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Soil quality — Determination of trace elements in extracts of soil by inductively coupled plasma - atomic emission spectrometry (ICP-AES)

Qualité du sol — Dosage des éléments traces dans des extraits de sol par spectrométrie d'émission atomique avec plasma induit par haute fréquence (ICP-AES)

Reference number
ISO 22036:2008(E)

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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 22036 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

INTERNATIONAL STANDARD **ISO 22036:2008(E)**

Soil quality — Determination of trace elements in extracts of soil by inductively coupled plasma - atomic emission spectrometry (ICP-AES)

WARNING — The procedures in this International Standard should be carried out by competent, trained persons. Some of the techniques and reagents, including the use of equipment, are potentially very dangerous. Users of this International Standard who are not thoroughly familiar with the potential dangers and related safe practices should take professional advice before commencing any operation.

1 Scope

This International Standard describes the determination of trace elements in digests or extraction solutions from soil by inductively coupled plasma - atomic emission spectrometry (ICP-AES) for 34 elements (see Table 1).

This multi-element determination method is applicable to soil extracts obtained with aqua regia in accordance with ISO 11466, with DTPA in accordance with ISO 14870 or other weak extractants, or soil extracts for the determination of total element contents using the acid digestion method of ISO 14869-1 or the fusion method of ISO 14869-2.

The choice of calibration method depends on the extractant and can be adapted to the extractant concentration.

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 32, *Calibration in analytical chemistry and use of certified reference materials*

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

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

ISO 11466, *Soil quality — Extraction of trace elements soluble in aqua regia*

ISO 14869-1, *Soil quality — Dissolution for the determination of total element content — Part 1: Dissolution with hydrofluoric and perchloric acids*

ISO 14869-2, *Soil quality — Dissolution for the determination of total element content — Part 2: Dissolution by alkaline fusion*

ISO 14870, *Soil quality — Extraction of trace elements by buffered DTPA solution*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5725-1, ISO 5725-2, ISO Guide 32 and the following apply.

3.1
analyte
element to be determined

3.2
blank calibration solution
solution prepared in the same way as the calibration solution but leaving out the analytes

3.3
blank test solution
solution prepared in the same way as the test sample solution but omitting the test portion

3.4
calibration solution
solution used to calibrate the instrument, prepared from stock solutions by adding acids, buffer, reference element and salts as needed

3.5
instrument detection limit
lowest concentration that can be detected with a defined statistical probability using a clean instrument and a clean solution

NOTE The clean solution is usually dilute nitric acid.

3.6
laboratory sample
sample sent to the laboratory for analysis

3.7
linearity
straight-line relationship between the mean result of measurement and the quantity (concentration) of the analyte

3.8
method detection limit
lowest concentration that can be detected using a specific analytical method with a defined statistical probability for defined maximum matrix element concentrations

3.9
pure chemical
chemical with the highest available purity and known stoichiometry

NOTE The content of analyte and contaminants should be known with an established degree of certainty.

3.10
stock solution
solution with accurately known analyte concentration(s), prepared from pure chemicals (3.9)

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

3.11
test sample
portion taken from the laboratory sample after homogenizing, grinding, dividing, etc.

3.12
test sample solution

solution prepared after extraction or dissolution of the test sample according to appropriate specifications

NOTE The test sample solution is intended for use for measurement.

4 Principle

Inductively coupled plasma - atomic emission spectrometry (ICP-AES) can be used to determine trace elements in solution. The solution is dispersed by a suitable nebulizer and the resulting aerosol is transported into the plasma torch. In a radio-frequency inductively coupled plasma the solvent is evaporated, the dried salts are then vaporized, dissociated, atomized and ionized. The atoms or ions are excited thermally and the number of photons emitted during transition to a lower energy level are measured with optical emission spectrometry. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. The identification of the element takes place by means of the wavelength of the radiation (energy of photons), while the concentration of the element is proportional to the intensity of the radiation (number of photons). The ICP-AES method can be used to perform multi-element determinations using sequential or simultaneous optical systems and axial or radial viewing of the plasma.

Table 1 shows examples of recommended wavelengths, and detection limits for one particular instrument. Data given are valid for water acidified with nitric acid with an optimized instrument. Using other instruments can lead to different detection limits. Adoption of other wavelengths is possible.

Table 1 — Recommended wavelengths and estimated detection limits for selected elements and wavelengths obtained using ICP-AES Varian, Vista-MPX megapixel (CD detector features) [9]

Element wavelengths and analytical lines			Axial viewing		Radial viewing	
Element	Wavelength nm	Lines I = atom II = ion	Detection limit µg/l [a]	Detection limit mg/kg [b]	Detection limit µg/l [a]	Detection limit mg/kg [b]
Aluminium	396,068		1	0,10	4	0,4
	308,215	I	2,6	0,26		
	309,271	I				
	396,152	I	0,1	0,01	4	0,4
	167,078	I	0,3	0,03	1	0,1
Antimony	206,833	I	0,5	0,5	16	1,6
	217,581	I	1,8	0,18	5	0,5
	231,146	I	2	0,2		
Arsenic	188,979		2	0,2	12	1,2
	193,696		1	0,1	11	1,1
	197,198	I	5	0,5		
	189,042	I				
	188,979	I	1,5	0,15	5	0,5
Barium	233,527	II	0,06	0,006	0,7	0,07
	455,403	II	0,01	0,001	0,15	0,02
	493,409	II	0,04	0,004	0,15	0,02
Beryllium	313,107	II	0,03	0,003	0,15	0,02
	313,402	II	0,01	0,001	0,15	0,02
	234,861	II	0,01	0,001	0,05	0,005
Bismuth	223,061	I	1,8	0,18	6	0,6
	306,771	I	17	1,7		
	315,887					
Boron	208,959	I	0,7	0,07	1,2	0,12
	249,678	I	1,1	0,11	1,5	0,15
	249,772	I	0,5	0,05	1	0,1

Table 1 (*continued*)

Element wavelengths and analytical lines			Axial viewing		Radial viewing	
Element	**Wavelength** nm	**Lines** I = atom II = ion	**Detection limit** µg/l [a]	**Detection limit** mg/kg [b]	**Detection limit** µg/l [a]	**Detection limit** mg/kg [b]
Cadmium	214,438	II	0,1	0,01	0,5	0,05
	226,502	II	0,11	0,011	0,6	0,06
	228,802	II	0,20	0,02	0,5	0,05
Calcium	396,847	II	0,5	0,05	0,3	0,03
	317,933	II	0,3	0,03	6,5	0,7
	393,366	II	0,5	0,05		
Chromium	267,716	II	0,1	0,01	1	0,1
	205,552	II	0,3	0,03		
	206,149	II				
	283,563	II	0,2	0,02		
	284,325	II				
Cobalt	238,892	II	0,4	0,04	1,2	0,1
	228,616	II	0,4	0,04	1	0,1
	230,786	II				
Copper	327,396	I	0,3	0,03	1,5	0,1
	224,700	II				
	324,754	I	0,6	0,06		
Iron	238,204	II	0,3	0,03	0,9	0,09
	239,562	II				
	259,940	II	0,5	0,05	0,7	0,07
Lead	220,353	II	0,4	0,04	8	0,8
	216,999	I				
	224,688	I				
	261,418	I				
	283,306	I	1,8	0,18		
Lithium	670,783	I	1,7	0,17	1	0,1
	460,286	I	67	6,7		
Magnesium	279,553	II	0,02	0,002	0,1	0,01
	279,079	II	1	0,1	4	0,4
	285,213	I	0,06	0,006	0,25	0,025
	279,806	II	1,5	0,15	10	1
Manganese	257,610	II	0,10	0,01	0,13	0,01
	260,569	II				
	279,482	II				
	293,306	II	0,4	0,04	1	0,1
	403,076	I	0,8	0,08		
	259,372	II	0,05	0,005		
Mercury	194,227	II	1,2	0,12	2,5	0,25
	253,652	I	1	0,1	2	0,20
	184,890	I				
Molybdenum	202,030	II	0,2	0,02	2	0,2
	204,598	II	0,6	0,06	3	0,3
Nickel	231,604	II	0,4	0,04	2,1	0,2
	221,647	II	0,3	0,03	1,4	0,14
	216,555	I	0,15	0,015		
	232,003	II				
Phosphorus	177,428	I	1,5	0,15	25	2,5
	178,222	I	7	0,7		
	213,618	I	1,3	0,13	5,3	0,53
	214,914	I	1	0,1	11	1,1

