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Measurement of radioactivity — Determination of beta emitters activities — Test method using liquid scintillation counting

Mesurage de la radioactivité — Détermination de l'activité des radionucléides émetteurs bêta — Méthode d'essai par comptage des scintillations en milieu liquide

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Contents

Foreword

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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).

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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 85, N*uclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 2, *Radiological protection*.

Introduction

Everyone is exposed to natural radiation. The natural sources of radiation are cosmic rays and naturally occurring radioactive substances which exist in the earth and within the human body. Human activities involving the use of radiation and radioactive substances add to the radiation exposure from this natural exposure. Some of those activities, such as the mining and use of ores containing naturally-occurring radioactive materials (NORM) and the production of energy by burning coal that contains such substances, simply enhance the exposure from natural radiation sources. Nuclear power plants and other nuclear installations use radioactive materials and produce radioactive effluent and waste during operation and on their decommissioning. The use of radioactive materials in industry, agriculture and research is expanding around the globe.

All these human activities give rise to radiation exposures that are only a small fraction of the global average level of natural exposure. The medical use of radiation is the largest and a growing man-made source of radiation exposure in developed countries. It includes diagnostic radiology, radiotherapy, nuclear medicine and interventional radiology.

Radiation exposure also occurs as a result of occupational activities. It is incurred by workers in industry, medicine and research using radiation or radioactive substances, as well as by passengers and crew during air travel and for astronauts. The average level of occupational exposures is generally below the global average level of natural radiation exposure[13].

As uses of radiation increase, so do the potential health risk and the public's concerns. Thus, all these exposures are regularly assessed in order to: (1) improve the understanding of global levels and temporal trends of public and worker exposure; (2) to evaluate the components of exposure so as to provide a measure of their relative importance, and; (3) to identify emerging issues that may warrant more attention and study. While doses to workers are mostly directly measured, doses to the public are usually assessed by indirect methods using radioactivity measurements performed on various sources: waste, effluent and/or environmental samples.

To ensure that the data obtained from radioactivity monitoring programs support their intended use, it is essential that the stakeholders (for example, nuclear site operators, regulatory and local authorities) agree on appropriate methods and procedures for obtaining representative samples and then handling, storing, preparing and measuring the test samples. An assessment of the overall measurement uncertainty need also to be carried out systematically. As reliable, comparable and 'fit for purpose' data are an essential requirement for any public health decision based on radioactivity measurements, international standards of tested and validated radionuclide test methods are an important tool for the production of such measurement results. The application of standards serves also to guarantee comparability over time of the test results and between different testing laboratories. Laboratories apply them to demonstrate their technical qualifications and to successfully complete proficiency tests during interlaboratory comparison, two prerequisites for obtaining national accreditation. Today, over a hundred international standards, prepared by Technical Committees of the International Standardization Organization, including those produced by ISO/TC 85, and the International Electrotechnical Commission (IEC), are available for application by testing laboratories to measure the main radionuclides.

Generic standards help testing laboratories to manage the measurement process by setting out the general requirements and methods to calibrate and validate techniques. These standards underpin specific standards which describe the test methods to be performed by staff, for example, for different types of sample. The specific standards cover test methods for:

- Naturally-occurring 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}Pb) which can be found in materials from natural sources 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, and curium), ^{3}H, ^{14}C, ^{90}Sr and gamma emitting radionuclides found in waste, liquid and gaseous

effluent, in environmental matrices (water, air, soil, biota) and food and feed as a result of authorized releases into the environment and of fallout resulting from the explosion in the atmosphere of nuclear devices and accidents, such as those that occurred in Chernobyl and Fukushima.

Many of these radionuclides are beta emitters that can be measured by liquid scintillation counting, following appropriate sample preparation.

A generic international standard on liquid scintillation counting is justified for test laboratories carrying out beta emitter measurements in fulfilment of national authority requirements. For example, testing laboratories need to obtain a specific accreditation for radionuclide measurement for the monitoring of drinking water, food, the environment or the discharges, as well as for biological samples for medical purpose.

This document describes (after appropriate sampling, sample handling and test sample preparation) the generic requirements to quantify the activity concentration of beta emitters using liquid scintillation counting. In the absence of a specific pre-treatment of the test sample (such as distillation for ^{3}H measurement, or after benzene synthesis for ^{14}C measurement), this document is to be used as a screening method unless the interference of beta emitters, others than those to be quantified, is considered negligible in the test portion.

This document is one of a set of generic International Standards on measurement of radioactivity.

Measurement of radioactivity — Determination of beta emitters activities — Test method using liquid scintillation counting

1 Scope

This document applies to liquid scintillation counters and requires the preparation of a scintillation source obtained by mixing the test sample and a scintillation cocktail. The test sample can be liquid (aqueous or organic), or solid (particles or filter or planchet).

This document describes the conditions for measuring the activity of beta emitter radionuclides by liquid scintillation counting[14][15].

