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

~~*Qualité de l'eau — Application de la spectrométrie de masse avec plasma à couplage
inductif (ICP-MS) — Partie 2: Dosage des éléments sélectionnés y compris les isotopes
d'uranium*~~

~~Third edition~~

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Contents	Page
Foreword.....	iv
1 Scope.....	1
2 Normative references	3
3 Terms and definitions.....	3
4 Principle	3
5 Interferences.....	4
6 Reagents	9
7 Apparatus.....	14
8 Sampling.....	15
9 Sample pre-treatment.....	17
10 Procedure	17
11 Calculation.....	19
12 Test report.....	20
Annex A (normative) Determination of the mass concentration of uranium isotopes	21
Annex B (informative) Description of the matrices of the samples used for the interlaboratory trial.....	32
Annex C (informative) Performance data.....	34
Bibliography.....	37

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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 Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 230, *Water analysis*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 17294-2:2016), which has been technically revised.

The main changes ~~compared to the previous edition~~ are as follows:

- with ~~the~~ incorporation of mercury in the previous edition, mercury ~~was included~~ has now been excluded as a hydrolysable ~~and has now become a non-hydrolysable~~ element ~~which because it~~ was not in line with the other existing standards for the determination of mercury;
- the addition of a modifier ~~is~~ has been clarified ~~in this edition~~;
- titanium ~~was~~ has been added to the scope;
- ~~Editorial revision to the ISO guidelines.~~

A list of all parts in the ISO 17294 series can be found on the ISO website.

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.

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

WARNING — Persons using this document should be familiar with normal laboratory practice. 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.

IMPORTANT — It is absolutely essential that tests, conducted in accordance with this document, be carried out by suitably qualified staff.

1 Scope

This document specifies a method for the determination of the elements aluminium, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, caesium, calcium, cerium, chromium, cobalt, copper, dysprosium, erbium, gadolinium, gallium, germanium, gold, hafnium, holmium, indium, iridium, iron, lanthanum, lead, lithium, lutetium, magnesium, manganese, mercury, molybdenum, neodymium, nickel, palladium, phosphorus, platinum, potassium, praseodymium, rubidium, rhenium, rhodium, ruthenium, samarium, scandium, selenium, silver, sodium, strontium, terbium, tellurium, thorium, thallium, thulium, tin, titanium, tungsten, uranium and its isotopes, vanadium, yttrium, ytterbium, zinc and zirconium in water (e.g. drinking water, surface water, ground water, waste water and eluates).

Taking into account the specific and additionally occurring interferences, these elements can be determined in water and digests of water and sludge (e.g. digests of water as described in ISO 15587-1 or ISO 15587-2).

The working range depends on the matrix and the interferences encountered. In drinking water and relatively unpolluted waters, the limit of quantification (L_{00}) lies between 0,002 µg/l and 1,0 µg/l for most elements (see Table 1). The working range typically covers concentrations between several ng/l and mg/l depending on the element and specified requirements.

The quantification limits of most elements are affected by blank contamination and depend predominantly on the laboratory air-handling facilities available on the purity of reagents and the cleanliness of glassware.

The lower limit of quantification is higher in cases where the determination suffers from interferences (see Clause 5) or memory effects (see ISO 17294-1).

~~Other elements~~ Elements other than those mentioned in this list the scope can also be determined according to this document provided that the user of the document is able to validate the method appropriately (e.g. interferences, sensitivity, repeatability, recovery).