Table 1 (*continued*)

Element wavelengths and analytical lines			Axial viewing		Radial viewing	
Element	**Wavelength** nm	**Lines** I = atom II = ion	**Detection limit** µg/l [a]	**Detection limit** mg/kg [b]	**Detection limit** µg/l [a]	**Detection limit** mg/kg [b]
Potassium	766,491 769,896	I I	0,2 23	0,02 2,3	4 12	0,4 1,2
Rubidium	780,03	I	1	0,1	5	0,5
Selenium	196,026 203,985	I I	0,8 2,8	0,08 0,28	16	1,6
Silicon	251,611 212,412 288,158	I I I	0,9 1,3 1	0,09 0,13 0,1	2,2 5	0,22 0,5
Silver	328,068 338,289	I I	0,4 1	0,04 0,1	1 2	0,1 0,2
Sodium	589,592 588,995 330,237	I I I	0,6 12 69	0,06 1,2 6,9	1,5 15	0,2 0,15
Strontium	407,771 421,552 460,733	II II I	0,01 0,01 0,3	0,001 0,001 0,03	0,1 0,1	0,01 0,01
Sulfur	181,962 182,036	I	4	0,4	13	1,3
Thallium	190,800 190,864	II II	2	0,2	13	0,1
Tin	189,933 235,484 283,998	II I I	6 23 11	0,6 2,3	8 20	0,8 2,0
Titanium	336,121 334,941 337,280	II II II	0,15 0,2 0,2	0,015 0,02 0,02	1 0,25 1	0,1 0,25 0,1
Vanadium	292,402 309,310 311,837 290,882 310,230	II II II II II	0,3 0,08 0,1	0,03 0,008 0,01	2	0,2
Zinc	213,856 202,548 206,200	I II II	0,05 0,03 0,15	0,005 0,003 0,015	0,8 0,7 2	0,08 0,07 0,02

[a] Typical 3-sigma detection limits using 30 s integration time.

[b] The detection limit (LOD), as a mass fraction of the soil sample in mg/kg dry matter, is given assuming that a test sample of 1 g is extracted and diluted to 100 ml. The LOD shown in Table 1 are only examples of a given equipment and laboratory conditions. Each laboratory shall select appropriate wavelengths and determine LOD under its specific laboratory conditions.

NOTE The wavelengths given in Table 1 are often used, but they are given here only as an example. Adoption of other wavelengths is possible. The limit of detection and the linear range vary for each element with the wavelength, spectrometer, operating conditions and matrix load in the sample solution. If solutions with high salt concentrations (typical for soil extract solutions) are measured, the LOD is substantially increased compared with water samples.

This International Standard refers specifically to the use of inductively coupled plasma - atomic emission spectrometry. Users of this International Standard are advised to operate their laboratories to accepted quality control procedures. Certified Reference Materials (CRM) should be used to establish the amounts of the relevant elements in in-house reference materials. The latter can be used for routine quality control of the procedures given in this International Standard.

Results shall be established with control charts, for each element, within the laboratory. No result shall be accepted which falls outside an agreed limit. Quality control procedures based on widely accepted statistical techniques shall be used to establish such limits, that these are stable and that no long-term drift is occurring. Certified Reference Materials should be used regularly to maintain the integrity of the in-house reference materials and, thereby, the quality control system.

5 Interferences

5.1 General

The presence of different matrix elements in the sample solution can cause severe interferences, which result in systematic errors of the analyte signal. Special techniques, e.g. background correction, matrix matching of the calibration solution or the standard addition technique, can be used to compensate such interferences.

Interferences are classified into spectral and non-spectral interferences. They can be specific for an analyte or non-specific.

Spectral interferences (see 5.2) are due to incomplete isolation of the radiation emitted by the analyte from other radiation sources detected and amplified by the detection system (additive interferences).

Non-spectral interferences (see 5.3) are interferences where the sensitivity changes due to the composition of the solutions to be measured (multiplicative interferences). The observed matrix effect is a composite interference due to all of the components in the sample solution.

Background correction is required for trace element determination. Background emission shall be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on one or both sides of the analytical line, is determined by the complexity of the spectrum adjacent to the analyte line. The position used should be as free as possible from spectral interference, and should reflect the same change in background intensity as occurs at the analyte wavelength measured. Increase in background is more intensive with axial-view instruments. Background correction is not required in cases of line broadening, where the analytical result is actually degraded by a background correction measurement.

5.2 Spectral interferences

Spectral interferences are, e.g.

- partially or complete overlap of an emission line of another element with that of the analyte; special case: increase of background caused by a wing of a strong emission line located nearby, e.g. sloping background shift at Pb 220,353 nm caused by Al 220,463 nm,
- overlap of a molecular band from a multi-atomic particle formed in the plasma from the solvent, the ambient air or the gases (e.g. N_2^+, NO, NH, OH, CN) with the emission line of an analyte,
- background increase caused by recombination phenomena, e.g. continuum emitted by Al between 190 nm to 220 nm,
- increase of background caused by stray light.

A spectral line overlap usually leads to the choice of an alternative line. If this is not possible, mathematical correction procedures (e.g. inter-element correction technique, multi-component spectral fitting) can be used to compensate the interference. A parallel background shift can be compensated by background correction. To correct a sloping background shift, two background correction points on each side of the peak are used.

For the investigation of spectral interferences of aqua regia extracts of soil, the most prominent lines of the analytes As, Cd, Co, Cr, Cu, Mn, Ni, Pb, Tl and Zn were used. The most important soil elements Al, As, Ca, Cr, Cu, Co, Cu, Fe, Mg, Mn, Mo, Ni, Ti, V and Zn were used as interference elements in two concentrations: 100 mg/l and 500 mg/l. These element concentrations are equal to 0,33 % and 1,67 % (mass fraction) in soils, for aqua regia extraction carried out in accordance with ISO 11466.

Tables B.1 and B.3 in Annex B give a summary of potential spectral interferences when analysing aqua regia extracts of soils. Both the interfering elements and the emission line of these elements are given. A Perkin-Elmer Optima 3000[1] instrument with a spectral resolution of 0,006 nm at 200 nm was employed for the study for Table B.1, and a Varian Vista-PRO[1] with axial plasma for Table B.3. Line coincidences, which are dependent on the spectral resolution of the spectrometer, only become perceptible when the concentration of the interfering element and analyte reach a critical level.

In Table B.2 the interference is expressed as analyte concentration equivalents (i.e. false positive increase of analyte concentrations) arising from 100 mg/l and 500 mg/l of the interfering element, respectively. The data are intended as a guide for indicating the extent of potential interference. The user should be aware that other instruments may exhibit somewhat different levels of interference than those shown in Table B.2, because the intensities vary with instrument construction and operating conditions, such as power, introduction gas flow rate, and observation height.

Some potential spectral interferences observed for the recommended wavelengths using an axial viewing instrument are given in Table B.3. For example, if Cr is to be determined at 267,716 nm in a sample containing approximately 100 mg/l of Al, a false positive signal is observed for a Cr level equivalent to approximately 0,06 mg/l. The user should take into account that other instruments may exhibit levels of interference somewhat different from those shown in Table B.3. The interference effects shall be evaluated for each individual instrument, whether configured as a sequential or simultaneous instrument. For each instrument, intensities vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences (see Table B.3) as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst should use a computer routine for automatic correction on all analysis.

