



DRAFT INTERNATIONAL STANDARD ISO/DIS 13166-1

ISO/TC 147/SC 3

Secretariat: AFNOR

Voting begins on
2012-10-22

Voting terminates on
2013-01-22

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Water quality — Uranium isotopes —

Part 1: Test method using alpha spectrometry

Qualité de l'eau — Uranium isotopes —

Partie 1: Méthode d'essai par spectrométrie alpha

ICS 13.060.60; 13.280

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

ISO 13166-1 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radiologicals methods*.

ISO 13166 consists of the following parts, under the general title *Water Quality — Uranium Isotopes*:

— *Part 1: Test method using alpha spectrometry*

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Introduction

Uranium is widespread in nature, occurring in granites and various other mineral deposits.

Uranium is used mainly as fuel in nuclear power plants.

Uranium present in the environmental waters is the result of leaching from natural deposits, release in mill tailing, emission from the nuclear industry, combustion of coal and other fuels and use of phosphate fertilizers.

The concentration in the earth's crust varies from $0,3 \mu\text{g.g}^{-1}$ to $12 \mu\text{g.g}^{-1}$ [1]. In surface, ground and ocean waters levels vary from less than $1 \mu\text{g.l}^{-1}$ to more than $10 \mu\text{g.l}^{-1}$ [2].

Uranium presents a chemical hazard [3].

All uranium isotopes are radioactive and it has three naturally occurring isotopes [4] [5]:

Uranium isotope	Mass Abundance	Isotopic ratio (activities)	
^{234}U	$0,00005363 \pm 0,00000016$	$^{238}\text{U}/^{234}\text{U}$	1
^{235}U	$0,007204 \pm 0,000006$	$^{238}\text{U}/^{235}\text{U}$	21
^{238}U	$0,992742 \pm 0,000010$		

Isotopic ratios different from above are frequently observed in water. [6] [7].

Others uranium isotopes such as uranium-232, uranium-233 and uranium-236 also exist. Uranium-232 is used as a tracer. Uranium-233 and Uranium-236 are not normally encountered in environmental samples.

Nuclear data can be used to transform uranium isotopes concentrations (in g.l^{-1} for example) to uranium isotopes activity concentrations (in Bq.l^{-1}) and vice versa [4]:

Uranium isotope	Half life (years)	Specific activity (Bq.g^{-1})
^{234}U	$2,455 (\pm 0,006) \times 10^5$	$2,312 \times 10^8$
^{235}U	$704 (\pm 1) \times 10^6$	$7,997 \times 10^4$
^{238}U	$4,468 (\pm 0,005) \times 10^9$	$1,244 \times 10^4$

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Water Quality — Uranium Isotopes — Part 1: Test method using alpha spectrometry

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this standard be carried out by suitably trained staff.

1 Scope

This International Standard describes the conditions for the determination of uranium isotopes activity concentration in samples of environmental water (including sea waters) using α spectrometry and ^{232}U as a yield tracer. A chemical separation shall be carried out to separate and purify uranium from the sample test portion.

Plutonium isotopes can interfere if present with detectable activities in the sample.

The detection limit for measurement of about 500 ml test portion is $\sim 5 \text{ mBq.l}^{-1}$ with a counting time around 200 000 s.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5667-1/AC1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*, Geneva

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples*, Geneva

ISO 11929, *Determination of the detection limit and decision threshold for ionizing radiation measurements*

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

ISO/IEC Guide 98-3, *Uncertainty in Measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM)*

3 Terms, definitions and symbols

For the purposes of this document, the definitions, symbols and abbreviations defined in ISO 31-9 and ISO 11929 and the following apply.

Parameter	Description
E_{α}	Energy of emitted α particle
V	Volume of test sample, in litre;
A	Activity of ^{232}U tracer added, in becquerels, at the date of measurement;
c_A	Activity concentration of ^{238}U or ^{235}U or ^{234}U in becquerel per litre;
t_0	Background counting time, in second;
t_g	Sample counting time, in second;
r_g, r_{gT}	Gross count rate per second from measured uranium and tracer, respectively;
r_0, r_{0T}	Background count rate per second from uranium and tracer, respectively;
R	Total measurement yield
R_c	Chemical yield
ε	Detection yield
$u(c_A)$	Standard uncertainty associated with the measurement result; in becquerel per litre;
U	Expanded uncertainty, calculated by $U = k \cdot u(c_A)$ with $k = 1, 2, \dots$, in becquerel per litre;
c_A^*	Decision threshold, in becquerel per litre;
$c_A^{\#}$	Detection limit, in becquerel per litre;
$c_A^{\triangleleft}, c_A^{\triangleright}$	Lower and upper limits of the confidence interval, in becquerel per litre;

4 Principle

The assay sample is mixed with an aliquot of ^{232}U tracer, followed by equilibration of the sample prior to analysis. After a valence cycle to adjust the oxidation states, chemical isolation of uranium is achieved by a concentration step (a precipitation for example) followed by a specific separation step (using ion exchange chromatography for example).