Table 1 — Lower limits of quantification for unpolluted water

Element	Isotope often used	Limit of quantification on L_{00}^a µg/l	Element	Isotope often used	Limit of quantification on L_{00}^a µg/l	Element	Isotope often used	Limit of quantification on L_{00}^a µg/l
Ag	¹⁰⁷ Ag	0,5	Hf	¹⁷⁸ Hf	0,1	Ru	¹⁰² Ru	0,1
	¹⁰⁹ Ag	0,5	Hg	²⁰² Hg	0,05	Sb	¹²¹ Sb	0,2

ISO/FDIS 17294-2:2023(E)

Element	Isotope often used	Limit of quantification on L_{00}^a	Element	Isotope often used	Limit of quantification on L_{00}^a	Element	Isotope often used	Limit of quantification on L_{00}^a
				²⁰¹ Hg	0,1			
Al	²⁷ Al	1	Ho	¹⁶⁵ Ho	0,1		¹²³ Sb	0,2
As	⁷⁵ As ^c	0,1	In	¹¹⁵ In	0,1	Sc	⁴⁵ Sc	5
Au	¹⁹⁷ Au	0,5	Ir	¹⁹³ Ir	0,1	Se	⁷⁷ Se ^c	1
B	¹⁰ B	1	K	³⁹ K ^c	5		⁷⁸ Se ^c	0,1
	¹¹ B	1	La	¹³⁹ La	0,1	⁸² Se	1	
Ba	¹³⁷ Ba	3	Li	⁶ Li	10	Sm	¹⁴⁷ Sm	0,1
	¹³⁸ Ba	0,5		⁷ Li	1	Sn	¹¹⁸ Sn	1
Be	⁹ Be	0,1	Lu	¹⁷⁵ Lu	0,1		¹²⁰ Sn	1
Bi	²⁰⁹ Bi	0,5	Mg	²⁴ Mg	1	Sr	⁸⁶ Sr	0,5
Ca	⁴³ Ca	100		²⁵ Mg	10		⁸⁸ Sr	0,3
	⁴⁴ Ca	50	Mn	⁵⁵ Mn	0,1	Tb	¹⁵⁹ Tb	0,1
	⁴⁰ Ca	10	Mo	⁹⁵ Mo	0,5	Te	¹²⁶ Te	2
Cd	¹¹¹ Cd	0,1		⁹⁸ Mo	0,3	Th	²³² Th	0,1
	¹¹⁴ Cd	0,5	Na	²³ Na	10	Tl	²⁰³ Tl	0,2
Ce	¹⁴⁰ Ce	0,1	Nd	¹⁴⁶ Nd	0,1	Tl	²⁰⁵ Tl	0,1
						Ti	⁴⁷ Ti	10
						Ti	⁴⁸ Ti	1
						Ti	⁴⁹ Ti	10
Co	⁵⁹ Co	0,2	Ni	⁵⁸ Ni ^c	0,1	Tm	¹⁶⁹ Tm	0,1
Cr	⁵² Cr ^c	0,1		⁶⁰ Ni	0,1	U	²³⁸ U	0,1
	⁵³ Cr	5	P	³¹ P	5	U	²³⁵ U	$1 \cdot 10^{-4}$
Cs	¹³³ Cs	0,1	Pb	²⁰⁶ Pb ^b	0,2	U	²³⁴ U	$1 \cdot 10^{-5}$
Cu	⁶³ Cu	0,1		²⁰⁷ Pb ^b	0,2	V	⁵¹ V ^c	0,1
	⁶⁵ Cu	0,1		²⁰⁸ Pb ^b	0,1	W	¹⁸² W	0,3
Dy	¹⁶³ Dy	0,1	Pd	¹⁰⁸ Pd	0,5		¹⁸⁴ W	0,3
Er	¹⁶⁶ Er	0,1	Pr	¹⁴¹ Pr	0,1	Y	⁸⁹ Y	0,1
Fe	⁵⁶ Fe ^c	5	Pt	¹⁹⁵ Pt	0,5	Yb	¹⁷² Yb	0,2
Ga	⁶⁹ Ga	0,3	Rb	⁸⁵ Rb	0,1		¹⁷⁴ Yb	0,2
	⁷¹ Ga	0,3	Re	¹⁸⁵ Re	0,1	Zn	⁶⁴ Zn	1
Gd	¹⁵⁷ Gd	0,1		¹⁸⁷ Re	0,1	⁶⁶ Zn	1	
	¹⁵⁸ Gd	0,1	Rh	¹⁰³ Rh	0,1	⁶⁸ Zn	1	
Ge	⁷⁴ Ge	0,3	Ru	¹⁰¹ Ru	0,2	Zr	⁹⁰ Zr	0,2

^a Depending on the instrumentation, significantly lower limits can be achieved.

