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Measurement of radioactivity in urine- ^{238}Pu , ^{239}Pu and ^{240}Pu — Test method using alpha spectrometry or ICP-MS

National foreword

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ISO 18990

**Measurement of radioactivity in
urine- ^{238}Pu , ^{239}Pu and ^{240}Pu — Test
method using alpha spectrometry
or ICP-MS**

*Mesurage de la radioactivité dans les urines- ^{238}Pu , ^{239}Pu et
 ^{240}Pu — Méthode d'essai utilisant la spectrométrie alpha ou
l'ICP-MS*

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Foreword

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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

In the course of employment, individuals might work with radioactive materials that, under certain circumstances, could be taken into the body. Minimising the risks to workers from incorporated radionuclides requires the monitoring of potential or actual intakes. This monitoring involves the measurement of the activity of the radionuclides in the body (in vivo measurement) and/or biological samples, e.g. urine or faeces (in vitro measurement).

Analytical methods for plutonium urine bioassay are addressed in this document because of:

- the convenience of urine sampling and relatively easy sample processing. Urine bioassay is the most commonly used in vitro measurement method for accurately assessing the magnitude of internal contamination;
- the high radiotoxicity of Pu isotopes (e.g. ^{238}Pu , ^{239}Pu and ^{240}Pu) due to their long half-lives, highly energetic alpha emission and ability to accumulate into bone and organs;
- the major contribution of Pu isotopes to the internal contamination in many situations, and the relatively low detection limits of Pu isotopes compared to the other highly radiotoxic actinides.

For routine individual monitoring, the detection of all exposures whose sum can lead to an annual dose exceeding 1 mSv should be ensured according to ISO 20553. In emergency situations, the urine bioassay techniques should be sufficiently sensitive to meet the maximum value for reference level of 0,1 Sv recommended by the International Commission on Radiological Protection (ICRP)^{[1][2][3]}.

This document offers general requirements for sample processing and measurement of the activity concentration of Pu isotopes in urine samples. Examples of detailed procedures are given in the annexes for different monitoring situations.

Measurement of radioactivity in urine- ^{238}Pu , ^{239}Pu and ^{240}Pu — Test method using alpha spectrometry or ICP-MS

1 Scope

This document specifies approaches for the determination of plutonium isotopes (^{238}Pu , ^{239}Pu and ^{240}Pu) in urine using alpha spectrometry or inductively coupled plasma mass spectrometry (ICP-MS).

It is applicable to the measurement of plutonium isotopes at levels which are appropriate for

- workers handling plutonium in planned exposure situations, where detection limits are sufficiently low to be in accordance with dose limits, and
- workers, members of the public and emergency responders in emergency exposure situations, where required detection limits can be much higher, and results need to be reported in a short timescale.

This document does not provide information on when monitoring is carried out or the interpretation of the results in terms of dose or biological effects.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes the 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 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 13167, *Water quality — Plutonium, americium, curium and neptunium — Test method using alpha spectrometry*

ISO 15189, *Medical laboratories — Requirements for quality and competence*

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

ISO 17294-1, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General requirements*

ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of selected elements including uranium isotopes*

ISO 20553, *Radiation protection — Monitoring of workers occupationally exposed to a risk of internal contamination with radioactive material*

ISO 20899, *Water quality — Plutonium and neptunium — Test method using ICP-MS*

ISO 28218, *Radiation protection — Performance criteria for radiobioassay*

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 28218, ISO 20553, ISO 80000-10, ISO 11929 (series), ISO 17924-1, ISO 20899 and the following apply.

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>

3.1

reference level

dose criterion used to guide the optimisation process in existing and emergency exposure situations

Note 1 to entry: Generally expressed in terms of individual annual dose ($\text{mSv}\cdot\text{year}^{-1}$), the value of a reference level should be selected considering the appropriate time frame, individual dose distribution of the affected people, and the tolerability of risk in the circumstances. An objective is to facilitate the identification of people for whom protective efforts should be given priority.

3.2

spot urine sample

urine sample where the collection duration is shorter than 24 h (normally a single voiding)

3.3

sample turn-around time

total time for sample processing and measurement

3.4

sample preparation volume

volume of sample taken from the collected sample for pretreatment and subsequent analysis

4 Symbols

For the purposes of this document, the symbols given in ISO/IEC Guide 98-3, ISO 80000-10, ISO 11929-1 and the following shall apply.

