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

Ta slovenski standard je istoveten z: **FprEN 16170**

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13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge
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**FINAL DRAFT
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Will supersede CEN/TS 16170:2012

English Version

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

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

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This draft European Standard is submitted to CEN members for unique acceptance procedure. It has been drawn up by the Technical Committee CEN/TC 400.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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FprEN 16170:2015 (E)

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European foreword

This document (FprEN 16170:2015) 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.

This document is currently submitted to the Unique Acceptance Procedure.

This document will supersede CEN/TS 16170:2012.

The preparation of the previous edition of this analytical method 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.

This document contains the following technical changes in comparison with the previous edition:

— repeatability and reproducibility data have been added from a European interlaboratory comparison organized by the German Federal Institute for Materials Research and Testing BAM in 2013 (see Annex A).

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Introduction

This European Standard is applicable and validated for several types of matrices as indicated in Table 1 (see Annex A for the results of validation).

Table 1 — Matrices for which this European Standard is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Compost
Soil	Sludge amended soils

WARNING — Persons using this European Standard should be familiar with usual laboratory practice. This European Standard 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 European Standard be carried out by suitably trained staff.

1   Scope

This European Standard specifies a method for the determination of the following elements in *aqua regia* or 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).

The method has been validated for the elements given in Table A.1. The method is applicable for the other elements listed above, provided the user has verified the applicability.

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)*

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.

NOTE   For the determination of tin only *aqua regia* extraction applies (EN 16174).

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.

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 B.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, $\rho(HNO_3)$ = 1,4g/ml, $c(HNO_3)$ = 15 mol/l, $w(HNO_3)$ = 650 g/kg.

5.3 Hydrochloric acid, HCl, ρ(HCl) = 1,18 g/ml, c(HCl) = 12 mol/l, w(HCl) = 370 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, ρ(element) = 1 000 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.

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.

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Applying digestion with aqua regia or nitric acid to sludge, treated biowaste or soil, ubiquitous elements like Al, Na, K, Ca, Mg, Ti and Fe can be co-extracted resulting in 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 control samples and interference control samples. Otherwise the sample matrix elements shall be adapted in calibration solutions for each batch of sample types.

Alternatively the standard addition method shall be used.

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 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 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 B.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.