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Water quality — Plutonium, americium, curium and neptunium — Test method using alpha spectrometry

Qualité de l'eau — Plutonium, américium, curium et neptunium — 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 ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

This second edition cancels and replaces the first edition (ISO 13167:2015), which has been technically revised.

The main changes are as follows:

— addition of a description for determination of bias in the chemical recoveries of americium and curium.

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 ^{3}H, ^{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, 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 (e.g. 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 in 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 Chornobyl 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 facilities during planned, existing and emergency exposure situations.[2],[3] Some drinking water sources can thus contain radionuclides at activity concentrations that can 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 ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Am, ^{243}Cm, ^{244}Cm, ^{237}Np in drinking water is 1 $Bq{\cdot}l^{-1}$[3]. For ^{242}Cm the guidance/guidelines level (GL) is 10 $Bq{\cdot}l^{-1}$.[3] 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 that, with an intake of 2 $l{\cdot}d^{-1}$ of drinking water for one year, results in an effective dose of 0,1 $mSv{\cdot}a^{-1}$ 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 methods to determine ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Am, ^{242}Cm, ^{243}Cm, ^{244}Cm, ^{237}Np in water samples. It has been developed to support laboratories that need either a certification or accreditation to determine these nuclides in water samples. A certification or accreditation is sometimes required by local and national authorities as well as some customers. The certification and accreditation are provided by an independent body.

The methods described in this document can be used for various types of waters. 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.

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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 test method be carried out by suitably trained staff.

1 Scope

This document specifies a test method for measuring actinides (^{238}Pu, $^{239+240}$Pu, ^{241}Am, ^{242}Cm, $^{243+244}$Cm and ^{237}Np) in water samples by alpha spectrometry following a chemical separation.

This method can be used for any type of environmental study or monitoring after appropriate sampling and handling, and test sample preparation.

The detection limit of the test method is 5×10^{-3} Bq·l^{-1} to 5×10^{-4} Bq·l^{-1} for a volume of test portion between 0,1 l to 5 l with a counting time of two to ten days. This is lower than the WHO criteria for safe consumption of drinking water (1 Bq·l^{-1} or 10 Bq·l^{-1} depending on radionuclide).[4]

The method described in this document is applicable in the event of an emergency situation.

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

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

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 given in ISO/IEC Guide 98-3, ISO/IEC Guide 99, ISO 80000-10, ISO 11929-1 and the following shall apply.

Symbol	Description	Unit
A	Activity of the tracer added	Bq
α	Probability of the false positive decision	
β	Probability of the false negative decision	
c_A	Activity concentration of the measurand measured in the sample	$Bq \cdot l^{-1}$
c_A^*	Decision threshold of the measurand	$Bq \cdot l^{-1}$
$c_A^\#$	Detection limit of the measurand	$Bq \cdot l^{-1}$
$c_A^\triangleleft, c_A^\triangleright$	Lower and upper limits of the probabilistically symmetric coverage interval of the measurand, respectively	$Bq \cdot l^{-1}$
$c_A^<, c_A^>$	Lower and upper limits of the shortest coverage interval of the measurand, respectively	$Bq \cdot l^{-1}$
$\widetilde{c_A}$	Possible or assumed true quantity values of the measurand	$Bq \cdot l^{-1}$
c_{AT}	Activity concentration of the tracer solution at the moment of separation	$Bq \cdot g^{-1}$
ε	Counting efficiency	
f	Correction factor for possible bias for curium isotopes using ^{243}Am as a tracer or for ^{237}Np using ^{236}Pu as a tracer. For plutonium isotopes or for ^{241}Am, f is equal to 1	
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 $1-\gamma/2$)	
λ	Decay constant of the isotope (ex: $\lambda_{215_{Po}}$ is the decay constant of ^{215}Po)	s^{-1}
m	Sample mass	kg
m_{ST}	Mass of tracer solution	g
N_0	Number of counts measured of the background on the alpha spectrum for a given time in the region of interest of the measurand.	Counts
N_{0T}	Number of counts measured of the background on the alpha spectrum for a given time in the region of interest of the tracer.	Counts
N_g	Number of counts measured on the alpha spectrum for a given time in the region of interest of the measurand.	Counts
N_T	Number of counts measured on the alpha spectrum for a given time in the region of interest of the tracer.	Counts
P_α	Probability of the isotope decaying by alpha particle emission (branching ratio)	
r_0	Background count rate in the region of interest of the measurand	s^{-1}
r_{0T}	Background count rate in the tracer region of interest of the tracer	s^{-1}

Symbol	Description	Unit
R	Total recovery	
R_c	Chemical recovery	
r_g	Gross count rate in the region of interest of the measurand	s^{-1}
r_{net}	Net count rate of the measurand	s^{-1}
r_{netT}	Net count rate of the tracer	s^{-1}
r_T	Gross count rate in the region of interest of the tracer	s^{-1}
$t_{1/2}$	Radiological half-life of the isotope of interest	s
t_0	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	
$u(c_A)$	Standard uncertainty of the activity concentration of the measurand	$Bq{\cdot}l^{-1}$
$\tilde{u}(\widetilde{c_A})$	Standard uncertainty of the estimator c_A as a function of an assumed true value c_A of the measurand	$Bq{\cdot}l^{-1}$
V	Sample volume	l

4 Principle

The actinide isotopes included in this document are deposited as a thin source for measurement by alpha spectrometry by means of a grid chamber detector or a semi-conductor detector type equipment. The sources are usually prepared by electrodeposition or co-precipitation after chemical separation and purification of the actinides isotopes present in the test portion.[9],[10],[11],[12]

Specific chemical separation and purification procedures are required to avoid interference from the presence of other α-emitters, and stable nuclides in the sample, in quantities that are often larger than the actinide isotopes of interest. Actinides can be pre-concentrated by iron hydroxide co-precipitation at pH 8. The resulting precipitate is dissolved with an acidic solution and passed through an ion exchange, or extraction chromatography resin (see Annexes A and B) to purify the analyte from potential interferences. The potential radiological interferences for the measurement of the various radionuclides relevant to this method are listed in Annex F.

