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## **Sludge, treated biowaste and soil — Trace elements — Method by inductively coupled plasma - Optical emission Spectrometry (ICP-OES)**

*Schlamm, behandelte Bio-Abfälle und boden — Spuren Elementen — Methode mit induktiv gekoppeltes Plasma mit Atomare Emission Spectrometer (ICP-AES)*

*Boue, bio déchet traité et sol — Éléments trace — Méthode de spectromètre d'émission optique avec plasma induit par haute fréquence (ICP-OES)*

ICS:

Descriptors:

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## Foreword

This document (BT/TF151 WI CSS99026) has been prepared by Technical Committee CEN/BT TF 151 "Horizontal standards in the field of Sludge, Biowaste and Soil", the secretariat of which is held by Danish Standards.

This standard is applicable and validated for several types of matrices. The table below indicates which ones.

Material	Validated	Document
Soil		[reference]
Sludge		
Biowaste		
Soil improvers		

## Introduction

This document is developed in the framework of the project 'Horizontal'. It is the result of a desk study "Determination of elements by ICP-OES and ICP-MS" in Phase I and "Trace elements with ICP-OES: ruggedness test" in Phase II and aims at evaluation of the latest developments in assessing measurement of trace elements in sludge, soil, treated biowaste and neighbouring fields. After an evaluation study, in which e.g. the ruggedness of the method was studied, a European wide validation of the draft standard has taken place. The results of the desk studies as well as the evaluation and validation studies have been subject to discussions with all parties concerned in CEN. The standard is part of a modular horizontal approach in which the standard belongs to the analytical step.

Until now test methods determining properties of materials were often prepared in Technical Committees (TCs) working on specific products or specific sectors. In those test methods often steps as sampling, extraction, release or other processing, analyses, etc were included. In this approach it was necessary to develop, edit and validate similar procedural steps over and over again for every material or product. Consequently this has resulted in duplication of work. To avoid such duplication of work for parts of a testing procedure references to parts of test methods from other TCs were introduced. However the following problems are often encountered while using references in this way: 1) The referenced parts are often not edited in a way that they could easily be referred to, 2) the referenced parts are often not validated for the other type of material and 3) the updates of such test standards on products might lead to inadequate references.

In the growing amount of product and sector oriented test methods it was recognised that many steps in test procedures are or could be used in test procedures for many products, materials and sectors. It was supposed that, by careful determination of these steps and selection of specific questions within these steps, elements of the test procedure could be described in a way that can be used for all materials and products or for all materials and products with certain specifications.

Based on this hypothesis a horizontal modular approach is being investigated and developed in the project 'Horizontal'. 'Horizontal' means that the methods can be used for a wide range of materials and products with certain properties. 'Modular' means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole "chain of measurement" (from sampling to analyses). **A beneficial feature of this approach is that "modules" can be replaced by better ones without jeopardizing the standard "chain".**

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the test procedure.

The modules that relates to this standard are specified in section XX Normative references.

An overview of modules and the manner, in which modules are selected will be worked out later, at which time proper reference in this standard will be provided.

## 1 Scope

This European Standard specifies the method for the determination of 44 elements in *aqua regia* and nitric acid digests of treated bio waste, soil, sludge and sediment:

aluminium, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, calcium, cerium, chromium, cobalt, copper, iron, lanthanum, lead, lithium, magnesium, manganese, mercury, molybdenum, neodymium, nickel, phosphorus, potassium, praseodymium, samarium, scandium, selenium, silicon, silver, sodium, strontium, sulphur, tellurium, thallium, thorium, tin, titanium, tungsten, uranium, vanadium, zinc and zirconium.

Table A.1 list elements for which this method applies along with the recommended wavelength and typical instrumental detection limits for clean matrices.

## 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.

EN ISO 3696: 1997, *Water for analytical laboratory use — Specification and test methods*.

<Aqua regia horizontal standard>, *Digestion for the determination of selected elements — Part 1: Aqua regia digestion*.

<Nitric acid horizontal standard>, *Digestion for the determination of selected elements — Part 2: Nitric acid digestion*.

## 3 Terms and definitions

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

### 3.1

#### **Instrumental limit of detection (IDL)**

The smallest amount or concentration of an analyte in the test sample that can be reliably distinguished from zero. The limit of detection shall be calculated as:

$$X_{LD} = 3 \cdot S_0$$

Where:

$X_{LD}$  limit of detection

$S_0$  is the standard deviation of the outlier free results of at least 3 measurements of a reagent blank so

## 4 Symbols and abbreviations

ICS Interference check solution

IEC Interelement correction factor

## 5 Principle

This method describes multi-elemental determinations by ICP-OES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Additional interferences and matrix effects must be recognized and appropriate corrections made; tests for their presence are described.

Alternatively, users may choose multivariate calibration methods (e.g. Multicomponent Spectral Fitting). In this case, point selections for background correction are superfluous since whole spectral regions are processed.

## 6 Interferences

*Spectral interferences* are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

*Background emission* and *stray light* can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

*Spectral overlaps* may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction **require** the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. The interferences are listed in Table A.1.

*Physical interferences* are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using an internal standard or a high solids nebulizer. They can also be minimized by matrix matching particularly by matching the acid concentration.

*Chemical interferences* include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (Rf power, observation position, gas flow rate and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

*Memory interferences* result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition in the uptake tubing or to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The occurrence memory effects depends on the element and can be minimized by flushing the system

with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis during method development.

## 7 Reagents

### 7.1 General

For the determination of elements at trace and ultratrace level, the reagents shall be of adequate purity. The concentration of the analyte or interfering substances in the reagents and the water should be negligible compared to the lowest concentration to be determined.

Use water according to Grade 1 of ISO 3696 for all sample preparation and dilutions.