Symbol	Description	Unit
A_0	Activity of the certified calibration source, on the date of the measurement	Bq
A_T	Activity of the tracer added on the measurement date	Bq
α	Probability of the false positive decision	—
β	Probability of the false negative decision	—
c_A	Activity concentration of plutonium isotopes	$\text{Bq}\cdot\text{l}^{-1}$
c_A^*	Decision threshold of the measurand	$\text{Bq}\cdot\text{l}^{-1}$
$c_A^\#$	Decision 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	$\text{Bq}\cdot\text{l}^{-1}$
$\tilde{c}_A$	Possible or assumed true quantity values of the measurand	$\text{Bq}\cdot\text{l}^{-1}$
c_{AT}	Activity concentration of the tracer solution at the moment of separation	$\text{Bq}\cdot\text{g}^{-1}$
ε	Counting efficiency	—
Φ	Distribution function of the standardized normal distribution	—
$1-\gamma$	Probability for the coverage interval of the measurand	—
I_α	Summation of alpha emission intensity	—
λ	The probability per unit time that an individual radioactive nucleus will decay, as defined by the exponential decay law (decay constant).	—

Symbol	Description	Unit
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	$1 \cdot s^{-1}$
$r_{0\varepsilon}$	Background count rate of the detection efficiency region	$1 \cdot s^{-1}$
r_{0T}	Background count rate in the tracer region of interest of the tracer	$1 \cdot s^{-1}$
R	Total measurement yield	—
R_C	Chemical recovery	—
r_g	Gross count rate per second for the tracer and plutonium isotopes	$1 \cdot s^{-1}$
$r_{g\varepsilon}$	Gross count rate of the detection efficiency region	$1 \cdot s^{-1}$
r_{net}	Net count rate of the measurand	$1 \cdot s^{-1}$
r_{netT}	Net count rate of the tracer	$1 \cdot s^{-1}$
r_T	Gross count rate in the region of interest of the tracer	$1 \cdot s^{-1}$
t_0	Counting time of the background by alpha spectrometry	s
t_g	Sample counting time by alpha spectrometry	s
$U(x)$	Expanded uncertainty	—
$u(x)$	Standard uncertainty	—
u_{rel}	Relative standard uncertainty	—
$u(c_A)$	Standard uncertainty of the activity concentration of the measurand	$Bq \cdot l^{-1}$
$\tilde{u}(\tilde{c}_A)$	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

5 Principle

This document introduces the methods for the determination of plutonium (^{238}Pu , ^{239}Pu and ^{240}Pu) in urine by alpha spectrometry or inductively coupled plasma mass spectrometry (ICP-MS). The sample preparation volume and measurement method can be selected according to the required detection limit, the sample turn-around time to be observed and isotopes to be determined.

Based on the reference level of 100 mSv for responders on-site (effective dose, during the early and during the intermediate phase in an accident emergencies, recommended by the ICRP[1][2][3]), the derived daily urinary excretions of ^{238}Pu , ^{239}Pu or ^{240}Pu on the third day after the intake would be $7 \times 10^{-3} \text{ Bq} \cdot l^{-1}$.

NOTE 1 Assuming the form of Pu is Type S, and 5 μm activity median aerodynamic diameter aerosols inhaled by a reference worker.

NOTE 2 Assuming 1,6 l daily urine excretion for a reference man.

Based on the recording level of 1 mSv (5 % of the annual dose limit of 20 mSv, 50 year committed effective dose) in routine monitoring^[4], the derived daily urinary excretions of the ²³⁸Pu, ²³⁹Pu and ²⁴⁰Pu would be $1,5 \times 10^{-4}$ Bq·l⁻¹ for ²³⁸Pu and $1,3 \times 10^{-4}$ Bq·l⁻¹ for ²³⁹Pu and ²⁴⁰Pu with an annual monitoring frequency (365 days $\pm$ 30 days monitoring interval).

NOTE 1 Assuming the form of Pu is Type M, and 5 μ m activity median aerodynamic diameter aerosols inhaled by a reference worker.

NOTE 2 Assuming 1,6 l daily urine excretion for a reference man.

