INTERNATIONAL STANDARD

ISO 13161

Second edition
2020-07

Water quality — Polonium 210 — Test method using alpha spectrometry

Qualité de l'eau — Polonium 210 — Méthode d'essai par spectrométrie alpha

Reference number
ISO 13161:2020(E)

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Published in Switzerland

Contents

Page

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, *Radiological methods.*

This second edition cancels and replaces the first edition (ISO 13161:2011), which has been technically revised. The main changes compared to the previous edition are as follows:

— addition of a common introduction;

— addition of a new option for the chemical preparation using precipitation on a filter.

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, and ^{210}Pb, 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 phosphate fertilizer 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 the 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]. Drinking-water may thus contain radionuclides at activity concentrations which could present a risk to human health.

The radionuclides present in liquid effluents are usually controlled before being discharged into the environment[2] and water bodies. Drinking water is monitored for its radioactivity content as recommended by the World Health Organization (WHO)[3] so that proper actions can be taken to ensure that there is no adverse health effects to the public. Following these international recommendations, national regulation usually specify radionuclide authorized concentration limits for liquid effluent discharged to the environment and radionuclide guidance levels for water bodies and drinking waters for planned, existing and emergency exposure situations. Conformance 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[4].

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 guidance level in drinking water is 0,1 Bq l^{-1} for polonium-210 activity concentration.

NOTE 1 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[3].

In the event of a nuclear emergency, the WHO Codex Guideline Levels[5] mentioned that the activity concentration might be greater.

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)[5].

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[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 could 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 may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

This document is one of a family of International Standards on test methods dealing with the measurement of the activity concentration of radionuclides in water samples.

Water quality — Polonium 210 — Test method using alpha spectrometry

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 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 measurement of ^{210}Po in all types of waters by alpha spectrometry.

The method is applicable to test samples of supply/drinking water, rainwater, surface and ground water, marine 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 may be required.

The detection limit depends on the sample volume, the instrument used, the counting time, the background count rate, the detection efficiency and the chemical yield. The method described in this document, using currently available alpha spectrometry apparatus, has a detection limit of approximately 5 mBq l^{-1}, which is lower than the WHO criteria for safe consumption of drinking water (100 mBq l^{-1}). This value can be achieved with a counting time of 24 h for a sample volume of 500 ml.

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

The analysis of ^{210}Po adsorbed to suspended matter in the sample is not covered by this method.

If suspended material has to be removed or analysed, filtration using a 0,45 µm filter is recommended. The analysis of the insoluble fraction requires a mineralization step that is not covered by this document [13]. In this case, the measurement is made on the different phases obtained. The final activity is the sum of all the measured activity concentrations.

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 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 waters*

ISO 11929-1, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 1: Elementary applications*

ISO 11929-3, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 3: Applications to unfolding methods*

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, symbols and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions 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 http://www.electropedia.org/

3.1.1
certified standard solution
solution of known concentration traceable to primary or secondary certified radioactivity standard solution

3.1.2
tracer solution
usually a secondary standard or reference material, such as ^{208}Po or ^{209}Po, employed to determine the chemical yield of the analysis

3.1.3
quality control standard
radioactive source used to demonstrate that the measurement equipment employed performs within defined limits

Note 1 to entry: Quality control is usually carried out by the regular measurement of a suitable radioactive source in accordance with ISO 7870-1[14], ISO 7870-2[15], and ISO 7870-4[16].

3.2 Symbols and abbreviated terms

For the purposes of this document, the symbols and abbreviated terms defined in ISO 80000-10 and the following apply.

A	activity of the tracer added	Bq
c_A	activity concentration of ^{210}Po	Bq l^{-1}
c_A^*	decision threshold	Bq l^{-1}
$c_A^\#$	detection limit	Bq l^{-1}
$c_A^<, c_A^>$	lower and upper limits of the shortest coverage interval	Bq l^{-1}
$c_A^\triangleleft, c_A^\triangleright$	lower and upper limits of the probabilistically symmetric coverage interval	Bq l^{-1}

R_c	chemical yield	/
R_T	total yield	/
r_0	background count rate in the ^{210}Po region of interest	s^{-1}
r_{0T}	background count rate in the tracer region of interest	s^{-1}
r_g	gross count rate of the sample in the ^{210}Po region of interest	s^{-1}
r_T	gross count rate in the tracer region of interest	s^{-1}
t_0	background counting time	s
t_g	sample counting time	s
U	expanded uncertainty calculated by $U = k \cdot u(c_A)$ with $k = 1, 2 \ldots$	Bq l^{-1}
$u(c_A)$	standard uncertainty associated with the initial measurement result	Bq l^{-1}
V	volume of the test sample aliquot	l
ε	counting efficiency	1