### 7.2 Nitric acid, $c(\text{HNO}_3) = 16 \text{ mol/l}$ , $\rho \sim 1,4 \text{ g/ml}$ .

NOTE Nitric acid is available both as  $c(\text{HNO}_3) = 1,40 \text{ g/ml}$  ( $w(\text{HNO}_3) = 650 \text{ g/kg}$ ) and  $c(\text{HNO}_3) = 1,42 \text{ g/ml}$  ( $w(\text{HNO}_3) = 690 \text{ g/kg}$ ). Both are suitable for use in this method provided there is minimal content of the interesting analytes.

### 7.3 Hydrochloric acid, $c(\text{HCl}) = 16 \text{ mol/l}$ , $\rho \sim 1,18 \text{ g/ml}$ .

### 7.4 Element stock solutions

Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cu, Fe, Hg, K, La, Li, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, Pr, Si, Sb, Sc, Se, Sm, Sn, Sr, Te, Ti, Th, Tl, U, V, W, Zn, Zr,  $\rho = 1\ 000 \text{ mg/l}$  each.

Both single-element stock solutions and multi-element stock solutions with adequate specification stating the acid used and the preparation technique are commercially available. Single element solutions can be made from high purity metals.

These solutions are considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered.

### 7.5 Anion stock solutions

Cl, P- $\text{PO}_4$ , S- $\text{SO}_4$   $\rho = 1\ 000 \text{ mg/l}$  each.

Prepare these solutions from the respective acids. The solutions are as well commercially available.

These solutions are considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered.

### 7.6 Multi-element calibration solutions

Depending on the scope, different multi-element standard solutions may be necessary. In general, when combining multi-element standard solutions, their chemical compatibility and the possible hydrolysis of the components shall be regarded. Care must be taken to prevent chemical reactions.

NOTE In multi element standards precipitation of Ag, Ba or Pb can occur; Ag is only stable in high hydrochloric acid concentrations or nitric acid.

The multi-element standard solutions are considered to be stable for several months, if stored in the dark. This does not apply to multi-element standard solutions that are prone to hydrolysis, in particular solutions of Bi, Mo, Sn, Sb, Te, W, and Zr.

Multi-element standard solution A may contain the following elements: Ag, Al, As, B, Ba, Be, Bi, Cd, Ce, Co, Cr, Cu, La, Li, Mn, Ni, Pb, Se, Sr, Tl, V, Zn. Use nitric acid for stabilization of standard solution A.

Multi-element standard solution B may contain the following elements: Mo, Sb, Sn, W, Zr. Standard solution B needs hydrochloric acid for stabilization

Multi-element standard solutions with more elements are allowed provided that these solutions are stable.

Prepare in one or more steps calibration solutions at the highest concentration of interest. If more concentration levels are needed prepare those similarly in an equidistant concentration range.

Add acids (7.3 – 7.4) to match the acid concentration of samples closely.

If traceability of the values is not established check the validity by comparison with a (traceable) independent standard.

Check the stability of the calibration solutions.

### **7.7 Calibration blank**

Prepare the calibration blank by acidifying water (7.2) to the same concentration of the acids used in the calibration solutions and samples.

### **7.8 Method blank**

The method blank must contain all of the reagents in the same concentrations as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

### **7.9 Calibration check solution**

Prepare the calibration check solution by acidifying water (7.2) to the same concentration of the acids used in the calibration solutions, using the same standards used for calibration at an upper concentration level.

### **7.10 Internal standard solution**

The use of internal standard can be a suitable method to correct for non spectral interferences. The approach involves the addition of a known amount of a substance to the sample and calibration solutions. The ratio of responses the determinant and the internal standard are measured in the sample and calibration solution. The observation for the internal standard is used to relate the determinant signal to the determinant concentration.

The choice of elements for the internal standard solution depends on the analytical problem. The solution of this/these internal standard(s) should cover the mass range of interest. The concentrations of the selected elements (used as internal standard) should be negligibly low in the digests of samples. The elements Sc, Y, In and Rh have been found suitable for this purpose.

Generally, a suitable concentration of the internal standard in samples and calibration solutions is 1 mg/l to 10 mg/l.

### **7.11 Interference check solution (ICS)**

If interference cannot be excluded (see Table A.1) prepare the interference check solution to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. The choice of the concentration and interfering element are matrix dependent.



Avoid two or more interferences for an analyte in the same interference check solution. Spike the sample with the analytes of interest, particularly those with known interferences at 0.5 to 1 mg/L.

In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

## **8 Apparatus**

### **8.1 Inductively coupled argon plasma emission spectrometer**

- Computer-controlled emission spectrometer with background correction.
- Radio-frequency generator
- Optional mass flow controller for argon nebulizer gas supply.
- Optional peristaltic pump.
- Optional auto sampler.
- Argon gas supply - high purity (e.g.  $\geq 99.95\%$ ).

**NOTE** The instruments for ICP-OES come in two different varieties i.e. axial or radial viewing or a combination of both. Axial viewing gives more signal intensity due to the increased observation path length of the normal analytical zone of the plasma. This will in most cases lead to a higher signal but an increase of interference relative to radial viewing is commonly observed for many elements. Most instruments however have special adaptations to overcome the excessive interference by radiation from the bullet zone and molecular region of the plasma. Especially for low UV (e.g. Pb, Zn, As, Cd, P, Se and S) the Signal to Background Ratio's are more or less the same for both types. Several instruments are equipped with dual view by using additional mirrors. This allows the choice by the operator to select the most suitable option but can give limitations use due to the lower light intensity and because of different torch design (longer neck), lead to faster blockage and decreased long term stability. For environmental samples axial viewing can be advantageous.

