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Water quality — Actinium-227 — Test method using alpha-spectrometry

Qualité de l'eau — Actinium-227 — Méthode d'essai par spectrométrie alpha

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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 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

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

Radionuclides are present throughout the environment; thus, water bodies (e.g. surface waters, ground waters, sea waters) contain radionuclides which can be of either natural, or anthropogenic origin:

- naturally-occurring radionuclides, including, ^3H , ^{14}C , ^{40}K and those originating from the thorium and uranium decay series, in particular ^{210}Pb , ^{210}Po , ^{222}Rn , ^{226}Ra , ^{228}Ra , ^{227}Ac , ^{231}Pa , ^{234}U and ^{238}U , can be found in water bodies due to either natural processes (e.g. desorption from the soil and runoff by rain water) or released from technological processes involving naturally-occurring radioactive materials (e.g. mining, mineral processing, oil, gas, and coal production, water treatment and the production and use of phosphate fertilisers);
- anthropogenic radionuclides such as ^{55}Fe , ^{59}Ni , ^{63}Ni , ^{90}Sr , ^{99}Tc , transuranic elements (Np, Pu, Am, and Cm) and some gamma emitting radionuclides such as ^{60}Co and ^{137}Cs can also be found in natural waters. Small quantities of anthropogenic radionuclides can be discharged from nuclear facilities to the environment as a result of authorized routine releases. The radionuclides present in liquid effluents are usually controlled before being discharged to the environment^[1] and water bodies. Anthropogenic radionuclides used for medical and industrial applications can be released to the environment after use. Anthropogenic radionuclides are also found in waters due to contamination from fallout resulting from above-ground nuclear detonations and accidents such as those that have occurred at the Chernobyl and Fukushima nuclear facilities.

Radionuclide activity concentrations in water bodies can vary according to local geological characteristics and climatic conditions and can be locally and temporally enhanced by releases from nuclear installations during planned, existing, and emergency exposure situations^{[2][3]}. Some drinking-water sources can thus contain radionuclides at activity concentrations that could present a human health risk. The World Health Organization (WHO) recommends to routinely monitor radioactivity in drinking waters^[4] and to take proper actions when needed to minimize the health risk.

National regulations usually specify the activity concentration limits that are authorized in drinking waters, water bodies, and liquid effluents to be discharged to the environment. These limits can vary for planned, existing, and emergency exposure situations. As an example, during either a planned or existing situation, the WHO guidance level for ^{227}Ac in drinking water is $0,1 \text{ Bq}\cdot\text{l}^{-1}$, see Notes 1 and 2. Compliance with these limits is assessed by measuring radioactivity in water samples and by comparing the results obtained with their associated uncertainties as specified by ISO/IEC Guide 98-3^[5] and ISO 5667-20^[6].

NOTE 1 If the value is not specified in Annex 6 of reference [4], the value has been calculated using the formula provided in reference [4] and the dose coefficient data from references [7] and [8].

NOTE 2 The guidance level calculated in Reference [4] is the activity concentration with an intake of $2 \text{ l}\cdot\text{d}^{-1}$ of drinking water for one year, results in an effective dose of $0,1 \text{ mSv}\cdot\text{a}^{-1}$ to members of the public. This is an effective dose that represents a very low level of risk to human health and which is not expected to give rise to any detectable adverse health effects^[4].

This document contains method(s) to support laboratories, which need to determine ^{227}Ac in water samples. The method(s) described in this document can be used for various types of waters (see Scope). Minor modifications such as sample volume and counting time can be made if needed to ensure that the characteristic limit, decision threshold, detection limit and uncertainties are below the required limits. This can be done for several reasons such as emergency situations, lower national guidance limits, and operational requirements.

Water quality — Actinium-227 — 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 essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies a test method to determine the activity concentration of ^{227}Ac in all types of waters by alpha spectrometry.

The test 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 (see ISO 5667-1, ISO 5667-3, ISO 5667-10). Filtration of the test sample is necessary.