^b Lead (Pb) is reported as the sum of the signal intensities of ²⁰⁶Pb, ²⁰⁷Pb and ²⁰⁸Pb.

^c These limits are achieved by the use of a collision/reaction cell.

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 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 8466-1, *Water quality — Calibration and evaluation of analytical methods — Part 1: Linear calibration function*

ISO 15587-1, *Water quality — Digestion for the determination of selected elements in water — Part 1: Aqua regia digestion*

ISO 15587-2, *Water quality — Digestion for the determination of selected elements in water — Part 2: Nitric acid digestion*

ISO 17294-1:2004, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 17294-1 and the following apply.

ISO and IEC maintain ~~terminological~~**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/>

3.1.1

analyte(s)

element(s) to be determined

3.1.2

background

N_0

counts for a given mass in the blank solution

Note 1 to entry: Background is expressed in Counts.

3.1.3

blank calibration solution

solution prepared in the same way as the *calibration solution* (3.1.4) but leaving out the *analyte* (3.1.1)

3.1.4

calibration solution

solution used to calibrate the instrument, prepared from a *stock solution(s)* (3.1.24) or from a certified standard

3.41.5

determination

entire process from preparing the *test sample solution* (3.471.26) up to and including the measurement and calculation of the final *result* (3.431.22)

3.51.6

expanded uncertainty

U

product of the standard uncertainty, $u(C)$, and the coverage factor, k , with $k=1, 2, \dots$, as follows:

$$U = k \cdot u(C)$$

Note 1 to entry: Expanded uncertainty is expressed in the unit of the quantity C .

3.1.7

instrument detection limit

L_{DI}

smallest concentration that can be detected with a defined statistical probability using a contaminant-free instrument and a *blank calibration solution* (3.21.3)

Note 1 to entry: It is the lowest value that can be measured by the instrument in the most optimal set up and is determined by three times the standard deviation obtained with 10 replicates of the blank.

Note 2 to entry: Instrument detection limit is expressed in $\mu\text{g/L}$.

3.61.8

instrumental limit of quantification

$L_{LOQ,ins}$

limit of quantification (3.1.13) expressed in counts for the chosen m/z , due to the blank and the instrument

Note 1 to entry: Instrumental limit of quantification is expressed in $\mu\text{g/L}$.

3.1.9

internal standard correction factor

C_{int}

sample matrix effect correction when an internal standard is added to the sample

3.1.10

internal standard mass

m_T

mass of the isotope dilution tracer added

Note 1 to entry: Internal standard mass is expressed in μg .

3.1.11

laboratory sample

sample as prepared for sending to the laboratory and intended for inspection or testing.

[SOURCE: ISO 6206:1979, 3.2.10]

3.71.12

limit of application

L_{OA}

lowest or highest concentration of an *analyte* (3.1.1) that can be determined with a defined level of accuracy and *precision* (3.1.18)

Note 1 to entry: Limit of application is expressed in µg/l.

3.1.13
limit of quantification

L_{00}
value determined by 10 times the standard deviation obtained with 10 replicates of the blank

Note 1 to entry: Limit of quantification is expressed in µg/l.

3.1.14
linearity

the functional relationship between the indicated values and the ~~contents~~ content

3.1.15
mass concentration

C
mass of *analyte* (3.1.1) per unit volume of the sample

Note 1 to entry: Mass concentration is expressed in µg/l.

