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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

Reference number
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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
Ag	328,068	0,02
	338,289	0,02
Al	308,215	0,1
	396,152	0,1
	167,08	0,04
As	193,696	0,1
	197,197	0,1
	189,042	0,08
B	208,959	0,005
	249,678	0,006
	247,773	0,01
Ba	233,527	0,004
	455,403	0,002
	493,409	0,003
	313,042	0,002
	234,861	0,005
	313,107	
Bi	223,061	0,04
	306,772	0,08
Ca	315,887	0,1
	317,933	0,01
	393,366	0,002
Cd	214,438	0,01
	226,502	0,01
	228,802	0,01
Co	228,616	0,01
Cr	205,552	0,01
	267,716	0,01
	283,563	0,01
	284,325	0,01
Cu	324,754	0,01
	327,396	0,01
Fe	259,940	0,02
	238,20	
K	766,490	
	769,90	2
Li	460,286	0,9
	670,784	0,002
Mg	279,079	0,03
	279,553	0,0005
	285,213	0,001
Mn	257,610	0,002
	293,306	0,02
Mo	202,030	0,03
	204,598	0,05
Na	589,592	0,1
	588,995	0,02
	330,237	0,02
Ni	231,604	
P	178,287	0,5
	213,618	0,1
	214,914	0,1
	177,428	0,5
Pb	220,353	0,2
	283,306	0,07
S	182,036	0,5
	180,669	0,5
Sb	206,833	0,1
	217,581	0,1
Se	196,026	0,1
	203,985	0,1
Si	251,611	0,02
	212,412	0,02
	288,158	0,03
Sn	235,848	0,1
	189,980	0,1
Sr	407,771	0,0005
	421,552	0,01
	460,733	0,1
Ti	334,941	0,005
	336,121	0,01
	337,280	0,01
	368,520	0,01
V	290,882	0,01
	292,402	0,01
	310,230	0,01
	311,071	0,01
W	207,911	0,03
	209,860	0,06
	239,709	0,06
	222,589	0,06
	202,998	0,06
Zn	206,191	0,01
	213,856	0,005
Zr	343,823	0,01
	354,262	0,05
	339,198	

Table 2 — Spectral interferences

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

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.

4 Principle

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, $\rho(HNO_3) = 1,40$ 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, $\rho(H_2SO_4) = 1,84$ g/ml.

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

5.6 Ammonium sulfate, $(NH_4)_2SO_4$.

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

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

Prepare this solution by adding (10 ± 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

ρ(Sn, Ti, As, Se, Sb) = 10 mg/l.

Prepare this solution by adding (10 ± 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

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

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

5.8.4 Element reference solution IV

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

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

5.8.5 Element reference solution V

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

Proceed as given in 5.8.2 using (10 ± 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

ρ (Ca, Mg, Na, K, S, P) = 10 mg/l.

Prepare this solution by adding (10 ± 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.

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.

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.

7 Sample handling and preservation

7.1 General

The requirements in ISO 5667-3 apply.

For the determination of trace elements, contamination and the loss of elements of interest are of prime concern. Dust in the laboratory environment, impurities in reagents and impurities on laboratory apparatus which the sample contacts are all sources of potential contamination. Sample containers can introduce either positive or negative errors in the measurement of trace elements by

a) leaching or surface desorption, and

b) by depleting concentrations through adsorption.

Laboratory glassware, including sample bottles, shall be rinsed before use with nitric acid followed by deionized water.

Perform the following preservation and pretreatment steps (filtration and acid preservation) at the time the sample is collected, or as soon as possible thereafter.

7.2 Prior to determination of dissolved elements

Filter the sample through a 0,45 µm membrane filter (6.3.2) as soon as possible after collection (glass or PTFE filtering apparatus are recommended to avoid possible contamination).

Use the first 50 ml to 100 ml to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with 0,5 ml of nitric acid (5.2) per 100 ml of sample to ensure that the pH of the sample is < 2.

7.3 Prior to determination of particulate elements

Filter a measured volume of unpreserved sample through a 0,45 µm membrane filter as soon as possible after collection. Transfer the filter containing particulate material to a container for storage and/or shipment. No preservative is required.

7.4 Prior to determination of total elements

Acidify the sample with 0,5 ml HNO_3 (5.2) per 100 ml sample to pH < 2 as soon as possible, preferably at the time of collection. Do not filter the sample before processing.

8 Instrumental method and analytical procedure

8.1 Instrumental method

8.1.1 General

Follow the instructions provided by the manufacturer of the particular instrument.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects should be investigated and established for each individual analyte line on that particular instrument.

Set up the instrument with the proper operating parameters established from the manufacturer's instruction manual. Allow the instrument to become thermally stable before beginning.

Initiate the appropriate operating configuration of the computer.

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions (5.8.1 to 5.8.6). Flush the system with the reagent blank (5.8.7) between each standard.

Before beginning the sample run, reanalyse the highest-concentration mixed reference standard as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 % (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

Begin the sample run by flushing the system with the reagent blank (5.8.7) solution and flush again between each sample. Analyse the instrument check standard (8.1.2) and the calibration blank every 10 samples.

