
**Water quality — Determination of
33 elements by inductively coupled
plasma atomic emission spectroscopy**

*Qualité de l'eau — Dosage de 33 éléments par spectroscopie
d'émission atomique avec plasma couplé par induction*

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ISO 11885:1996

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Foreword

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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.

International Standard ISO 11885 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Annex A of this International Standard is for information only.

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Water quality — Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy

1 Scope

1.1 Field of application

This International Standard specifies a method for the determination of dissolved, particulate or total elements in raw, potable and waste water for the following elements:

aluminium, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, calcium, chromium, cobalt, copper, iron, lead, lithium, magnesium, manganese, molybdenum, nickel, phosphorus, potassium, selenium, silicon, silver, sodium, strontium, sulfur, tin, titanium, tungsten, vanadium, zinc, zirconium.

Table 1 lists these elements along with the recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample-dependent and as the sample matrix varies, these concentrations can also vary.

Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst will need to refer to the instructions provided by the manufacturer of the particular instrument.

1.2 Interferences

Table 2 lists elements and the most important spectral interferences at the wavelengths recommended for analysis.

Several types of interference effects can contribute to inaccuracies in the determination of trace elements. They can be summarized as follows.

a) Spectral interferences, categorized as:

- 1) overlap of a spectral line from another element; these effects can be compensated by utilizing computer correction of the raw data;
- 2) unresolved overlap of molecular band spectra; these effects can possibly be overcome by selection of an alternative wavelength.

If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

b) Background influences, categorized as:

- 2) background contribution from continuous or recombination phenomena;
- 3) background contribution from stray light from the line emission of elements in high concentration.

The effect of background interferences can usually be compensated by background correction adjacent to the analyte line.

Table 1 — Recommended wavelengths and typical detection limits

Element	Wavelength nm	Limit of detection mg/l	Element	Wavelength nm	Limit of detection mg/l
Ag	328,068	0,02	Mo	202,030	0,03
	338,289	0,02		204,598	0,05
Al	308,215	0,1	Na	589,592	0,1
	396,152	0,1		588,995	0,02
	167,08	0,04		330,237	0,02
As	193,696	0,1	Ni	231,604	
	197,197	0,1			
	189,042	0,08	P	178,287	0,5
B	208,959	0,005		213,618	0,1
	249,678	0,006		214,914	0,1
	247,773	0,01		177,428	0,5
Ba	233,527	0,004	Pb	220,353	0,2
	455,403	0,002		283,306	0,07
	493,409	0,003	S	182,036	0,5
	313,042	0,002		180,669	0,5
	234,861	0,005			
Bi	223,061	0,04	Sb	206,833	0,1
	306,772	0,08		217,581	0,1
Ca	315,887	0,1	Se	196,026	0,1
	317,933	0,01		203,985	0,1
	393,366	0,002	Si	251,611	0,02
Cd	214,438	0,01		212,412	0,02
	226,502	0,01		288,158	0,03
	228,802	0,01	Sn	235,848	0,1
		189,980		0,1	
Co	228,616	0,01	Sr	407,771	0,0005
				421,552	0,01
Cr	205,552	0,01			460,733
	267,716	0,01	Ti	334,941	0,005
	283,563	0,01		336,121	0,01
	284,325	0,01		337,280	0,01
		368,520		0,01	
Cu	324,754	0,01	V	290,882	0,01
	327,396	0,01		292,402	0,01
Fe	259,940	0,02		310,230	0,01
	238,20			311,071	0,01
K	766,490		W	207,911	0,03
	769,90	2		209,860	0,06
Li	460,286	0,9		239,709	0,06
	670,784	0,002		222,589	0,06
				202,998	0,06
Mg	279,079	0,03	Zn	206,191	0,01
	279,553	0,0005		213,856	0,005
	285,213	0,001			
Mn	257,610	0,002	Zr	343,823	0,01
	293,306	0,02		354,262	0,05
				339,198	

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Table 2 — Spectral interferences

