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Water quality — Polonium 210 — Test method using alpha spectrometry

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

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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).

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 …	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.
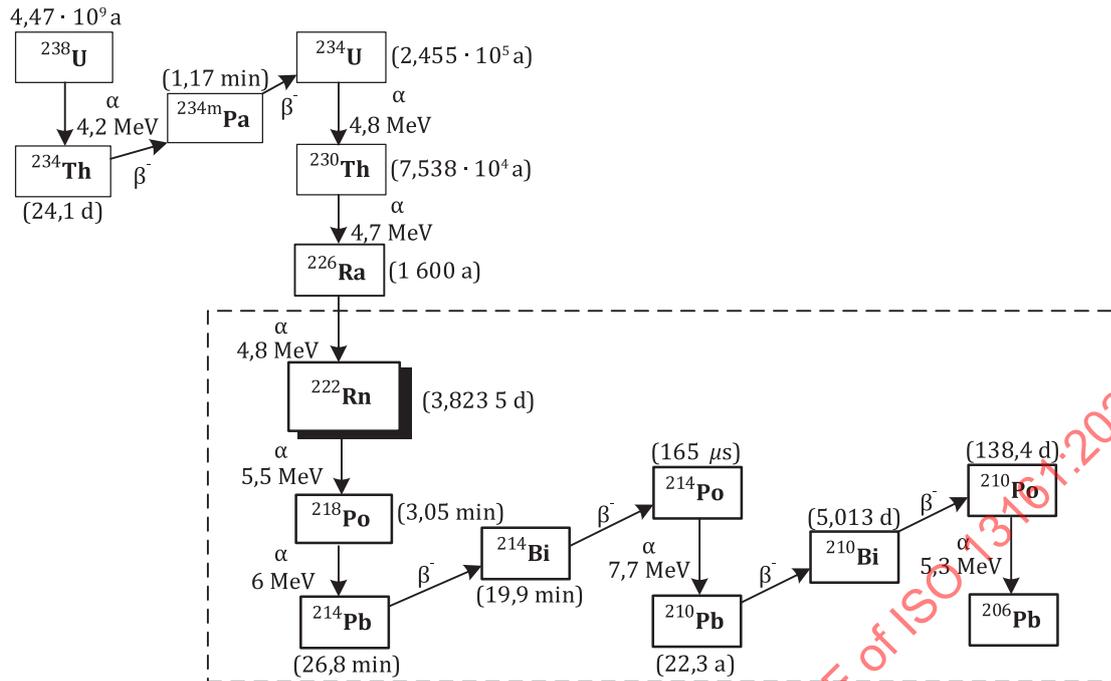
NOTE ^{206}Pb is stable.

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;

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.

— evaporate to dryness;

— dissolve with 10 ml of 1 mol·l^{-1} HCl;

— filter the solution;

— add a co-precipitation agent (e.g. 50 µg of copper as copper chloride);

— micro-precipitate polonium with sulfide;

— filter on a filter.

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

4.3   Principle of alpha spectrometry

The thin layer deposited on the metal disc or on a filter allows the detection of alpha particles without excessive attenuation by the sample matrix. The interaction of alpha particles and detector results in a change in (bias) current in the detector that is proportional to the energy of the particles.

The electronic pulses generated by the detector are amplified, and displayed as an energy spectrum using an analogue-digital converter, multiple channel analyser, and computer processing. The spectrum display enables the radionuclides present in the source to be identified and integration of counts enables the determination of activity of the test sample, taking into account the background counting rates and/or the blank test and the total yield.

A blank test should be carried out with the same reagents replacing the water sample by water conforming with ISO 3696, grade 3 previously used for the preparation of the reagents without tracer.

To ensure an acceptable performance of the detector system, a quality control standard shall be measured.

The chemical yield of [210]Po measurement is determined by adding a radioactive tracer. The total yield is a product of the chemical yield and the detection efficiency.

5   Reagents and equipment

5.1   Reagents

During chemical treatment and cleaning of the metal disc, use only reagents of recognized analytical grade. Use only reagents with no measurable [210]Po concentration.

