INTERNATIONAL STANDARD

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Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)

Qualité de l'eau — Dosage d'éléments choisis par spectroscopie d'émission optique avec plasma induit par haute fréquence (ICP-OES)

Reference number
ISO 11885:2007(E)

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web 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.

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.

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.

ISO 11885 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 11885:1996), which has been technically revised.

Introduction

When applying this International Standard, it is necessary in each case, depending on the range to be tested, to determine if and to what extent additional conditions should be established.

Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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 International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method for the determination of dissolved elements, elements bound to particles ("particulate") and total content of elements in different types of water (e.g. ground, surface, raw, potable and waste water) for the following elements: aluminium, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, calcium, chromium, cobalt, copper, gallium, indium, iron, lead, lithium, magnesium, manganese, molybdenum, nickel, phosphorus, potassium, selenium, silicon, silver, sodium, strontium, sulfur, tin, titanium, tungsten, vanadium, zinc and zirconium.

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 specified in ISO 15587-1 or ISO 15587-2). The method is suitable for mass concentrations of particulate matter in waste water below 2 g/l. The scope of this method may be extended to other matrices or to higher amounts of particulate matter if it can be shown that additionally occurring interferences are considered and corrected for carefully. It is up to the user to demonstrate the fitness for purpose.

Recommended wavelengths, limits of quantification and important spectral interferences for the selected elements are given in Table 1.

2 Normative references

The following referenced documents are indispensable for the application 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 Guide 30, *Terms and definitions used in connection with reference materials*

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: Guidance on the preservation and handling of water samples*

ISO 7027, *Water quality — Determination of turbidity*

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*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
accuracy
closeness of agreement between test result and the accepted reference value

NOTE The term accuracy, when applied to a set of observed values, describes a combination of random error components and common systematic error components. Accuracy includes precision and trueness.

3.2
analyte
element(s) to be determined

3.3
background equivalent concentration
BEC
elemental concentration required to produce an analyte signal with the same intensity as a background signal

3.4
calibration blank solution
prepared in the same way as the calibration solution but leaving out the analyte

3.5
calibration solution
solution used to calibrate the instrument, prepared from (a) stock solution(s) or from a certified standard

3.6
calibration check solution
solution of known composition within the range of the calibration solutions, but prepared independently

3.7
determination
entire process from preparing the test sample solution up to and including measurement and calculation of the final result

3.8
instrument performance check solution
solution used to determine and control the instrument drift for relevant analytes

3.9
linearity
straight line relationship between the (mean) result of measurement (signal) and the quantity (concentration) of the component to be determined

3.10
limit of detection

X_{LD}

smallest amount or concentration of an analyte in the test sample that can be reliably distinguished from zero

NOTE The limit of detection shall be calculated as:

$$X_{LD} = 3\, s_0$$

where

X_{LD} is the limit of detection;

s_0 is the standard deviation of the outlier-free results of at least 3 measurements of a reagent blank solution (3.14)

[ISO 13530]

3.11
limit of quantification

X_{LQ}

the smallest amount or concentration of an analyte in the test sample which can be determined with a fixed precision

EXAMPLE Relative standard deviation s_{rel} = 33,3 %

$$X_{LQ} = 3\, X_{LD} = 9\, s_0$$

[ISO 13530]

3.12
mean result

mean value of n results, calculated as intensity (ratio) or as mass concentration (ρ)

NOTE The mass concentration is expressed in units of milligrams per litre, mg/l.

3.13
precision

closeness of agreement between independent test results obtained under prescribed conditions

NOTE Precision depends only on the distribution of random errors and does not relate to true value or the specified value.