8.1.2 Instrument performance check procedures

Analyse an appropriate instrument check standard containing the elements of interest with each batch of 10 samples. This check standard is used to determine instrument drift. If agreement is not within ± 5 % of the expected values or within the established control limits, whichever is lower, the analysis is out of control. Terminate the analysis, correct the problem, and recalibrate the instrument.

Analyse the reference standard with each batch of 10 samples. The result shall be within the established control limits of two standard deviations of the mean value. If not, repeat the analysis two more times and average the three results. If the average is not within the control limit, terminate the analysis, correct the problem and recalibrate the instrument.

To verify interelement and background correction factors, analyse the instrument check standard at the beginning, end and at periodic intervals throughout the sample run. Results should fall within the established control limits of 2,0 times the standard deviation of the mean value. If not, terminate the analysis, correct the problem and recalibrate the instrument.

8.1.3 Independent calibration check

8.1.3.1 General requirement

An independent calibration standard obtained from an outside source shall first be used for the initial verification of the calibration standards. A fresh dilution of this sample shall be analysed every week thereafter to monitor their stability. If the results are not within ± 5 % of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

Prepare calibration check standards for all elements from stock solutions reserved for this purpose consisting of the same matrix as the unknown samples and carried through the entire analytical process.

Analyse one calibration standard and one blank solution with each batch of 25 samples and take them through the entire analytical processes (including dilution, filtering and digestion, etc.).

8.1.3.2 Standard addition method of analysis

NOTE — The standard addition technique involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interference. The simplest version of this technique is the single-addition method.

Take two identical aliquots of the sample solution, each of volume V_x. To the first (labelled A) add a small volume V_S of a standard analyte solution of concentration ρ_S of the solvent. Measure the analytical signals of A and B and correct for nonanalyte signals. Calculate the unknown sample concentration ρ_x as follows:

$$\rho_x = \frac{S_B \cdot V_S \cdot \rho_S}{(S_A - S_B) \cdot V_x}$$

where

S_A, S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively;

V_S is the added volume of standard analyte, in millilitres;

V_x is the volume of identical aliquots of sample solution, in millilitres;

ρ_S is the mass concentration of standard analyte solution, in milligrams per litre.

Choose V_S and ρ_S so that S_A is on average twice S_B. It is best if V_S is made much less than V_x and thus ρ_S is much greater than ρ_x, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, first make the addition and carry out the entire procedure. For the results from this technique to be valid, all of the following requirements shall apply:

- the analytical response shall be linear;
- the chemical form of the analyte added shall respond the same as the analyte in the sample;
- the interference effect shall be constant over the working range of concern;
- the signal shall be corrected for any additive interference.

8.2 Analytical procedure

8.2.1 Determination of dissolved elements

Analyse the filtered, preserved sample (7.2) as-received in accordance with 8.1.1. The acid matrix and concentration of the samples and calibration standards shall be the same. If a precipitate has formed upon acidification of the sample or during transit or storage, it shall be redissolved before analysis by adding additional acid and/or by heating on a hot plate.

8.2.2 Determination of particulate elements

Transfer the membrane filter containing the insoluble material (see 7.3) to a glass beaker and add 4 ml of nitric acid (5.2). Cover the beaker with a watch glass and heat gently. The warm acid will soon dissolve the membrane.

Increase the temperature of the hot plate and digest the material. When the acid has nearly evaporated, cool the beaker and watch glass and add another 3 ml of nitric acid (5.2). Cover and continue heating until the digestion is complete, generally indicated by a light-coloured digestate. Evaporate to near dryness (2 ml), cool, add 10 ml of hydrochloric acid (0,2 mol/l) and 15 ml of deionized, distilled water per 100 ml dilution and warm the beaker gently for 15 min to dissolve any precipitated or residue material. Allow to cool, wash down the watch glass and beaker walls with deionized or distilled water and filter the sample to remove insoluble material which can block the nebulizer. Adjust the volume based on the expected concentrations of elements present. This volume will vary depending on the elements to be determined. Analyse the sample in accordance with 8.1.1. Concentrations so determined shall be reported as "particulate".

NOTES

1 Instead of filtering, the sample after diluting and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

2 Calibration standards/Analytical Quality Control solutions should be prepared at the same acid concentration.

3 A filter blank should be taken through the identical procedure as samples and subtracted from the sample analytical results at the calculation stage.

8.2.3 Determination of total elements

Acidify 100 ml of sample (see 7.4) with 0,5 ml of nitric acid (5.2).

Evaporate the mixture to near dryness, making sure that no area at the bottom of the beaker goes dry (a complete drying may lead to low results).

In case of an incomplete digestion (undissolved material), add some water and repeat the treatment.

Dissolve the residue in 1 ml of nitric acid (5.2) and some water and make up to 100 ml volume with water. Analyse the resulting solution in accordance with 8.1.1.

NOTE — Some elements or their compounds (e.g. antimony, silicon, alumina, tin, titanium) are only partially dissolved by this digestion procedure and care should be taken in the interpretation of results.