5.1.1   Water, conforming with ISO 3696, grade 3.

5.1.2   Tracer solution.

Use a tracer solution of known activity. [208]Po ($T_{1/2}$ = 1 058,5 d ± 0,7 d) or [209]Po ($T_{1/2}$ = 115 y ± 13 y) [17] are suitable isotopes. The activity of tracer added shall be close to the [210]Po activity concentration expected in the test sample[20][21].

Tracer decay shall be taken into account in accordance with the information given on the calibration certificate.

NOTE   The half-life of [209]Po was subjected to a recent investigation[22].

5.1.3 **Concentrated hydrochloric acid**, 37 % mass fraction, or concentrated nitric acid.

5.1.4 **Dilute hydrochloric acid** or **dilute nitric acid**, to adjust the pH at the beginning of the treatment.

5.1.5 **Ascorbic acid (**or **hydroxylamine hydrochloride)**.

5.1.6 **Ethanol**.

5.1.7 **Copper chloride solution** (500 mg·l^{-1} in HCl 1 % v/v).

5.1.8 **Sodium sulfide** solution for precipitation (10 % m/v).

5.2 Equipment

The laboratory equipment should be appropriate for the chemical treatment used (Clause 7).

5.2.1 **Standard laboratory equipment**, including filtration material, hot plate, pH meter or pH paper.

5.2.2 **Electronic balance**, with a readability of 0,1 mg.

5.2.3 **Stirrer**.

5.2.4 **Equipment for the preparation of the thin layer deposit**.

5.2.5 **Metal disc**, of a metal that allows reductive deposition of polonium (e.g. stainless steel [304L type], silver, nickel or another metal displaying this property). Depending on the device used for deposition, it can be useful to cover one side of the disc (e.g. with spray paint, or equivalent) to prevent deposition on the side that is not measured.

5.2.6 **Filters with 0,1 μm porosity** (e.g. cellulose nitrate or polypropylene filters[1]).

5.3 Alpha spectrometry measuring equipment

The alpha spectrometry counting can be carried out using either gridded ionization chambers or thin semiconductor detectors.

A spectrum display is essential.

6 Sampling and samples

Sampling, handling, and storage of the water shall be done as specified in ISO 5667-1 and ISO 5667-3, ISO 5667-10 and guidance is given for the different types of water in References [23] to [30].

It is important that the laboratory receives a sample that is truly representative and has not been damaged or modified during transportation or storage.

If necessary, the sample is filtered using a 0,45 μm filter to remove suspended matter before acidification (the use of a single-use filtering device is recommended).

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1) Eichrom Resolve® Filters is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.

7   Chemical treatment and deposit process

7.1   General

The volume of the test sample is variable; the usual quantity is between 150 ml and 2 000 ml.

All reasonable precautions should be taken during handling and storage of the test sample to avoid contamination or degradation.

The verification that any contamination of reagents and residual contamination in the analytical equipment are below the detection limit for the analysis shall be carried out and documented (blank analysis).

The steps of the chemical treatment where a hot plate is used shall be carried out under a fume-extracting hood.

7.2   Chemical treatment

7.2.1   Autodeposition of polonium on a disc

7.2.1.1   Treatment

Take an aliquot of volume V from the test sample, e.g. 500 ml.

If large test samples are used, or if a special study is being carried out, it is possible that a preliminary concentration prior to the first step of chemical treatment is necessary, e.g. co-precipitation of $Fe(OH)_3$ adding Fe^{3+} can be used. Co-precipitation from large samples decreases the detection limit.

Adjust the pH with hydrochloric acid preferably or nitric acid (5.1.4) in order to reduce the pH below 1,5.

According to ISO 5667-3, it is recommended to store the samples after acidification with concentrated nitric acid (5.1.3). In this case if hydrochloric acid is to be used for the chemical treatment, the sample shall be evaporated to dryness before dilution in hydrochloric acid.

Add a sufficient, known quantity of tracer solution (5.1.2) to enable the determination of the chemical yield without introducing large measurement uncertainties or potentially contaminating the laboratory equipment. Ideally the ^{210}Po peak and the tracer peak should be of the same magnitude (an ideal aim is the acquisition of between 400 counts and 10 000 counts in the tracer peak).