After purification, a co-precipitation with cerium fluoride (CeF_3) is performed or the analytes are electrodeposited. The actinides of interest are measured by alpha spectrometry for a suitable counting period. The activity concentrations of the actinides of interest are calculated and reported (see Clause 9 for more details).

These procedures allow the main sources of interference to be removed, namely:

— the salt content of the water sample, especially hydrolysable elements, in order to prepare the thinnest deposited source;

— other α-emitting radionuclides, such as uranium and thorium isotopes, whose emissions can interfere with those of actinide isotopes of interest.

The total recovery for each analysis (product of chemical separation yield and detection efficiency) is determined by adding a standard solution of tracer: ^{236}Pu can be used for plutonium isotopes and ^{237}Np, ^{242}Pu can be used for plutonium isotopes only and ^{243}Am can be used for americium and curium isotopes. Enough tracer is added to obtain a good statistical precision and be easily distinguished from a blank sample (e.g. about 100 mBq is often suitable).

The procedure shall include a reduction/oxidation cycle to adjust the tracer and the analytes to the same oxidation state.

It is possible to quantify curium isotopes and ^{237}Np using ^{243}Am and ^{236}Pu tracer recovery respectively. This can lead to a potential bias that shall be quantified using a standard solution, participation in inter-laboratory comparison tests, or establishment of the bias factor (see Annex E).

NOTE ^{235}Np, ^{236}Np, ^{238}Np and ^{239}Np can be used as yield tracers for ^{237}Np (if available), and ^{245}Cm as a yield tracer for other Cm isotopes but the test method of this document does not cover these measurements.

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, and guidance is given for the different types of water in References [13] to [20]. 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 pH < 2 with HNO_3.

6 Reagents and apparatus

6.1 Reagents

The chemical reagents and equipment are described in Annexes A and B for chemical separation and in Annexes C and D for the preparation of the deposited source.

Except for the certified standard solutions, all the chemical reagents needed to carry out this procedure shall be analytical grade.

6.2 Laboratory equipment

The usual laboratory equipment and, in particular, the following shall be used.

6.2.1 Vacuum filtration system.

6.2.2 Filters, of pore size 0,45 µm or smaller.

6.2.3 Glass beakers.

6.2.4 Centrifuge.

6.2.5 Multi-hole vacuum box, for example, 12 positions. (optional)

6.2.6 Analytical balance, accuracy 0,1 mg.

6.2.7 Centrifuge tubes/bottles, for example, 50 ml and 500 ml in volume.

6.2.8 Pipettes.

6.2.9 Hot plate.

6.2.10 Magnetic stirring plate.

6.2.11 Magnetic stirrer bars.

6.2.12 Metal discs with a sticky side.

6.2.13 Alpha spectrometer.

7 Procedure

Filter and acidify the sample and a blank sample prepared with ultrapure water as specified in Clause 5. A minimum of one blank sample is required for all the tests presented. However, the average of several blanks can be used and is preferred. Also, measuring blank samples at regular interval enables to rapidly detect a background issue when measuring the samples (see quality assurance and quality control program in Clause 8).

The radioactive tracers are added during this initial treatment phase.

If required, actinides can be concentrated by either evaporation or co-precipitation. If an evaporation step is performed, the resultant residue is dissolved with an acidic solution. If a co-precipitation is performed, it is often useful to add a carrier to the sample to aid collection of the precipitate. For example, iron nitrate or chloride can be added to precipitate the actinides along with $Fe(OH)_3$ at pH = 9. After centrifugation or filtration, the precipitate is dissolved with an acidic solution.

The procedure shall include an oxidation/reduction cycle to equilibrate the tracer(s) and the actinide isotopes. For example, a primarily reduction step can be carried out by adding $NH_2OH.HCl$, $NaHSO_3$ or $Na_2S_2O_3$, then an oxidation step can be done with $NaNO_2$ or H_2O_2.

7.1 Chemical separation

There are two commonly used techniques for the chemical separation of actinides: extraction on an ion exchange resin or specific extraction chromatographic resin. One method from each technique is presented in Annexes A and B: separation by anionic resin[21] or by extraction chromatographic resins.[22],[23]

It is also possible to use a combination of these techniques, for example, use an ion exchange resin followed by a specific extraction chromatographic resin for americium separation.

7.2 Preparation of the counting source

7.2.1 General

The counting source can be prepared by electrodeposition on a stainless-steel disc or by co-precipitation.

7.2.2 Electrodeposition method

Electrodeposition is carried out after the chemical separation of the actinides from interfering elements. It allows the electrochemical deposition of the actinides in an ultra-thin layer onto a stainless-steel disc. The procedure, described in Annex C, can follow either of the two chemical separation methods described in Annexes A and B.

NOTE Electrodeposition is not a selective method because some stable metal cations are likely to form insoluble hydroxides, which can be deposited at the same time as the actinides.

7.2.3 Co-precipitation method

Co-precipitation, using fluoride compounds, can be carried out after the chemical separation of the actinides from other interfering elements. The procedure, described in Annex D, can follow either of the two chemical separation methods described in Annexes A and B.

7.3 Background determination

Measure the background activity using a blank sample (e.g. laboratory water) prepared following the selected method. This blank sample should be prepared without tracer to be used for obtaining the background count rates.