**8.2 Volumetric flasks**, of suitable precision and accuracy.

**8.3 Volumetric pipets**, of suitable precision and accuracy.

**NOTE** Dispensing of a volume of less than 50  $\mu\text{l}$  by means of a pipet should be avoided.

## **9 Procedure**

### **9.1 Test sample**

The test sample is particle free digest obtained by CSS99025A (Horizontal standard nitric acid digestion) or B (*Horizontal standard aqua regia digestion*).

### **9.2 Test portion**

The test portion may be directly obtained for the test sample or may be diluted from the test sample to accommodate the measurement range or to dilute the matrix.

The acidity of the test portion must match the acidity of calibration solutions.

### 9.3 Set up of the procedure

#### 9.3.1 General requirements

Adjust the instrumental parameters of the ICP-OES system in accordance with the manufacturer's manual. Develop a method (set of instrument parameters) depending on the type of samples and matrices to be measured.

Define the wavelengths of the analytes of interest and the need for corresponding corrections according to Table A.1. Alternatively, apply multivariate calibration procedures.

Define the rinsing times depending on the length of the flow path; The time for rinsing the sample introduction system between the subsequent samples must be long enough to rinse all the analytes of interest from the system.

A guideline for method development and instrument set up is given in [1].

NOTE ICP-OES has excellent multi-element capability. Nevertheless it does not mean that all elements can be analyzed during one measurement run. The sensitivity of determination depends on numerous parameters (nebulizer flow, radio-frequency power, viewing height etc.). The optimal instrument settings can not be reached for all elements at once.

#### 9.3.2 Inter-element correction

Investigate whether the interfering elements in Table A.1 may results in measured values higher than three times the instrumental limit of detection or 0,5 times the lowest concentration to be reported. If so, correct for interference. See Annex C for determining the IEC factors.

#### 9.3.3 Internal standard

If the type of samples to be measured strongly varies, matrix matching may not be possible. Investigate the application of an internal standard to reduce the effect of the mismatch on analyte sensitivity (6). The internal standard method can be applied independently from the calibration procedure.

If the internal standard procedure insufficiently reduces matrix effects, apply standard addition calibration (9.4.3).

The use of an internal standard is recommended. Add the internal standard solution (7.9) to all solutions to be measured. On-line dilution and mixing of the sample flow with internal standard solution by means of the peristaltic pump of the nebulizer is common practice. In such case the calibration solutions are diluted the same way as the sample solutions.

The mass concentration of the reference-elements shall be the same in all solutions. Divide the response of the analyte by the response of the internal standard in the calibration solution(s) and use this ratio in the calibration function.

#### 9.3.4 Instrument performance check

About 30 min prior to measurement, adjust the instrument to working condition.

Before each series of measurement check the sensitivity and the stability of the system and minimize interference e.g. by using the optimization solution (7.12).

Check the resolution and the mass calibration as often as required by the manufacturer.

Check the wavelength calibration as often as required by the manufacturer.

## 9.4 Calibration

### 9.4.1 Linear calibration function

If more than two concentration levels, including zero, are used apply weighted linear regression to obtain the linear calibration function.

**NOTE** ICP-OES provide a large measurement range. The dispersion of blank measurements is usually much smaller than the dispersion at full scale. Ordinary linear regression assumes that the dispersion is constant over the entire range. As a consequence a much higher percentage of the calculated intercepts is out of the range expected from the spread of blanks: a non-zero blank value is calculated that is actually not there. Weighted linear regression forces the line through points of low dispersion, resulting in the expected intercept dispersion. Unfortunately, many data systems cannot handle weighted regression. An alternative, but less efficient, approach is ordinary linear regression where the line is forced through the blank value or through zero. The most inexpensive way however is a (single or multiple) two point calibration. If calibration lines are linear, as they are usually in ICP-OES, this procedure is valid and efficient.

If weighted linear regression is not possible apply linear regression forced through the blank value or through zero. In the latter case check regularly by running a blank sample whether the assumption on the absence of a blank value is justified.

A two point calibration is allowed if the calibration function is linear, which usually is the case. Check regularly with a calibration solution of known dilution whether the assumption on linearity is justified.

Instead of one measurement per level more measurements can be performed to reduce the uncertainty of the calibration line.

### 9.4.2 Non-linear calibration function

Alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects.

Calculate the calibration function from weighted polynomial regression (second degree).

If this option is not possible use polynomial regression under the condition that the function is forced through the blank or through zero. In the latter case check regularly whether the assumption on the absence of a blank value is justified.

### 9.4.3 Standard addition calibration

Add a known amount of standard solution ( $V_s$ ) of the analyte and an equal amount of blank solution ( $V_b$ ) to two separate but equal portions of the sample solution (or its dilution). Minimise dilution or correct for spike dilution. The added amount of standard solution should be between 0.4 and 2 times the expected sample mass concentration. Measure both solutions as a sample solution. Determine the 'measured spike concentration' as the difference in mass concentration between the two spiked sample portions. Use the ratio 'true spike concentration' versus 'measured spike concentration' as a correction factor for the initially measured concentration of the sample portion.

## 9.5 Sample measurement

Run one or more calibration solutions and calibration blanks.

Run the interference check solution(s) to establish interference correction or to check presence of interference.

Run all samples including one or more method blanks.

Every 25 samples or less and at the end, run a calibration blank and a calibration check solution.

Run at least one spiked sample (post digest) from the series to check recovery.

NOTE 1 If standard addition calibration is applied to all samples leave out the recovery check.

Whenever a new or usual sample matrix is encountered check:

- matrix effects by running the spike sample or
- matrix effects by running a fivefold diluted sample and
- inter-element interference analysing at a different wavelength.

