ISO/FDIS 23548~~:2024(en)~~

ISO/TC 85/SC 2~~/WG 17~~

Secretariat: AFNOR

Date: 2024-~~03-29~~xx

Measurement of radioactivity — Alpha emitting radionuclides — Generic test method using alpha spectrometry

Mesurage de la radioactivité — Radionucléides émetteurs alpha — Méthode d'essai générique par spectrométrie alpha

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Contents

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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 document 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).

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

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~~www.iso.org/members.html.

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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 flora and fauna, including the human body. Human activities involving the use of radiation and radioactive substances add to additional source of exposure to radiation. 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 effluents and wastes during operation and decommissioning. The use of radioactive materials in industry, agriculture, medicine 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. The average level of occupational exposures is generally similar to the global average level of natural radiation exposure (see Reference ~~[1]).~~[1]).

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

a) ~~a)~~ improve the understanding of global levels and temporal trends of public and worker exposure,

b) ~~b)~~ evaluate the components of exposure so as to provide a measure of their relative importance, and

c) ~~c)~~ identify emerging issues that may warrant more attention and study.

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While doses to workers are mostly directly measured, doses to the public are usually assessed by indirect methods using the results of radioactivity measurements of waste, effluent and/or environmental samples.

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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 for handling, storing, preparing and measuring the test samples. An assessment of the overall measurement uncertainty also needs 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 of the test results over time and between different testing laboratories. Laboratories apply them to demonstrate their technical competences and to complete proficiency tests successfully during interlaboratory comparisons, two prerequisites for obtaining accreditation.

Today, over a hundred International Standards are available to testing laboratories for measuring radionuclides in different matrices.

Generic standards help testing laboratories to manage the measurement process by setting out the general requirements and methods to calibrate equipment 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

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- ~~—~~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 the environmental components for natural reasons 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), and

- ~~—~~human-made radionuclides, such as transuranium elements (americium, plutonium, neptunium, and curium), ^{3}H, ^{14}C, ^{90}Sr and gamma-ray emitting radionuclides found in waste, liquid and gaseous effluents, in environmental matrices (water, air, soil and biota), in food and in animal feed as a result of authorized releases into the environment, fallout from the explosion in the atmosphere of nuclear devices and fallout from accidents, such as those that occurred in Chernobyl and Fukushima. Natural components and foodstuff may thus contain radionuclides at activity concentrations which could present a risk to human health. In order to assess the radiological levels of environment and food, including the quality of drinking-water (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, natural resources and food are monitored for their radioactivity content as recommended by the World Health Organization (WHO) and the International Atomic Energy Agency (IAEA).

An international standard on a generic test method using alpha spectrometry for the determination of the activity concentration of alpha emitting radionuclides in natural and food samples or other kind of samples from nuclear facilities 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 measurements in natural, food or nuclear facilities samples.

Artificial alpha emitting radionuclide activity concentrations can vary according to authorized local effluent discharges from nuclear plant and environmental characteristics.

This document is one of a set of generic International Standards on measurement of radioactivity such as ISO 19361~~[2]~~[2] and ISO 20042~~[3].~~[3].

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Measurement of radioactivity ~~–~~ — Alpha emitting radionuclides — Generic test method using alpha spectrometry

1 Scope

This document describes a generic test method for measuring alpha emitting radionuclides, for all types of sampling (soil, sediment, construction material, foodstuff, water, airborne, environmental bio-indicator, human biological samples as urine, faeces etc.) by alpha spectrometry. This method can be used for any type of environmental study or monitoring of alpha emitting radionuclides activities.

If relevant, this test method requires appropriate sample pre-treatment followed by specific chemical separation of the test portion in order to obtain a thin source proper to alpha spectrometry measurement.

This test method can be used to determine the activity, specific activity or activity concentration of a sample containing alpha emitting radionuclides such as ^{210}Po, ^{226}Ra, ^{228}Th, ^{229}Th, ^{230}Th, ^{232}Th, ^{232}U,^{234}U, ^{235}U, ^{238}U, ^{238}Pu, $^{239+240}$Pu, ^{241}Am or $^{243+244}$Cm.

This test method can be used to measure very low levels of activity, one or two orders of magnitude less than the usual natural levels of alpha emitting radionuclides. Annexes B of UNSCEAR 2000 and UNSCEAR 2008 (References ~~[4]~~[4] and ~~[5])~~[5]) give, respectively, typical natural activity concentrations for air, foods, drinking waters and, soils and building materials. The detection limit of the test method depends on the amount of the sample material analysed (mass or volume) after concentration, chemical yield, thickness of measurement source and counting time.