The detection limit depends on the sample volume, the instrument used, the background count rate, the detection efficiency, the counting time, the chemical yield, and the progeny ingrowth. The method described in this document, using currently available alpha spectrometry apparatus, has a detection limit of approximately $0,03 \text{ Bq}\cdot\text{l}^{-1}$, when directly measuring the alpha peak of ^{227}Ac . This detection limit is lower than the WHO criteria for safe consumption of drinking water for any actinide alpha emitter ($0,1 \text{ Bq}\cdot\text{l}^{-1}$).^[4] This value can be achieved with a counting time of 48 h for a sample volume of 1 l.

Only a small fraction of ^{227}Ac decays through alpha emissions (~1,42 %). An option to lower the detection limit of the method is to wait, let the progenies of ^{227}Ac grow in, and measure an alpha progeny peak of ^{227}Ac (e.g. ^{215}Po). This is a longer technique, but a lower detection limit of approximately $0,000 2 \text{ Bq}\cdot\text{l}^{-1}$ can be obtained by re-counting the sample approximately 90 days after purification. The sample can be re-counted before 90 days, but with a higher detection limit.

The test method(s) described in this document can be used during planned, existing and emergency exposure situations as well as for wastewaters and liquid effluents with specific modifications that can increase the overall uncertainty, detection limit and threshold. For an emergency situation, it is preferable to reduce the counting time rather than the sample volume.

The analysis of ^{227}Ac adsorbed to suspended matter is not covered by this document.

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/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 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 water*

ISO 11929 (all parts), *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application*

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

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

3 Terms, definitions and symbols

3.1 Terms and definitions

No terms and definitions are listed in this document.

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>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.2 Symbols

For the purposes of this document, the symbols and designations given in ISO/IEC Guide 98-3, ISO/IEC Guide 99, ISO 11929 (all parts), ISO 80000-10 and the following shall apply.

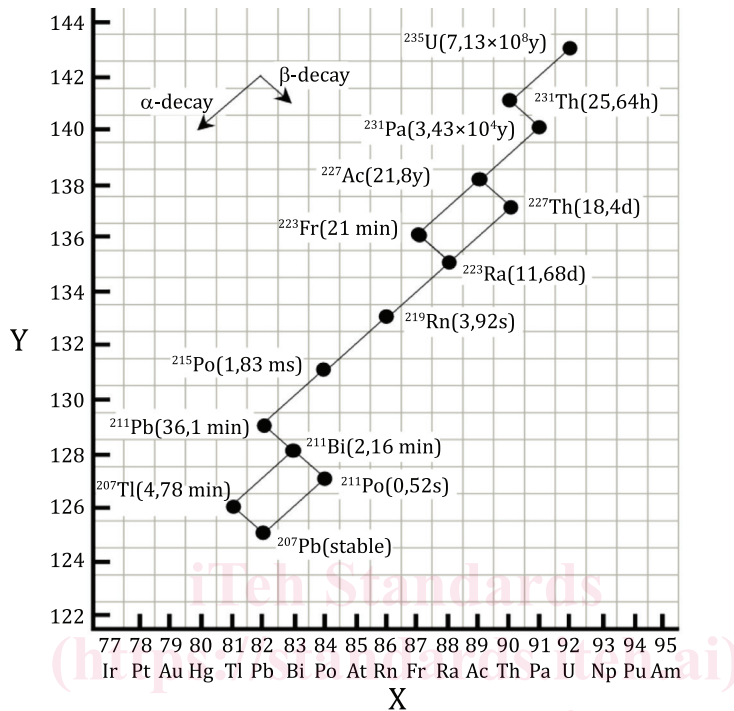
Symbol	Definition	Unit
A	Activity of ^{225}Ac tracer added	Bq
α	Probability of the false positive decision	
β	Probability of the false negative decision	
C_A	Activity concentration of ^{227}Ac measured in the sample	$\text{Bq}\cdot\text{l}^{-1}$
C_A^*	Decision threshold of the measurand	$\text{Bq}\cdot\text{l}^{-1}$
$C_A^\#$	Detection limit of the measurand	$\text{Bq}\cdot\text{l}^{-1}$
$C_A^<, C_A^>$	Lower and upper limits of the probabilistically symmetric coverage interval of the measurand, respectively	$\text{Bq}\cdot\text{l}^{-1}$
$C_A^<, C_A^>$	Lower and upper limits of the shortest coverage interval of the measurand, respectively	$\text{Bq}\cdot\text{l}^{-1}$
\widetilde{C}_A	Possible or assumed true quantity values of the measurand	$\text{Bq}\cdot\text{l}^{-1}$
C_{AT}	Activity concentration of ^{225}Ac tracer solution at the moment of separation	$\text{Bq}\cdot\text{l}^{-1}$
ε	Counting efficiency	
ε_1	Counting efficiency for the first measurement of the indirect method	
ε_2	Counting efficiency for the second measurement of the indirect method	
F	Bias correction factor for the losses of ^{219}Rn	
Φ	Distribution function of the standardized normal distribution; $\Phi(k p) = p$ applies	
$1-\gamma$	Probability for the coverage interval of the measurand	
k_p	Quantiles of the standardized normal distribution for the probabilities p (for instance $p = 1-\alpha, 1-\beta$ or $1-\gamma/2$)	