7.4 Counting efficiency determination

The counting efficiency is estimated by measuring a calibration source. It is required in order to determine the chemical recovery.

7.5 Measurement

The actinide activity concentration is calculated by counting the sample source for an appropriate counting time. The same instrumental parameters should be used for the sample, the background and the calibration source measurements.

The counting time required depends on the sample and background count rates and also the detection limit and decision threshold required.

It should be verified on the spectrum that no interference is present, and an adequate energy resolution is obtained.

8 Quality assurance and quality control program

8.1 General

Quality control operations shall meet the requirements of ISO/IEC 17025. Measurement methods shall be performed by suitably skilled staff under a quality assurance program.

8.2 Variables that can influence the measurement

Special care shall be taken in order to limit as much as possible the influence of parameters that can bias the measurement and lead to a non-representative result. Failure to take sufficient precautions during the different steps of the measurement process such as sampling, transportation and storage, reagents, transfer, instrument can require corrective factors to be applied to the measured results.

8.3 Instrument verification

Major instrumental parameters (e.g. detection efficiency, calibration, background signal) shall be periodically verified within a quality assurance program established by the laboratory and in accordance with the manufacturer's instructions.

Usually, a thin alpha source with a radionuclide of long radiological half-life of known activity such as a $^{239+240}$Pu source is employed to estimate the detector efficiency because there is no appreciable decay over the working life of the source. The efficiency source should have a similar geometry to the sample sources. The alpha peak energy is calibrated using a multi-isotope source, which can be obtained commercially. The background rate of each detector is determined with an empty source support (clean disc); this shall take at least as much time as the counting of a sample source.

8.4 Contamination

Verify the absence of reagent contamination through the periodic performance of reagent blank analysis. Laboratory procedures shall ensure that laboratory and equipment contamination as well as sample cross contamination are avoided.

8.5 Interference control

It is the user's responsibility to ensure that all potential interferences have been removed. The removal of potential interferences is limited by the decontamination factor of the method. The main interferences in the measurement to the various isotopes of interest are listed in Table F.1.

8.6 Method verification

Periodic verification of the method accuracy should be performed. This can be accomplished by:

— participating in intercomparison exercises;

— analysing reference materials;

— analysing spiked samples.

The repeatability of the method should be verified (e.g. by replicate measurements).

The chemical recovery (R_c) can be calculated for quality control. It can be calculated using Formula (1):

$$R_c = \frac{R}{\varepsilon} = \frac{r_T - r_{0T}}{A \cdot \varepsilon} = \frac{r_{netT}}{A \cdot \varepsilon} \quad (1)$$

8.7 Demonstration of analyst capability

If an analyst has not performed this procedure before, a precision and bias test should be performed by running a duplicate measurement of a reference or spiked material. Acceptance limits should be within limits specified by the laboratory.

A similar evaluation should be performed by the analyst who routinely applies this procedure, with a periodicity defined by the laboratory. Acceptance limits should be within limits specified by the laboratory.

9 Expression of results

9.1 General

Measurement results are expressed as activity concentrations in $Bq \cdot l^{-1}$ or $Bq \cdot kg^{-1}$ with associated uncertainties, presented in a test report. The coverage factor for the expanded uncertainty is specified in the presentation of results.

9.2 Tracer activity added

The activity of the tracer added (e.g., ^{243}Am, ^{242}Pu) (A) is calculated using Formula (2):

$$A = m_{ST} \cdot c_{AT} \quad (2)$$

9.3 Count rate and net count rate

The count rates are calculated using Formulae (3) to (6):

$$r_g = N_g / t_g \quad (3)$$

$$r_T = N_T / t_g \quad (4)$$

$$r_0 = N_0 / t_0 \quad (5)$$

$$r_{0T} = N_{0T} / t_0 \tag{6}$$

It is recommended to count the background for up to the same amount of time as for the sample.

The net count rate of the sample (r_{net}) and the tracer ($r_{\text{net}T}$) are calculated using Formulae (7) and (8), respectively.

$$r_{\text{net}} = r_{\text{g}} - r_0 \tag{7}$$

$$r_{\text{net}T} = r_T - r_{0T} \tag{8}$$

9.4 Total recovery

The total recovery (R) is the product of the chemical recovery (R_c) and counting efficiency (ε). It is calculated using Formula (9):

$$R = R_{\text{c}} \cdot \varepsilon = r_{\text{net}T} / A \tag{9}$$

9.5 Activity concentration of the measurand

The activity concentration (c_A) of the measurand in the test sample is calculated using Formula (10):

$$c_A = \frac{r_{\text{net}}}{P_\alpha \cdot f \cdot V \cdot R} = w \cdot r_{\text{net}} \tag{10}$$

The term w in Formula (10) is isolated [see Formula (11)] to calculate the decision threshold, the detection limit and the probabilistic symmetric coverage interval.

$$w = \frac{1}{P_\alpha \cdot f \cdot V \cdot R} \tag{11}$$

When the tracer is not the same element as the analyte (for example, when ^{243}Am is used in the determination of curium isotope activity concentration, or a Pu tracer is used in the determination of ^{237}Np activity concentrations), a correction factor, f, needs to be applied to correct for the possible bias. f is the ratio of the Pu/Np or Am/Cm chemical yield. A procedure for determining the value of f in the case of using ^{243}Am as a yield tracer for Cm isotopes is given in Annex E. The method can be readily adapted to provide a value of f in the case of using a Pu yield tracer to assess Np isotope recovery.

The contribution of analytes from the reagents is not considered in Formula (10). To determine the contribution of any given actinide from the reagents, method blanks should be prepared in parallel. The average amount of the nuclide measured from the method blanks is then subtracted from each sample measured before reporting the results.