In case of exposure to more than one radionuclide, the required detection limit and the measurement frequency for ²³⁸Pu, ²³⁹Pu and ²⁴⁰Pu should be adjusted accordingly.

The detection limit depends on the sample preparation volume, the efficiency of chemical separation, the performance of the measurement device, and the counting time of alpha spectrometry.

In accident situations, a large number of workers, members of the public and emergency responders may need to be assessed in a short timescale. In that case, spot urine samples can be collected and processed to quickly estimate the level of contamination for screening purposes i.e. to provide information for the initiation and support of any appropriate health surveillance.

Spot sampling should not be done within 4 h after the contamination incident as sufficient time is needed for radionuclides to pass through the systemic circulation into the urine.

In the individual monitoring of occupational exposure (e.g. routine monitoring, special monitoring, confirmatory monitoring, and task-related monitoring), 24 h urine excretion can be collected.

NOTE If medical interventions are administered, the first post-intervention urine sample should be collected preferentially.

Since internal dose calculations with biokinetic models require excreted activity per day, it will be necessary to have a method to normalise the results from spot urine samples to a 24 h period. ICRP recommends^[4] that if a 24 h sample is less than 500 ml, it is doubtful that it has been collected over a full 24 h period and normalization should be considered.

Methods for normalization include by measuring creatinine^{[5][6]} concentration, by volume (with or without a correction for specific gravity^[7]) and by the length of the sampling interval^[4]. The reference volumes are 1,6 l and 1,2 l for males and females respectively, and the reference values of creatinine excreted are 1,7 g·d⁻¹ and 1,0 g·d⁻¹ for males and females, respectively^[8].

The urine sample should be subjected to chemical separation before measurement to eliminate potential interferences, including

- the salt content of the urine sample (in order to prepare a thin α counting source and achieve the desired energy resolution and counting efficiency for alpha spectrometry, or to avoid signal suppression for ICP-MS measurement).
- other potential α -emitters radionuclides, such as ²⁴¹Am (5,44 to 5,49) MeV and ²²⁸Th (5,35 to 5,42) MeV, whose emissions can overlap with ²³⁸Pu (5,46 to 5,50) MeV.
- the organic matrix of urine, which can seriously suppress the detection efficiency of Pu isotopes in both alpha spectrometry and ICP-MS measurement.
- the interferences at $m/z = 239, 240$ from U⁺ peak tailing, UH⁺, UH₂⁺ and other isobaric or polyatomic ions when measuring plutonium isotopes with ICP-MS.

Due to its relatively short half-life and the isobaric interference of ²³⁸U, measurement of ²³⁸Pu by ICP-MS would not in practice meet the required detection limits, and alpha spectrometer measurement is recommended.

Given that the alpha particle energies of ²³⁹Pu (5,11 to 5,16) MeV and ²⁴⁰Pu (5,12 to 5,17) MeV are similar, and their spectral peaks overlap significantly, the alpha spectrometry method is only applicable to the analysis of ²³⁹⁺²⁴⁰Pu activity. Where this is appropriate, the alpha spectrometry measurement method may

be selected. If the individual activities of ^{239}Pu and ^{240}Pu are required, the ICP-MS measurement method shall be selected.

The mass concentrations obtained by ICP-MS can be converted into activity concentrations of the different isotopes.

6 Chemical reagents and apparatus

6.1 Chemical reagents

The chemical reagents for specific chemical separations are described in [Annexes A, B and C](#), for the preparation of the deposited sources in [Annexes D and E](#), and for alpha spectrometry measurement and ICP-MS measurement in [Annexes F and G](#).

6.1.1 Tracer, ^{242}Pu is commonly used, but ^{236}Pu and ^{244}Pu can also be chosen. The tracer solution should be traceable to national or international measurement certified standards and can be obtained from a number of commercial suppliers and national measurement institutes.

6.1.2 Ultrapure water, resistivity $>18,2 \Omega\cdot\text{cm}$.

6.2 Apparatus

Usual laboratory apparatus and in particular, the following equipment.

6.2.1 Alpha spectrometer, semiconductor detector equipped with vacuum pump. For obtaining a well-resolved spectrum, the chamber should be maintained at a high vacuum during the measurement, and the distance between the detector and the source should be appropriate.