NOTE 2 The analyte concentrations after dilution must at least be higher than twice the lowest concentration to be reported.

## 10 Calculation

Calculate the element concentration in the digested solid sample:

$$w = (\rho_1 - \rho_0)f_a V \cdot 100 / (m \cdot dw)$$

where:

$\rho_1$  is the concentration of the element in the test sample in mg/l;

$\rho_0$  is the concentration of the element in the blank in mg/l;

$f_d$  is the dilution factor due to digestion of an aqueous sample;

in all other cases  $f_d = 1$ ;

$f_a$  is the dilution factor of the test portion;

$w$  is the mass fraction of the element in the solid sample in mg/kg;

$V$  is the volume of the test sample (digest) in litres;

$m$  is the mass of the digested sample in kg.

$dw$  is the dry weight percentage of the sample.

## 11 Expression of results

State as many significant figures as are acceptable according to the precision of the measuring values, but not more than three significant figures.

## 12 Performance characteristics

The performance of this analytical method has been verified in connection with the digestion methods for soil, sludge and treated biowaste using nitric acid and aqua regia. A separate validation of digests has not been carried out apart from the within lab variability in the ruggedness phase. The reproducibility is generally a factor 2 – 2.5 larger than the repeatability.

### 12.1 Blank

Result of the calibration blank check shall be within 3 times the instrumental limit of detection.

### 12.2 Calibration check

The result of the calibration check solution shall not deviate more than 10% from the previous measurement or within 10% of the theoretical concentration. Recalibrate the instrument in case of exceeding of this limit.

### 12.3 Interference

Magnitude of uncorrected background and spectral interference shall not be higher than three times the instrumental detection limit or 0.5 times the lowest concentration to be reported.

Successive values of a correction factor shall not differ more than 20%.

### 12.4 Recovery

Post digestion spike recovery shall be between 75% and 125% or difference between results for the original sample and the fivefold-diluted sample shall be less than 10% when the concentration after dilution is higher than ten times the instrumental detection limit or higher than twice the lowest concentration to be reported. Spike concentration should be within 0.4 and 2 times the analyte concentration. If the recovery is not within these limits, a matrix effect should be suspected.

### 12.5 Precision

The performance characteristics of the method data have been evaluated based on the repeatability results (Annex B) and the performance data of digests (see CSS99025 a and b). Table 2 gives typical values for repeatability and reproducibility limits that can be expected taking into consideration the different sensitivities of the different elements.

**Table 2 — Typical values and observed ranges of the repeatability and reproducibility limits**

The reproducibility limit provides a determination of the differences (positive and negative) that can be found (with a 95 % statistical confidence) between a single test result obtained by a laboratory using its own facilities and another test result obtained by another laboratory using its own facilities, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard and the two laboratory samples are obtained from the same primary field sample and prepared under identical procedures. Conversely, the repeatability limit refers to measurements obtained from the same laboratory, all other conditions being identical. The reproducibility limit and the repeatability limit do not cover sampling but cover all activities carried out on the laboratory sample including its preparation from the primary sample.

Results of the validation of the determination of elements in aqua regia extract from, sludge by Inductively coupled plasma – mass spectrometry (ICP MS) method	Typical value %	Observed range %
Repeatability limit, r	5	3 - 10
Reproducibility limit, R	10	6 - 20

NOTE 1. The above results refer to the difference that may be found between two test results performed on two laboratory samples obtained under the same conditions. In the case when reference is made to the dispersion of the values that could reasonably be attributed to the parameter being measured, the above typical reproducibility values and observed reproducibility ranges should be divided by  $\sqrt{2}$  to obtain the corresponding typical dispersion limit and its observed range.

NOTE 2. A limited number of materials and parameters were tested. Consequently, for other materials and parameters, performance characteristics may fall outside the limits as derived from the validation of the determination of elements in aqua regia digest of sludge by Inductively coupled plasma with optical emissions spectrometry (ICP OES) method. In particular for relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Table D.2 (Annex D) and this table.

NOTE 3. The repeatability and reproducibility are at least better than the performance characteristics reported for the aqua regia and nitric acid digestion as specified in respectively BT\_TF151\_WI\_CSS99025B\_Aqua\_regia digestion and BT\_TF151\_WI\_CSS99025A\_Nitric acid digestion.

### 13 Test report

The test report shall contain the following information:

- a) a reference to this European Standard including its date of publication;
- b) sampling report including precise identification of the sample;
- c) pre-treatment method;
- d) specific reporting on the considered quantification standard;
- e) expression of results, according to 11;
- f) any deviation from this standard, and any facts which may have influenced the result. Where the test is not carried out in accordance with this standard, reference may only be made to EN xxxx in the report in case all deviations from the procedures prescribed in this standard are indicated in the report stating the reason for deviation.

**Annex A**  
(informative)

**Recommended  
wavelengths,  
spectral  
interferences  
and estimated  
instrumental  
detection limits**

**Table A.1 — Recommended wavelengths, spectral interferences and estimated instrumental detection limits (in µg/l)**

Element	Line (nm)	Interfering elements	Instrumental detection limit (µg/l)
Ag	328,068	Mn	7
	338,289	Cr,Ti	15
Al	308,215	Mn, V, Fe	50
	396,152	Mo, Cu	10
	167,08	Fe	1
As	193,696	Fe, Al, Mo	50
	197,197	Fe, Al, V	75
	189,042	Al	
B	208,959	Al, Mo	10
	249,678	Fe, Cr	5
	249,773	Fe	5
Ba	233,527	Fe, V	5
	455,403		1
	493,409		1
Be	313,042	V,Ti	2
	234,861	Fe	30
	313,107		