Element	Wavelength nm	Interfering elements	Element	Wavelength nm	Interfering elements
Ag	328,068 338,289	Cr	Mo	202,030 204,598	Al, Fe
Al	308,215 396,152 167,08	Mn, V, Fe Mo, Cu Fe	Na	589,592 588,995 330,237	Ar
As	193,696 197,197 189,042	Fe, Al Fe, Al Al	Ni	231,604	Co
B	208,959 249,678 247,773	Al, Mo Fe, Cr Fe	P	178,287 213,618 214,914 177,428	I Cu, Fe, Mo, Zn Cu, Al, Mg Cu
Ba	233,527 455,403 493,409 313,042 234,861 313,107	Fe, V V Fe	Pb	220,353 283,306	Al, Co, Ti
Bi	223,061 306,772	Cu Fe, V	S	182,036 180,669	Cr, Mo Ca
Ca	315,887 317,933 393,366	Co Fe, V	Sb	206,833 217,581	Cr, Mg, Co, Mn
Cd	214,438 226,502 228,802	Fe Fe As, Co	Se	196,026 203,985	
Co	228,616	Ti	Si	251,611 212,412 288,158	
Cr	205,552 267,716 283,563 284,325	Fe, Mo Mn, V Fe, Mo Fe	Sn	235,848 189,980	Mo, Co
Cu	324,754 327,396	Ti, Fe	Sr	407,771 421,552 460,733	
Fe	259,940 238,20	Co	Ti	334,941 336,121 337,280 368,520	Ca, Cr, Si
K	766,490 769,90	Mg, Ar	V	290,882 292,402 310,230 311,071	Co, Cr Fe, Mo Fe, Mo, Cr Fe, Mn, Ti, Cr
Li	460,286 670,784	Fe	W	207,911 209,860 239,709 222,589 202,998	Cu
Mg	279,079 279,553 285,213	Fe	Zn	206,191 213,856	Cr Cu, Ni, Fe
Mn	257,610 293,306	Fe, Mo, Cr Al, Fe	Zr	343,823 354,262 339,198	

Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies, especially in samples which may contain high concentrations of dissolved solids and/or acid. If these types of interference are operative, they will be reduced by dilution of the sample and/or utilization of standard addition techniques.

Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. These effects are overcome by buffering of the sample matrix and by standard addition procedures (see 8.1.3.2).

Whenever a new or unusual sample matrix is encountered, a series of tests should be performed prior to reporting concentration data for analyte elements. When investigating a new sample matrix, comparison tests can be performed using other analytical techniques, such as atomic absorption spectrometry.

Serial dilution — If the analyte concentration is sufficiently high (minimally 10 x the instrumental detection limit after dilution), an analysis of a dilution needs to agree within 5 % of the original determination (or within some acceptable control limit that has been established for that matrix). If not, a chemical or physical interference effect could be responsible.

Calibration by standard addition (see 8.1.3.2) — A spike added to the original determination at a minimum level of 10 x the instrumental detection limit (maximum 100 x) needs to be recovered to within 90 % to 110 %, or within the established control limit for that matrix. If not, a matrix effect should be suspected. The use of a standard addition analysis procedure can usually compensate for this effect.

2 Normative reference iTeh STANDARD PREVIEW

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

3 Definitions

For the purposes of this International Standard, the following definitions apply:

3.1 dissolved metals:

Those metals in a water sample which will pass through a 0,45 µm membrane filter.

3.2 particulate metals:

Those metals in a water sample which are retained by a 0,45 µm membrane filter.

3.3 total metals:

Concentration of metals determined on an unfiltered sample following digestion (see 8.1.3), or the sum of the dissolved (see 8.1.1) plus particulate metal concentrations (see 8.1.2).

3.4 instrumental detection limit:

Concentration, equivalent to a signal due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

3.5 sensitivity:

Slope of the curve of the relationship between signal emission intensity and sample concentration.

3.6 instrument check standard:

Multielement standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis (see 5.7).

3.7 interference check sample:

Solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

3.8 independent calibration check:

Solution, obtained from an outside source, having known concentration values to be used to verify the calibration standards.

3.9 reference standards:

Series of known standard solutions used by the analyst for calibration of the instrument (i.e. preparation of the calibration curve).

3.10 linear dynamic range:

Concentration range over which the analytical response remains linear.

3.11 reagent blank:

Deionized water, containing the same acid matrix as the calibration standards, which is carried through the entire analytical scheme.

3.12 calibration blank:

Deionized water acidified with nitric or hydrochloric acid.

3.13 method of standard addition:

Analytical technique involving the use of the unknown analyte and the unknown analyte plus a known amount of standard analyte (see 8.1.3.2).

3.14 sample duplication:

Analysis of two portions of the same sample.

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4 Principle

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The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line 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 detectors. The signals from the detectors are processed and controlled by a computer system. A background correction technique is used to compensate for variable background contributions to the determination of trace elements.

5 Reagents

WARNING — The toxicity of each reagent used in this method has not been precisely defined; however, each chemical compound needs to be treated as a potential health hazard. From this viewpoint, exposure to these chemicals to the lowest possible level by whatever means available is recommended.

Preparation methods involve the use of strong acids, which are corrosive and cause burns. Laboratory coats, gloves and safety spectacles should be worn when handling acids.

Toxic fumes are evolved by nitric acid. Always carry out digestion in a fume cupboard, as well as addition of acid to samples because of the possibility of toxic gases being released.

The exhaust gases from the plasma should be ducted away by an efficient fume extraction system.

5.1 General requirements

NOTE — Standard stock solutions may be purchased or prepared from ultrahigh-purity grade chemicals.

The content of the determinants in the water and reagents shall be negligibly low, compared with the concentrations to be determined.

All salts shall be dried for 1 h at 105 °C unless otherwise specified.