The quantity of the sample to be collected and analysed depends on the expected activity of the sample and the detection limit to achieve.

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.

~~<std>ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*</std>~~

~~<std>ISO 661, *Animal and vegetable fats and oils — Preparation of test sample*</std>~~

~~<std>ISO 707, *Milk and milk products — Guidance on sampling*</std>~~

~~<std>ISO 874, *Fresh fruits and vegetables — Sampling*</std>~~

~~<std>ISO 3696, *Water for analytical laboratory use — Specification and test methods*</std>~~

~~<std>ISO 5500, *Oilseed residues — Sampling*</std>~~

~~<std>ISO 5538, *Milk and milk products — Sampling — Inspection by attributes*</std>~~

~~<std>ISO 5555, *Animal and vegetable fats and oils — Sampling*</std>~~

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~~<std>ISO 5667 (all parts), *Water quality — Sampling*</std>~~

~~<std>ISO 10381 (all parts), *Soil quality - Sampling*</std>~~

~~<std>~~ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

ISO 661, *Animal and vegetable fats and oils — Preparation of test sample*

ISO 707, *Milk and milk products — Guidance on sampling*

ISO 874, *Fresh fruits and vegetables — Sampling*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5500, *Oilseed residues — Sampling*

ISO 5538, *Milk and milk products — Sampling — Inspection by attributes*

ISO 5555, *Animal and vegetable fats and oils — Sampling*

ISO 5667 (all parts), *Water quality — Sampling*

ISO 10381 (all parts), *Soil quality - Sampling*

ISO 11929 (all parts), Determination of the characteristic limits (decision threshold, detection limit and limits of the confidence interval) for measurements of ionizing radiation — Fundamentals and application~~</std>~~

~~<std>ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*</std>~~

~~<std>ISO 17604, *Microbiology of the food chain — Carcass sampling for microbiological analysis*</std>~~

~~<std>ISO 18589-~~ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 17604, *Microbiology of the food chain — Carcass sampling for microbiological analysis*

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~~</std>~~

~~<std>ISO 24333, *Cereals and cereal products — Sampling*</std>~~

~~<std>ISO 80000-10, *Quantities and units — Part 10: Atomic and nuclear physics*</std>~~

ISO 24333, *Cereals and cereal products — Sampling*

ISO 80000-10, *Quantities and units — Part 10: Atomic and nuclear physics*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 80000-10 and ISO 11929 series and the following apply.

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ISO 11929-4, Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 4: Guidelines to applications

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 ...

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

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

— IEC Electropedia: available at ~~https://www.electropedia.org/~~https://www.electropedia.org/

3.1
thin test source
measurement source with a very thin layer deposited on a substrate containing the radionuclides of interest in order to obtain an optimal spectral resolution previously defined by the laboratory according to the expected level of activity

3.2
substrate
medium containing the source to be measured on its surface e.g. stainless steel disk, test dish or membrane filter

3.3
reference source
radioactive secondary standard source for use in the calibration of the measuring instruments i.e. material (solid, liquid) containing one or more radionuclides of known activity (Bq or Bq g^{-1} or Bq ml^{-1}), prepared such that the activity is traceable to national or international primary standards of radioactivity

3.4
alpha emitter radionuclide
radioactive nuclide with a specified atomic number and mass which emits alpha particle

3.5
energy resolution
FWHM
full width at half of the maximum of the alpha peak distribution

Note 1 to entry: The width is given in kiloelectronvolts (keV).

3.6
adsorption
process in which a substance forms a very thin layer onto the surface of a solid by attraction of the molecules

3.7
resolution
ratio between *energy resolution* ~~(3.5)~~(3.5) and energy corresponding to the maximum of the peak distribution

Note 1 to entry: The resolution is the spectral resolution and it is given in per cent.