At this stage, a concentration step [dry evaporation and addition of dilute hydrochloric acid (5.1.4)] can be carried out by slow evaporation to dryness at a temperature lower than 80 °C (to avoid losses of Po). Add, HCl and water to produce a final solution of between 0,1 mol l^{-1} and 3 mol l^{-1} and an appropriate volume for the deposition phase. This volume depends on the laboratory's equipment for undertaking the deposition step.

The consistent use of the same acid concentration and total volume are recommended to maintain the repeatability of the method.

A chemical treatment procedure using extraction chromatographic resin can be used when both ^{210}Pb and ^{210}Po are to be measured. The measurement of ^{210}Pb is beyond the scope of this document[31].

7.2.1.2   Disc cleaning

The disc shall be thoroughly cleaned in order to remove deposits of organic impurities, e.g. by washing with ethanol or acetone or both. It may also be necessary to remove surface oxide deposits with dilute hydrochloric acid.

In the case of unprotected silver discs, the oxide and sulfur deposits present on the surface can be eliminated by polishing or by washing with dilute ammonia solution (or any silver polish equivalent).

Depending on the deposition equipment used (e.g. hanging the disc in the solution), it is important to get the deposition on only one side of the disc. Cover one side of the disc, e.g. with tape, if necessary.

7.2.1.3 Deposition phase

Transfer the solution to the deposition equipment (Annex A) and add an excess of the reducing agent, e.g. 0,1 g of ascorbic acid or hydroxylamine hydrochloride (5.1.5).

During the deposition phase, the metal disc catalyses the reduction of the polonium from Po^{4+} or Po^{2+} to metallic polonium. The addition of a reducing agent prevents the reoxidation (and therefore redissolution) of the deposited polonium on the disc by any oxidants that may be present in the solution, e.g. Fe^{3+}.

Stir the solution continuously using, for example, an automatic stirrer during the entire deposition phase.

The deposition of Po^{4+} or Po^{2+} to metallic polonium is slow at room temperature. The deposition rate can be accelerated by heating the solution to a maximum of 90 °C, making sure that the solution is in permanent contact with the metal disc, without excessive bubbling. It is advisable to limit polonium evaporation losses by keeping the acidity of the solution low. This phase shall be long enough to allow a good deposition yield and in normal circumstances is completed within 3 h to 4 h[8][9].

Add ascorbic acid carefully as it can interfere with the spectral resolution. The quantity needed depends on the amount of Fe present in the sample, especially when an $Fe(OH)_3$ co-precipitation has been used.

On completion, polonium is deposited as a thin source on the disc. Rinse the disc with water (5.1.1) and wait until it is dry before measuring it by alpha spectrometry.

To avoid contamination of the detector, it is recommended to put the disc on a hot plate between 200 °C and 300 °C for a few minutes.

7.2.2 Microprecipitation on a filter

7.2.2.1 Coprecipitation of polonium sulfide with copper sulfide

Take an aliquot of volume V from the filtered test sample, e.g. 500 ml.

If large volume samples need to be tested, or if a special study is being carried out, it is possible that a preliminary concentration prior to the first step of chemical treatment is necessary, e.g. co-precipitation with $CaHPO_4$ can be used. If a co-precipitation is performed, the volume of analysed sample can be large and the detection limit lowered in the same proportion.

Add a sufficient, known quantity of tracer solution (5.1.2) to enable the determination of the chemical yield without introducing large measurement uncertainties or potentially contaminating the laboratory equipment. Ideally the ^{210}Po peak and the tracer peak should be of the same magnitude (an ideal aim is the acquisition of between 400 counts and 10 000 counts in the tracer peak).

Acidified to approximately 1 mol l^{-1} HCl by adding 0,8 ml of concentrated HCl.

A volume of 0,05 ml of the copper solution (5.1.7) is added and a volume of 0,1 ml of the sodium sulfide (5.1.8) is then added.

The solution obtained is vigorously shaken and allow to react for 10 min.

Polonium sulfide coprecipitates with CuS.

7.2.2.2 Filtration step

Filter the solution through a filter of 0,1 µm porosity (5.2.6) with an appropriate diameter size for alpha spectrometer measurement.

After filtration, the precipitate is rinsed with 1 ml to 2 ml of 80 % ethanol (5.1.6) and air-dried for a few minutes.