Symbol	Definition	Unit
k_q	Quantiles of the standardized normal distribution for the probabilities q (for instance $q = 1-\alpha, 1-\beta$ or $1-\gamma/2$)	
p, q	Probability for the coverage interval	
λ	Decay constant of the isotope (ex: $\lambda_{215\text{Po}}$ is the decay constant of ^{215}Po)	
m	Sample mass	kg
m_{ST}	Mass of tracer solution	g
N_o	Number of counts measured of the background on the alpha spectrum for a given time in the region of interest of ^{227}Ac , the measurand.	
N_{oT}	Number of counts measured of the background on the alpha spectrum for a given time in the region of interest of ^{225}Ac , the tracer.	
N_g	Number of counts measured on the alpha spectrum for a given time in the region of interest of ^{227}Ac , the measurand.	
N_T	Number of counts measured on the alpha spectrum for a given time in the region of interest of ^{225}Ac , the tracer.	
p, q	Probability for the coverage interval	
P_α	Probability of the isotope to decay through alpha particle emission (branching ratio)	
r_o	Background count rate in the region of interest of the measurand (^{227}Ac)	s^{-1}
r_{oT}	Background count rate in the tracer region of interest of the tracer (^{225}Ac)	s^{-1}
R	Total recovery	
R_c	Chemical recovery	
r_g	Gross count rate in the region of interest of the measurand (^{227}Ac)	s^{-1}
r_{net}	Net count rate of the isotope to measure (^{227}Ac)	s^{-1}
r_{netT}	Net count rate of the tracer (^{225}Ac)	s^{-1}
$r_{\text{netT}(1)}$	Net count rate of the tracer (^{225}Ac) for the first measurement of the indirect method	
$r_{\text{netT}(2)}$	Net count rate of the tracer (^{225}Ac) for the second measurement of the indirect method	
r_T	Gross count rate in the region of interest of the tracer (^{225}Ac)	s^{-1}
$t_{1/2}$	Radiological half-life of the isotope (ex: $t_{1/2}^{215\text{Po}}$ is the radiological half-life of ^{215}Po)	s
t	Counting time	s
t_o	Counting time of the background by alpha spectrometry	s
t_1	Time elapsed between separation and counting	s
t_g	Sample counting time by alpha spectrometry	s
U	Expanded uncertainty	
u	Standard uncertainty	
u_{rel}^2	Relative uncertainty	
$u(c_A)$	Standard uncertainty of the activity concentration of ^{227}Ac	$\text{Bq}\cdot\text{l}^{-1}$
$\tilde{u}(\tilde{c}_A)$	Standard uncertainty of the estimator C_A as a function of an assumed true value \tilde{c}_A of the measurand	$\text{Bq}\cdot\text{l}^{-1}$
$\tilde{u}(c_A^\#)$	Standard uncertainty of an estimate of the measurand when the true value is equivalent to the detection limit	
V	Sample volume	l
w	Ratio of activity concentration (C_A) on net count rate (r_{net}) (C_A/r_{net})	l^{-1}
ω	Distribution function of the standardized normal distribution	
$X_1, X_2, X_3, X_4, X_5, Z$	Terms for Formula 13	

If the results are expressed in mass activity, C_A is replaced by A and the volume, V , is replaced by the sample mass, m .