9.6 Combined uncertainties

This subclause contains the formulae needed to calculate the uncertainty on c_A. The uncertainties on ε, λ, $t_{1/2}$, t_g and t_0 are considered negligible for the calculation of $u(c_A)$. According to ISO/IEC Guide 98-3, the combined uncertainty of c_A is calculated using Formula (12):

$$u(c_A)=\sqrt{w^2\cdot\left(r_g/t_g+r_0/t_0\right)+c_A^2\cdot u_{rel}^2(w)} \tag{12}$$

The relative standard uncertainty of w is calculated using Formula (13):

$$u_{rel}^2(w)=u_{rel}^2(R)+u_{rel}^2(V)+u_{rel}^2(f) \tag{13}$$

The relative standard uncertainty of R is calculated using Formula (14):

$$u_{rel}^2(R)=\left(r_T/t_g+r_{0T}/t_0\right)/(r_T-r_{0T})^2+u_{rel}^2(A) \tag{14}$$

The relative standard uncertainty of A is calculated using Formula (15):

$$u_{rel}^2(A)=u_{rel}^2(m_{ST})+u_{rel}^2(c_{AT}) \tag{15}$$

For quality assurance, the combined uncertainty of R_c, $u(R_c)$, is calculated using Formula (16):

$$u(R_c)=R_c\sqrt{u_{rel}^2(R)+u_{rel}^2(\varepsilon)} \tag{16}$$

If needed, calculate the standard uncertainty of c_A as a function of its true value, noted $\tilde{u}(\tilde{c}_A)$, using Formula (17):

$$\tilde{u}(\tilde{c}_A)=\sqrt{w^2\cdot\left((\tilde{c}_A/w+r_0)/t_g+r_0/t_0\right)+\tilde{c}_A^2\cdot u_{rel}^2(w)} \tag{17}$$

9.7 Decision threshold

The decision threshold c_A^*, expressed in $Bq\cdot l^{-1}$, is obtained from Formula (18) (see ISO 11929-1).

This yields:

$$c_A^*=k_{1-\alpha}\cdot w\cdot\sqrt{\left(r_0/t_g\right)+\left(r_0/t_0\right)} \tag{18}$$

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

When $t_g=t_0=t$, c_A^* is calculated using Formula (19):

$$c_A^*=k_{1-\alpha}\cdot w\cdot\sqrt{2N_0}/t \tag{19}$$

When the background is very low, or when $N_0=0$, c_A^* is calculated with Formula (20) according to ISO 11929-2:

$$c_A^*=k_{1-\alpha}\cdot w\cdot\sqrt{2(N_0+1)}/t \tag{20}$$

9.8 Detection limit

The detection limit, $c_A^{\#}$, is calculated using the implicit Formula (21) according to ISO 11929-1:

$$c_A^{\#} = c_A^{*} + k_{1-\beta} \cdot \tilde{u}\left(c_A^{\#}\right) = c_A^{*} + k_{1-\beta} \cdot \sqrt{w^2 \cdot \left(\left(c_A^{\#} / w + r_0\right) / t_g + r_0 / t_0\right) + c_A^{\#2} \cdot u_{rel}^2(w)} \tag{21}$$

$\beta = 0{,}05$ and then, $k_{1-\beta} = 1{,}65$ is often chosen by default.

The detection limit can be calculated by solving Formula (22) for $c_A^{\#}$ or, more simply, by iteration with a starting approximation $c_A^{\#} = 2 \cdot c_A^{*}$.

When taking $k_{1-\alpha} = k_{1-\beta} = k$, the solution of Formula (21) is given by Formula (22):

$$c_A^{\#} = \left(2c_A^{*} + \left(w \cdot k_{1-\alpha}^2\right) / t_g\right) / \left(1 - k_{1-\alpha}^2 \cdot u_{rel}^2(w)\right) \tag{22}$$

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

9.9 Probabilistically symmetric coverage interval

9.9.1 Limits of the probabilistically symmetric coverage interval

The lower, $c_A^{\triangleleft}$, and upper, $c_A^{\triangleright}$, coverage limits are calculated using Formulae (23) and (24) according to ISO 11929-1:

$$c_A^{\triangleleft} = c_A - k_p \cdot u(c_A) \text{ with } p = \omega \cdot (1 - \gamma / 2) \tag{23}$$

$$c_A^{\triangleright} = c_A + k_q \cdot u(c_A) \text{ with } q = 1 - \omega \cdot \gamma / 2 \tag{24}$$

where

$\omega = \Phi[y / u(y)]$, Φ being the distribution function of the standardized normal distribution;

$(1 - \gamma)$ is the probability for the coverage interval of the measurand;

$\omega = 1$ may be set if $c_A \cdot \geq 4\, u(c_A)$.

In this case:

$$c_A^{\triangleleft}, c_A^{\triangleright} = c_A \pm k_{1-\gamma/2} \cdot u(c_A) \tag{25}$$

γ = 0,05 and then, $k_{1-\gamma/2}$ = 1,96 are values often chosen by default.

9.9.2 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 Formula (26):

$$c_A^<, c_A^> = c_A \pm k_p \cdot u(c_A); p = (1 + \omega \cdot (1 - \gamma)/2) \tag{26}$$

or in the case where $c_A^< < 0$, by Formula (27):

$$c_A^< = 0, c_A^> = c_A \pm k_q \cdot u(c_A); q = 1 - \omega \cdot \gamma \tag{27}$$

$\omega = \Phi[y/u(y)]$, Φ being the distribution function of the standardized normal distribution.

The relations $0 \leq c_A^< < c_A^>$ apply and the approximation of Formula (25) is valid.