The filter is then mounted on a stainless-steel disc for alpha spectrometry measurement.

8 Measurement by alpha spectrometry

8.1 General

The counting time depends on the data quality objectives, uncertainty and detection limit to be achieved.

8.2 Quality control

Equipment quality control sources shall be measured (see 3.1.3) to verify that the measurement equipment is performing within agreed limits[16].

A thin source of $^{239/240}$Pu may be employed for this purpose as well to estimate the detection efficiency; the alpha emissions are in the 5,10 MeV to 5,20 MeV energy region, and there is no appreciable decay over the working life of the source.

The chemical yield of the process can be calculated using Formula (1):

$$R_c = \frac{R_T}{\varepsilon} \tag{1}$$

In general, the chemical yield obtained is greater than 90 %.

— The background rate of each detector is determined with an empty source support (cleaned disc); The background count time must be at least equal to the sample count time.

   NOTE   The optimum time for the measurement of the background source can be shown to be equal to that of the source from very low activity sources[21].

— The blank analysis [i.e. analysis carried out with water (5.1.1) containing no detectable, or very low levels of, ^{210}Po without adding tracer solution (5.1.2)] value shall be compared to the previous background values obtained from the same detector. This value shall be similar to the background value measured with an empty source support (cleaned disc) in the energy region of ^{210}Po and of the tracer to make sure that there is no reagent or laboratory equipment contamination.

— A blank count rate significantly larger than the background count rate, r_0, demonstrates either the presence of polonium in the reagents and/or equipment, or cross-contamination.

8.3 Measurement

The source is measured using grid ionization chambers or thin semiconductor detectors (See Annex B).

9 Expression of results

9.1 General

Measurement results presented in a test report are expressed as activity concentrations, in becquerels per litre, with an associated uncertainty. The coverage factor for the expanded uncertainty is specified in the presentation of results.

The reported result is an estimation of the "true" value with an associated uncertainty, which is itself a combination of elementary uncertainties.

Where alpha spectrometry is used to measure radionuclide activity concentration, the main uncertainty components to be taken into account are the following:

a) gross counts in the analyte and background signals;

b) mass of the added tracer (or volume of the tracer solution);

c) activity of the tracer;

d) volume of the test sample.

Other uncertainties are small in comparison to these parameters and may be disregarded, unless there is reason to include them.

When ^{209}Po is used as a chemical yield tracer, ^{209}Po activity should be corrected for the alpha decay probability of $(99,52 \pm 0,04)$ % and for decay from its reference date. When the tracer quantity is added by a volumetric method, it is necessary to know the exact volumetric activity of the tracer solution. Use of a gravimetric method to add the tracer is recommended.

When ^{210}Pb is known to be present, the calculation of the activity at the sample reference date can be complex. When ^{210}Po deposition and measurement are not done immediately, ^{210}Pb and/or ^{210}Bi activities should also be measured in order to make the proper corrections.

9.2 Total yield

The total yield is the product of the chemical yield and the counting efficiency.

The chemical yield can be considered as a quality control parameter.

Total yield, R_T, is calculated from the sample spectrum using Formula (2):

$$R_T = \frac{(r_T - r_{0T})}{A} \tag{2}$$

Decay correction is not taken into account in Formula (2), but shall be done if necessary.

9.3 Activity concentration of ^{210}Po in the sample

In all the formulae, counting time is taken to be identical for the measurement of the sample and the measurement of the background.

The activity concentration, in becquerels per litre at the date of measurement, of ^{210}Po of the sample is calculated using Formula (3):

$$c_A = \frac{(r_g - r_0)}{V\,R_T} = (r_g - r_0)w \tag{3}$$

where $w = \dfrac{1}{V\,R_T}$.

If ^{208}Po or ^{209}Po are used as tracers, a correction due to impurities is necessary if these have been declared by the supplier or are otherwise known to be present.

The decay correction of the ^{210}Po activity concentration is made if necessary, e.g. for a very long counting time or a significant delay between the deposition date and the counting time.