Element	Line (nm)	Interfering elements	Instrumental detection limit (µg/l)
Bi	223,061	Cu	30
	306,772	Fe, V	75
Ca	315,887	Co	30
	317,933	Fe, V	20
	393,366		0,2
Cd	214,438	Fe	3
	226,502	Fe	3
	228,802	As, Fe, Co, Cs	3
Ce	394,275	Co,Mn,Mo,Nd,Rh	70
	413,380	Zn,Fe,Mg,Ni,Ti	50
Co	228,616	Ti	7
Cr	205,552	Fe, Mo	6
	206.149	Al,Fe,Ti,Zn	6
	267,716	Mn, V	7
	283,563	Fe, Mo	7
	284,325	Fe	7
Cu	324,754	Ti, Fe	5
	327,396		10
Fe	259,940		6
	238,20	Co	1
K	766,490	Mg, Ar	6
	769,90		
Hg	184.887		5
	194.164	V	5
La	333,749	?	10
	379,478	?	10
Li	460,286	Fe	



Element	Line (nm)	Interfering elements	Instrumental detection limit (µg/l)
	670,784		6
Mg	279,079	Fe	30
	279,553		0,1
	285,213	Fe	
Mn	257,610	Fe, Mo, Cr	1
	293,306	Al, Fe	1
Mo	202,030	Al, Fe	8
	204,598		10
Na	588,995	Ar	70
	589,592	Ba	100
Na	330,237		
Nd	406,109	Sm,Pr	100
	430,358	Pr,V	75
Ni	231,604	Co,Fe	25
Ni	232,003	Cr	5
P	178,287	I	15
	213,618	Cu, Fe, Mo, Zn	7
	214,914	Cu, Al, Mg	7
	177,428	Cu	
Pb	220,353	Al, Co, Ti	40
	283,306	Fe	
Pr	390,844	Ce,U	40
	417,939	Cr,CeV,Fe,Sm,Nd	40
S	182,036	Cr, Mo	15
	180,669	Ca	13
Sb	206,833	Cr, Mg, Co, Mn	30
	217,581	Nb, V, W	40

Element	Line (nm)	Interfering elements	Instrumental detection limit (µg/l)
Sc	357,635	Ce	0,3
	361,384	Ce,W	0,3
Se	196,026	Ti	75
	203,985	Cr, Mn, Sb	100
Si	251,611		10
	212,412		15
	288,158		30
Sm	359,260	?	40
	388,529	?	80
Sn	235,848	Mo, Co	100
	240,170	Fe	100
	283,998	Cr	100
	189,980		25
Sr	407,771		0,3
	421,552		0,3
	460,733		70
Te	214,281	V,Sc,Re	40
	225,902	W	100
Ti	334,941	Ca, Cr, Si	4
	336,121		5
	337,280		7
	368,520	Co, Cr	10
Tl	190,807	W	100
	190,794	Co	100
U	385,957	Fe, V,Sc,Sm,Pr,Th	10
	409.013		10
V	290,882	Fe, Mo	8

Element	Line (nm)	Interfering elements	Instrumental detection limit (µg/l)
	292,402	Fe, Mo, Cr,Ti	8
	310,230	Ni	7
	311,071	Fe, Mn, Ti, Cr	10
W	207,911		30
	209,860		40
	239,709		60
	222,589	Cu	60
	202,998		75
Zn	206,191	Cr	6
	213,856	Cu, Ni, Fe	7
Zr	343,823		10
	354,262		7
	339,198		7

## Annex B (informative)

### Precision data

**Table B.1 — Method Limit of detection (MLOD) <sup>a</sup>**

MLOD in matrix						
Element	wavelength			matrix solution (mg/l)	mg/kg dw (2,5g/100 ml)	Reference (informative)
Ag	328,1	atom	axial	0,003	0,10	-
Ag	338,3	atom	axial	0,002	0,09	
As	189,0	atom	axial	0,034	1,72	
As	193,7	atom	axial	0,072	2,88	4
As	197,2	atom	axial	0,066	2,64	
Ba	234,5	ion	rad	0,012	0,46	15
Ba	455,4	ion	rad	0,008	0,31	
Ba	493,4	ion	rad	0,007	0,36	
Be	234,9	atom	axial	0,003	0,12	
Be	313,0	ion	axial	0,003	0,10	0,1
Be	313,1	ion	axial	0,002	0,11	
Cd	214,4	atom	axial	0,003	0,12	0,17
Cd	226,5	ion	axial	0,004	0,17	
Cd	228,8	atom	axial	0,003	0,16	
Co	228,6	ion	axial	0,003	0,12	1
Co	230,8	ion	axial	0,003	0,12	
Co	238,9	ion	axial	0,025	1,27	
Cr	205,6	ion	axial	0,031	1,22	15
Cr	206,2	ion	axial	0,036	1,42	
Cr	267,7	ion	axial	0,031	1,53	

