FINAL DRAFT

INTERNATIONAL STANDARD

ISO/FDIS 17294-2

ISO/TC 147/SC 2

Secretariat: DIN

Voting begins on: **2015-10-29**

Voting terminates on:

2015-12-29

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

Partie 2: Dosag d'uranium

Please see the administrative notes on page iii

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.



Reference number ISO/FDIS 17294-2:2015(E)

ISO/CEN PARALLEL PROCESSING

This final draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO-lead** mode of collaboration as defined in the Vienna Agreement. The final draft was established on the basis of comments received during a parallel enquiry on the draft.

This final draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel two-month approval vote in ISO and formal vote in CEN.

Positive votes shall not be accompanied by comments.

Negative votes shall be accompanied by the relevant technical reasons.

Helps://standards.itelt.adphac.infl.standards.itelt.son.ing.da.2.2.2016



COPYRIGHT PROTECTED DOCUMENT

© ISO 2015, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office Ch. de Blandonnet 8 • CP 401 CH-1214 Vernier, Geneva, Switzerland Tel. +41 22 749 01 11 Fax +41 22 749 09 47 copyright@iso.org www.iso.org

Con	tent	S	Page							
Forev	vord		iv							
Intro	ductio	n	v							
1	Scope	e	1							
2	•	native references								
3		s and definitions								
4	Principle									
5		Interferences								
3	5.1 5.2 5.3	General Spectral interferences 5.2.1 General 5.2.2 Isobaric elemental 5.2.3 Polyatomic interferences	4 4 4 6							
	5.3	Non-spectral interferences	0							
6	Reagents Apparatus Sampling Sample pre-treatment									
7	Appa	ratus	11							
8	Samp	oling	12							
9	Samp	ole pre-treatment	13							
	9.1 9.2	Determination of the mass concentration of dissolved elements without digestion	13							
		Determination of the total mass concentration after digestion	13							
10	Proce	General Calibration of the ICP-MS system in the Calibration of the ICP-MS system in the Calibration of the C	13							
	10.1	Calibration of the ICP-MS system	13 14							
	10.3	Measurement of the matrix solution for evaluation of the correction factors	14							
	10.4	Measurement of the samples	14							
11	Calcu	llation Alaft Ala	14							
12	Test	Measurement of the samples to the samples of the sample of the samples of the sam	15							
Anne	x A (no	ormative) Determination of the mass concentration of uranium isotopes	16							
	-	formative) Description of the matrices of the samples used for the								
1111110		laboratory trial	26							
Anne		formative) Performance data								
	•	y								
	0 1	v								

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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This second edition cancels and replaces the first edition (ISO 17294-2:2003), which has been technically revised.

ISO 17294 consists of the following parts, under the general title *Water quality — Application of inductively coupled plasma mass spectrometry (IGP-MS)*:

- Part 1: General guidelines
- Part 2: Determination of selected elements including uranium isotopes

Introduction

When applying this part of ISO 17294, 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.

Introsil gerdance and a state of the land and a state

Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) —

Part 2:

Determination of selected elements including uranium isotopes

WARNING — Persons using this part of ISO 17294 should be familiar with normal laboratory practice. This part of ISO 17294 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 in accordance with this part of ISO 17294, be carried out by suitably qualified staff.

1 Scope

This part of ISO 17294 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, europium, 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 (xLQ) lies between 0,002 μ g/l and 1,0 μ g/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 <u>Clause 5</u>) or memory effects (see ISO 17294-1:2004, 8.2).

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

Element	Isotope often used	Limit of quantification ^a	Element	Isotope often used	Limit of quantification ^a	Element	Isotope often used	Limit of quantification ^a
		μg/l			μg/l			μg/l
Δ.	¹⁰⁷ Ag	0,5	Hf	¹⁷⁸ Hf	0,1	Ru	¹⁰² Ru	0,1
Ag	109Ag	0,5	Hg	202Hg	0,05	CI.	121Sb	0,2
Al	27A1	1	Но	165Ho	0,1	Sb	123Sb	0,2
As	75Asc	0,1	In	115In	0,1	Sc	⁴⁵ Sc	5
Au	¹⁹⁷ Au	0,5	Ir	193 _{Ir}	0,1		⁷⁷ Se ^c	1
Б	10B	1	К	39Kc	5	Se	⁷⁸ Sec	0,1
В	11B	1	La	¹³⁹ La	0,1		⁸² Se	1
Б	¹³⁷ Ba	3		⁶ Li	10	Sm	¹⁴⁷ Sm	0,1
Ва	138Ba	0,5	Li	⁷ Li	1		118Sn	1
Ве	⁹ Be	0,1	Lu	175Lu	0,1	Sn	120Sn	1
Bi	²⁰⁹ Bi	0,5		²⁴ Mg	1		86Sr	0,5
	⁴³ Ca	100	Mg	²⁵ Mg	10	Sr	88Sr	0,3
Ca	⁴⁴ Ca	50	Mn	55Mn	0,1	Tb 🙌	159Tb	0,1
	⁴⁰ Ca	10		95Mo	0,5	Te	126Te	2
6.1	¹¹¹ Cd	0,1	Мо	⁹⁸ Mo	0,3	St Th O	²³² Th	0,1
Cd	¹¹⁴ Cd	0,5	Na	23Na	10	TI TI	203Tl	0,2
Се	140Ce	0,1	Nd	146Nd	S. Od nda	11	205Tl	0,1
Со	59 C o	0,2	N:	58Nic	and of 1staliso	Tm	169Tm	0,1
C -	52Crc	0,1	Ni	60Nic	11 51 210,17 100		238U	0,1
Cr	53Cr	5	A)	31p 😵	11co 35,0	U [235U	10-4
Cs	133Cs	0,1		206Рbb	0,2		234U	10-5
C	⁶³ Cu	0,1	Pb	207 p b	0,2	V	51 y c	0,1
Cu	⁶⁵ Cu	0,1		208Ррь	0,1	YAZ	182W	0,3
Dy	163Dy	0,1	Pd \\	¹⁰⁸ Pd	0,5	W	184W	0,3
Er	166Er	0,1	Pro	141Pr	0,1	Y	89Y	0,1
Fe	56Fec	5	Pt	¹⁹⁵ Pt	0,5	VI	¹⁷² Yb	0,2
G -	⁶⁹ Ga	0,3	Rb	⁸⁵ Rb	0,1	Yb	174Yb	0,2
Ga	⁷¹ Ga	0,3	D.a.	¹⁸⁵ Re	0,1		64Zn	1
C4	¹⁵⁷ Gd	0,1	Re	¹⁸⁷ Re	0,1	Zn	66Zn	1
Gd	¹⁵⁸ Gd	0,1	Rh	103Rh	0,1		68Zn	1
Ge	⁷⁴ Ge	0,3	Ru	101Ru	0,2	Zr	90Zr	0,2