4 Principle

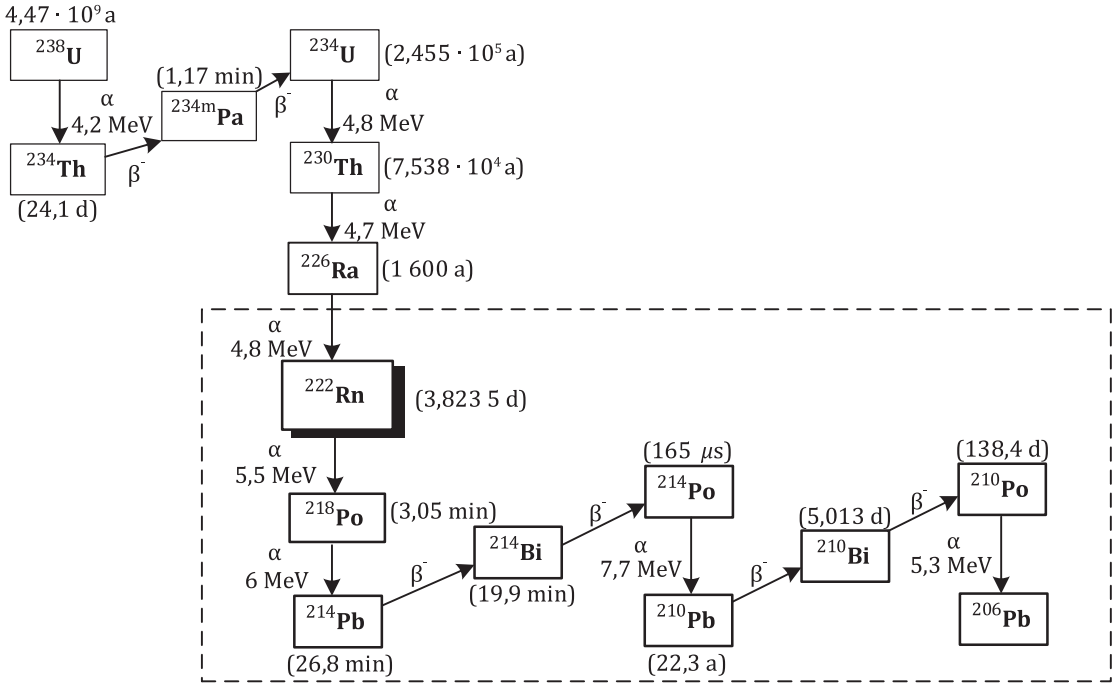
4.1 General

Polonium-210 is a natural alpha-emitting radionuclide with a half-life of (138,376 ± 0,002) days[17]. It appears in the natural chain of ^{238}U (see Figure 1). It is a long-life decay product of ^{222}Rn (Figure 1) through ^{210}Pb[8][12].

There are different techniques to measure ^{210}Po activity concentration in water: alpha spectrometry, liquid scintillation counting, and alpha proportional counting. This document describes the alpha spectrometry technique.

After sampling, the test sample undergoes treatment to produce an extremely thin deposit of the polonium on a metal disc or on a filter for measurement by alpha spectrometry.

The sample shall be analysed as soon as possible in order to evaluate the activity concentration at the sampling date. If the time elapsed between sampling and measurement is long, the activity concentration measured requires correction. It is then necessary to know the ^{210}Pb and ^{210}Bi activity concentrations in the sample in order to adjust the ^{210}Po activity concentration to the sampling date.



NOTE ^{206}Pb is stable.

Figure 1 — Uranium-238 and its decay products

4.2 Treatment

4.2.1 Treatment for a deposition on a disc

The main steps of the sample treatment are:

— filtration if necessary;

— acidification with concentrated hydrochloric acid (or nitric acid);

— addition of a polonium tracer (^{208}Po or ^{209}Po) solution;

The polonium isotopes ^{208}Po (5,11 MeV alpha emission) or ^{209}Po (4,88 MeV alpha emission) can be used as tracers since interference with ^{210}Po (5,31 MeV alpha emission) is minimal for sources displaying good resolution (<50 keV FWHM); ^{209}Po is preferred, but ^{208}Po is acceptable.

— addition of a reducing agent (e.g. ascorbic acid);

— spontaneous deposition of a thin layer on to a metal disc.

The activity concentration measurement as well as the determination of the total yield is carried out by alpha spectrometry.

4.2.2 Treatment for a precipitation on a filter

The main steps of the sample treatment are[18][19]:

— filtration;

— acidification with concentrated hydrochloric acid;

— addition of a polonium tracer (^{208}Po or ^{209}Po) solution;