4 Principle

Actinium-227 is a naturally occurring radionuclide from the ^{235}U decay series (see [Figure 1](#)). It has a half-life of $21,772 \pm 0,003$ a^[9], which is by far the longest half-life among Ac isotopes. Actinium-227 mainly decays through beta emission (98,58 %) to ^{227}Th and slightly through alpha emission (1,42 %) to ^{223}Fr (calculated based on the sum of alpha probabilities in Reference [\[9\]](#)).



Key

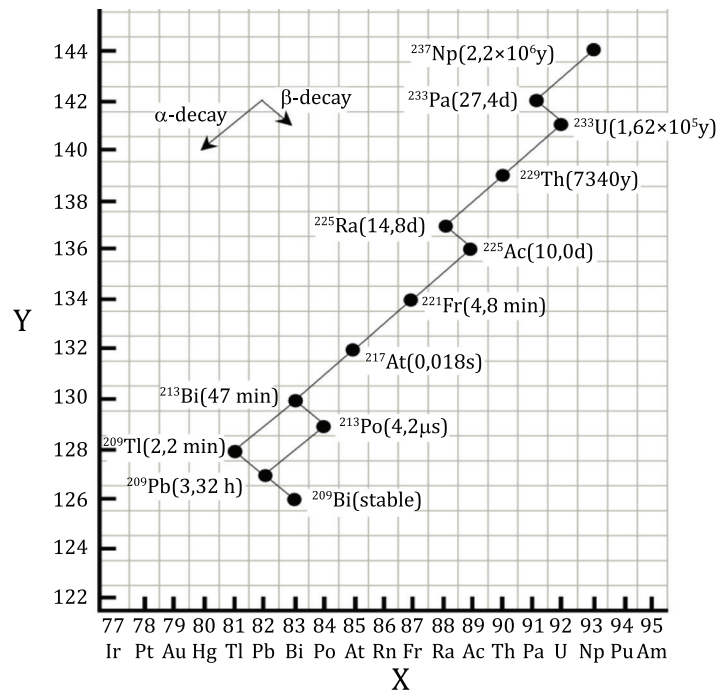
- X atomic number (Z)
- Y neutron number (N)

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Figure 1 — Decay series of ^{235}U

To determine ^{227}Ac in water, a water sample of 1 l is collected, filtered, and acidified (see [Clause 5](#)).

The ^{225}Ac tracer is added to the sample from a ^{229}Th solution (see [Figure 2](#)). Given the relatively short radiological half-life of ^{225}Ac ($10,0 \pm 0,1$ d)^[9], it is more practical to add ^{225}Ac tracer via a ^{229}Th solution of certified activity, which is in radiological equilibrium with its ^{225}Ac progeny. The parent ^{229}Th is separated from ^{225}Ac during the purification process. Enough tracer is added to obtain a good statistical precision and be easily distinguished from a blank sample (e.g. 15 mBq).

**Key**

- X atomic number (Z)
Y neutron number (N)

Figure 2 — Decay series of ^{237}Np

Actinium is preconcentrated by coprecipitation at pH 3,5. The resulting precipitate is dissolved with an acidic solution. The solution is passed through an extraction chromatography resin (EXC) to purify Ac from potential interferences. The potential radioactivity interferences for the measurement of ^{227}Ac and the ^{225}Ac tracer, are listed in Annex A. The main potential chemical interference is Ca^{2+} , which can precipitate with F^- and degrade the alpha resolution.

After purification, either a micro-precipitation with cerium fluoride (CeF_3) or an electrodeposition is performed and ^{227}Ac is measured by alpha spectrometry for 48 h. The activity concentration of ^{227}Ac is calculated and reported (see Clause 9).

5 Sampling, handling and storage

Sampling, handling and storage of the water shall be done as specified in ISO 5667-1, ISO 5667-3 and ISO 5667-10. Guidance is given for the different types of water in References [10] to [17]. It is important that the laboratory receives a sample that is truly representative and has not been damaged or modified during transportation or storage.

The sample is filtered to remove suspended matter using a 0,45 μm filter. A smaller pore size filter can also be used, but the filtration can be more tedious and time consuming. The sample shall be acidified after filtration to 1 % HNO_3 .

6 Reagents and apparatus

6.1 Reagents

Use only reagents of recognized analytical grade. It is recommended to use acids and bases of trace metal grade or equivalent (a better purity grade can also be used).