3.8
tracer
radionuclide, which has the same chemical properties as the alpha radionuclides of interest and whose activity in the test source is used to determine the chemical yield or the total efficiency

3.9
carrier
a stable chemical element which is added to the test sample to ensure that the radionuclide of interest will behave normally in the radiochemical separation procedures

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4 Symbols and units

Table 1 — Symbols and definitions

Symbol	Definition	Unit
~~A_0~~ A_0	activity of certified calibration source at the date of the measurement	Bq
~~A_T~~ A_T	activity of the tracer added, at the date of measurement	Bq
~~a~~ a	activity of the measured radionuclides on thin test source, at the date of measurement	Bq
~~$\tilde{a}$~~ $\tilde{a}$	true activity (see ISO 11929-1)	Bq
~~a^*~~ a^*	decision threshold of the activity	Bq
~~$a^\#$~~ $a^\#$	detection limit of the activity	Bq
~~a_j~~ a_j	derived activity measured from the thin test source in the unit of ~~w_1~~ w_1	(see ~~w_1)~~ w_1)
~~a_j^*~~ a_j^*	decision threshold of the derived activity in the unit of ~~w_1~~ w_1	(see ~~w_1)~~ w_1)
~~$a_j^\#$~~ $a_j^\#$	detection limit of the derived activity in the unit of ~~w_1~~ w_1	(see ~~w_1)~~ w_1)
~~$a_j^\triangleleft$, $a_j^\triangleright$~~ $a_j^\triangleleft$, $a_j^\triangleright$	lower and upper limits of the probabilistically symmetric coverage interval of derived activity in the unit of ~~w_1~~ w_1	(see ~~w_1)~~ w_1)
~~$a_j^<$, $a_j^>$~~ $a_j^<$, $a_j^>$	lower and upper limits of the shortest coverage interval of derived activity in the unit of ~~w_1~~ w_1	(see ~~w_1)~~ w_1)
~~ε~~ ε	detection efficiency	
G	geometrical factor of alpha detection as a function of the effective solid angle of measurement, ~~Ω~~ Ω with ~~$\Omega = 4\pi \cdot G$~~ $\Omega = 4\pi \cdot G$	
~~I_α~~ I_α	summation of alpha emission intensity	
~~k~~ k	coverage factor with ~~$k = 1, 2, 3...$~~ $k = 1, 2, 3 \ldots$	
~~$k_{1-\alpha}$, $k_{1-\beta}$ and $k_{1-\gamma/2}$~~ $k_{1-\alpha}$, $k_{1-\beta}$ and $k_{1-\gamma/2}$	quantiles of the standardized normal distribution for the probabilities: ~~$1-\alpha$, $1-\beta$~~ $1-\alpha$, $1-\beta$ and ~~$1-\gamma/2$~~ $1-\gamma/2$	
~~n~~ n	number of counts in the region of interest of the spectrum	
~~n_g~~ n_g	number of gross counts in the region of interest of the spectrum	
~~n_0~~ n_0	number of counts of the global background counts in the region of interest of the spectrum	
N	number of channels of alpha peak integration	
R	total efficiency with ~~$R = R_c \cdot \varepsilon$~~ $R = R_c \cdot \varepsilon$	

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Symbol	**Definition**	**Unit**
~~R_c~~ R_c	chemical yield	
~~r~~ r	count rate per second in the region of interest of the spectrum	s^{-1}
~~r_0~~ r_0	background count rate per second of the test sample region	s^{-1}
~~r_{0T}~~ r_{0T}	background count rate per second of the blank sample region	s^{-1}
~~$r_{0\varepsilon}$~~ $r_{0\varepsilon}$	background count rate per second of the detection efficiency region	s^{-1}
~~r_g~~ r_g	gross count rate per second of the test sample region	s^{-1}
~~$r_{g\varepsilon}$~~ $r_{g\varepsilon}$	gross count rate per second of the detection efficiency region	s^{-1}
~~r_{gT}~~ r_{gT}	gross count rate per second of the tracer region	s^{-1}
S_{ref}	reference area of alpha peak integration	
t	counting time in second	s
~~t_0~~ t_0	background counting time of the test sample in second	s
~~$t_{0\varepsilon}$~~ $t_{0\varepsilon}$	background counting time of detection efficiency in second	s
~~t_{0T}~~ t_{0T}	background counting time of tracer in second	s
~~t_g~~ t_g	counting time of the test sample in second	s
~~$t_{g\varepsilon}$~~ $t_{g\varepsilon}$	counting time of detection efficiency in second	s
~~t_{gT}~~ t_{gT}	counting time of tracer in second	s
~~$u(y)$~~ $u(y)$	standard uncertainty associated with parameter ~~y~~ y result ~~$(k=1)$~~ $(k = 1)$ in unit of the measurand ~~y~~ y	
~~$U(y)$~~ $U(y)$	expanded uncertainty calculated by ~~$U(y)=k \cdot u(y)$~~ $U(y) = k \cdot u(y)$ with ~~$k>1$,~~ $k > 1$, in unit of the measurand ~~y~~ y	
~~$u_{rel}(y)$,~~ $u_{rel}(y)$,	relative standard uncertainty associated with parameter ~~y~~ y result calculated by ~~$u_{rel}(y)=u(y) \cdot y^{-1}$~~ $u_{rel}(y) = u(y) \cdot y^{-1}$	
~~$U_{rel}(y)$~~ $U_{rel}(y)$	relative expanded uncertainty calculated by ~~$U_{rel}(y)=k \cdot u_{rel}(y)$ with k > 1~~ $U_{rel}(y) = k \cdot u_{rel}(y)$ with $k > 1$	
~~w~~ w	correction factor for activity of the thin test source	Bq
~~w_1~~ w_1	correction factor for derived activity of the test sample in the inverse of the unit sample i.e.: — ~~—~~per kilogram for a solid sample with a specific activity; — ~~—~~per litre for a liquid sample with a volume activity; — ~~—~~per square metre for a contaminated surface sampled with a wipe-test; — ~~—~~per cubic metre for a volume activity of particles in air or gases sampled; — ~~—~~per one unit for specific thin test sample directly measured.	 kg^{-1} l^{-1} m^{-2} m^{-3}

