

Sludge, treated biowaste and soil - Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS)

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

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English Version

**Sludge, treated biowaste and soil - Determination of
elements using inductively coupled plasma mass
spectrometry (ICP-MS)**

Boues, bio-déchets traités et sols - Détermination des
éléments en traces par spectrométrie de masse avec
plasma induit par haute fréquence (ICP-MS)

Schlamm, behandelter Bioabfall und Boden -
Bestimmung von Elementen mittels
Massenspektrometrie mit induktiv gekoppeltem
Plasma (ICP-MS)

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

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European foreword

This document (EN 16171:2016) 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of an identical text or by endorsement, at the latest by April 2017, and conflicting national standards shall be withdrawn at the latest by April 2017.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 16171:2012.

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 digests 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), cesium (Cs), chromium (Cr), cobalt (Co), copper (Cu), dysprosium (Dy), erbium (Er), europium (Eu), gadolinium (Gd), gallium (Ga), germanium (Ge), gold (Au), hafnium (Hf), holmium (Ho), indium (In), iridium (Ir), iron (Fe), lanthanum (La), lead (Pb), lithium (Li), lutetium (Lu), magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), neodymium (Nd), nickel (Ni), palladium (Pd), phosphorus (P), platinum (Pt), potassium (K), praseodymium (Pr), rhenium (Re), rhodium (Rh), rubidium (Rb), ruthenium (Ru), samarium (Sm), scandium (Sc), selenium (Se), silicon (Si), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), tellurium (Te), terbium (Tb), thallium (Tl), thorium (Th), thulium (Tm), tin (Sn), titanium (Ti), tungsten (W), uranium (U), vanadium (V), ytterbium (Yb), yttrium (Y), zinc (Zn), and zirconium (Zr).

The working range depends on the matrix and the interferences encountered.

The method detection limit of the method is between 0,1 mg/kg dry matter and 2,0 mg/kg dry matter for most elements. The limit of detection will be higher in cases where the determination is likely to be interfered (see Clause 4) or in case of memory effects (see e.g. EN ISO 17294-1:2006, 8.3).

The method has been validated for the elements given in Table A.1 (sludge), Table A.2 (compost) and Table A.3 (soil). 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)*

EN ISO 17294-1:2006, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines (ISO 17294-1:2004)*

3 Principle

Digests of sludge, treated biowaste or soil with nitric acid or *aqua regia* (see EN 16173 and EN 16174) are analysed by ICP-MS to get a multi-elemental determination of analytes.

The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in the digest solution are nebulised and the resulting aerosol is transported by argon gas into the plasma. The ions produced by the high temperatures of the plasma are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer, sorted according to their mass-to-charge ratios and quantified with a detector (e.g. channel electron multiplier).