The choice of the test method using liquid scintillation counting involves the consideration of the potential presence of other beta emitter radionuclides in the test sample. In this case, a specific sample treatment by separation or extraction is implemented to isolate the radionuclide of interest in order to avoid any interference with other beta-, alpha- and gamma-emitting radionuclides during the counting phase.

This document is applicable to all types of liquid samples having an activity concentration ranging from a few $Bq \cdot l^{-1}$ to 10^6 $Bq \cdot l^{-1}$. For a liquid test sample, it is possible to dilute liquid test samples in order to obtain a solution having an activity compatible with the measuring instrument. For solid samples, the activity of the prepared scintillation source shall be compatible with the measuring instrument.

The measurement range is related to the test method used: nature of test portion, preparation of the scintillator - test portion mixture, measuring assembly as well as to the presence of the co-existing activities due to interfering radionuclides.

Test portion preparations (such as distillation for ^3H measurement, or benzene synthesis for ^{14}C measurement, etc.) are outside the scope of this document and are described in specific test methods using liquid scintillation[2][3][4][5][6][7][8][9].

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

ISO 18589-2, *Measurement of radioactivity in the environment — Soil — Part 2: Guidance for the selection of the sampling strategy, sampling and pre-treatment of samples*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Symbols, abbreviations and units

For the purposes of this document, the symbols and abbreviations defined in ISO 80000-10[10], ISO/IEC Guide 98-3[11], ISO/IEC Guide 99[12] and the following apply.

β_{max}	Maximum energy for the beta emission, in keV
V	Volume of test portion, in litre
m	Mass of test portion, in kilogram
ρ	Density of the sample, in kilogram per litre
ε_{p}	Preparation efficiency
a	Activity per unit of mass, in becquerel per kilogram
c_A	Activity concentration, in becquerel per litre
A	Activity of the calibration source, in becquerel
t_0	Background counting time, in second
t_{g}	Portion counting time, in second
t_{s}	Calibration counting time, in second
r_0	Background count rate, per second
r_{g}	Portion count rate, per second
r_{s}	Calibration count rate, per second
ε	Detection efficiency
ε_{q}	Quenched efficiency
f_{q}	Quench factor
$u(c_{\mathrm{A}})$	Standard uncertainty associated with the measurement result; in becquerel per litre
U	Expanded uncertainty, calculated by $U = k \cdot u(c_{\mathrm{A}})$ with k = 1, 2,…, in becquerel per litre
c_{A}^{*}	Decision threshold, in becquerel per litre
$c_{\mathrm{A}}^{\#}$	Detection limit, in becquerel per litre
$c_{\mathrm{A}}^{<}$, $c_{\mathrm{A}}^{>}$	Lower and upper limits of the confidence interval, in becquerel per litre

5 Principle

The aqueous, organic or particles portion is mixed with the scintillation cocktail in a counting vial to obtain a homogeneous medium (scintillation source). Electrons emitted from beta disintegration transfer their energy to the scintillation cocktail molecules that are excited by this process before returning to their ground state by emitting photons that are detected by photoelectron multiplier tubes (phototubes).

The electronic pulses emitted by the phototubes are amplified. The peak pulse amplitude is converted to a digital value by an analogue-to-digital convertor (ADC) and the pulse height stored using a multichannel analyser (MCA). The pulses are analysed (in order to remove random events) by the electronic systems and the data analysis software. The count rate of these photons allows the determination of the activity in the test portion, after correcting for the background count rate and detection efficiency, taking account of the quench correction. The requirements of the specific test method for specific beta emitting radionuclides, including test portion preparation and scintillation source preparation, shall be determined according to the intended use of the measurement results and the associated data quality objectives.

In order to determine the background count rate, a blank portion shall be prepared in the same way as the test portion.

The conditions to be met for the blank sample, the test portion and the calibration source are:

- same scintillation cocktail;
- same type of counting vial;
- same filling geometry;
- same ratio between test portion and scintillation cocktail;
- same preparation conditions, minimizing photoluminescence and static electricity effects;

In addition, the quench indicating parameter should be within the range of the quench calibration curve. An alternative method using the Cerenkov effect[16][17] is treated in Annex C.

6 Reagents and equipment

Use only reagents of recognized analytical grade.

6.1 Reagents

6.1.1 Blank material

Blank material is used to prepare the blank portion. For direct counting of test portion, it shall be as free as possible of chemical impurities to avoid quenching, and with radioactive impurities negligible in comparison with the test portion activities to be measured.

If some preparation is required for the test portion, the blank portion shall be prepared with a reference material of the lowest activity available.

This blank sample shall be kept physically remote from any radioactive material to avoid cross-contamination.

For example, a water sample with a low tritium and carbon 14 activity concentration can be obtained from (deep) subterranean water kept in a well-sealed borosilicate glass bottle in the dark at a controlled temperature (see ISO 5667-3). When the volume of blank water is sufficiently large (e.g. 10 l to 20 l) and well-sealed, tritium and carbon 14 activity concentrations remain stable for years, although it is advisable to determine these activity concentrations at predetermined intervals (e.g. every year).

6.1.2 Calibration source solutions

To avoid cross-contamination, preparation of samples and calibration source solution shall be segregated.