5.3 Non-spectral interferences

Non-spectral interferences can occur during nebulization or sample introduction (physical nature) or in the plasma itself (both physical and chemical natures).

Transport interferences are due to differences in the physical properties (viscosity, surface tension, density) between the sample solutions and the calibration solutions. They are caused by differences in the dissolved solid content (e.g. salts, organic substances) as well as in the type or concentration of acid. As a consequence, the supply of solution to the nebulizer, the efficiency of nebulization and the droplet size distribution of the aerosol are altered, and hence the sensitivity changes. Errors due to these interferences can be overcome by dilution of the solutions, by matrix matching, by standard addition or by the reference element technique (internal standardization).

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[1] Perkin-Elmer Optima 3000 and Varian Vista-Pro are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Excitation interferences cause changes in the sensitivity as a result of changed plasma conditions due to introduction of the matrix. These changes are attributed to a change in the excitation conditions in the plasma caused by easily ionizable elements like alkali metals. Enhancement or depressant effect of easily ionizable elements on analyte emission depends on the operating conditions of the plasma (e.g. power, sample introduction gas flow rate, observation height), and differ from element to element. Improvement of the plasma conditions can therefore reduce excitation interferences. Other possibilities are dilution of the solutions, matrix matching or the standard addition technique.

6 Reagents

All reagents shall be of recognized analytical grade.

6.1 Water.

Use demineralized water or water distilled from an all-glass apparatus, conforming to Grade 2 of ISO 3696. The water used for blank determinations, and for preparing reagents and standard solutions, shall have element concentrations that are negligible compared with the lowest concentration to be determined in the sample solutions.

An example of reagents used for aqua regia extractions in accordance with ISO 11466 is given in the following. Reagents based on other International Standards or other documents should be prepared accordingly.

6.2 Nitric acid, $w(HNO_3) = 65$ %; ~ 1,40 g/ml.

The same batch of nitric acid shall be used throughout the procedure.

6.3 Nitric acid (1+1).

Add 500 ml nitric acid (6.2) to 400 ml water, mix and dilute to 1 l.

6.4 Hydrochloric acid, $w(HCl) = 37$ %; ~ 1,18 g/ml.

The same batch of hydrochloric acid shall be used throughout the procedure.

6.5 Hydrochloric acid (1+1).

Add 500 ml hydrochloric acid (6.4) to 400 ml water (6.1), mix and dilute to 1 l.

Other reagents used for dissolution or extraction of soil samples are described in the relevant standards.

6.6 Preparation of stock solutions and standard solutions of individual elements.

Two sources of stock solutions are available:

- commercially available stock solutions;
- stock solutions prepared in the laboratory from pure elements or stoichiometrically defined salts or oxides. The concentrations of single-element solutions are 1 000 mg/l.

NOTE Commercially available stock solutions have the advantage that they remove the need to handle directly toxic metals, especially thallium. However, special care needs to be taken that these solutions are supplied with a certified composition from a reputable source and are checked on a regular basis.

6.7 Intermediate standard solutions.

Intermediate standard solutions may be prepared for each individual analyte, or for multi-element standard solutions by dilution of stock solutions. These solutions should be stabilized by adding 10 ml nitric acid (6.3) to 100 ml of solution. The intermediate solutions have only limited stability and should be discarded after three months, depending on the solution concentration.

6.8 Multi-element standard solutions.

If several elements are to be determined on each sample, it can be more convenient to prepare multi-element standard solutions.

Suggested multi-element mixed standard solutions are as follows:

- **Mixed standard solution 1**:

  Al, B, Be, Cd, Co, Cr, Cu, Fe, Pb, Li, Mn, Mo, Ni, V, Bi, Sr, Zn, Zr and 10 ml nitric acid (6.3) in 100 ml.

- **Mixed standard solution 2**:

  As, Se, Sb, Sn and Tl with 5 ml nitric acid (6.3) and 40 ml hydrochloric acid (6.5) in 100 ml.

- **Mixed standard solution 3**:

  Ca, Mg, Na, K, Ba, Ti and 10 ml nitric acid (6.3) in 100 ml.

Matrix matching should be used if high concentrations of co-extracted elements like Al, Ca, Fe, Mg, Na or K are observed. The stability of calibration solution can be checked by comparison with freshly prepared solutions on a regular basis.

Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Other elements combinations are also possible and depend on the analytical task. To avoid cross-contamination, only pure chemicals should be used. The diluted solutions should be stored in clean PFA-, FEP-fluorocarbon, HDPE or PP bottles. For concentrations below 1 mg/l, the stability is limited and shall be checked.

6.9 Reference element solution.

Sc, Y or Lu is commonly used as a reference element (internal standard). A concentration of 5 mg/l in nitric acid solution (6.3) should be used. This concentration should be added to blank calibration solutions, quality control solutions and test solutions.

7 Instrumentation

The ICP atomic emission spectrometer consists of a sample introduction system, the plasma (as an excitation source), an optical system, a detector and a computer. The sample introduction system usually consists of a rotation tube pump for sample transport to the nebulizer, the nebulizer and a spray chamber. The most common nebulizers are the concentric nebulizer [e.g. Meinhardt[2]], the cross-flow nebulizer, the V-groove nebulizer [Babington[3]] with self-aspiration capabilities for the first two, but usually a pump is used for assisting the sample transport. They are made from glass, quartz or plastics.

Also many types of spray chambers are applied in commercial systems. The most common, beside the impact bead and the cyclonic type, is the Scott[4] type where double concentric tubes separate larger droplets from the fine aerosol which is introduced into the plasma torch.

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[2] Meinhardt is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

[3] Babington is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

[4] Scott is an example of a suitable product type available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

The torch consists of three concentric tubes [Fassel[5] type]. Quartz is the most commonly used material. The sample introduction tube or injector tube can be made of HF-acid resistant material, e.g. aluminium oxide or platinum. The plasma gas flow and the auxiliary gas flow rates depend on the type of construction and are around 10 l/min to 20 l/min and 0 l/min to 3 l/min, respectively.

At the end of the torch a water-cooled coil with up to five windings is placed, through which a high alternating current flows to excite the plasma. The frequency used by manufacturers differs from around 27 MHz to 56 MHz with a power of 600 W to 2 000 W.

The emission from the plasma can be observed either from the side (radial view) or from the torch central symmetrical axis (axial view). For many elements, axial-viewing instruments provide lower detection limits (up to a factor of 10, but interferences can be more severe).

Spectral lines are measured and registered in either a sequential or a simultaneous manner. Classical grating polychromators as well as computer-controlled devices are used.

NOTE 1 The computerized registration of light intensities by several element lines is converted into concentrations using appropriate software packages from the instrument manufacturers.

NOTE 2 Details of various instrumentation construction can be found in References [9], [10], [11].

8 Procedure

8.1 Cleaning of glassware

All glassware used in the determination of trace element concentrations shall be cleaned carefully before use, e.g. by immersion in 5 % (*v*/*v*) aqueous nitric acid solution for a minimum of 6 h, followed by rinsing with water (6.1) before use. The nitric acid shall be replaced each week.

8.2 Instrument performance parameters

Due to differences between various models of instruments, no detailed instructions can be given to operate the specific instrument. The instruction provided by the manufacturer for waiting time, for instrument stability, gas flow rates, plasma conditions, nebulizer conditions, sample uptake rates, etc., should be followed.

The following performance parameters should be assessed with typical matrix concentrations:

- selection of appropriate analyte wavelength;
- working range and linearity;
- long- and short-term stabilities, relative standard deviation (RSD) of measurements;
- limit of detection of the method for each analyte and of each type of sample (MDL);
- verification of inter-element corrections;
- verification of routines for correcting spectral interferences;
- rinsing time between samples and standards;
- background equivalent concentration (BEC).

The performance of the measuring equipment should be checked with appropriate quality control solutions before measuring test samples (tuning).