10 Test report

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

a) a reference to this document, i.e. ISO 13167:2023;

b) the method used;

c) identification of the sample;

d) units in which the results are expressed;

e) the test result:

   1) when the activity concentration, c_A, is compared with the decision threshold (see the ISO 11929 series);

      — if the result is less than the decision threshold, the result of the measurement is expressed as $\leq c_A^*$,

      — if the result is greater than the decision threshold, the result of the measurement is expressed as $c_A \pm u_c(c_A)$ or $c_A \pm U$ with the associated k value,

   2) when the activity concentration, c_A is compared with the detection limit;

      — if the result is less than the detection limit, the result of the measurement is expressed as $\leq c_A^\#$,

      — if the result is greater than the detection limit, the result of the measurement is expressed as $c_A \pm u_c(c_A)$ or $c_A \pm U$ with the associated k value;

f) any deviation from the procedure;

g) any unusual features observed;

h) the date of the test.

Complementary information can be provided such as:

a) 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 $c_A^<$, $c_A^>$;

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

c) decision threshold and the detection limit;

d) if the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose;

e) mention of any relevant information likely to affect the results.

NOTE It is occasionally requested by the customer or regulator to compare the primary measurement result, c_A, with the detection limit, $c_A^{\#}$, in order to decide whether the physical effect is recognized or not. Such stipulations are not in accordance with the ISO 11929 series. They have the consequence that it is decided too frequently that the physical effect is absent when in fact it is not absent.

Annex A
(normative)

Chemical separation of actinides on anionic resin

A.1 Principle

The actinide isotopes are separated from the other radionuclides and from the matrix in an anionic complex form using an ion exchange resin in a column in a nitric acid medium and hydrochloric one.

A.2 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

A.2.1 Hot plate.

A.2.2 Glass or plastic column.

A.3 Reagents

A.3.1 Tracer solutions.

A.3.2 Hydrochloric acid, concentrated, $c(HCl) = 12\ mol \cdot l^{-1}$.

A.3.3 Nitric acid, concentrated, $c(HNO_3) = 15{,}7\ mol \cdot l^{-1}$.

A.3.4 Ammonia, concentrated, $w(NH_4OH)$: 25 %.

A.3.5 Sodium nitrite ($NaNO_2$).

A.3.6 Anionic resin 1 × 4 50/100 mesh or 1 × 8 100/200 mesh.

A.3.7 Nitric acid, solution $c(HNO_3) = 8\ mol \cdot l^{-1}$

A.3.8 Hydrochloric acid, solution $c(HCl) = 0{,}2\ mol \cdot l^{-1}$.

A.3.9 Hydrochloric acid, solution, $c(HCl) = 2{,}0\ mol \cdot l^{-1}$.

A.3.10 Hydrochloric acid, solution, $c(HCl) = 8\ mol \cdot l^{-1}$.

A.3.11 Hydrochloric acid, solution, $c(HCl) = 10\ mol \cdot l^{-1}$.

A.3.12 Hydroxylamine hydrochloride, $c(NH_2OH.HCl) = 0{,}2\ mol \cdot l^{-1}$ in hydrochloric acid, $c(HCl) = 2\ mol \cdot l^{-1}$.

A.3.13 Methanol.

A.3.14 Solution A: 10 % (vol) 10 mol·l^{-1} HNO_3 and 90 % (vol) of methanol, freshly made.

A.3.15 Solution B: 30 % (vol) 0,5 mol·l^{-1} HNO_3 and 70 % (vol) of methanol, freshly made.

A.4 Procedure

A.4.1 General

Add a known activity of tracers to the water sample.

This procedure is carried out with two main steps: chemical separation and elution.

For chromatographic columns the flow rate should be approximately 1 ml·min^{-1}, unless otherwise specified.

NOTE In some cases, the flow through resin columns is very poor because of sample composition. Working with vacuum box systems is helpful to support the flow rate.

A.4.2 Separation of plutonium and neptunium

a) Prepare the sample test solution according to Clause 7. Evaporate the solution to dryness and dissolve the residue in about 50 – 100 ml of 8 mol·l^{-1} HNO_3 (A.3.7).

b) Add 0,6 g of $NaNO_2$ (A.3.5). Cover the beaker with a watch glass and heat until brown fumes are no longer evolved.

c) Fill the column with the resin (A.3.6). (For example, a 50 ml volume column can be filled with about 10 ml of prepared resin. This is suitable for load samples of about 50 ml to 100 ml. Column size can be scaled for different volumes of test solution.)

d) Convert the resin to the nitrate form by passing 50 ml of 8 mol·l^{-1} HNO_3 (A.3.7).

e) Place a clean beaker under the column and load the test solution on the column.

f) Wash the load beaker and column with portions of 8 mol·l^{-1} HNO_3 (A.3.7) up to 50 ml with a flow rate of approximately 1 ml·min^{-1}. This eluate (E1) contains Am, Cm, Fe and U.

g) Place a clean beaker under the column and wash the column with 100 ml of 8 mol·l^{-1} HCl (A.3.10). This eluate (E2) contains Th.

A.4.3 Elution of plutonium and neptunium

a) Place a clean beaker under the column.

b) Elute the plutonium and neptunium (E3) with 50 ml of 0,2 mol·l^{-1} $NH_2OH.HCl$ in 2 mol·l^{-1} HCl (A.3.9) with a flow rate of approximately 0,5 ml·min^{-1}.

NOTE Elution can also be achieved by washing the column with 0,1 mol·l^{-1} ammonium iodide in 8,5 mol·l^{-1} HCl.[9]

c) Evaporate the eluate E3 slowly until the volume is less than 25 ml and carefully add 2 ml of concentrated HNO_3 (A.3.3).

