquots or both. The method presented in this document is not intended for the determination of an ultra-trace amount of ^{210}Pb.

The range of application depends on the amount of dissolved material in the water and on the performance characteristics of the measurement equipment (background count rate and counting efficiency).

The method described in this document is applicable to an emergency situation.

The analysis of Pb adsorbed to suspended matter is not covered by this method.

It is the user’s responsibility to ensure the validity 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/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 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 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste water*

ISO 11929 (all parts), *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application*

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

ISO 19361, *Measurement of radioactivity — Determination of beta emitters activities — Test method using liquid scintillation counting*

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

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 80000-10, the ISO 11929 series, ISO/IEC Guide 98-3 and ISO/IEC Guide 99 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/

3.2 Symbols and abbreviated terms

Symbol	Definition	Unit
c_{A}	Activity concentration in the sample	Bq l^{-1}
c_{A0}	Activity concentration of the standard	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 probabilistically symmetric coverage interval	Bq l^{-1}
$c_A^{<}, c_A^{>}$	Lower and upper limits of the shortest coverage interval	Bq l^{-1}
C_{coeff}	Coefficient of ^{210}Bi ingrowth in the sample from the end of bismuth elution to time of counting	n/a
DPM	Disintegrations per minute	n/a
βmax	Maximum Beta particle energy	keV
R_{c}	Chemical recovery	n/a
r_{b}	count rate of reagent blanks	s^{-1} or counts s^{-1}
r_{g}	Sample count rates	s^{-1}
r_{s}	Calibration count rates	s^{-1}
r_0	Background count rate	s^{-1}
S	Eluted solution containing lead	n/a
SQPE	Spectral quench parameter of the external standard	n/a
TDCR	Triple to double counts ratio	n/a
t_{g}	Sample counting time	s
t_{s}	Calibration counting time	s
t_0	Background counting time	s
n/a Not applicable.		

Symbol	**Definition**	**Unit**
tSIE	Transformed spectral index of the external standard	n/a
U	Expanded uncertainty, calculated by $U = ku(c_A)$ with $k = 1, 2…$	Bq l^{-1}
$u(c_A)$	Standard uncertainty associated with the measurement result	Bq l^{-1}
V	Volume of the eluted phase S containing lead	l
V_e	Total volume of the test sample plus carrier	l
V_s	Volume of the standard test sample	l
V_{sample}	Volume of the sample	l
V_1	Volume of the aliquot from S for ^{210}Pb counting	l
V_2	Volume of the aliquot from S for determination of chemical recovery of lead	l
ε	General term for detection efficiency	s^{-1} Bq^{-1}
C_{Pb}	Concentration of lead in the eluted solution S	mg l^{-1}
$C_{Pb,e}$	Concentration of lead in the sample after addition of carrier	mg l^{-1}
n/a Not applicable.		

4 Principle

Lead-210 is a naturally occurring beta-emitting radionuclide with a maximum beta-energy of 63,5(5) keV and a half-life of 22,23(12) years [8][9]. It appears in the ^{238}U decay series (4n+2) as a long-lived decay product of ^{222}Rn (see Figure 1).

This document describes the measurement of ^{210}Pb after separation from its progeny, ^{210}Bi and ^{210}Po and its activity is measured by liquid scintillation counting, either immediately after its separation or indirectly after ingrowth of its progeny ^{210}Bi [10][26] to[34].

Lead-210 is chemically purified from potential interferents, which consist of any isotope that can make the liquid scintillator emits light in the region of interest (ROI) of ^{210}Pb. Different methods for the purification of ^{210}Pb are presented in Annex A.

After removal of the potential interferents, the chemical recovery of lead (R_c) is determined. The purified sample is mixed with the scintillation cocktail in a counting vial to obtain a homogenous medium. The vial is counted by LSC.

Because of their identical separation behaviour in the extraction chromatographic procedure and their half-lives, ^{214}Pb, ^{211}Pb, and ^{212}Pb are potential interferences (Table 1).

To avoid the possible interferences of the isotopes with short half-lives such as ^{211}Pb and ^{214}Pb and their progeny during the liquid scintillation counting, it is recommended to wait at least 3 h between elution of lead and sample counting to allow these radionuclides to substantially decay.