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[5] Fassel is an example of a suitable product type available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

8.3 Instrument optimization

The manufacturer's instructions for operating conditions should be followed to assess maximum signal-to-background ratio of the least sensitive elements, such as As, Se Pb and Tl. All gas flow rates should be stabilized and controlled, e.g. with mass flow controllers.

8.4 Alignment of the spectrometer

8.4.1 General

Set up the instrument according to the manufacturer's instructions and ignite the plasma. Verify that the instrument configuration and performance criteria satisfy the safety and analytical requirements (e.g. laboratory environment conditions, power, exhaustion requirements). The plasma shall be allowed to become thermally stable before starting the measurement (usually at least 30 min stabilization time prior to calibration).

8.4.2 Software method development, wavelength selection

Develop a method following the instructions provided by the instrument manufacturer. Select the spectral lines on the basis of information (detection limits, spectral interferences) given in the literature (e.g. atlas for emission lines), the computer library of the spectrometer and in this International Standard (Table 1 and Tables B.1 to B.3).

NOTE The information given is intended as guide for indicating potential interferences. Other instruments with other spectral resolution can exhibit different interferences.

When working with an unknown matrix, check the wavelengths of every element to be measured for possible interferences by studying the region of the line. If spectral interferences (partial line overlap, line coincidence) occur, the measurement shall be carried out at another wavelength. If this is not possible, correction of the interference by mathematical correction procedures (e.g. inter-element correction technique, multi-component spectral fitting) should be carried out.

Background correction is required for trace element determination with ICP-AES. Background shall be measured adjacent to analyte lines on the sample during analysis. The position selected for the background-intensity measurement, on one or both sides of the analyte line, is determined by the complexity of the spectrum adjacent to the analyte line. The position shall be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelengths measured.

8.4.3 Wavelength correctness, optimization of measurement conditions

Instrument optics often require calibration of wavelengths (e.g. reprofiling by using a reference line or by calculating the offset of special set-up-lines) after switching on the ICP-AES. The procedure is inherent to the instrument in use, and therefore shall follow the manufacturer's recommended practice.

The aim of the optimization of the measurement conditions is to find the best sensitivity and best precision for the set of lines to be used. Emission efficiency is related, amongst other parameters, to the plasma temperature, which is a function of RF power, argon gas flow rates and observation height (for radially viewed plasmas). Also, the type of nebulizer and sample uptake rate has an impact on the signal and the background. Optimization for a multi-element analysis involves a change in the parameters that influence the intensity of signal and background (e.g. RF power), resulting in compromise conditions. For special procedures (e.g. alignment of the plasma viewing position), refer to the instrument manufacturer's manual.

8.4.4 Long-term stability

Long-term stability (one day, several hours) assessment is a measurement of the instrument drift. A common procedure is to compensate the drift by the reference element technique (using an internal standard). A possible drift can also be detected by analysing a calibration standard or the quality control sample at regular intervals between samples. With these measured values, an apparent instrument drift can be compensated by a mathematical procedure. The individual behaviour of each instrument shall be checked.

8.4.5 Preliminary instrument check

The reproducibility and sensitivity of the system shall be checked on a daily basis. The precision of replicates should be below RSD of 1 %.

NOTE This can be done by measuring a 1 mg/l solution of manganese, with a minimum of five replicates, with the selected integration time.

Nebulizer condition, steady even flow rate of the peristaltic pump, gas flow rate, observation height, torch conditions, etc., shall be controlled before measurement of samples.

8.5 Calibration methods

Three calibration techniques are available:

a) the standard calibration method, with or without matrix matching in the calibration solutions;

b) the bracketing method;

c) the standard addition method.

Most important is the standard calibration method, where the light emission intensities of analyte lines are measured in the calibration and test sample solutions. With assistance of a linear calibration curve, the concentrations are calculated in the unknown sample solutions. The linearity over a broad concentration range shall be checked for setting the calibration range. Alkaline element lines in particular suffer from non-linear calibration curves, due to ionization and self-absorption effects, and can be calculated by second-order curve fit with appropriate regression lines.

Calibration standards should be prepared freshly each time a batch of samples is analysed. All acids, salts, buffers, detergents and releasers which are present in the test sample solution shall also be present in the calibration solutions in the same concentration. The use of a reference element is highly recommended, and should be added to the calibration solutions. The reference element shall not be present in the sample. Therefore, only negligible or very low concentrations should originally be present in extract or digestion solutions.

A minimum of four calibration standards with equidistant analyte concentrations should cover the calibration range. If only a two-point calibration procedure is applied, using a blank calibration and a high calibration standard solution, the linearity shall be checked. The accuracy of the concentration of this calibration solution shall be assured.

In soil extracts, especially in aqua regia extracts of soil, ubiquitous elements such as Al, Na, K, Ca, Mg, Ti and Fe can be co-extracted, resulting in element concentrations of several hundreds of mg/l. The efficiency of the method selected to compensate spectral interferences, background subtraction, transport interference shall be checked by analysis of control samples and interference control samples. Otherwise the sample matrix elements shall be adapted in calibration solutions for each batch of sample types. If this is not practicable, the standard addition method shall be used.

8.6 Solutions to be prepared

8.6.1 General

Two types of blank solution are required for the analysis: the calibration blank solution (8.6.2) and the blank test solution (8.6.3) prepared during test sample processing.

8.6.2 Blank calibration solution

This solution is prepared by adding the same amount of acids, buffer concentration or salt concentration as in the calibration and test sample solutions. A sufficient quantity should be prepared to flush the system between standards and samples, and to be used as a quality control sample. If a reference element is applied, add the same concentration as used in standards and samples (5 mg/l).

8.6.3 Blank test solution

This solution is prepared during the extraction or dissolution process of the sample. Carry out a blank test at the same time as the extraction or dissolution of soil samples, and follow the sample procedure, using the same quantities of all the reagents for the determination, but omitting the test portion. If a reference element is used, add the same concentration as in standards and samples.

Carry out a blank test at the same time as the extraction or digestion of the soil sample, following the same procedure.

8.6.4 Calibration solutions

Prepare mixed calibration solutions covering the range of concentrations to be measured by combining appropriate volumes of the stock solutions in volumetric flasks. Care should be taken when preparing the mixed calibration solutions to ensure that the elements are compatible and stable together. Add appropriate volumes of acids and/or the solutions used for the soil extraction so that the matrix of the calibration solutions corresponds with the matrix of the sample solutions. Fill up the volumetric flasks to the mark with water or acid to adjust the same acidity as in the sample solutions.

Matrix matching of the blank calibration solution and the calibration solutions concerning the main components such as Al, Ca, Fe, K, Mg and Na is recommended, if high concentrations of these elements are present in the sample solution.

Sc, Y or Lu is commonly used as an internal standard. If the reference element technique is to be used, the internal standard shall be added to all calibration solutions, the quality control solution and the sample solutions, so that all solutions contain the reference element in the same concentration.

8.6.5 Test sample solutions

Carry out the sample preparation in accordance with ISO 11466, ISO 14870, ISO 14869-1 or ISO 14869-2 or other standards, as appropriate, to obtain a test sample.

8.7 Measurement procedure

After stabilization of the instrument and verification of stable conditions (8.4), carry out measurements on blank calibration and calibration solutions, calibration verification solution, blank test and sample solutions and quality control solutions. The rinse time between solutions shall be long enough not to contaminate the next solution.

The temperature of all calibration, quality control and test solutions should be within 2 °C of each other at the time of ICP-AES measurement.

After a sufficient delay (depending on sample flow rate stability), read and record the emission intensity of the solution at least twice and, if the values fall within an acceptable range, average the values.

NOTE The definition of an acceptable range is outside the scope of this International Standard. However, users are reminded to review Clause 4 concerning quality control procedures. Whatever the basis for the latter in the laboratory, it should conform to well-established practices, such as those based on Certified Reference Materials, in-house reference materials, verification of calibration accuracy, recovery rate of spiked samples, linearity check, control charts and other measures.

