
Blato, obdelani biološki odpadki in tla - Določevanje elementov z optično emisijsko spektrometrijo z induktivno sklopljeno plazmo (ICP/OES)

Sludge, treated biowaste and soil - Determination of elements using inductively coupled plasma optical emission spectrometry (ICP-OES)

Schlamm, behandelter Bioabfall und Boden - Bestimmung von Elementen mittels optischer Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES)

Boues, bio-déchets et sols - Détermination des éléments en traces par spectrométrie d'émission optique avec plasma induit par haute fréquence (ICP-OES)

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**Sludge, treated biowaste and soil - Determination of elements
using inductively coupled plasma optical emission spectrometry
(ICP-OES)**

Boues, bio-déchets et sols - Détermination des éléments
en traces par spectrométrie d'émission optique avec
plasma induit par haute fréquence (ICP-OES)

Schlamm, behandelter Bioabfall und Boden - Bestimmung
von Elementen mittels optischer Emissionsspektrometrie
mit induktiv gekoppeltem Plasma (ICP-OES)

This Technical Specification (CEN/TS) was approved by CEN on 16 July 2012 for provisional application.

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Foreword

This document (CEN/TS 16170:2012) has been prepared by Technical Committee CEN/TC 400 "Project Committee - Horizontal standards in the fields of sludge, biowaste and soil", the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

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Introduction

This Technical Specification is applicable for several types of matrices as indicated in Table 1.

Table 1 — Matrices for which this Technical Specification is applicable

Matrix	Materials tested
Sludge	Municipal sludge
Biowaste	Compost
Soil	Sludge amended soils

WARNING — Persons using this Technical Specification should be familiar with usual laboratory practice. This Technical Specification does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this Technical Specification be carried out by suitably trained staff.

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1 Scope

This Technical Specification specifies a method for the determination of the following elements in *aqua regia*, nitric acid digest solutions of sludge, treated biowaste and soil:

Aluminium (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), bismuth (Bi), boron (B), cadmium (Cd), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), gallium (Ga), indium (In), iron (Fe), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), nickel (Ni), phosphorus (P), potassium (K), selenium (Se), silicon (Si), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), thallium (Tl), tin (Sn), titanium (Ti), tungsten (W), uranium (U), vanadium (V), zinc (Zn) and zirconium (Zr).

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

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN 16173, *Sludge, treated biowaste and soil — Digestion of nitric acid soluble fractions of elements*

EN 16174, *Sludge, treated biowaste and soil — Digestion of aqua regia soluble fractions of elements*

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

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3 Principle

Digests of sludge, treated biowaste or soil with nitric acid or *aqua regia* (see EN 16173 and EN 16174) are analysed by inductively coupled plasma optical emission spectrometry (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. Analyte species originating in the digest solution are nebulised 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.

4 Interferences

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 shall be recognised and appropriate corrections made. Tests for their presence are described below.

Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light which causes background increase or overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

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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 compared with 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 shall 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. Alternatively they can be corrected by multiple dimensional spectra fitting methods or by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements to be analysed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and would be reported as analyte concentrations. The interferences are listed in Table A.1.

If available, the user should apply multiple dimensional spectra fitting methods provided by the manufacturer, as a corrective action. In this case, the selection of background points for correction is not necessary, since all adjacent wavelengths are processed.

Physical interferences are effects associated with the sample nebulisation 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 shall be reduced by diluting the sample, matching the acid concentration, matrix-matching, or a high solid nebuliser. They can be corrected for by using an internal standard.

Chemical interferences include molecular compound formation, ionisation effects, and solute vaporisation effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimised by careful selection of operating conditions (e. g. radio frequency (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 nebuliser and from the build-up of sample material in the plasma torch and spray chamber. The occurrence memory effects depend on the element and can be minimised by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognised within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element shall be estimated prior to analysis during method development.

5 Reagents

For the determination of elements at trace and ultra-trace 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.

- 5.1 **Water**, grade 2 as specified in EN ISO 3696 for all sample preparations and dilutions.
- 5.2 **Nitric acid**, HNO_3 , $\rho(\text{HNO}_3) = 1,4\text{g/ml}$, $c(\text{HNO}_3) = 15\text{ mol/l}$, $w(\text{HNO}_3) = 650\text{ g/kg}$.
- 5.3 **Hydrochloric acid**, HCl , $\rho(\text{HCl}) = 1,18\text{ g/ml}$, $c(\text{HCl}) = 12\text{ mol/l}$, $w(\text{HCl}) = 370\text{ g/kg}$.

5.4 Single-element standard stock solutions

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

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

For stability of the solutions refer to manufacturer guarantee statement.