The standardized solution used to prepare the calibration source solution shall be provided with a calibration certificate confirming traceability to a national or international standard of radioactivity.

Weigh and pour into a weighed volumetric flask (for example 100 ml) the required quantity of a standardized solution of the radionuclide to be measured, so that the activity concentration generates sufficient counts to reach the required measurement uncertainty after dilution with the blank solution and thorough mixing. Calculate the activity concentrations of the resulting calibration source solution (A). Note the date at which the standard solution was made up ($t = 0$).

The radionuclide activity concentration of the calibration source solution at time *t* at which the samples are measured shall be corrected for radioactive decay.

6.1.3 Scintillation solution

The scintillation cocktail is chosen according to the characteristics of the sample to be analysed and according to the properties of the detection equipment[18][19].

For the measurement of usual environmental and drinking water sample or for test sample prepared as an aqueous solution, it is recommended to use a hydrophilic scintillation cocktail.

For the direct measurement of particles in suspension, it is recommended to use a scintillation cocktail that leads to a gel type mixture.

In all cases, the characteristics of the scintillation cocktail when mixed with the sample shall result in a scintillation source with the form of a homogeneous and stable medium.

It is recommended to:

- store all samples in the dark and, particularly just before use, avoiding exposure to direct sunlight or fluorescent light in order to prevent interfering luminescence;
- comply with storage conditions specified by the scintillation cocktail supplier.

The mixtures (scintillation cocktail and test sample) should be disposed of as chemical waste, and, depending on the radioactivity, may require disposal as radioactive waste.

6.1.4 Quenching agent

Water, as well as dissolved oxygen, is a quenching agent for the scintillation cocktail.

Examples of chemical quenching agents include acetone, organochloride compounds, nitromethane, etc.

Some quenching agents are dangerous or toxic and shall be handled and disposed properly.

6.2 Equipment

6.2.1 General

Laboratory equipment, such as pipettes and balances, shall be employed that enables the expected/agreed data quality objectives to be achieved, including the uncertainty attached to the measurement.

Control of the quantity of liquid scintillation cocktail used in source preparation is essential to achieve consistent data quality.

6.2.2 Liquid scintillation counter

Liquid scintillation counter with an automatic sample transfer is preferable. Operation at constant temperature is recommended following the manufacturer's instructions.

The generic method specified in this document relates to the widely used liquid scintillation counters with vials that hold about 20 ml. When other vials are used with appropriate counters, the described method shall be adapted accordingly.

It is recommended to use a liquid scintillation counter using an external source, so that the level of quench can be determined. Otherwise, an LSC-counter with three photomultipliers and appropriate software[20][21] may enable the activity to be determined directly (see Annex B).

For low activity measurements, a counter with low background photomultipliers, electronic equipment with the option of background correction and suitable shielding is recommended.

6.2.3 Counting vials

Different types of scintillation vials exist, manufactured using a large range of materials. The most common are glass vials and polyethylene vials. Glass vials allow visual inspection of the scintillation medium, but have an inherent background, due to the presence of ^{40}K. However, some organic solvents contained in scintillation cocktails diffuse through the polyethylene, accelerating the degradation of the scintillation source.

Other types of vials exist:

— glass vials with low level of ^{40}K, exhibit a lower background than ‘normal’ glass vials;

— for the determination of very low concentration of low energy beta emitters (for example, tritium), the use of polytetrafluoroethylene vials (PTFE) or polyethylene vials with an inner layer of PTFE on inside vial wall is recommended. Diffusion of organic solvents is then slower through PTFE than through polyethylene. These vials are used for long counting times with very low-level activity to be measured.

Generally, the vials are single use. If vials are re-used, it is necessary to apply an efficient cleaning procedure.

To prevent interfering luminescence, the counting vials should be kept in the dark and should not be exposed to direct sunlight or fluorescent light, particularly just before use.

Toluene-based scintillation solutions may physically distort polyethylene and should therefore not be used in combination with polyethylene counting vials. Diffusion of organic solvents into and through the polyethylene walls is also a serious drawback of polyethylene vials.

7 Sampling and samples

7.1 Sampling

It is important that the laboratory receives a representative sample, unmodified during the transport or storage and in an undamaged container.

For example, for water and soil, conditions of sampling shall comply with ISO 5667-1 and ISO 18589-2 respectively.

If carbonated species are to be measured, water sample shall not be acidified in order to avoid changing the equilibrium of carbonated species.

For water, it is recommended to use a glass flask and to fill it to the maximum, to minimize tritium exchange with the atmospheric moisture. For low level activity measurements, it is important to avoid any contact between sample and atmosphere during the sampling.

7.2 Sample storage

If storage of samples is required, the sample shall be stored to avoid oxidation, fermentation or any modification of its properties.

For water, if storage is required, the sample shall be stored in accordance with ISO 5667-3. If the storage duration exceeds that specified in ISO 5667-3, it is advisable to store the samples in glass flasks.

8 Procedure

8.1 Determination of background

In order to determine the background count rate, a blank sample is prepared in the same way as the test sample.