If an unknown type of sample is to be handled, determine the element concentration by the standard addition method. If the analytical results according to the standard addition method and the standard calibration method are equal, the calibration curve method can be applied.

9 Calculation of results

By reference to the calibration graph obtained, the software calculates the concentration of each element corresponding to the intensities of the test sample solution (see 8.6.5) and of the blank test solution (8.6.3). Calculate the mass fraction (w) of the element (E) of the sample for each element using Equation (1):

$$w_{\mathrm{E}} = \frac{(\rho_1 - \rho_0)}{m} \cdot f \cdot V \cdot C \qquad (1)$$

where

w_{E} is the mass fraction of the element E in the sample, in milligrams per kilogram dry matter;

ρ_1 is the element concentration, in milligrams per litre, corresponding to the test sample solution;

ρ_0 is the element concentration, in milligrams per litre, corresponding to the blank test solution;

f is the dilution factor of test sample, if applicable;

V is the volume, in millilitres, of the test portion taken for analysis, e.g. 100 ml for aqua regia extraction in accordance with ISO 11466;

m is the mass of sample, in grams;

C is the correction factor for the dry soil sample: $C = 100/w_{\mathrm{dm}}$;

w_{dm} is the dry matter content of soil, expressed as a mass fraction (%) in accordance with ISO 11465.

10 Precision

An interlaboratory test carried out in the Netherlands in 2005 yielded the data given in Table A.1. Table A.2 and Table A.3 contain data from proficiency tests carried out in Germany in 2004 and 2005. Repeatability and reproducibility were calculated according to the principles of ISO 5725-2.

11 Expression of results

The measurement uncertainty reported for the results should reflect the results from quality control measurements, and incorporate the deviation between the individual readings for the sample in question. In general, values shall not be expressed to a degree of accuracy greater than three significant figures. The rounding of values will depend on the statistics of the quality control procedures mentioned earlier, and the requirements of the analysis.

EXAMPLE $w_{\mathrm{Cd}} = 8{,}54$ mg/kg

$w_{\mathrm{Cd}} = 12{,}6$ mg/kg

12 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) complete identification of the sample;

c) a reference to the method used;

d) the results of the determination;

e) any details not specified in this International Standard or which are optional, as well as any factor which may have affected the results.

Annex A
(informative)

Repeatability and precision results

Table A.1 — Repeatability and precision results for the determination of trace elements in aqua regia extracts of soil measured by ICP-AES

Sample	Element	N_{lab}	N_{res}	w_{E}	V_r	V_R	S_r	S_R
PD 152b	As	8	474	43,4	6,2	9,5	2,7	4,1
PD 152b	Ba	7	406	800,0	6,0	9,1	48	73
PD 152b	Cd	8	475	8,0	5,8	8,0	0,5	0,6
PD 152b	Co	6	357	18,4	7,4	9,8	1,4	1,8
PD 152b	Cr	8	474	180	7,0	8,9	13	16
PD 152b	Cu	8	475	154	5,5	7,1	8	11
PD 152b	Ni	8	476	52	6,4	8,9	3,3	5
PD 152b	Pb	8	454	268	5,2	8,7	14	23
PD 152b	Zn	8	475	969	5,4	7,2	52	69

N_{lab} is the number of laboratories after elimination of outliers;

N_{res} is the number of results;

w_{E} is the content of element E in the sample, in milligrams per kilogram (mg/kg) dry matter;

V_r is the relative repeatability standard deviation, in percent (%);

V_R is the relative reproducibility standard deviation, in percent (%);

S_r is the repeatability limit, in milligrams per kilogram (mg/kg);

S_R is the reproducibility limit, in milligrams per kilogram (mg/kg).

Table A.2 — Repeatability and precision results of 10th BAM PT-scheme 2004 for the determination of trace elements in aqua regia extracts of soil by ICP-AES, calculated in accordance with ISO 5725-2

Sample	Element	N_{lab}	N_{res}	w_{E}	V_r	V_R	S_r	S_R
10-N1	As	32	64	32,9	2,2	12,2	0,71	4,01
10-N2	As	24	48	28,6	2,3	7,1	0,66	2,02
10-N3	As	56	112	15,9	2,6	7,2	0,41	1,15
10-N1	Cd	34	68	25,7	1,6	6,2	0,4	1,58
10-N2	Cd	31	62	21,2	2,6	6,4	0,54	1,35
10-N3	Cd	53	106	0,45	5,8	21,2	0,026	0,095
10-N1	Cr	48	96	395	1,6	8,3	6,4	32,74
10-N2	Cr	40	80	311	2,0	6,1	6,07	19,01
10-N3	Cr	87	174	124	2,5	9,0	3,09	11,11
10-N1	Cu	45	90	599	1,3	4,2	7,7	24,91
10-N2	Cu	40	80	784	1,4	5,0	10,94	39,16
10-N3	Cu	90	184	79	3,3	12,0	2,61	9,41
10-N1	Ni	46	92	206	2,0	5,9	4,02	12,2
10-N2	Ni	39	78	169	2,0	6,0	3,47	10,18
10-N3	Ni	84	168	32	3,7	23,5	1,17	7,52
10-N1	Pb	41	82	261	1,6	5,8	4,04	15,2
10-N2	Pb	39	78	235	2,5	6,4	5,79	15,06
10-N3	Pb	87	174	201	3,3	7,1	6,61	14,2
10-N1	Zn	45	90	248	1,5	6,9	35,89	170,4
10-N2	Zn	39	78	2113	2,0	6,0	41,4	127,52
10-N3	Zn	89	178	203	2,5	6,4	5,13	12,93

N_{lab} is the number of laboratories after elimination of outliers;

N_{res} is the number of results;

w_{E} is the content of element E in the sample, in milligrams per kilogram (mg/kg) dry matter;

V_r is the relative repeatability standard deviation, in percent (%);

V_R is the relative reproducibility standard deviation, in percent (%);

S_r is the repeatability limit, in milligrams per kilogram (mg/kg);

S_R is the reproducibility limit, in milligrams per kilogram (mg/kg).

Table A.3 — Repeatability and precision results of 11th BAM PT-scheme 2005 for the determination of trace elements in aqua regia extracts of soil by ICP-AES, calculated in accordance with ISO 5725-2

Sample	Element	N_{lab}	N_{res}	w_{E}	V_r	V_R	S_r	S_R
11-N1	As	62	124	5,9	4,3	27,7	0,25	1,62
11-N2	As	27	54	3,9	4,0	14,8	0,156	0,574
11-N3	As	38	74	2,7	5,7	25,3	0,156	0,689
11-N1	Cd	90	180	12,1	2,0	6,7	0,24	0,81
11-N2	Cd	47	94	8,0	2,1	6,6	0,166	0,522
11-N3	Cd	43	86	6,3	2,1	9,9	0,135	0,624
11-N1	Cr	96	192	899	1,5	6,4	13,09	57,53
11-N2	Cr	51	102	461	2,1	6,0	9,88	27,86
11-N3	Cr	46	92	253	2,9	8,2	7,38	20,77
11-N1	Cu	96	192	271	1,7	5,4	4,67	14,72
11-N2	Cu	49	98	144	1,8	5,9	2,59	8,56
11-N3	Cu	46	92	87	2,8	7,3	2,43	6,29
11-N1	Mn	100	200	83	2,4	9,3	1,97	7,73
11-N2	Mn	53	106	54	2,7	10,6	1,43	5,72
11-N3	Mn	45	90	43	2,9	18,0	1,21	7,64
11-N1	Ni	93	196	43,0	1,8	7,1	0,77	3,07
11-N2	Ni	51	102	25,3	2,7	8,3	0,67	2,09
11-N3	Ni	44	88	17,4	2,4	8,8	0,416	1,52
11-N1	Pb	94	188	227	1,8	7,7	4,03	17,56
11-N2	Pb	48	96	149	2,3	7,9	3,36	11,79
11-N3	Pb	46	92	104	3,0	9,0	3,18	9,42
11-N1	Zn	97	194	690	1,8	6,5	12,06	44,91
11-N2	Zn	50	100	419	2,0	6,5	8,27	27,15
11-N3	Zn	46	92	273	2,4	9,5	6,64	25,81

N_{lab} is the number of laboratories after elimination of outliers;

N_{res} is the number of results;

w_{E} is the content of element E in the sample, in milligrams per kilogram (mg/kg) dry matter;

V_r is the relative repeatability standard deviation, in percent (%);

V_R is the relative reproducibility standard deviation, in percent (%);

S_r is the repeatability limit, in milligrams per kilogram (mg/kg);

S_R is the reproducibility limit, in milligrams per kilogram (mg/kg).