6.2.2 ICP-MS apparatus with associated software, installed in an air-conditioned room with argon gas supply.

6.2.3 Balance, accurate to 0,1 mg.

6.2.4 Vacuum filtration system.

6.2.5 Filters, of pore size $0,45 \mu\text{m}$ or smaller.

6.2.6 Metal discs with a sticky side.

6.2.7 Centrifuge.

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

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

6.2.10 Hot plate.

6.2.11 Magnetic stirring plate.

6.2.12 Magnetic stirrer bars.

In addition, the apparatuses for specific chemical separation and sample measurement are listed in [Annexes A, B, C, D and E](#).

7 Sample preparation procedure

7.1 General

The urine sample shall be acidified to ensure that the pH is less than 2, and shall be analysed as soon as possible or stored in the refrigerator at 0-5 °C.

NOTE Specimens should be handled in a safe manner and according to applicable legal requirements or guidance.

The volume of urine sample preparation can be chosen based on the monitoring situation (e.g. emergency situations, the individual monitoring of occupational exposure), the requirements of detection limits and sample turn-around time.

In emergency situations, spot urine samples (<50 ml) can be collected for Pu concentration measurement for screening purposes. Two examples of sample preparation procedures for emergency monitoring using 20 ml and 100 ml of spot urine sample are described in [Annex A](#) and [Annex B](#), respectively.

In the individual monitoring of occupational exposure (routine monitoring, special monitoring, confirmatory monitoring, and task-related monitoring), a 24 h urine sample can be used. An example sample preparation method for routine monitoring using 24 h urine sample (approximately 1,6 l) is shown in [Annex C](#).

7.2 Sample pre-concentration

The chemical recovery tracer is added during this initial sample pre-treatment phase.

The sample should be well mixed, and the isotopic equilibrium between the analyte radionuclides and the tracer should be ensured.

For small-volume urine samples (<50 ml), pre-concentration may not be necessary.

For samples of greater than 50 ml volume, pre-concentration steps are necessary, which can be carried out through 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. After centrifugation or filtration, the precipitate is dissolved with an acidic solution.

Co-precipitation is an effective method for processing large-volume samples. For example, calcium phosphates [$\text{Ca}_3(\text{PO}_4)_2$], bismuth phosphate (BiPO_4), iron hydroxide [$\text{Fe}(\text{OH})_2$ or $\text{Fe}(\text{OH})_3$], calcium oxalate (CaC_2O_4), titanium hydroxide [$\text{Ti}(\text{OH})_3$], manganese oxide (MnO_2), and fluorides can be added to co-precipitate the plutonium isotopes. Two examples of co-precipitation with titanium hydroxide for different volumes of urine samples is described in [Annex B](#) and [Annex C](#).

7.3 Organics decomposition

Organics decomposition is necessary for urine samples of greater than 100 ml in order to achieve a good chemical recovery and method performance. This step is recommended to be performed after sample pre-concentration, which can reduce reagent usage and shorten sample preparation time.

Acid digestion methods, such as Fenton's reaction using H_2O_2 and an Fe^{2+} catalyst, heating with HNO_3 or $\text{HNO}_3 + \text{H}_2\text{O}_2$, or microwave mineralization, are commonly used to decompose organics in urine. An example of organics decomposition by heating with $\text{HNO}_3 + \text{H}_2\text{O}_2$ is described in [Annex C](#).

7.4 Chemical separation

To achieve identical chemical behaviour for the tracer and the target radionuclides, a valence adjustment step is necessary to bring the Pu isotopes to a suitable oxidation state. Various reagents, such as H_2O_2 , NaNO_2 , hydroxylamine hydrochloride ($\text{NH}_2\text{OH} \cdot \text{HCl}$), silver oxide (Ag_2O), ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$), titanium chloride (TiCl_3), sodium sulfite (Na_2SO_3), potassium metabisulfite ($\text{K}_2\text{S}_2\text{O}_5$), ferrous sulfate (FeSO_4), ferrous sulfamate [$\text{Fe}(\text{NH}_2\text{SO}_3)_2$], and ammonium persulfate [$(\text{NH}_4)_2\text{S}_2\text{O}_8$], have been used to convert Pu to a suitable oxidation state, e.g. the (+IV) oxidation state or the (+III) oxidation state.