The beta-energies of ^{211}Pb, ^{212}Pb and ^{214}Pb and their progeny are higher than the maximum energy of ^{210}Pb. The 3 h delay time before counting can be reduced by setting appropriate counting windows different from the one set for ^{210}Pb to eliminate these interferences. In this approach, it is possible to start counting without a 3 h delay to neglect ^{210}Bi ingrowth during counting.

It is necessary to know the content of stable lead in the sample in order to adjust the quantity of the lead carrier to add to avoid resin saturation and to allow for the chemical recovery of ^{210}Pb. Total content of stable lead in samples should not exceed 10 mg Pb per gram of extraction chromatographic resin 18C6 to be used for the lead separation.

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

Suspended material is removed prior to analysis by filtration through a 0,45 µm filter membrane. The analysis of the insoluble fraction requires a mineralization step that is not covered by this document.

NOTE A suitable mineralization step is specified in ISO 18589-2 [11].

The measurement of stable lead for the determination of the chemical recovery can be carried out according to protocols such as:

- ICP-AES according to ISO 11885 [12];
- ICP-MS according to ISO 17294-2 [13]; or
- AAS according to ISO 15586.[14][15]

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

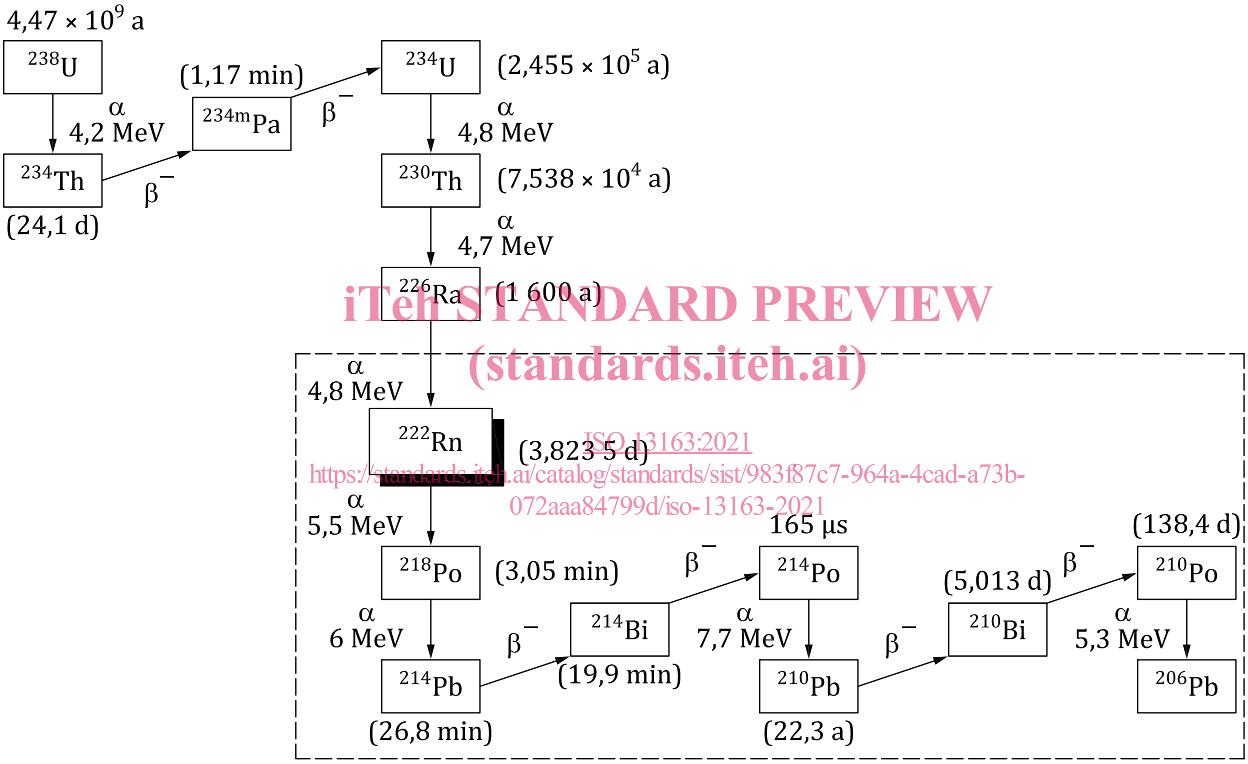

Figure 1 — Uranium-238 and its decay products

Table 1 — Decay data for lead radioisotopes and their progenies[9]