Annex B
(informative)

Interferences

Interference data are given in Tables B.1 to B.3.

To obtain the data for Tables B.1 and B.2, a Perkin-Elmer Optima 3000 instrument with a spectral resolution of 0,006 nm at 200 nm was employed for the study. Line coincidences, which are dependent on the spectral resolution of the spectrometer, only become perceptible when the concentration of interfering element and analyte reach a critical level.

The data given in Table B.3 as analyte concentration equivalents were obtained on a Varian Vista-PRO instrument under standard conditions with axial plasma. Each laboratory should determine these figures, as necessary, for their specific application of the method, since interferences are affected by background correction choice and are highly dependent on operating conditions and plasma position.

Table B.1 — Spectral interferences

Element	λ nm	Spectral overlap		Background shift		Low resolution: potential interference	
		Interfering element	Description of interference	Interfering element	Description of interference	Interfering element	Description of interference
As	193,696			Al	Sloping BG [a] shift	Fe	Line at 193,663 nm
				V	Strong BG due to line at 193,68 nm		
				Ca, Mg, Ti	Strong BG		
				Cr, Fe, Mn, Mo	BG		
As	197,197			Al, Ca, Mg	Strong BG		
				Cr, Cu, Fe, Mo, Ti, V	BG		
Cd	214,438	Fe	Line coincidence with Fe 214,445 nm	Ca, Cr, Cu, Ni	Low BG	As	Line at 214,410 nm
						Fe	Lines at 214,390 nm and 214,519 nm
						Mo	Line at 214,407 nm
						Ti	Lines at 214,361 nm and 214,390 nm
						V	Line at 214,371 nm
Cd	226,502	Fe	Line coincidence with Fe 226,505 nm	Ni	BG due to lines at 226,446 nm and 226,535 nm nearby	Fe	Lines at 226,439 nm, 226,459 nm and 226,599 nm
		Ti	Partial line overlap with Ti 226,514 nm	Al, As, Ca	Low BG	Mo	Line at 226,474 nm

Table B.1 (*continued*)

Element	λ nm	Spectral overlap		Background shift		Low resolution: potential interference	
		Interfering element	**Description of interference**	**Interfering element**	**Description of interference**	**Interfering element**	**Description of interference**
Cd	228,802	As	Partial line overlap with As 228,812 nm	Ni	BG due to lines at 228,765 nm and 228,839 nm	Fe	Line at 228,763 nm
				Ca, Mg, Ti, V	BG		
Co	228,616	Ni	Line coincidence with Ni[b]	Al, Ca, Cu, Fe, Mg, Mn, Ti, V	BG		
		Ti	Line coincidence with Ti 228,618 nm	Cr	BG due to lines nearby		
Co	230,786	Ni	Partial line overlap with Ni 230,779 nm	Al, Ca, Cr, Cu, Mg, Mn, Ti, V	BG	Cr	Line at 230,72 nm
				Fe	Sloping background shift due to line at 230,731 nm	Ni	Line at 230,817 nm
Co	238,892	Fe	Partial line overlap with Fe 238,863 nm	Ca, Cr, Cu, Mg, Mn, Ti	Low BG		
		Ni	Line coincidence with Ni 238,892 nm				
		V	Line coincidence with V 238,892 nm				
Cr	205,552	Mo	Partial line overlap with Mo 205,567 nm	Fe	BG due to line at 205,529 nm		
		Ni	Partial line overlap with Ni 205,550 nm	Al, Ca, Mg, Mn, Ti, V	BG		
Cr	206,149	Zn	Partial line overlap with Zn 206,200 nm	Al, Ca, Fe, Mg, Ti, V	Low BG		
Cr	267,716	Mn	Partial line overlap with Mn 267,725 nm	Mo	BG and wing overlap due to line at 267,648 nm	Fe	Lines at 267,611 nm, 267,688 nm and 267,805 nm
				V	sloping background shift due to line at 267,780 nm		
				Al, Ca, Fe, Mg, Ni, Ti	BG		
Cu	224,700	Fe	Line coincidence with Fe 224,691 nm	Al, Ca, Mg, Mn, Ni, V	BG	Fe	Lines at 224,746 nm and 224,769 nm
		Ti	Line overlap[b]			Ni	Line at 224,723 nm nearby[b]

Table B.1 (*continued*)

Element	λ nm	Spectral overlap		Background shift		Low resolution: potential interference	
		Interfering element	**Description of interference**	**Interfering element**	**Description of interference**	**Interfering element**	**Description of interference**
Cu	324,754			Ca, Fe, Mn, Ti, V	BG	Fe	Wing overlap with lines at 324,696 nm, 324,717 nm and 324,739 nm; line at 324,821 nm
						Cr	Wing overlap with line at 324,727 nm (at 500 mg/l)
						V	Line at about 324,72 nm [b]
Cu	327,396			Al, Cr, Mg, Mn, Ti, V	BG	Ca	Wing overlap with line at 327,396 nm (at 500 mg/l)
						Fe	Line at 327,445 nm nearby
						Ti	Wing overlap with line at 327,405 nm; line at 327,529 nm (at 500 mg/l)
						V	Lines at about 327,42 nm and 327,44 nm [b]
Mn	257,610			Co, Fe	Low BG	Al	Line at 257,510 nm
						Cr	Line at 257,580 nm
						Fe	Line at 257,574 nm
						V	Line at 257,729 nm
Mn	260,568	Co	Line coincidence with Co 260,568 nm	Fe	BG	Co	Line at 260,612 nm
		Cr	Line coincidence with Cr 260,561 nm			Fe	Lines at 260,565 nm and 260,542 nm
		Mo	Partial line overlap with Mo 260,593 nm			Ti	Line at 260,515 nm

Table B.1 (*continued*)

Element	λ nm	Spectral overlap		Background shift		Low resolution: potential interference	
		Interfering element	**Description of interference**	**Interfering element**	**Description of interference**	**Interfering element**	**Description of interference**
Mn	279,482	Mg	Line coincidence with Mg 279,553 nm	Ca	Sloping background shift due to line at about 279,55 nm [b]	Mo	Lines at about 279,53 nm [b] and 279,55 nm [b]
		Mo	Partial line overlap with Mo 279,457 nm	Co, Cr, Mo, Ti	BG	V	Line at 279,430 nm
		V	Line coincidence with V 279,483 nm	Fe	BG due to lines at 279,470 nm, 279,500 nm and 279,554 nm		
Mn	294,92	Cr	Partial line overlap with Cr 294,944 nm	Cr, Mg, Mo, Ti	BG	V	Line at 294,963 nm
		Fe	Line coincidence with Fe 294,921 nm				
		Mo	Partial line overlap with Mo line [b]				
		V	Line coincidence with V 294,917 nm				
Mn	403,075	Cr	Partial line overlap with Cr 403,068 nm	Al, Ca, V	Small BG	Cr	Line at 403,113 nm nearby
		Fe	Partial line overlap with Fe 403,049 nm			Mo	Line at 402,994 nm
		Mo	Partial line overlap with Mo line (?)			V	Line at 403,122 nm nearby
		Ti	Partial line overlap with Ti 403,051 nm				
Ni	221,647			Fe	BG due to lines at 221,706 nm and about 221,6 nm [b]	V	Line at 221,603 nm
				Al, Ca, Cr, Cu, Mg, Mn, Ti, V	BG		
Ni	231,604			Fe	BG due to lines at 231,485 nm and 231,738 nm	V	Line at 231,563 nm
				Al, Ca, Cu, Mg, Mn, Ti, V	BG		