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5 Principle

Generally, the test sample solution, containing alpha emitter radionuclides of interest, is mixed with an aliquot of a solution of similar physico-chemical properties and containing a radioactive tracer. This solution has at least one isotope of the radionuclides of interest contained in the sample, except if the chemical properties of the tracing radionuclide are close enough to the alpha radionuclides of interest, for example as in the case of curium and americium (see ~~Table 2).~~Table 2). This is followed by a specific preparation according to the type of sample matrix by dissolution entailing either dissolution with an acid or fusion by melting an inorganic base (e.g. alkaline fusion).

After a valence cycle to adjust the oxidation states, chemical separation of the radionuclide is achieved by a concentration step (e.g. a precipitation) followed by one or more specific separation steps (e.g. ion exchange chromatography, extraction chromatography, liquid-liquid extraction or selective precipitation).

The sample can also be measured directly by alpha spectrometry, i.e. without sample preparation or pre-treatment, no addition of carrier or radionuclide tracer, no chemical separation, or some combination of these considerations. If relevant, in this case, the user shall ensure that the quality of the measurement source allows to quantify its activity or to qualitatively determine the energies of the alpha-emitter present with a sufficient resolution previously defined by the user without degrading the performance of the detector by radioactive contamination due to a poor quality of the deposit or by volatile decay parent products, e.g. ^{210}Po, radon isotopes and their daughter nuclides. Other alpha emitting radionuclides can be present in the sample and can interfere with the counting of the radionuclide of interest to measure if no chemical separation is carried out to remove these interfering radionuclides from the test sample.

The thin test source is usually prepared by electro-deposition, by coprecipitation or by spontaneous deposition and assayed by alpha spectrometry using a grid chamber or a semiconductor-type device. The measurement relies on the interaction of the alpha particles with the detecting medium. This interaction creates an electric charge, which is amplified and output as a voltage pulse proportional to the deposited energy of the incoming alpha particle.

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The electric pulse from the detector is analysed by the electronic systems and stored by a multichannel ~~analyzer~~analyser (MCA). Data analysis software shows the alpha emitter radionuclides spectrum which is the distribution of number of pulses (counts) as a function of energy.

The analysis of the count rates in the region of interest (ROI) of the alpha spectra for the radionuclide considered allows the determination of the test sample activity concentration after correcting by blank count rate, chemical yield and detection efficiency.

The chemical yield and the detection efficiency are not necessarily determined separately. In this case, they are often obtained as of the total efficiency combined from the net count rate of the radionuclide added as a chemical yield tracer. In order to quantify any potential interference coming from the reagents, a blank sample is prepared in the same way as the test sample. This blank sample is prepared using an appropriate blank material.

For quality control, in order to quantify potential impurities in the tracer solution, another blank sample shall be prepared with addition of the tracer.

Even with a good energy resolution of ~(20 to 30) keV (FWHM) several isotopes of one radioactive element cannot easily be resolved by alpha spectrometry due to the very close similarity of their alpha emission energies, e.g.^{233}U and ^{234}U, ^{235}U and ^{236}U, ^{239}Pu and ^{240}Pu or even ^{243}Cm and ^{244}Cm.

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