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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)

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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*.

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.