Table B.1 (*continued*)

Element	λ nm	Spectral overlap		Background shift		Low resolution: potential interference	
		Interfering element	**Description of interference**	**Interfering element**	**Description of interference**	**Interfering element**	**Description of interference**
Ni	232,003	Cr	Line coincidence with Cr 232,008 nm	Fe	Strong BG due to lines at 232,031 nm (possible wing overlap) and 231,854 nm	Mn	Line at 232,045 nm
		V	Partial line overlap with V 232,016 nm	Al, Ca, Mg, Mn, Ti	BG		
Pb	216,999	Fe	Line coincidence with Fe 216,995 nm	Al, Ca, Co, Cr, Fe, Mg, Mn, Ti, V, Zn	Strong BG	Fe	Line at 217,019 nm
		Mo	Partial line overlap with Mo 217,02 nm (?)	Cu	Sloping background shift due to line at 216,953 nm	Mo	Line at 216,951 nm
		Ni	Partial line overlap with Ni 216,961 nm				
		V	Partial line overlap with V 216,985 nm and V 217,007 nm				
Pb	220,353	Co	Partial line overlap with Co 220,343 nm	Al	Sloping background due to line at 220,463 nm	Co	Line at 220,296 nm
		Ti	Partial line overlap with Ti 220,361 nm	Ca, Cu, Ni	Strong BG	Cr	Lines at 220,28 nm [b] and 220,391 nm
				Cr, Fe, Mg, Mn, Mo, V, Zn	Low BG		
Pb	224,688	Co	Line coincidence with Co 224,65 nm [b]	Al, Ca, Cr, Mg, Mn, Ni, Ti, V, Zn	Strong BG	Ni	Line at 224,723 nm nearby
		Cu	Line coincidence with Cu 224,700 nm				
		Fe	Line coincidence with Fe 224,691 nm				
		Mo	Line coincidence with Mo 224,695 nm				
		Ti	Partial line overlap with Ti 224,70 nm (?)				

Table B.1 (*continued*)

Element	λ nm	Spectral overlap		Background shift		Low resolution: potential interference	
		Interfering element	**Description of interference**	**Interfering element**	**Description of interference**	**Interfering element**	**Description of interference**
Pb	261,418	Co	Line coincidence with Co 261,436 nm	Al, Ni, Ti, Zn	Low BG	Al	Line at 261,39 nm [b]
		Fe	Line coincidence with Fe 261,418 nm	Ca, Cr, Cu, Mg, Mn, Mo	Strong BG	Cr	Lines at 261,350 nm, 261,457 nm and 261,463 nm nearby
						Mn	Lines at 261,359 nm and 261,404 nm
						Mo	Lines at 261,372 nm and 261,539 nm
						Ni	Line at 261,519 nm
						Ti	Line at 261,37 nm [b]
						V	Lines at 261,441 nm, 261,540 nm and about 261,40 nm [b]
Pb	283,306	Fe	Line coincidence with Fe 283,310 nm	Al, Co, Cu, Ni, Zn	Low BG	Cr	Lines at 283,246 nm, 283,279 nm and 283,339 nm
				Ca, Cr, Mg, Mn, Mo, Ti, V	Strong BG	Fe	Line at 283,244 nm
						Mo	Lines at 283,266 nm and 283,379 nm
						Ti	Line at 283,266 nm (BG)
Tl	190,801	Co	Partial line overlap with Co 190,79 nm (?)	Al, Ca	Strong BG	Mn	Line at λ > 190,83 nm (BG)
		Mo	Partial line overlap with Mo 190,806 nm	Cr, Cu, Fe, Mg, Mn, Mo, Ni, Zn	BG	V	Line at 190,768 nm
		V	Partial line overlap with V 190,779 nm	Ti	Sloping background shift due to line at about 190,76 nm (?)		

Table B.1 (*continued*)

Element	λ nm	Spectral overlap		Background shift		Low resolution: potential interference	
		Interfering element	**Description of interference**	**Interfering element**	**Description of interference**	**Interfering element**	**Description of interference**
Tl	276,787	Mn	Line coincidence with Mn 276,79 nm [b]	Ca, Co, Cu, Ti	BG	Al	Line at about 276,75 nm (?) nearby
		Ni	Partial line overlap with Ni 276,76 nm [b]	Cr	Sloping background shift due to line at 276,754 nm	Cr	Lines at 276,728 nm and 276,815 nm nearby
				Fe	Sloping background shift due to line at 276,752 nm	Mn	Line at 276,745 nm
				Mg	Sloping background shift due to lines at about 276,76 nm [b], 276,835 nm, 276,846 nm	Ti	Line at 276,820 nm nearby
				Mo	Structured background due to lines at about 276,76 nm [b] and 276,809 nm		
				V	BG due to line at 276,813 nm		
Tl	351,924	Ti	Line coincidence with Ti 351,924 nm	Ca	Strong BG	Cr	Line at 351,945 nm nearby
		V	Line coincidence with V 351,917 nm	Co	Sloping background shift due to line at 351,835 nm	Fe	Line at 351,888 nm
				Cr, Fe, Mg, Mn, Mo	BG	Mo	Line at 351,857 nm
				Ni	Strong BG due to lines at 351,863 nm and 351,977 nm		
Zn	202,548	Cr	Partial line overlap with Cr 202,559 nm	Mg	Sloping background due to line at 202,582 nm	Mo	Line at about 202,58 nm [b]
		Cu	Line coincidence with Cu 202,548 nm	Al, Ca, Fe, Mn, Mo, Ti, V	Low BG		
		Ni	Partial line overlap with Ni 202,538 nm				

Table B.1 (*continued*)

Element	λ nm	Spectral overlap		Background shift		Low resolution: potential interference	
		Interfering element	Description of interference	Interfering element	Description of interference	Interfering element	Description of interference
Zn	206,191	Cr	Partial line overlap with Zn 206,149 nm	Al, Ca, Fe, Mg, Ti, V	Low BG		
		Mo	Line coincidence with Mo line at 500 mg/l (b)				
Zn	213,856	Cu	Partial line overlap with Cu 213,853 nm	Fe	Small BG due to lines at 213,801 nm and 213,970 nm	Ti	Line at 213,837 nm
		Fe	Line coincidence with Fe 213,859 nm	Ca, Mg, Ti, V	Low BG	V	Line at 213,816 nm
		Ni	Line coincidence with Ni 213,858 nm				

[a] BG is the parallel background shift, unless stated otherwise.

[b] No line given in wavelength tables.

