
**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*





COPYRIGHT PROTECTED DOCUMENT

© ISO 2021

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

| | |
|--|-----------|
| Foreword | iv |
| Introduction | v |
| 1 Scope | 1 |
| 2 Normative references | 1 |
| 3 Terms, definitions and symbols | 2 |
| 3.1 Terms and definitions..... | 2 |
| 3.2 Symbols and abbreviated terms..... | 2 |
| 4 Principle | 3 |
| 5 Sampling and storage | 5 |
| 6 Procedure | 6 |
| 6.1 Sample preparation for measurement..... | 6 |
| 6.2 Sample measurement..... | 6 |
| 7 Quality assurance and quality control program | 6 |
| 7.1 General..... | 6 |
| 7.2 Variables that could influence the measurement..... | 6 |
| 7.3 Instrument quality control..... | 6 |
| 7.4 Reagent interferences..... | 7 |
| 7.5 Interference control..... | 7 |
| 7.6 Method verification..... | 7 |
| 7.7 Demonstration of analyst capability..... | 7 |
| 7.8 Calibration..... | 7 |
| 8 Expression of results | 8 |
| 8.1 General..... | 8 |
| 8.2 Sample recovery, activity and uncertainties..... | 9 |
| 8.3 Decision threshold..... | 10 |
| 8.4 Detection limit..... | 10 |
| 8.5 Limits of the coverage intervals..... | 11 |
| 8.5.1 Limits of the probabilistically symmetric coverage interval..... | 11 |
| 8.5.2 The shortest coverage interval..... | 11 |
| 9 Test report | 11 |
| Annex A (informative) Separation and purification of ²¹⁰Pb | 13 |
| Annex B (informative) Spectra examples | 18 |
| Bibliography | 20 |

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 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 , ^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][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 $0,1 \text{ Bq}\cdot\text{l}^{-1}$ for ^{210}Pb activity concentration.

NOTE 1 The guidance level is the activity concentration with an intake of $2 \text{ l}\cdot\text{d}^{-1}$ of drinking water for one year that results in an effective dose of $0,1 \text{ mSv}\cdot\text{a}^{-1}$ 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 $0,1 \text{ Bq}\cdot\text{l}^{-1}$ 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.

ISO 13163:2021(E)

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*