When the presence of significant amounts of other actinides is suspected, it is necessary to repeat the purification process from A.4.2 a) to A.4.3 c).

d) Prepare the source by electrodeposition or co-precipitation, as described in Annex C and Annex D, respectively.

A.4.4 Separation of americium and curium

a) Evaporate eluate E1 to dryness.

b) Dissolve the residue with 50 ml of 10 $mol \cdot l^{-1}$ HCl (A.3.11).

c) Fill the column with 15 ml of anionic resin (A.3.6).

d) Prepare 50 ml of a 10 $mol \cdot l^{-1}$ HCl (A.3.11) solution and wash the column to convert the resin to the chloride form. Discard the washings.

e) Place a clean beaker under the column and transfer the solution obtained in b) into the top of the column.

f) Wash the beaker and column with portions of 10 $mol \cdot l^{-1}$ HCl (A.3.11) up to 50 ml. Eluate E4 is obtained from combining the eluates from e) and f).

g) Evaporate eluate E4 to dryness.

A.4.5 Purification of americium and curium

Carry out the following steps.

a) Dissolve the residue of eluate E4 with 10 ml of 8 $mol \cdot l^{-1}$ HNO_3 (A.3.7).

b) Stir and add 90 ml of methanol.

c) Fill the column with 15 ml of anionic resin (A.3.6).

d) Prepare 50 ml of a fresh solution A (A.3.14) and add to the resin.

e) Pour the solution into the top of the column.

f) Wash the beaker and column with 50 ml of solution A (A.3.14).

g) Place a clean beaker under the column and elute americium and curium with 100 ml of a fresh solution B (A.3.15) as E5.

h) Evaporate to dryness the eluate E5.

i) Dissolve the residue with:

   1) 1 ml of concentrated HNO_3 (A.3.3) in case of electrodeposition;

   2) 50 ml of 0,2 $mol \cdot l^{-1}$ HCl (A.3.12) in case of co-precipitation.

j) Prepare the source by electrodeposition or co-precipitation, as described in Annexes C and D, respectively.

Annex B
(normative)

Chemical separation of actinides by specific resins

B.1 Principle

This technique is based on the selective chromatographic extraction of plutonium isotopes, ^{241}Am, curium isotopes and ^{237}Np using a resin coated with a specific extractant CMPO/TBP. The chemical separation is fast and well suited for monitoring plutonium and americium activity in the environment.

B.2 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

B.2.1 Scales, to an accuracy of 0,1 mg.

B.2.2 Evaporator or hot plate.

B.2.3 CMPO/TBP extractant-coated resin columns (2 ml volume in general).

B.3 Reagents

B.3.1 Tracer solutions.

B.3.2 Aluminium nitrate $c(Al(NO_3)_3)$ = 0,5 mol·l^{-1} in nitric acid $c(HNO_3)$ = 3 mol·l^{-1}.

B.3.3 Sodium nitrite, $c(NaNO_2)$ = 0,1 mol·l^{-1} in nitric acid, $c(HNO_3)$ = 2 mol·l^{-1}.

B.3.4 Nitric acid, concentrated, $c(HNO_3)$ = 15,7 mol·l^{-1}.

B.3.5 Nitric acid (HNO_3) solution: $c(HNO_3)$ = 3 mol·l^{-1}.

B.3.6 Nitric acid (HNO_3) solution: $c(HNO_3)$ = 2 mol·l^{-1}.

B.3.7 Nitric acid (HNO_3) solution: $c(HNO_3)$ = 1 mol·l^{-1}.

B.3.8 Nitric acid (HNO_3) solution: $c(HNO_3)$ = 0,5 mol·l^{-1}.

B.3.9 Ascorbic acid.

B.3.10 Hydrogen peroxide $w(H_2O_2)$: 30 %.

B.3.11 Hydrochloric acid, concentrated, $c(HCl)$ = 12 mol·l^{-1}.

B.3.12 Hydrochloric acid solution, $c(HCl)$ = 0,2 mol·l^{-1}.

B.3.13 Hydrochloric acid solution, $c(HCl) = 9\ mol \cdot l^{-1}$.

B.3.14 Hydrochloric acid solution, $c(HCl) = 4\ mol \cdot l^{-1}$.

B.3.15 Ammonium hydrogen oxalate, $c(NH_4HC_2O_4) = 0{,}1\ mol \cdot l^{-1}$ in hydrochloric acid, $c(HCl) = 1\ mol \cdot l^{-1}$.

B.3.16 Hydrofluoric acid, $c(HF) = 0{,}1\ mol \cdot l^{-1}$ in hydrochloric acid, $c(HCl) = 4\ mol \cdot l^{-1}$.

B.4 Procedure

B.4.1 General

Add a known activity of tracers to the water sample acidified with nitric acid.

This procedure is carried out with two main steps: chromatographic extraction and elution of americium, curium, plutonium and neptunium. For chromatographic columns, the flow rate should be approximately 1 ml min^{-1} unless otherwise specified.

A ferric hydroxide co-precipitation can be added before the separation steps to concentrate actinides.

NOTE In some case, the flow through resin columns is very poor because of sample composition. Working with vacuum box systems is helpful to support the flow.