Table B.2 — Selected analyte concentration equivalents arising from interference at the 100 mg/l and the 500 mg/l levels

Analyte	λ nm	Interfering element	Spectral interference	Analyte concentration equivalent	
				100 mg/l interfering element	500 mg/l interfering element
Cd	214,438	Fe	Line coincidence	0,02 mg/l	0,10 mg/l
Cd	226,502	Fe	Line coincidence	0,024 mg/l	0,12 mg/l
Cd	226,502	Ti	Partial line overlap	0,03 mg/l	0,13 mg/l
Cd	228,802	As	Partial line overlap	3,27 mg/l	15,18 mg/l
Co	228,616	Ti	Line coincidence	0,18 mg/l	0,86 mg/l
Co	230,786	Ni	Partial line overlap	0,71 mg/l	3,57 mg/l
Co	238,892	Fe	Partial line overlap	0,87 mg/l	4,44 mg/l
Co	238,892	Ni	Line coincidence	0,16 mg/l	0,76 mg/l
Co	238,892	V	Line coincidence	0,06 mg/l	0,26 mg/l
Cr	205,559	Mo	Partial line overlap	0,14 mg/l	0,72 mg/l
Cr	205,559	Ni	Partial line overlap	0,21 mg/l	1,06 mg/l
Cr	267,716	Mn	Partial line overlap	0,03 mg/l	0,18 mg/l
Cu	224,700	Fe	Line coincidence	0,10 mg/l	0,53 mg/l
Cu	224,700	Ti	Line coincidence	0,03 mg/l	0,13 mg/l
Cu	324,754	Cr	Partial line overlap	0,002 mg/l	0,02 mg/l
Cu	327,396	Ti	Partial line overlap	0,05 mg/l	0,25 mg/l
Mn	260,568	Co	Line coincidence	0,10 mg/l	0,45 mg/l
Mn	260,568	Cr	Line coincidence	0,02 mg/l	0,05 mg/l
Mn	260,568	Mo	Partial line overlap	0,03 mg/l	0,12 mg/l
Mn	279,482	Mg	Line coincidence	9,36 mg/l	38,56 mg/l
Mn	279,482	Mo	Partial line overlap	0,04 mg/l	0,14 mg/l
Mn	279,482	V	Line coincidence	0,25 mg/l	1,20 mg/l
Mn	294,920	Cr	Partial line overlap	0,06 mg/l	0,26 mg/l
Mn	294,920	Fe	Line coincidence	0,06 mg/l	0,23 mg/l
Mn	294,920	Mo	Partial line overlap	0,02 mg/l	0,07 mg/l
Mn	294,920	V	Line coincidence	0,57 mg/l	2,72 mg/l
Mn	403,075	Cr	Partial line overlap	0,11 mg/l	0,53 mg/l
Mn	403,075	Fe	Partial line overlap	0,011 mg/l	0,53 mg/l
Mn	403,075	Mo	Partial line overlap	0,02 mg/l	0,06 mg/l

Table B.2 (*continued*)

Analyte	λ nm	Interfering element	Spectral interference	Analyte concentration equivalent	
				100 mg/l interfering element	500 mg/l interfering element
Mn	403,075	Ti	Partial line overlap	0,73 mg/l	3,54 mg/l
Ni	232,003	Cr	Line coincidence	2,77 mg/l	13,87 mg/l
Ni	232,003	V	Partial line overlap	0,12 mg/l	0,48 mg/l
Pb	216,999	Fe	Line coincidence	0,25 mg/l	1,19 mg/l
Pb	216,999	Mo	Partial line overlap	1,77 mg/l	8,81 mg/l
Pb	216,999	Ni	Partial line overlap	0,21 mg/l	1,01 mg/l
Pb	216,999	V	Partial line overlap	0,36 mg/l	1,62 mg/l
Pb	220,353	Co	Partial line overlap	0,26 mg/l	1,01 mg/l
Pb	220,353	Ti	Partial line overlap	0,17 mg/l	0,39 mg/l
Pb	224,688	Co	Line coincidence	0,31 mg/l	1,30 mg/l
Pb	224,688	Cu	Line coincidence	5903 mg/l	28980 mg/l
Pb	224,688	Fe	Line coincidence	6,28 mg/l	32,75 mg/l
Pb	224,688	Mo	Line coincidence	7,04 mg/l	35,58 mg/l
Pb	224,688	Ti	Partial line overlap	1,50 mg/l	7,39 mg/l
Pb	261,418	Co	Line coincidence	23,64 mg/l	117,84 mg/l
Pb	261,418	Fe	Line coincidence	5,06 mg/l	25,32 mg/l
Pb	283,306	Fe	Line coincidence	0,18 mg/l	1,32 mg/l
Tl	190,801	Co	Partial line overlap	0,76 mg/l	3,83 mg/l
Tl	190,801	Mo	Partial line overlap	0,81 mg/l	4,05 mg/l
Tl	190,801	V	Partial line overlap	0,40 mg/l	1,94 mg/l
Tl	276,787	Mn	Line coincidence	1,73 mg/l	8,37 mg/l
Tl	276,787	Ni	Partial line overlap	0,02 mg/l	0,06 mg/l
Tl	351,924	Ti	Line coincidence	0,19 mg/l	1,06 mg/l
Tl	351,924	V	Line coincidence	0,29 mg/l	1,50 mg/l
Zn	202,548	Cr	Partial line overlap	1,00 mg/l	5,11 mg/l
Zn	202,548	Cu	Line coincidence	1,08 mg/l	5,43 mg/l
Zn	202,548	Ni	Partial line overlap	0,18 mg/l	0,90 mg/l
Zn	213,856	Cu	Partial line overlap	0,43 mg/l	2,18 mg/l
Zn	213,856	Ni	Line coincidence	0,36 mg/l	1,83 mg/l

Table B.3 — Potential interferences and analyte concentration equivalents (mg/l) arising from interference at several mg/l levels for axial plasma (Varian Vista-PRO™)

Analyte	λ nm	**LOQ** [a] mg/l	**Al** 1 000 mg/l	**Ca** 1 000 mg/l	**Cr** 200 mg/l	**Cu** 200 mg/l	**Fe** 1 000 mg/l	**Mg** 1 000 mg/l	**Mn** 200 mg/l	**Ti** 200 mg/l	**V** 200 mg/l
Aluminium	257,509	2,0	n.a.	—	—	—	—	—	—	—	—
Antimony	206,834	0,5	—	—	2,5	—	—	—	—	—	—
Arsenic	188,980	0,5	—	—	—	—	—	—	—	—	—
Barium	455,403	0,02	—	—	—	—	—	—	—	—	—
Beryllium	313,042	0,02	—	—	—	—	—	—	—	—	0,02
Cadmium	214,439	0,005	—	—	—	—	—	—	—	—	—
Calcium	315,887	5	—	n.a.	—	—	—	—	—	—	—
Chromium	267,716	0,02	—	—	n.a.	—	0,06	—	0,02	—	—
Cobalt	238,892	0,02	—	—	—	—	—	—	—	—	0,05
Copper	324,754	0,02	—	—	—	n.a.	—	—	—	—	—
Iron	238,204	1	—	—	—	—	n.a.	—	—	—	—
Lead	220,353	0,02	—	—	—	—	—	—	0,03	—	—
Magnesium	279,553	1	—	—	—	—	—	n.a.	—	—	—
Manganese	257,610	0,2	—	—	—	—	—	—	n.a.	—	—
Molybdenum	202,032	0,02	—	—	—	—	—	—	—	—	0,06
Nickel	231,604	0,02	—	—	—	—	—	—	—	—	—
Phosphorus	213,618	0,5	—	—	—	—	—	—	—	—	—
Potassium	766,491	1	—	—	—	—	—	—	—	—	—
Sodium	589,592	0,5	—	—	—	—	—	—	—	—	1,4
Selenium	196,026	0,5	—	—	—	—	—	—	—	—	—
Strontium	407,771	0,02	—	0,03	—	—	—	—	—	—	—
Thallium	190,794	0,1	—	—	—	—	—	—	0,3	—	0,3
Titanium	336,122	0,02	—	—	—	0,04	—	—	—	n.a.	—
Vanadium	292,401	0,02	—	—	—	—	—	—	—	0,21	n.a.
Zinc	213,857	0,05	0,08	—	—	0,12	—	—	—	—	—

n.a. not applicable.

[a] LOQ = limit of quantification.

Dashes indicate that no interference was observed, even when interferents were introduced at the following levels:

Al at 1 000 mg/l, Cu at 200 mg/l, Mn at 200 mg/l

Ca at 1 000 mg/l, Fe at 1 000 mg/l, Ti at 200 mg/l

Cr at 200 mg/l, Mg at 1 000 mg/l, V at 200 mg/l

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