MLOD in matrix						
Element	wavelength			matrix solution (mg/l)	mg/kg dw (2,5g/100 ml)	Reference (informative)
<b>Cu</b>	224,7	ion	axial	0,014	0,72	
<b>Cu</b>	324,7	atom	axial	0,015	0,62	5
<b>Cu</b>	327,4	atom	axial	0,018	0,71	
<b>Mo</b>	202,0	ion	axial	0,005	0,19	1,5
<b>Mo</b>	203,8	ion	axial	0,022	1,10	
<b>Mo</b>	204,6	ion	axial	0,015	0,59	
<b>Ni</b>	221,6	ion	axial	0,008	0,31	
<b>Ni</b>	231,6	ion	axial	0,008	0,31	3
<b>Ni</b>	233,0	atom	axial	0,019	0,94	
<b>Pb</b>	217,0	atom	axial	0,074	2,97	
<b>Pb</b>	220,3	ion	axial	0,019	0,77	13
<b>Pb</b>	283,3	atom	axial	0,085	4,25	
<b>Sb</b>	206,8	atom	axial	0,037	1,49	
<b>Sb</b>	217,6	atom	axial	0,028	1,12	
<b>Se</b>	196,0	atom	axial	0,080	3,19	
<b>Sn</b>	189,9	ion	axial	0,021	0,83	6
<b>Sn</b>	242,2	atom	axial	0,065	3,24	
<b>Tl</b>	190,8	ion	axial	0,033	1,31	0,3
<b>Tl</b>	351,9	atom	axial	0,014	0,69	
<b>V</b>	290,9	ion	rad	0,010	0,41	4
<b>V</b>	292,4	ion	rad	0,009	0,35	
<b>V</b>	310,2	ion	rad	0,003	0,16	
<b>Zn</b>	202,5	ion	rad	0,022	0,89	17
<b>Zn</b>	206,2	ion	rad	0,065	3,27	
<b>Zn</b>	213,8	atom	rad	0,029	1,15	

<sup>a</sup> Taken from ICP-OES: Ruggedness study Horizontal W6-19 [4].

**Table B.2 — Content of the matrix solution**

<b>Component</b>	<b>Concentration</b>
Aluminium	1200 mg/L
Iron	950 mg/L
Potassium	2000 mg/L
Sodium	230 mg/L
Phosphorus	150 mg/L
Calcium	1800 mg/L
Magnesium	320 mg/L
Sulphur	430 mg/L
Manganese	20 mg/L
Titanium	60 mg/L

Tabel B.2 — Measurement of precision (in VC<sub>w</sub> %) <sup>a</sup>

Element and specifications			Sample type										Average (VC <sub>w</sub> %)			
			1	2	3	4	5	6	7	8	9	10				
<b>Ag</b>	atom	Axial	10,5					14,4								<b>12,5</b>
<b>Al</b>	atom	rad	5,0	5,4	6,9	4,2	6,0	6,6	7,2	7,0	6,9	5,9				<b>6,1</b>
<b>As</b>	atom	axial	17,8	9,9	7,4	10,2	18,1			19,0						<b>13,7</b>
<b>Ba</b>	ion	rad	18,5	6,7	6,5	5,1	5,4	5,7	6,4	31,8	6,9	6,9				<b>10,0</b>
<b>Be</b>	ion	axial	9,3	4,9	14,1	3,3	5,4	14,3	3,6	5,1						<b>7,5</b>
<b>Ca</b>	ion	rad	5,4	4,9	4,5	3,6	4,8	6,6	4,6	6,7	5,3	4,1				<b>5,0</b>
<b>Cd</b>	atom	axial	7,5	6,5	17,2			11,7	3,6							<b>9,3</b>
<b>Co</b>	ion	axial	6,5	5,4	7,5	8,5	9,1	4,9	8,0	8,1		11,6				<b>7,7</b>
<b>Co</b>	ion	axial	IEC	7,2	5,5	9,4	10,6	11,8	5,0	8,7	8,7					<b>8,4</b>
<b>Cr</b>	ion	axial	3,9	3,2	8,1	5,4	5,1	4,2	3,9	5,8		5,2				<b>5,0</b>
<b>Cu</b>	atom	axial	4,7	8,2	4,0	4,6	3,8	5,5	4,3	5,9		8,1				<b>5,4</b>
<b>Fe</b>	ion	rad	3,8	1,6	3,4	3,0	2,3	4,0	3,6	2,6	3,3	1,6				<b>2,9</b>
<b>Mg</b>	atom	rad	5,2	3,7	5,9	4,4	5,4	5,1	5,7	7,1	6,7	5,9				<b>5,5</b>
<b>Mn</b>	ion	rad	4,7	5,5	6,1	7,7	8,1	7,4	3,3	14,7	14,8	15,4				<b>8,8</b>
<b>Mo</b>	ion	axial	8,1		5,4	5,0		15,8								<b>8,6</b>
<b>Ni</b>	ion	axial	3,8	4,8	6,3	4,0	5,0	4,1	5,4	7,9						<b>5,2</b>
<b>Ni</b>	ion	axial	IEC	3,9	6,1		5,8	6,7	4,2	5,9	10,4					<b>6,1</b>
<b>P</b>	atom	axial	2,9	3,0	3,3	4,0	3,8	2,8	3,1	5,3		4,3				<b>3,6</b>
<b>Pb</b>	ion	axial	3,8	4,7	4,5	3,5	4,8	6,2	4,3	5,8		7,0				<b>4,9</b>
<b>S</b>	atom	axial	1,7	4,4	1,8	5,3	3,3	1,6	22,7	4,1		3,4				<b>5,4</b>
<b>Sb</b>	atom	axial	12,6													<b>12,6</b>
<b>Se</b>	atom	axial														
<b>Sn</b>	ion	axial	5,2	3,3				6,6								<b>5,0</b>
<b>Ti</b>	ion	rad	5,2	3,4	4,8	3,3	4,6	5,0	3,6	3,8	5,5	5,6				<b>4,5</b>
<b>Tl</b>	ion	axial														

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<b>V</b>	ion	rad		7,1	6,1	3,9	2,8	3,5	5,2	3,0	4,3	8,6	<b>4,9</b>
<b>Zn</b>	ion	rad		5,4	2,7	3,8	2,8	4,0	5,0	4,9	5,5	5,4	<b>4,4</b>
<b>Zn</b>	ion	rad	IEC	5,4	2,7	3,8	2,8	4,0	5,0	4,9	5,5	5,4	<b>4,4</b>
<b>Over all</b>													<b>6,8</b>

<sup>a</sup> Taken from ICP-OES: Ruggedness study Horizontal W6-19 [4].

