SLOVENSKI STANDARD
oSIST prEN ISO 17294-2:2022

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Kakovost vode - Uporaba masne spektrometrije z induktivno sklopljeno plazmo (ICP/MS) - 2. del: Določevanje izbranih elementov, vključno z izotopi urana (ISO/DIS 17294-2:2022)

Water quality - Application of inductively coupled plasma mass spectrometry (ICP-MS) - Part 2: Determination of selected elements including uranium isotopes (ISO/DIS 17294-2:2022)

Wasserbeschaffenheit - Anwendung der induktiv gekoppelten Plasma-Massenspektrometrie (ICP-MS) - Teil 2: Bestimmung von ausgewählten Elementen einschließlich Uran-Isotope (ISO/DIS 17294-2:2022)

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 (ISO/DIS 17294-2:2022)

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13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances

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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

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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*.

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

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

- with incorporation of mercury in the previous edition, mercury was included as a hydrolysable element which was not in line with the other existing standards for the determination of mercury;
- the addition of a modifier is calcified in this edition.

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, tungsten, uranium and its isotopes, vanadium, yttrium, ytterbium, zinc and zirconium in water (for example, drinking water, surface water, ground water, waste water and eluates.

Taking into account the specific and additionally occurring interferences, these elements can also be determined in digests of water, sludges and sediments (for example, 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 (LOQ) 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 pg/l and mg/l depending on the element and pre-defined 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 17294-1:2004, 8.2).

Table 1 — Lower limits of quantification (LOQ) for unpolluted water

Element	Isotope often used	Limit of quantification[a] µg/l	Element	Isotope often used	Limit of quantification[a] µg/l	Element	Isotope often used	Limit of quantification[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
				^{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}KC	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		^{205}Tl	0,1
Co	^{59}Co	0,2	Ni	^{58}Nic	0,1	Tm	^{169}Tm	0,1
Cr	^{52}Cr[c]	0,1		^{60}Nic	0,1	U	^{238}U	0,1
	^{53}Cr	5	P	^{31}P	5		^{235}U	10-4
Cs	^{133}Cs	0,1	Pb	^{206}Pb[b]	0,2		^{234}U	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 ^{206}Pb, ^{207}Pb and ^{208}Pb.

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

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 3696, *Water for analytical laboratory use — Specification and test methods*

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

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 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 and definitions

For the purposes of this document, the terms and definitions given in ISO 17294-1 and in Annex A.2 apply.

ISO and IEC maintain terminological 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/

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 are to be established.

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 (for example, by pneumatic nebulization) where energy transfer processes from the plasma cause desolvation, decomposition, atomization and ionization of elements;
- as an additional option, collision and 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 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.

5 Interferences

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). 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 has to be applied. For the determination of uranium isotopes, the specific procedure detailed in Annex A has to 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 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.

5.2 Spectral interferences

5.2.1 General

For more detailed information on spectral interferences, see ISO 17294-1:2004, 6.2.

5.2.2 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 (for example, ^{114}Cd and ^{114}Sn).

Element interferences from isobars may 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^{++}	CNO
	^{44}Ca	Sr^{++}	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
	^{153}Eu	—	BaO
Fe	^{54}Fe	—	$^{37}Cl^{16}O^{1}H+$ $^{40}Ar^{14}N$
	^{56}Fe	—	$^{40}Ar^{16}O+$ $^{40}Ca^{16}O+$
	^{57}Fe	—	$^{40}Ar^{16}O^{1}H+$ $^{40}Ca^{16}O^{1}H+$ $^{40}Ar^{17}O+$
Ga	^{69}Ga	Ba^{++}	CrO, ArP, ClOO
Ge	^{74}Ge	Se^+	ArS, ClCl
Hg	^{201}Hg		$^{184}W^{17}O$
	^{202}Hg		$^{186}W^{16}O$
In	^{115}In	Sn^+	—
Ir	^{193}Ir	—	HfO
Mg	^{24}Mg	—	CC
	^{25}Mg	—	CC
Mn	^{55}Mn	—	NaS, ArOH, ArNH
Mo	^{98}Mo	Ru^+	—
Ni	^{58}Ni	Fe^+	CaO, CaN, NaCl, MgS
	^{60}Ni	—	CaO, CaOH, MgCl, NaCl
Pd	^{108}Pd	Cd^+	MoO, ZrO
Pt	^{195}Pt	—	HfO
Re	^{187}Re	Os^+	—
Ru	^{102}Ru	$Pd+$	—
Sb	^{123}Sb	$Te+$	—
Sc	^{45}Sc	—	COO, COOH