3.1.16
mass concentration of the internal standard solution

C_T
mass of internal standard element per unit volume of the internal standard solution

3.1.17
optimization solution

solution serving for mass calibration and for the optimization of the apparatus conditions

EXAMPLE Adjustment of maximal *sensitivity* (3.1.23) with respect to minimal oxide formation rate and minimal formation of doubly charged ions.

3.1.18
precision

closeness of agreement between independent test results (3.1.22) obtained under ~~prescribed~~ stipulated conditions

Note 1 to entry: Precision depends only on the distribution of random errors and does not relate to true value or the specified value.

[SOURCE: ISO 5725-1:1994, 3.12, modified — Notes 2 and 3 to entry have been deleted.]

3.1.19
pure chemical

chemical with the highest available purity and known stoichiometry and for which the content of *analyte* (3.1.1) and contaminants ~~should be~~ known with an established degree of certainty

3.1.20
repeatability

r
precision (3.1.18) under repeatability conditions:

[SOURCE: ISO 5725-1:1994, 3.13]

3.1.21
reproducibility

ISO/FDIS 17294-2:2023(E)

R
precision (3.91.18) under reproducibility conditions-

[SOURCE: ISO 5725-1:1994, 3.17]

~~3.131.22~~
result
outcome of a measurement

Note 1 to entry: The result is typically calculated as *mass concentration* (*C*) (3.1.15), expressed in ~~micrograms per litre~~ or milligrams per litre.

~~3.141.23~~
sensitivity
S

ratio of the variation of the magnitude of the signal (ΔI) to the corresponding variation in the concentration of the *analyte* (3.1.1) (ΔC) expressed by Formula (1):

$$S = \frac{\Delta I}{\Delta C} \quad (1)$$

~~3.151.24~~
stock solution

solution with accurately known *analyte* (3.1.1) concentration(s), prepared from *pure chemicals* (3.1.19)

Note 1 to entry: Stock solutions are reference materials within the meaning of as explained in ISO Guide 30.

~~3.161.25~~
test sample

sample prepared from the *laboratory sample* (3.61.11), for example, by grinding or homogenizing

~~3.171.26~~
test sample solution

solution prepared with the fraction (test portion) of the *test sample* (3.161.25) according to the appropriate specifications, such that it can be used for the envisaged measurement

Note 1 to entry: *Mass concentration of the internal standard solution* (3.1.16) is expressed in $\mu\text{g/l}$.

3.2 Symbols

<i>K</i>	coverage factor	
<i>N</i>	raw counts	
<i>N_{dl}</i>	counts calculated when using isotopic dilution	Counts
<i>N_{net}</i>	net counts ($N - N_0$). $N_{\text{net}} = a \cdot C + b$	Counts
	where	
	<i>a</i> is the regression line slope, expressed in $\text{Counts} \cdot \mu\text{g}^{-1} \cdot \text{l}$;	
	<i>b</i> is the coordinate at the origin of the regression line, expressed in Counts	
<i>R</i>	measured isotopic ratio	Counts
<i>R</i>	true isotopic ratio	-
<i>s_{W0}</i>	blank standard deviation	-
<i>T</i>	isotope distribution in the standard solution of ²³³ U (used for isotopic dilution)	

<u>$u(C)$</u>	<u>standard uncertainty associated with the measurement result</u>	<u>unit of the quantity C</u>
<u>V</u>	<u>volume of the sample</u>	<u>l</u>
<u>A</u>	<u>bias per unit mass</u>	<u>-</u>
<u>B</u>	<u>fractionation coefficient deviation</u>	<u>-</u>

3.4 Principle

When applying this document, it is necessary in each case, depending on the range to be tested, to determine if and to what extent additional conditions shall be established. Guidance is given in ISO 17294-1.