^{230}Th , ^{226}Ra and ^{228}Th may be present in water and could interfere with the counting of Uranium isotopes if no chemical separation is carried out to remove these radionuclides from the sample test portion.

The measured thin source is prepared by electro-deposition or co-precipitation and assayed by alpha spectrometry using a grid chamber or a semi-conductor type apparatus. Measurements rely on the interaction of alpha particles with the detecting medium. This interaction creates a charge, which is amplified and output as a voltage pulse proportional to the deposited energy of the incoming α particle.

The electric pulse from the detector is analysed by the electronic systems and the data analysis software providing a spectrum, in which the number of pulses (counts) recorded in each energy interval is showed.

The analysis of the count rates in the uranium isotopes alpha energy windows allows the determination of the test sample activity concentration for uranium-238, uranium-235 and uranium-234, after correcting for the blank count rate, volume of the test sample and the total measurement yield (chemical yield and detection efficiency).

The chemical yield and detection efficiency are not necessarily determined separately, but are determined together by measuring the total measurement yield from the net count rate of ^{232}U , 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 assay sample. This blank sample is prepared using a reference water.

In order to quantify potential impurities in the tracer solution, another blank sample is prepared with addition of tracer.

The radioactive characteristics of the main uranium isotopes are [4] [5]

Uranium isotope	Half life (years)	Main emission energy (keV) and intensity (%)
232	70,6 ($\pm 1,1$)	5263,48 ; 30,6
		5320,24 ; 69,1
233	159,1 ($\pm 0,2$)	4783,5 ; 13,2
		4824,2 ; 84,3
234	$2,455 (\pm 0,006) \times 10^5$	4722,4 ; 28,42
		4774,6 ; 71,37
235	$704 (\pm 1) \times 10^6$	4366,1 ; 18,8
		4397,8 ; 57,19
		4414,9 ; 3,01
236	$23,43 (\pm 0,06) \times 10^6$	4445 ; 26,1
		4494 ; 73,8
238	$4,468 (\pm 0,005) \times 10^9$	4151 ; 22,33
		4198 ; 77,54

With a spectra resolution of full width half maximum of > 20 keV, alpha spectrometry cannot easily resolve ^{233}U and ^{234}U , nor ^{235}U and ^{236}U , due to the similarity in their respective emission energies; however, ^{233}U and ^{236}U are not normally encountered in environmental samples.

5 Chemical reagents and equipment

5.1 General

The chemical reagents and equipment used for chemical treatment and preparation of the source are described in Annexes A to C as there are different possibilities to perform them. Some shall be used, whatever the option chosen is.

Use only reagents of recognized analytical grade.

5.2 Chemical reagents

5.2.1 Laboratory water

The laboratory water, used as a blank, ought to be as free as possible of chemical or radioactive impurities (such as uranium isotopes), complying with grade 3 of ISO 3696.

For example, one can obtain water with a uranium activity concentration very low in fresh rain-water. Uranium activity concentration of this water can be evaluated at the same time as interferences coming from reagents or using another type of precise measurement such as thermal ionisation or inductively coupled plasma mass spectrometry for example.

5.2.2 Uranium-232 tracer solution

A tracer solution of ^{232}U shall be used to determine the total yield. It can also be used to calculate the chemical yield. The solution shall be prepared by the dilution of a suitable standard that provides traceability to national and international standards. The tracer solution shall be homogeneous and stable.

The tracer solution concentration should be calculated to allow adding a small amount of this solution in order to be in the range of activity contained in the test portion. For example, the tracer solution concentration could be between $0,05 \text{ Bq.g}^{-1}$ and 1 Bq.g^{-1} .

It is recommended to check the activity and the purity of the tracer solution dilution before use and at regular intervals after preparation. This can be done, for example, by liquid scintillation counting, but account needs to be taken of progeny radionuclide ingrowth. Performing a blank analysis with tracer is a potential way to identify any presence of trace analyte in the tracer.

^{228}Th can be present in the ^{232}U tracer solution and has very close energy with its parent ^{232}U . Therefore, good separation between Th and U is required to minimize the interference of ^{228}Th so that the counting yield of ^{232}U is not over-estimated.

5.3 Equipment

Usual laboratory apparatus and:

5.3.1 Alpha spectrometer

This apparatus can be a grid chamber (with higher detection yield but lower resolution) or a semi-conductor type apparatus (with lower detection yield but higher resolution). Operation at constant temperature is recommended. Follow the manufacturer's instructions.

For semi-conductor type apparatus, the measurements using α -spectrometry depend on the interaction of α particles with ion-implanted silicon. This interaction changes instantly the conductivity of the silicon, proportional to the energy of the incoming α particle. To achieve well-resolved spectra, the detection system needs to be maintained at a pressure $< 1 \text{ Pa}$. Resolution can be further enhanced through increasing distance between source and detector.

5.3.2 Pipette

Suitable for the accurate transfer of (for example, $100 \mu\text{l}$) of Uranium-232 tracer solution with a total precision less than or equal to $\pm 1 \%$, if this is used as a transfer device.

5.3.3 Balance

For example, capable of achieving $\pm 0,1 \text{ mg}$ precision, if this is used as a transfer device.