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Kakovost vode - Svinec Pb-210 - Preskusna metoda s štetjem s tekočinskim scintilatorjem

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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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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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

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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

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

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 $mBq \cdot l^{-1}$ to 300 $mBq \cdot l^{-1}$ (References [12][13]). The guidance level for lead-210 in drinking water, as recommended by WHO, is 100 $mBq \cdot l^{-1}$ (Reference [14]).

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

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 mBq·l^{-1} to 50 mBq·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 $k = 1, 2...$, 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

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]

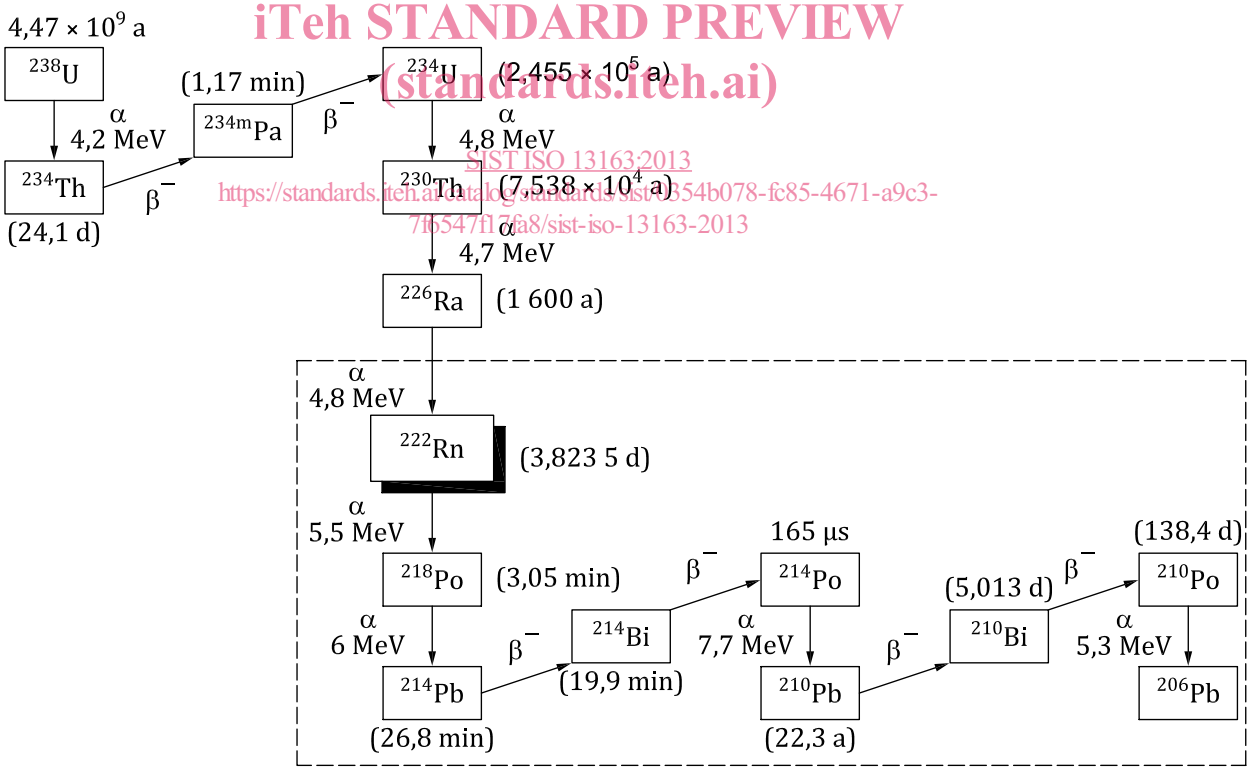

Figure 1 — Uranium-238 and its decay products (see ISO 13164-1)

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.