Multi-element determination of selected elements, including uranium isotopes, by inductively coupled plasma mass spectrometry (ICP-MS) consists of the following steps:

- introduction of a measuring solution into a radiofrequency plasma (e.g. by pneumatic nebulization) where energy transfer processes from the plasma cause desolvation, decomposition, atomization and ionization of elements;
- as an additional option, collision or reaction cell technology may be used to overcome several interferences (see 5.1);
- extraction of the ions from plasma through a differentially pumped vacuum interface with integrated ion optics and separation on the basis of their mass-to-charge ratio by a mass spectrometer (for instance, a quadrupole MS);
- transmission of the ions through the mass separation unit (for instance, a quadrupole) and detection, usually by a continuous dynode electron multiplier assembly, and ion information processing by a data handling system;
- quantitative determination after calibration with suitable calibration solutions.

The relationship between signal intensity and mass concentration is usually a linear one over a broad range (usually over more than several orders of magnitude).

The method ~~to be~~ used for the determination of uranium isotopes is described in Annex A.

With instruments equipped with a magnetic sector field, higher mass resolution spectra can be obtained. This can help to separate isotopes of interest from interfering species.

4.5 Interferences

4.5.1 General

In certain cases, isobaric and non-isobaric interferences can occur. The most important interferences in this respect are coinciding masses and physical interferences from the sample matrix. For more detailed information, see ISO 17294-1.

Common isobaric interferences are given in Table 2 (for additional information, see ISO 17294-1~~3-1~~). It is recommended that different isotopes of an element be determined in order to select an isotope that does not suffer from interference. If there are none that meet this requirement, a mathematical correction shall be applied. For the determination of uranium isotopes, the specific procedure detailed in Annex A shall be followed.

Small drifts or variations in intensities should be corrected by the application of the internal standard correction. In general, in order to avoid physical and spectral interferences, the mass concentration of

ISO/FDIS 17294-2:2023(E)

dissolved matter (salt content) should not exceed 2 g/l (corresponding to a conductivity of less than 2 700 µS/cm).

NOTE With the use of collision and reaction cell technology, it is possible to overcome several interferences. As the various options and parameters of those techniques cannot be described in detail in this document, the user is responsible for demonstrating that the chosen approach is fit for purpose and achieves the necessary performance.

4.2 Spectral interferences

4.2.1 General

For more detailed information on spectral interferences, see ISO 17294-1¹, 6.2.

4.2.2 Isobaric elemental

Isobaric elemental interferences are caused by isotopes of different elements of the same nominal mass-to-charge ratio and which cannot be separated due to an insufficient resolution of the mass spectrometer in use (e.g. ¹¹⁴Cd and ¹¹⁴Sn).

Element interferences from isobars can be corrected for taking into account the influence from the interfering element (see Table 3). In this case, the isotopes used for correction shall be determinable without any interference and with sufficient precision. Possible proposals for correction are often included in the instrument software.

Table 2 — Important isobaric and polyatomic interferences

Element	Isotope	Inter-element interferences caused by isobars and doubly charged ions	Interferences caused by polyatomic ions
Ag	¹⁰⁷ Ag ¹⁰⁹ Ag	—	ZrO NbO, ZrOH
As	⁷⁵ As	—	ArCl, CaCl
Au	¹⁹⁷ Au	—	TaO
B	¹⁰ B	—	—
	¹¹ B	—	BH
Ba	¹³⁸ Ba	La ⁺ , Ce ⁺	—
Be	⁹ Be	—	¹⁸ O ₂
Ca	⁴³ Ca	Sr⁺⁺ Sr²⁺	CNO
	⁴⁴ Ca	Sr⁺⁺ Sr²⁺	COO
Cd	¹¹¹ Cd	—	MoO, MoOH, ZrOH
	¹¹⁴ Cd	Sn ⁺	MoO, MoOH
Co	⁵⁹ Co	—	CaO, CaOH, MgCl
Cr	⁵² Cr	—	ArO, ArC, ClOH
	⁵³ Cr	Fe ⁺	ClO, ArOH,
Cu	⁶³ Cu	—	ArNa, POO, MgCl
	⁶⁵ Cu	—	SOOH
Eu	¹⁵¹ Eu	—	BaO

1 Second edition to be published.