5.5 Multi-element standard stock solutions

5.5.1 General

Depending on the scope, different multi-element standard stock 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 shall be taken to prevent chemical reactions (e.g. precipitation).

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

5.5.2 Multi-element standard stock solution A at the mg/l level may contain the following elements:

Ag, Al, As, B, Ba, Be, Bi, Cd, Co, Cr, Cu, Fe, Ga, Hg, In, Li, Mn, Ni, Pb, Se, Sr, Ti, Tl, U, V, Zn.

Use nitric acid (5.2) for stabilisation of multi-element standard stock solution A.

5.5.3 Multi-element standard stock solution B at the mg/l level may contain the following elements:

Mo, Sb, Si, Sn, W, Zr.
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Use hydrochloric acid (5.3) for stabilisation of multi-element standard stock solution B.

Other elements of interest may be added to the standard stock solution, provided that the resulting multi-element solution is stable.

5.5.4 Multi-element standard stock solution C at the mg/l level may contain the following elements:

Ca, Mg, Na, K, P, S.

Use nitric acid (5.2) for stabilisation of multi-element standard stock solution C.

5.6 Multi-element calibration solutions

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 (5.2 and/or 5.3) 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.

CEN/TS 16170:2012 (E)**5.7 Internal standard solution**

The choice of elements for the internal standard solution depends on the analytical problem. The internal standards elements shall not be analytes and the concentrations of the selected elements should be negligibly low in the digests of samples. The elements Sc, Y and Lu 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.

5.8 Calibration blank solution

Prepare the calibration blank solution by diluting acids (5.2, 5.3) with water (5.1) to the same concentrations as used in the calibration solutions and test sample solutions.

5.9 Test blank solution

The test blank solution shall contain all of the reagents in the same concentrations and shall be handled in the same way throughout the procedure as the samples. The test blank solution contains the same acid concentration in the final solution as the test sample solution after the digestion method is applied.

5.10 Calibration check solution

Prepare the calibration check solution using an independent multi-element standard stock solution, adapted to the same acid concentrations at an upper concentration level.

5.11 Interference check solution

If interferences cannot be excluded (see Table A.1), prepare an interference check solution to detect the interference, with known concentrations of interfering elements. The choice of the concentration and interfering element are matrix dependent.

Avoid two or more interferents 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 mg/l 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.

6 Apparatus**6.1 Inductively coupled argon plasma emission spectrometer**

NOTE The instruments for ICP-OES come in 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. To gain a higher sensitivity a higher signal to background ratio is necessary.

6.1.1 Computer-controlled emission spectrometer with background correction.

6.1.2 Radio-frequency generator

6.1.3 Mass flow controller, for argon nebuliser gas supply (optional).

6.1.4 Peristaltic pump (optional).

6.1.5 Auto sampler (optional).

6.1.6 Argon gas supply of high purity, e.g. $\geq 99,95\%$.

6.2 Volumetric flasks

6.3 Volumetric pipettes

7 Procedure

7.1 Test sample solution

The test sample solution is a particle-free digest or extraction solution prepared according to EN 16173 or EN 16174.

7.2 Test solution

The test solution is an aliquot of the test sample solution and may be directly obtained from the test sample solution or may be diluted to accommodate the measurement range or to dilute the matrix.

The acidity of calibration solutions shall match the acid concentration in test solutions.

7.3 Instrument set-up

7.3.1 General requirements

Adjust the instrumental parameters of the ICP-OES system in accordance with the manufacturer's instructions. 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. Define the rinsing times to blank level depending on the length of the flow path. The time for rinsing the sample introduction system between the subsequent samples shall be long enough to rinse all the analytes of interest from the system.

NOTE 1 A guideline for method development and instrument set up is given in EN ISO 11885.

NOTE 2 ICP-OES has excellent multi-element capability. Nevertheless, it does not mean that all elements can be analysed during one measurement run. The sensitivity of determination depends on numerous parameters (nebuliser flow, radio-frequency power, viewing height etc). The optimal instrument settings cannot be reached for all elements at once.

7.3.2 Inter-element correction

Investigate whether the interfering elements in Table A.1 result in measured values higher than three times the instrumental limit of detection or 0,5 times the lowest concentration to be reported. If this is the case, correct for interference. See Annex C for determining the inter-element correction factors (IEC factors).

7.3.3 Internal standard

If the type of samples to be measured strongly varies, matrix matching may not be possible. The internal standard correction method can be applied independently from the calibration procedure.

If the internal standard procedure insufficiently reduces matrix effects, apply standard addition calibration (see 7.4.2).

The use of an internal standard is recommended. Add the internal standard solution (5.7) to all solutions to be measured. One possibility is the on-line dilution and mixing the sample flow with a flow of internal standard solution by the peristaltic pump of the nebuliser.

The mass concentration of the internal standard 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.