5.2 Nitric acid, $\rho(\text{HNO}_3) = 1,40 \text{ g/ml}$

5.3 Dihydrogen dioxide (hydrogen peroxide), H_2O_2 , volume fraction 30 %.

NOTE — On the determination of phosphorus, attention should be paid to a possible stabilization of hydrogen peroxide with phosphoric acid, as this will affect the phosphorous determination.

5.4 Sulfuric acid, $\rho(\text{H}_2\text{SO}_4) = 1,84 \text{ g/ml}$.

5.5 Hydrochloric acid, $c(\text{HCl}) = 0,2 \text{ mol/l}$.

5.6 Ammonium sulfate, $(\text{NH}_4)_2\text{SO}_4$.

5.7 Element stock solutions

Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Se, Si, Sn, Sr, Ti, V, W and Zn at mass concentrations of 1 000 mg/l each.

NOTE — ICP-AES element standard reference solutions with respective specifications which can be used as stock solutions are commercially available. They have been prepared according to particulars of the manufacturer. Normally, these stock solutions contain hydrochloric acid or nitric acid and have a shelf life of several months. Formulations for the preparation of element stock solutions are identified below.

5.8 Intermediate mixed standard solutions

Prepare these as mixed element standards in 5 % nitric acid or 5 % hydrochloric acid (volume fraction). To ensure chemical compatibility use the combinations given in 5.8.1 to 5.8.6.

On composing multielement reference solutions, the chemical compatibility and the possible hydrolysis of the initial compounds, as well as spectral interferences, shall be taken into account. In order to avoid interferences, the digestion reagents (e.g. nitric acid, sulfuric acid, aqua regia) shall be added to the reference solutions.

5.8.1 Multielement reference solution I

$\rho(\text{Al, Be, Cd, Co, Cu, Fe, Pb, Li, Mn, Mo, Ni, V, Zn, Bi, Si, Sr, W and Zr}) = 10 \text{ mg/l}$.

Prepare this solution by adding $(10 \pm 0,1)$ ml of each of the commercial 1 000 mg/l stock standard solutions (5.7) of reference I elements in a 1-litre volumetric flask.

Add 50 ml of nitric acid (5.2).

Make up to the volume with water.

5.8.2 Multielement reference solution II

$\rho(\text{Sn, Ti, As, Se, Sb}) = 10 \text{ mg/l}$.

Prepare this solution by adding $(10 \pm 0,1)$ ml of each of the commercial 1 000 mg/l stock standard solutions (5.7) of reference II elements in a 1-litre volumetric flask.

Add 50 ml of nitric acid (5.2).

Make up to the volume with water.

5.8.3 Element reference solution III

$\rho(\text{Ba}) = 10 \text{ mg/l}$.

Proceed as given in 5.8.2 using $(10 \pm 0,1)$ ml of the commercial 1 000 mg/l stock standard solution of barium.

5.8.4 Element reference solution IV

$\rho(\text{Ag}) = 10 \text{ mg/l}$.

Proceed as given in 5.8.2 using $(10 \pm 0,1)$ ml of the commercial 1 000 mg/l stock standard solution of silver.

5.8.5 Element reference solution V

$\rho(\text{B}) = 10 \text{ mg/l}$.

Proceed as given in 5.8.2 using $(10 \pm 0,1)$ ml of the 1 000 mg/l stock standard solution of boron.

Prepare this standard in polymethylpentene (PMP) flasks.

NOTE — Element reference solutions III, IV, V, i.e. Ba, Ag, B, are known to cause problems by precipitation under certain circumstances, and it is recommended that separate standard solutions are prepared.

5.8.6 Multielement reference solution VI

$\rho(\text{Ca, Mg, Na, K, S, P}) = 10 \text{ mg/l}$.

Prepare this solution by adding $(10 \pm 0,1)$ ml of each of the commercial 1 000 mg/l stock solutions (5.7) of reference VI elements in a 1-litre volumetric flask.

Add 10 ml of nitric acid (5.2).

Make up to the volume with water.

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5.9 Reagent blank solution

Place 1 ml of nitric acid (5.2) and 100 ml of water into a polyethylene or polytetrafluorethylene (PTFE) container.

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6 Apparatus

NOTE — Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided.

6.1 Inductively coupled plasma-atomic emission spectrometer, including:

- computer-controlled atomic emission spectrometer with background correction;
- radiofrequency generator;
- argon gas supply (welding grade or better).

6.2 Sample bottles, 250 ml or 500 ml plastics (such as high density polyethylene or PTFE), for sample collection and storage. Containers shall have been previously cleaned with 10 % volume fraction nitric acid before use.

6.3 Glassware (beakers, filter funnels, volumetric flasks and pipettes) reserved for element preparation only. All glassware shall be soaked in 10 % volume fraction nitric acid overnight before use.

6.4 Acid dispensers, variable volume.

6.5 Membrane filtration equipment and filters of pore size 0,45 μm reserved for element preparation.