Lead radioisotopes	$T_{1/2}$	Decay mode	Emission energy (keV)	Emission probability (%)	Progeny
^{210}Pb	22,23(12) y	β-	63,5(5)	19,8(13) %	^{210}Bi
		β-	17,0(5)	80,2(13) %	^{210}Bi
		α	3792(20)	0,0000019(4) %	^{206}Hg
^{210}Bi	5,012(5) d	β-	1161,2(8)	99,99986(2) %	^{210}Po
		α	4778(4)	0,000056(6) %	^{206}Tl
		α	4740(4)	0,000084(9) %	^{206}Tl
^{210}Po	138,3763(17) d	α	5604,40(9) 5407,45(7)	0,00124(4) % 99,99876(4) %	^{206}Pb (stable)

Table 1 *(continued)*

Lead radioisotopes	**$T_{1/2}$**	**Decay mode**	**Emission energy (keV)**	**Emission probability (%)**	**Progeny**
^{206}Tl	4,202(11) min	β-	1532,4(6)	99,885(14) %	^{206}Pb (stable)
^{211}Pb	36,1(2) min	β-	1367(6) 962(6) 535(6) 257(6)	91,28(12) % 1,57(9) % 6,32(9) % 1,06(4) %	^{211}Bi
^{211}Bi	2,15(2) min	α α β-	6750,4(6) 6399,8(9) 574(5)	83,56(23) % 16,16(23) % 0,276(4) %	^{207}Tl ^{207}Tl ^{211}Po
^{207}Tl	4,774(12) min	β-	1418(5)	99,729(10) %	^{207}Pb (stable)
^{211}Po	0,516(3) s	α	7594,48(51)	100 %	^{207}Pb (stable)
^{212}Pb	10,64(1) h	β-	569,9(19) 331,3(19) 154,6(19)	13,3(11) % 81,7(11) % 4,99(21) %	^{212}Bi
^{212}Bi	60,54(6) min	β- α	2252 1(17) 6207 26(3)	64,07(7) % 35,93(7) %	^{208}Tl ^{212}Po
^{208}Tl	3,058(6) min	β-	1801,3(17) 1523,9(17) 1290,5(17)	49,2(6) % 22,1(5) % 24,1(2) %	^{208}Pb (stable)
^{212}Po	300(2) ns	α	8954 12(11)	100 %	^{208}Pb (stable)
^{214}Pb	26,916(44) min	β-	1019(11) 729(11) 667(11) 485(11) 180(11)	9,2(7) % 41,09(39) % 46,52(37) % 1,047(17) % 2,762(22) %	^{214}Bi
^{214}Bi	19,8(1) min	β- α	3270(11) 5621(3)	99,979(13) % 0,0210(13) %	^{214}Po ^{210}Tl
^{214}Po	162,3(12) µs	α	7833,46(6)	99,9895(7) %	^{210}Pb

5 Sampling and storage

Sampling, handling, and storage of the water shall be done according to ISO 5667-1, ISO 5667-3 and ISO 5667-10 and guidance is given for the different types of water in Reference [16] to Reference [23]. It is important that the laboratory receives a sample that is representative of the material being tested and has not been damaged or modified during transportation or storage.

The sample is filtered to remove suspended matter using a 0,45 µm filter membrane. A smaller pore size filter can also be used, but the filtration might be more time consuming. After filtration, the sample is acidified with nitric acid (HNO_3) to 0,01 $mol \cdot l^{-1}$ HNO_3.

An activity concentration of 100 Bq l^{-1} of ^{222}Rn in a sealed water sample with no airspace generates approximately 40 $mBq \cdot l^{-1}$ of ^{210}Pb for a storage time of 10 days. Thus, the storage time of samples dedicated to ^{210}Pb measurement shall be taken into consideration when the sample contains radon.