NOTE In the presence of elements in high mass concentrations, interferences can be caused by the formation of polyatoms or doubly charged ions which are not listed above.

Table 2 *(continued)*

Element	Isotope	Inter-element interferences caused by isobars and doubly charged ions	Interferences caused by polyatomic ions
Se	^{77}Se	—	CaCl, ArCl, ArArH
	^{78}Se	Kr^+	ArAr, CaCl
	^{82}Se	Kr^+	HBr
Sn	^{120}Sn	Te^+	—
V	^{51}V	—	ClO, SOH, ClN, ArNH
W	^{184}W	Os^+	—
Zn	^{64}Zn	Ni^+	AlCl, SS, SOO, CaO
	^{66}Zn	Ba^{++}	PCl, SS, FeC, SOO
	^{68}Zn	Ba^{++}, Ce^{++}	FeN, PCl, ArS, FeC, SS, ArNN, SOO
NOTE In the presence of elements in high mass concentrations, interferences can be caused by the formation of polyatoms or doubly charged ions which are not listed above.			

Table 3 — Examples for suitable isotopes with their relative atomic masses and formulae for correction

Element	Recommended isotope and inter-element correction	
As	^{75}As	$-3{,}127\ (^{77}Se - 0{,}815\ ^{82}Se)$ or
	^{75}As	$-3{,}127\ (^{77}Se + 0{,}322\ 0\ ^{78}Se)$
Ba	^{138}Ba	$-0{,}000\ 900\ 8\ ^{139}La - 0{,}002\ 825\ ^{140}Ce$
Cd	^{114}Cd	$-0{,}026\ 84\ ^{118}Sn$
Ge	^{74}Ge	$-0{,}138\ 5\ ^{82}Se$
In	^{115}In	$-0{,}014\ 86\ ^{118}Sn$
Mo	^{98}Mo	$-0{,}110\ 6\ ^{101}Ru$
Ni	^{58}Ni	$-0{,}048\ 25\ ^{54}Fe$
Pb	^{208}Pb	$+^{207}Pb + ^{206}Pb$
Se	^{82}Se	$-1{,}009\ ^{83}Kr$
Sn	^{120}Sn	$-0{,}013\ 44\ ^{125}Te$
V	^{51}V	$^{51}V -3{,}127\ (^{53}Cr -0{,}113\ 4\ ^{52}Cr)$
W	^{184}W	$-0{,}001\ 242\ ^{189}Os$
NOTE When using collision or reaction cell technology some of these interferences can be overcome.		

5.2.3 Polyatomic interferences

Polyatomic ions are formed by coincidence of plasma gas components, reagents and sample matrix (for example, interference of the relative mass ^{75}As by $^{40}Ar^{35}Cl$ and $^{40}Ca^{35}Cl$). Examples for correction formulae are given in Table 3 and information on the magnitude of interferences are stated in Table 4. This interference is of particular relevance for several elements (for example, As, Cr, Se, V).

It is recommended that the analyst checks the magnitude of this interference regularly for the particular instrument.

In the case of mathematical corrections, it shall be taken into account that the magnitude of interference depends both on the plasma adjustment (for example, oxide formation rate) and on the mass concentration of the interfering element, which will usually be a variable component of the sample solution.