B.4.2 Extraction of americium and curium

a) Following preparation according to Clause 7, evaporate the solution to be analysed to dryness.

b) Add 10 ml of 0,5 mol l^{-1} $Al(NO_3)_3$ in 3 $mol \cdot l^{-1}$ HNO_3 (B.3.2) to dissolve the precipitate.

c) Add 0,1 g of ascorbic acid (B.3.9) to keep all Fe as Fe(II) (to ensure reduction of Pu and Np but also to be sure no Fe(III) can interfere with Pu(III) and Am(III) uptake), heat until total dissolution and leave to cool.

d) Set up a CMPO/TBP column of 2 ml volume.

e) Prepare the resin by passing 25 ml 0,5 mol l^{-1} $Al(NO_3)_3$ in 3 $mol \cdot l^{-1}$ HNO3 (B.3.2).

f) Transfer the solution into the top of the column.

g) Wash the beaker with 5 ml of 3 $mol \cdot l^{-1}$ HNO_3, (B.3.5) transfer into the top of the column.

h) Wash the column with 5 ml of 2 $mol \cdot l^{-1}$ HNO_3 (B.3.6), 5 ml of 0,1 $mol \cdot l^{-1}$ $NaNO_2$ in 2 $mol \cdot l^{-1}$ HNO_3 (B.3.3). Discard the washings.

i) Place a clean beaker under the column and elute americium and curium with 5 ml 0,5 $mol \cdot l^{-1}$ HNO_3 (B.3.8), 3 ml 9 $mol \cdot l^{-1}$ HCl (B.3.13) and 20 ml of 4 $mol \cdot l^{-1}$ HCl (B.3.14) (eluate E6).

j) Evaporate the eluate E6 to dryness.

k) Dissolve the residue with:

   1) 1 ml of concentrated HNO_3 (B.3.4) in case of electrodeposition;

   2) 50 ml of 0,2 $mol \cdot l^{-1}$ HCl (B.3.12) in case of co-precipitation.

l) Prepare the source by electrodeposition or co-precipitation, as described in Annex C and Annex D, respectively.

B.4.3 Elution of plutonium and neptunium

a) Place a clean beaker under the column.

b) Elute plutonium and neptunium (eluate E7) with 20 ml of 0,1 mol·l^{-1} $NH_4HC_2O_4$ in 1 mol·l^{-1} HCl (B.3.15).

NOTE U is not eluted.

c) Evaporate eluate E7 to dryness. The white precipitate is in the oxalate form.

Thorium is known to be very insoluble in water. If the presence of thorium is suspected in the sample, it is necessary, prior to Pu/Np elution, to add a step to wash the column with 20 ml of 0,1 mol·l^{-1} HF in 4 mol·l^{-1} HCl (B.3.16) to elute thorium as it interferes with plutonium in α-spectrometry.

B.4.4 Oxalate decomposition

Carry out the following operations.

a) Add 1 ml of concentrated HNO_3 (B.3.4), and five drops of 30 % H_2O_2 (B.3.10) evaporate to dryness.

b) Add 1 ml of concentrated HNO_3 (B.3.4), and 1 ml of concentrated HCl (B.3.11), evaporate to dryness.

c) Add 1 ml of concentrated HNO_3 (B.3.4), evaporate to dryness.

d) Repeat step c) until the precipitate disappears.

e) Dissolve the residue in 4 ml of 1 mol·l^{-1} HNO_3 (B.3.7) (eluate E8).

f) Evaporate eluate E8 to dryness.

g) Dissolve the residue with:

1) 1 ml of concentrated HNO_3 (B.3.4) in case of electrodeposition;

2) 50 ml of 0,2 mol·l^{-1} HCl (B.3.12) in case of co-precipitation.

h) Prepare the source by electrodeposition or co-precipitation, as described in Annex C and Annex D, respectively.

Annex C
(normative)

Preparation of the source by electrodeposition

C.1 Principle

Using a DC generator to apply a voltage between two electrodes leads to the reduction of the metal cations dissolved in the electrolyte. The reduction that takes place at the cathode leads to the formation of a deposit of actinides.

C.2 Apparatus

C.2.1 Electrodeposition apparatus, generally composed of the following.

C.2.1.1 Glass or polyethylene electrodeposition cell.

C.2.1.2 Platinum wire (anode).

C.2.1.3 Stainless-steel disc (cathode) with a diameter adapted to the electrodeposition cell.

C.2.1.4 DC power supply.

C.2.2 Hot plate.

C.2.3 Petri dish (glass or plastic).

C.3 Reagents

C.3.1 Nitric acid, concentrated $w(HNO_3)$: 65 %.

C.3.2 Sodium sulfate $c(Na_2SO_4)$: 0,3 $mol \cdot l^{-1}$.

C.3.3 Sulfuric acid, concentrated $w(H_2SO_4)$: 95 % to 97 %.

C.3.4 Thymol blue $w(C_{27}H_{30}O_5S)$: 0,04 %.

C.3.5 Ammonia, concentrated $w(NH_4OH)$: 25 %.

C.3.6 Sulfuric acid $w(H_2SO_4)$: 1 %.

C.3.7 Ammonia $w(NH_4OH)$: 1 %.

C.4 Procedure

C.4.1 Assembly of the electrodeposition cell

This procedure refers to the electrodeposition apparatus designed for small diameter stainless-steel disks (see Figure C.1). The electrodeposition procedure for this type of apparatus is as follows.

a) Place the clean and degreased stainless-steel disk (C.2.1.3) in the cap assembly.

b) Assemble the cell with the cap.

c) Fill the cell with water to check for leaks.

d) Empty the cell.

e) Fix the platinum anode (C.2.1.2) vertically to its support. The distance between the anode and the disk (cathode) is approximately 3 mm.