9.4 Combined uncertainties

According to ISO/IEC Guide 98-3, the combined uncertainty of c_A is calculated using Formula (4):

$$u(c_A) = \sqrt{w^2 \left[u^2(r_g) + u^2(r_0) \right] + c_A^2 \, u_{rel}^2(w)} = \sqrt{w^2 \left(\frac{r_g}{t_g} + \frac{r_0}{t_0} \right) + c_A^2 \, u_{rel}^2(w)} \tag{4}$$

where the uncertainty of the counting time is neglected and the standard uncertainty of w, $u_{rel}(w)$, is calculated using Formula (5):

$$u_{rel}^2(w) = u_{rel}^2(R_T) + u_{rel}^2(V) \tag{5}$$

The relative standard uncertainty of R_T, $u_{rel}(R_T)$, is calculated using Formula (6):

$$u_{rel}^2(R_T) = u_{rel}^2(r_T - r_{0T}) + u_{rel}^2(A) = \frac{(r_T / t_g) + (r_{0T} / t_0)}{(r_T - r_{0T})^2} + u_{rel}^2(A) \tag{6}$$

where $u_{rel}^2(A)$ includes all the uncertainties related to the tracer activity, i.e. in the standard solution, preparation of the tracer solution, and the addition of the tracer solution to the sample.

For the calculation of the characteristic limits, $u(c_A)$ is required, i.e. the combined uncertainty of c_A as a function of its true value (according to ISO 11929-1), calculated using Formula (7):

$$\tilde{u}(\tilde{c}_A) = \sqrt{w^2 \left[\frac{(\tilde{c}_A / w + r_0)}{t_g} + \frac{r_0}{t_0} \right] + \tilde{c}_A^2 \, u_{rel}^2(w)} \tag{7}$$

9.5 Decision threshold

In accordance with ISO 11929-1 and ISO 11929-3, the decision threshold c_A^*, expressed in becquerels per litre, is obtained from Formula (7) for $c_A = 0$.

This yields:

$$c_A^* = k_{1-\alpha} \, \tilde{u}(0) = k_{1-\alpha} \, w \sqrt{\frac{r_0}{t_g} + \frac{r_0}{t_0}} \tag{8}$$

where $\alpha = 0{,}05$ with $k_{1-\alpha} = 1{,}65$, a value often chosen by default.

9.6 Detection limit

In accordance with ISO 11929-1 and ISO 11929-3, the detection limit $c_A^\#$, expressed in becquerels per litre, can be calculated using Formula (9):

$$c_A^\# = c_A^* + k_{1-\beta} \, \tilde{u}(c_A^\#) = c_A^* + k_{1-\beta} \sqrt{w^2 \left[\frac{(c_A^\# / w + r_0)}{t_g} + \frac{r_0}{t_0} \right] + c_A^{\#2} \, u_{rel}^2(w)} \tag{9}$$

The detection limit can be calculated by solving Formula (8) for $c_A^\#$ or more simply by iteration with a starting approximation $c_A^\# = 2c_A^*$ in terms of the right side of Formula (9).

$c_A^\#$ is obtained with $k_{1-\alpha} = k_{1-\beta} = k$:

$$c_A^\# = \frac{2c_A^* + \left(k^2\,w\right)/t_g}{1 - k^2\,u_{rel}^2\,(w)} \tag{10}$$

Values $\alpha = \beta = 0{,}05$ and therefore $k_{1-\alpha} = k_{1-\beta} = 1{,}65$ are often chosen by default.

9.7 Limits of the coverage interval

9.7.1 Limits of the probabilistically symmetric coverage interval

The lower, $c_A^\triangleleft$, and upper, $c_A^\triangleright$, coverage limits, are calculated using (according to ISO 11929-1 and ISO 11929-3) Formulae (11) and (12):

$$c_A^\triangleleft = c_A - k_p\,u\left(c_A\right)\ ;\ p = \omega\left(1 - \frac{\gamma}{2}\right) \tag{11}$$

$$c_A^\triangleright = c_A + k_q\,u\left(c_A\right)\ ;\ q = 1 - \frac{\omega\gamma}{2} \tag{12}$$

where $\omega = \Phi\left[\dfrac{c_A}{u(c_A)}\right]$.

Φ being the distribution function of the standardized normal distribution and $1 - \gamma$, the probability that the true value of c_A is situated in the coverage interval.