3.14
reagent blank solution

prepared by adding to the solvent the same amounts of reagents as those added to the test sample solution (same final volume)

3.15
reproducibility

precision under reproducibility conditions

[ISO 3534-2:2006, definition 3.3.10]

3.16
reproducibility conditions

observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in different test or measurement facilities with different operators using different equipment

[ISO 3534-2:2006, definition 3.3.11]

3.17
reproducibility standard deviation
standard deviation of test results or measurement results obtained under reproducibility conditions

[ISO 3534-2:2006, definition 3.3.12]

3.18
reproducibility limit
R
reproducibility critical difference for a specified probability of 95 %

[ISO 3534-2:2006, definition 3.3.14]

3.19
repeatability
precision under repeatability conditions

[ISO 3534-2:2006, definition 3.3.5]

3.20
repeatability conditions
observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in the same test or measuring facility by the same operator using the same equipment within short intervals of time

[ISO 3534-2:2006, definition 3.3.6]

3.21
repeatability standard deviation
standard deviation of test results or measurement results obtained under repeatability conditions

[ISO 3534-2:2006, definition 3.3.7]

3.22
repeatability limit
r
repeatability critical difference for a specified probability of 95 %

[ISO 3534-2:2006, definition 3.3.9]

3.23
stock solution
solution with accurately known analyte concentration(s), prepared from chemicals with an appropriate purity

NOTE Stock solutions are reference materials within the meaning of ISO Guide 30.

3.24
test sample
prepared from the laboratory sample (for example by grinding, homogenizing)

3.25
test sample solution
solution prepared with the fraction (test portion) of the test sample according to the appropriate specifications, such that it can be used for the envisaged measurement

3.26
total element concentration
concentration of elements determined on an unfiltered sample following digestion or the sum of concentrations of elements as determined in the dissolved state (9.5.1) and bound in the particulate fraction (9.5.2) of a sample

3.27
trueness
bias
closeness of agreement between the average value obtained from a large series of test results and an accepted reference value

NOTE The measure of trueness is usually expressed in terms of bias (bias = sum of systematic error components).

4 Principle

The basis of the method is the measurement of emission of light by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by a detector. The signals from the detector(s) are processed and controlled by a computer system. A suitable background correction technique is used to compensate for variable background contributions to the determination of trace elements.

5 Recommended wavelengths, limits of quantification and important spectral interferences

Elements for which this International Standard applies along with the recommended wavelengths and typical estimated limits of quantification (LOQ) are listed in Table 1 as far as data are available from the interlaboratory trial (see Annex B). Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, Table 1 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 1 — Recommended wavelengths [a], achievable limits of quantification (X_{LQ} [b]) for different types of instruments and important spectral Interferences

Element	Wavelength	Approx. X_{LQ}		Interfering elements
		Radial viewing	Axial viewing	
	nm	µg/l	µg/l	
Ag	328,068	(20)	(4)	Fe, Mn, Zr
	338,289	(20)	(10)	Cr, Fe, Zr, Mn
Al	167,079	1	2	Fe, Pb
	308,215	100	17	Fe, Mn, OH, V
	396,152	10	6	Cu, Fe, Mo, Zr
As	188,979	18	14	Al, Cr, Fe, Ti
	193,696	5	14	Al, Co, Fe, W, V
	197,197	(100)	31	Al, Co, Fe, Pb, Ti
B	182,528	(6)	—	S
	208,957	(5)	(7)	Al, Mo
	249,677	10	5	Co, Cr, Fe
	249,772	4	24	Co, Fe
Ba	230,425	—	3	—
	233,527	2	0,5	Fe, V
	455,403	6	0,7	Zr
	493,408	(3)	0,4	-
Be	313,042	(2)	(0,1)	Fe
	313,107	—	(0,3)	V
	234,861	(5)	(0,1)	—
Bi	223,060	(40)	(17)	Co, Cu, Ti, V
	306,770	(80)	(165)	Fe, Mo, V
Ca	315,887	100	13	Co, Mo
	317,933	26	4	Fe, V
	393,366	0,4	25	V, Zr
	422,673	—	—	V, Mo, Zr
Cd	214,441	1	0,9	As, Cr, Fe, Sc, Sb
	226,502	4	0,2	As, Co, Fe, Ni
	228,802	2	0,5	As, Co, Sc
Co	228,616	6	1	Ti
	238,892	10	3	Fe