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Slam, behandlat bioavfall och mark – Bestämning av grundämnen med induktivit kopplad plasma och optisk emissionsspektrometri

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

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Europastandarden EN 16170:2016 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN 16170:2016.

Denna standard ersätter SIS-CEN/TS 16170:2013, utgåva 1.

The European Standard EN 16170:2016 has the status of a Swedish Standard. This document contains the official English version of EN 16170:2016.

This standard supersedes the Swedish Standard SIS-CEN/TS 16170:2013, edition 1.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Karaktärisering av avfall, mark och slam, SIS/TK 535.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 16170

October 2016

ICS 13.030.01; 13.080.10

Supersedes 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)

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

This European Standard was approved by CEN on 19 March 2016.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

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European foreword

This document (EN 16170:2016) has been prepared by Technical Committee CEN/TC 444 “Test methods for environmental characterization of solid matrices”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2017, and conflicting national standards shall be withdrawn at the latest by April 2017.

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 supersedes 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).

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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	Soil

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), cerium (Ce), chromium (Cr), cobalt (Co), copper (Cu), gallium (Ga), indium (In), iron (Fe), lanthanum (La), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), neodymium (Nd), nickel (Ni), phosphorus (P), potassium (K), praseodymium (Pr), samarium (Sm), scandium (Sc), selenium (Se), silicon (Si), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), tellurium (Te), thallium (Tl), thorium (Th), 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, Ce, Cd, Co, Cr, Cu, Fe, Ga, Hg, In, K, La, Li, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, Pr, S, Sb, Sc, 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 or 5.3 or a mixture of 5.2 and 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.