Set $\omega = 1$, if $c_A \geq 4\,u(c_A)$. In this case:

$$c_A^{\triangleleft\triangleright} = c_A \pm k_{1-\gamma/2}\,u\left(c_A\right) \tag{13}$$

Values $\gamma = 0{,}05$ with $k_{1-\gamma/2} = 1{,}96$ are often chosen by default.

9.7.2 The shortest coverage interval

As described in detail in ISO 11929-1 the lower limit of the shortest coverage interval, $c_A^<$, and the upper limit of the shortest coverage interval, $c_A^>$, are calculated from a primary measurement result, c_A, of the measurand and the standard uncertainty, $u(c_A)$, associated with c_A, either by:

$$c_A^<,c_A^> = c_A \pm kp \cdot u\left(c_A\right)\,;\,p = \left(1 + \omega \cdot (1-\gamma)\right)/2 \tag{14}$$

or if $c_A^< < 0$, by:

$$c_A^<,c_A^> = 0\,;\,c_A \pm kq \cdot u\left(c_A\right)\ \text{with}\ q = 1 - \omega \cdot \gamma \tag{15}$$

where $\omega = \Phi\left[\dfrac{c_A}{u(c_A)}\right]$.

Φ being the distribution function of the standardized normal distribution.

The relation: $0 \leq c_A^< < c_A^>$ applies and the approximation of Formulae (11) and (12) is valid.

10 Test report

The test report shall conform to ISO/IEC 17025 requirements. It shall contain at least the following information:

a) the test method used, with reference to this document (ISO 13161:2020);

b) all information necessary for the complete identification of the sample;

c) units in which the results are expressed;

d) ^{210}Po activity concentration reference date — if the reference date is not the deposition date, then the equilibrium between ^{210}Po, ^{210}Pb and ^{210}Bi shall be taken into account to correct the activity concentration;

e) test result, $c_A \pm u(c_A)$ or $c_A \pm U$, with the associated k value. The uncertainty can also be expressed as the limits of the probabilistically symmetric coverage interval, $c_A^\triangleleft$, $c_A^\triangleright$, and/or the limits of the shortest coverage interval $c_A^<, c_A^>$;

Note that $U = k\, u(c_A)$ with $k = 1$ or 2.

Complementary information can be provided such as:

f) probabilities α, β, and $(1 - \gamma)$;

g) decision threshold and detection limit;

h) date of deposition or microprecipitation and date of measurement;

i) depending on the customer request, there are different ways to present the result:

1) when the activity concentration c_A is compared with the decision threshold following ISO 11929-1, the results of the measurement should be expressed as $\leq c_A^*$ when the result is below or equal to the decision threshold;

2) when the activity concentration c_A is compared with the detection limit, the result of the measurement can be expressed as $\leq c_A^\#$ when the result is below or equal to the detection limit.
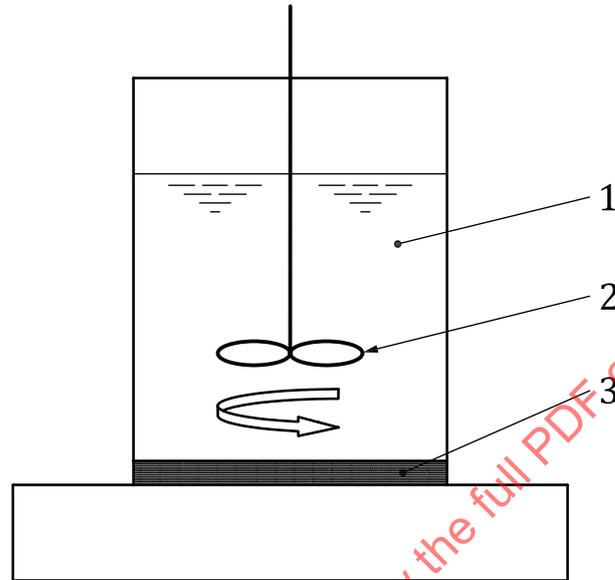
If the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose.

j) any relevant information likely to affect the results.

In accordance with ISO/IEC 17025, additional information can be provided, e.g. sampling details.

Annex A
(informative)

Cell deposit examples

Key

1   solution
2   stirrer
3   metal disc

Figure A.1 — Room temperature deposit system