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

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

b In order to avoid incorrect results due to the varying isotop ratios in the environment, the signal intensities of ²⁰⁶Pb, ²⁰⁷Pb and ²⁰⁸Pb shall be added.

c In order to reach these limits, depending on interferences, the use of a collision/reaction cell is recommended

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 and estimation of performance characteristics — Part 1: Statistical evaluation of the 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 the following apply.

3.1

limit of application

lowest concentration of an analyte that can be determined with a defined level of accuracy and precision

4 Principle

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 to overcome several interferences (see <u>5.1</u>);
- 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 <u>Annex A</u>. 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.

Interferences 5

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 $2700 \,\mu S/cm$).

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 part of ISO 17294, the user is responsible for demonstrating that the chosen approach is fit for purpose and achieves the necessary performance.

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 isotal spectrometer in the first spectrome 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

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.

Element Isotope Inter-element interferences caused Interferences caused by by isobars and doubly charged ions polyatomic ions 107Ag7r0Ag 109Ag NbO, ZrOH As 75AsArCl, CaCl 197Au Ta₀ An 9B 1802+ В 11_R BH 138Ba Ba La+, Ce+

Table 2 — Important isobaric and polyatomic interferences

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		
C	⁴³ Ca	_	CNO		
Ca	⁴⁴ Ca	_	C00		
0.1	111Cd	_	MoO, MoOH, ZrOH		
Cd	114Cd	Sn+	MoO, MoOH		
Со	⁵⁹ Co	_	CaO, CaOH, MgCl		
0	52Cr	_	ArO, ArC, ClOH		
Cr	⁵³ Cr	_	ClO, ArOH,		
	63Cu	_	ArNa, POO, MgCl		
Cu	65Cu	_	SOOH		
_	151Eu	_	BaO		
Eu	153Eu	_	BaO		
	⁵⁴ Fe	- 🖈	37Cl16O1H+ 40Ar14N		
Fe	56Fe	<u> </u>	40Ar160+ 40Ca160+		
ге	⁵⁷ Fe	PRE Sil Sulfand	40Ar16O1H+ 40Ca16O1H+ 40Ar17O+		
Ga	⁶⁹ Ga	Ba++ ds A	CrO, ArP, ClOO		
Ge	⁷⁴ Ge	De de Set de udar 1779	ArS, ClCl		
In	115In	Sh+ olstalist	_		
Ir	¹⁹³ Ir	ST KAITE III SE TAIGHTUE	HfO		
	²⁴ Mg	ail de Ch	CC		
Mg	25Mg	Site 2.83 —	CC		
Mn	55Mn	ndard 8463 —	NaS, ArOH, ArNH		
Мо	⁹⁸ Mo	Ru+	_		
	58Ni	rsi defe	CaO, CaN, NaCl, MgS		
Ni	60Ni	_	CaO, CaOH, MgCl, NaCl		
Pd	¹⁰⁸ Pd	Cd+	MoO, ZrO		
Pt	195Pt	_	HfO		
Re	¹⁸⁷ Re	0s+			
Ru	¹⁰² Ru	Pd+	_		
Sb	123Sb	Te+	_		
Sc	⁴⁵ Sc	_	СОО, СООН		
	77Se	_	CaCl, ArCl, ArArH		
Se	78Se	Kr+	ArAr, CaCl		
	82Se	Kr+	HBr		
Sn	120Sn	Te+	_		
V	51 V	_	ClO, SOH, ClN, ArNH		
W	184W	Os+	. , ,		

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.