**Key**

1. CRM 145R: sewage sludge.
  2. FeNeLab: internal reference material, river clay.
  3. Lksd-1: lake sediment.
  4. Stsd-2: stream sediment.
  5. CRM 7001: light sandy soil.
  6. CRM 144R: sewage sludge from domestic origin.
  7. CRM 141R: Calcareous loam soil.
  8. AMB WB: internal reference material: lake sediment.
  9. AMB SZ: internal reference material: 'playground sand'.
  10. AMB Compost: internal reference material; biowaste/garden waste.
  11. Average: averaged precision of the 10 test samples.
- VC<sub>w</sub> Variation coefficient in % based on the within laboratory reproducibility standard deviation.



## Annex C (informative)

### Inter element correction

Depending of the matrix of the samples that are to be determined, the possible interference should be investigated for each individual instrument.

Single element stock solutions should be used to determine the major interferences for ICP OES method.

From these single element stock solutions a series of nine standard solutions with a concentration of 200 mg/l of the interfering elements can be made and measured.

An example for environmental samples within the scope of this standard is given below.

Group I	Interferent standard I	V, Al, Ba, Ca, Ce
	Interferent standard II	La, Cu, Cd, Co, Cr
	Interferent standard III	Fe, Mo, Nd, Ti, Zn
	Interferent standard IV	Ni, S, As, Mg, Mn

Group II	Interferent standard V	V, Ti, Cu
	Interferent standard VI	Fe, Cr, Nd
	Interferent standard VII	Mn, Mo
	Interferent standard VIII	Ni, Zn, Nd
	Interferent standard IX	La, Ce

Standard solutions in diluted acid are aspirated into the plasma and for all line of the elements of interest the possible interference is measured.

The Inter-Element-Correction-factors (IEC) are calculated from these measurements.

$$IEC = \frac{\text{Apparent Analyte concentration}}{\text{Interferent concentration}}$$

$$\text{and } Z = X - (Y \times IEC)$$

where:

**X** is the measured analyte concentration

**Y** is the measured interferent concentration

$Z$  is the corrected analyte concentration

The conditions for applying an IEC are:

- Peak setting and selection must be stable and/or reproducible;
- Assuming linear response of interferent on analyte;
- No multiple overlap is allowed.

Beside the measurement of the IEC the background correction profile should be optimized by measuring the ICS solution followed by evaluation of the interference by other lines caused by elements present in the ICS.

Non spectral interferences (like physical, chemical and memory effects) are not investigated in this study. The effect of these interferences is minimized by the use of an internal standard and through matrix matching.

It is important, that inter element correction (IEC) can only be used, if there is a linear relationship between the concentration of the interfering element and the apparent analyte concentration. This is only true for direct spectral overlap (line coincidence) and in a limited concentration range of the interfering element. Furthermore the plasma conditions should remain unchanged. In many cases a partial line overlap is observed. Such interference can not be compensated by IEC. For partial line or multiple overlap multivariate calibration methods (e.g. MSF) are necessary. It is important, that inter element correction (IEC) can only be used, if there is a linear relationship between the concentration of the interfering element and the apparent analyte concentration. This is only true for direct spectral overlap (line coincidence) and in a limited concentration range of the interfering element. Furthermore the plasma conditions should remain unchanged. In many cases a partial line overlap is observed. Such interference can not be compensated by IEC. For partial line or multiple overlap multivariate calibration methods (e.g. MSF) are necessary.

In such a case use the procedure described in the manufacturers manual.

**Annex D (informative)**

**Comparison of OES and MS analysis on the same extracts for different matrices in one lab - Comparison of a standard AR extract by MS and OES and comparison of individual AR extracts for different matrices by MS and OES by different labs.**

There are different levels of comparison of OES and MS analysis. First there is the measurement by OES and MS on the same digest of different matrices within one lab (table C.1)

Table C.1 Comparison of the ratio of OES/MS for different matrices and different digestion methods within one lab

Matrix Digestion	Element	Compost 2		Sludge 1		Sludge 2		Soil 1		Soil 2		Digestion & Element
		Average Ratio OES/MS	Stdev Ratio OES/MS	Average Ratio OES/MS	Stdev Ratio OES/MS	Average Ratio OES/MS	Stdev Ratio OES/MS	Average Ratio OES/MS	Stdev Ratio OES/MS	Average Ratio OES/MS	Stdev Ratio OES/MS	Average
AR reflux	Pb	1.107	0.036	0.938	0.005	0.967	0.025	1.23	0.04	0.96	0.09	1.04
AR MW 115	Pb	0.940	0.032	1.023	0.011	0.920	0.022	1.04	0.13	0.83	0.02	0.95
AR MW 175	Pb	1.037	0.025	1.035	0.012	0.942	0.011	1.07	0.15	0.96	0.10	1.01
NA	Pb	1.244	0.135	1.070	0.029	0.943	0.093	1.16	0.02	0.94	0.03	1.07
AR reflux	Cr	1.07	0.03	0.81	0.01	1.11	0.03	1.15	0.02	1.07	0.01	1.04
AR MW 115	Cr	1.18	0.07	0.87	0.02	1.03	0.02	1.14	0.01	1.12	0.02	1.07
AR MW 175	Cr	1.15	0.04	0.90	0.01	0.99	0.01	1.05	0.01	1.09	0.01	1.03
NA	Cr	1.08	0.13	0.92	0.03	1.01	0.01	1.09	0.04	1.07	0.06	1.03
AR reflux	V	0.98	0.02	1.11	0.02	1.09	0.13	1.05	0.01	0.93	0.01	1.03
AR MW 115	V	1.19	0.01	1.25	0.03	1.05	0.02	1.03	0.01	0.93	0.01	1.09
AR MW 175	V	1.06	0.02	1.24	0.04	1.00	0.02	0.98	0.01	0.92	0.01	1.04
NA	V	1.04	0.08	1.29	0.03	1.02	0.02	1.06	0.02	0.92	0.02	1.06
AR reflux	Zn	1.24	0.04	1.05	0.01	1.12	0.02	1.08	0.02	0.88	0.01	1.07
AR MW 115	Zn	1.18	0.01	1.15	0.02	1.05	0.02	1.14	0.01	0.91	0.02	1.09
AR MW 175	Zn	1.16	0.03	1.18	0.02	1.06	0.01	1.18	0.01	0.95	0.01	1.11