C.4.2 Analyte deposition

Using the preparation obtained from the elution and purification phase (see A.4.3 and A.4.5) or from the extraction and oxalate decomposition step (see B.4.2 and B.4.4), carry out the following operations.

a) Evaporate to near dryness.

b) Add 1 ml of concentrated HNO_3 (C.3.1) and evaporate near dryness. Repeat this step three times.

c) Add 1 ml of 0,3 mol·l^{-1} Na_2SO_4 (C.3.2) and evaporate to dryness.

d) Add 0,5 ml of concentrated H_2SO_4 (C.3.3).

e) Add 10 ml of distilled water and 3 drops of 0,04 % thymol blue (C.3.4) and stir.

f) Adjust the pH between 2,1 and 2,3 by adding concentrated ammonia solution (colour changes from red to orange, a pH-meter can also be used).

g) Introduce this solution in the electrodeposition cell.

h) Rinse the beaker with a few millilitres of 1 % H_2SO_4 (C.3.6), whose pH is previously adjusted to 2,3; add to the electrodeposition cell.

i) Set up the anode and carry out the electrodeposition at a constant current (about 1,0 A) for approximately 1 h. Low temperatures improve the efficiency of electrodeposition.

NOTE Lower currents can be used if electrodeposition time is increased (e.g. 0,5A for 2 h). This helps reduce overheating of the cell.

j) One minute before switching off the generator, add 1 ml of concentrated NH_4OH (C.3.5).

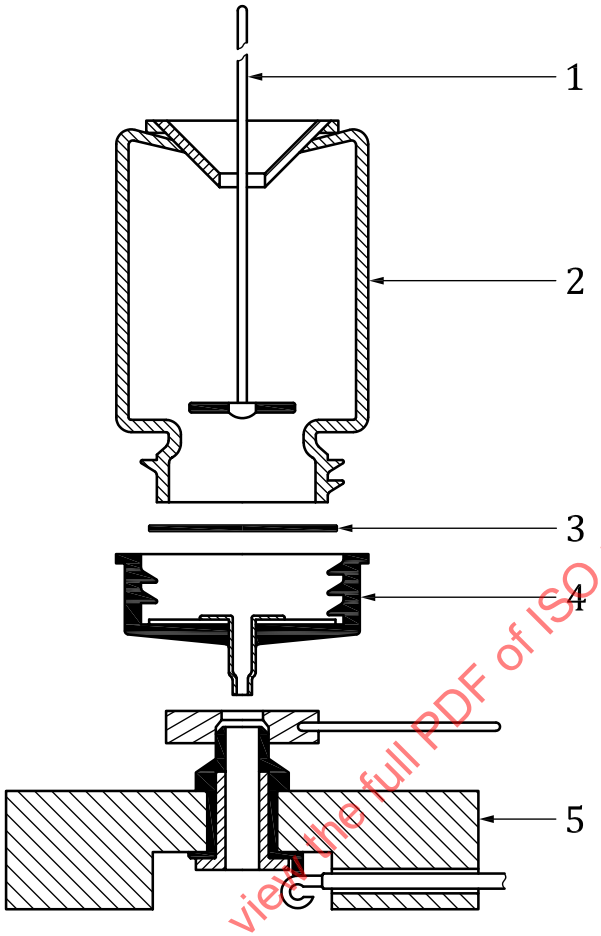
k) Wait 1 min, remove the anode before switching off the generator and quickly empty the cell.

l) Dismantle the apparatus, rinse the disk in water, then in 1 % NH_4OH, and dry it using flow of hot air or on a hot plate.

m) Identify the disk and place it in a Petri dish.

n) The source is ready to be measured by α-spectrometry.

Cooling the cell during electrodeposition can avoid an increase of temperature that can lead to excessive bubbling.



Key

1 anode
2 scintillation vial
3 disk
4 cap assembly
5 base

Figure C.1 — Diagram of an electrodeposition cell (reproduced with permission from Talvitie) [24]

Annex D
(normative)

Preparation of the alpha source by lanthanide fluoride co-precipitation

D.1 Principle

A thin source layer of the actinide isotopes is prepared by co-precipitation with a trivalent lanthanide (Ln) fluoride (e.g. lanthanum or cerium).

D.2 Apparatus

The equipment used for the co-precipitation method generally includes the following.

D.2.1 Cellulose ester membrane filter (pore diameter: 0,22 µm – 0,1 µm, 25 mm diameter).

D.2.2 Stainless-steel disc with a sticky side (the diameter should be the same as for the filter).

D.2.3 Filtration system.

D.3 Reagents

D.3.1 Lanthanide solution, $c(Ln^{3+}) = 500\ \mu g \cdot ml^{-1}$ (e.g. Ce or La) (nitrate or chloride salt).

D.3.2 Titanium(III) trichloride in HCl solution (e.g. 12M HCl), $c(TiCl_3) = 0{,}78\ mol \cdot l^{-1}$.

D.3.3 Ammonia concentrated (NH_4OH).

D.3.4 Hydrofluoric acid solution, c(HF): 28,9 $mol \cdot l^{-1}$ (a solution of ammonium fluoride (NH_4F), with the same molarity, can be used instead).

D.3.5 Ethanol.

D.4 Procedure

D.4.1 Ensure the sample is dissolved with a solution of HCl and that the acid molarity is between 0,01 and 0,1 $mol \cdot l^{-1}$. Add NH_4OH to adjust the molarity if needed. The sample shall be in a plastic container that can be closed with a cap.

NOTE 1 The acid molarity can be higher, but the recovery will be lower.

NOTE 2 HNO_3 can be used instead of HCl when only interested in actinides that are usually stable at oxidation states +3 and +4 such as Ac^{3+}, Th^{4+}, Am^{3+}, and Cm^{3+}.

D.4.2 Add 0,1 ml of titanium trichloride solution (D.3.2).

NOTE There is no need to add this solution when only interested in actinides that are usually stable at oxidation states +3 and +4 such as Ac^{3+}, Th^{4+}, Am^{3+}, and Cm^{3+}.