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NA	Zn	1.15	0.03	1.12	0.01	0.97	0.02	1.19	0.01	0.94	0.02	1.07
AR reflux	As	0.86	0.05	1.02	0.00	1.04	0.05	0.96	0.03	0.93	0.01	0.96
AR MW 115	As	0.83	0.10	1.05	0.03	1.01	0.04	1.01	0.05	0.95	0.01	0.97
AR MW 175	As	0.84	0.04	1.10	0.01	1.02	0.05	1.06	0.05	0.91	0.04	0.99
NA	As	0.78	0.06	1.06	0.02	0.90	0.02	1.05	0.04	0.97	0.03	0.95
AR reflux	Ni	1.15	0.02	1.04	0.01	1.14	0.07	1.11	0.01	1.21	0.01	1.13
AR MW 115	Ni	1.23	0.04	1.11	0.02	1.09	0.01	1.10	0.02	1.46	0.11	1.20
AR MW 175	Ni	1.21	0.09	1.15	0.02	1.07	0.01	1.11	0.02	1.53	0.03	1.21
NA	Ni	1.10	0.05	1.21	0.02	1.09	0.01	1.16	0.00	1.41	0.02	1.19
AR reflux	Co	1.18	0.03	0.96	0.01	1.27	0.01	1.24	0.01	1.07	0.01	1.14
AR MW 115	Co	1.39	0.10	1.00	0.00	1.21	0.01	1.24	0.01	1.03	0.04	1.18
AR MW 175	Co	1.36	0.04	1.04	0.01	1.22	0.00	1.21	0.12	1.16	0.02	1.20
NA	Co	1.36	0.05	1.08	0.01	1.10	0.02	1.19	0.15	1.05	0.02	1.16
	Matrix	Average	stdev	Average	stdev	Average	stdev	Average	stdev	Average	stdev	
		1.111	0.050	1.063	0.017	1.051	0.029	1.110	0.037	1.039	0.027	
										Overall average		1.075
										Overall st dev		0.13
										Var %		12.15

Conclusion 1: There appears to be a systematic difference between OES and MS

Conclusion 2: If labs can not be forced to use either MS or OES or both, this uncertainty has to be considered in the comparison of digestion methods

Conclusion 3: ICP OES/ MS difference is independent of the digestion method

Conclusion 4: ICP/MS difference seems element specific (e.g. As, Ni, Co).

Further more there is the comparison of analysis of the same extract from different matrices by OES and MS by different labs (Table C.2).

Table C.2 Comparison of ratios ICP OES/MS for the same digest for different matrices analysed by different labs

		Compost 2	Sewage Sludge 1	Soil 2	
Lab		Ratio OES/MS	Ratio OES/MS	Ratio OES/MS	
<b>Cd</b>	1	0.91	1.07		
	2	1.45	1.16	1.60	
	3	0.99	0.76	0.94	
	4	0.95	1.08	0.95	
	Avg	1.08	1.02	1.17	
	Stdev	0.25	0.17	0.38	
		23.62 %	17.11 %	32.28 %	
Labs		Compost 2	Sewage Sludge 1	Soil 1	
		Ratio OES/MS	Ratio OES/MS	Ratio OES/MS	
<b>Cr</b>	1	0.99		1.06	
	2	0.95	0.92	0.93	
	3	1.00	0.93	1.05	
	4	0.97	0.98	0.98	
	5	1.04	1.06	1.07	
	Avg	0.99	0.97	1.02	
	Stdev	0.03	0.06	0.06	
	3.53 %	6.28 %	6.23 %		
Labs		Compost 2	Sewage Sludge 1	Soil 1	
		Ratio OES/MS	Ratio OES/MS	Ratio OES/MS	
<b>Cu</b>	1	1.07	1.17	1.17	
	2	1.19	1.24	1.15	
	3	0.97	1.02	0.96	
	4	0.98	1.03	0.96	
	5		0.93	0.95	
	Avg	1.05	1.08	1.04	
	Stdev	0.10	0.13	0.11	
	9.9 %	11.6 %	10.84 %		

Finally, an aqua regia extract was circulated for analysis in parallel with the samples. The number of labs performing this step was limited.(Table C.3)

Table Table C.3 Within and between lab variability for an AR extract (No distinction between OES and MS)

<b>Matrix</b>	<b>p</b>	<b>Mean (ug/l)</b>	<b>sr</b>	<b>sR</b>	<b>VCr</b>	<b>VCR</b>	<b>Outliers</b>	<b>No of test data</b>	<b>BD #</b>
ICP Cd	8	10.4	0.8	0.9	7.2%	9.0%	0	19	0
ICP Co	7	72	2.3	19	3.2%	26%	0	15	0
ICP Cr	8	361	25	43.6	7.0%	12%	0	17	0
ICP Cu	9	1968	61	202	3.1%	10%	0	17	0
ICP Ni	9	258	7.2	29	2.8%	11%	0	18	0
ICP Pb	8	754	37	52	4.9%	7.0%	0	15	0
ICP Zn	9	6746	60	361	0.88%	5.4%	0	16	0

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