

Sludge, treated biowaste and soil - Digestion of nitric acid soluble fractions of elements

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ICS 13.080.10; 13.030.01

English Version

Sludge, treated biowaste and soil - Digestion of nitric acid soluble fractions of elements

Boues, biodéchets traités et sols - Digestion des éléments
solubles dans l'acide nitrique

Schlamm, behandelter Bioabfall und Boden - Aufschluss
von mit Salpetersäure löslichen Anteilen von Elementen

This European Standard was approved by CEN on 24 May 2012.

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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.

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Foreword

This document (EN 16173:2012) 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.

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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

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.

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 method is intended to provide a multi-element digestion of sludge, treated biowaste and soil prior to analysis. It is known that the digestion of environmental samples with nitric acid does not necessarily lead to a complete element breakdown, and the extract from a test sample may not reflect the total concentrations of the target analytes. However, for most environmental applications the result is fit for the purpose.

This European Standard is applicable and validated for several types of matrices as indicated in Table 1 (see also Annex A for the results of the 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. The reagents used in this European Standard are strongly corrosive and partly very toxic. Safety precautions are absolutely necessary, not only due to the strong corrosive reagents, but also to high temperature and high pressure.

The use of laboratory-grade microwave equipment with isolated and corrosion resistant safety devices is required. Domestic (kitchen) type microwave ovens shall not be used, as corrosion by acid vapours may compromise the function of the safety devices and prevent the microwave magnetron from shutting off when the door is open, which could result in operator exposure to microwave energy.

All procedures shall be performed in a fume hood or in closed force-ventilated equipment. By the use of strong oxidising reagents, the formation of explosive organic intermediates is possible, especially when dealing with samples with a high organic content. Do not open pressurized vessels before they have cooled down. Avoid contact with the chemicals and the gaseous reaction products.

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 microwave digestion of sludge, treated biowaste and soil using nitric acid.

This method is applicable for microwave-assisted nitric acid digestion of sludge, treated biowaste and soils for the following elements:

Aluminium (Al), 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), rubidium (Rb), rhenium (Re), rhodium (Rh), ruthenium (Ru), samarium (Sm), scandium (Sc), selenium (Se), silicon (Si), sodium (Na), strontium (Sr), sulphur (S), tellurium (Te), terbium (Tb), thallium (Tl), thorium (Th), thulium (Tm), titanium (Ti), tungsten (W), uranium (U), vanadium (V), ytterbium (Yb), yttrium (Y), zinc (Zn), and zirconium (Zr).

This European Standard may also be applicable for the digestion of other elements.

Digestion with nitric acid will not necessarily accomplish total decomposition of the sample. The extracted analyte concentrations may not necessarily reflect the total content in the sample.

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 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

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

3 Principle

A test portion is digested in concentrated nitric acid by means of microwave heating with a suitable laboratory microwave unit. The sample and the nitric acid are placed in a fluorocarbon polymer or quartz microwave vessel. The vessel is sealed and heated in the microwave unit. After cooling, the vessel contents are filtered, centrifuged or allowed to settle and the clear solution is then transferred into a volumetric flask and diluted to volume and analysed by the appropriate measurement method.

4 Interferences and sources of errors

Due to the volatility of some compounds care shall be taken that the sample is not heated before the digestion and that any volatile reaction products formed during the digestion do not escape.

The container in which the sample is delivered and stored can be a source of errors. The material shall be chosen according to the elements to be determined; e.g. elemental Hg can penetrate polyethylene walls very fast in both directions. Glass containing e.g. B, Na, K, Al can contaminate samples.

A few refractory sample matrix constituents, such as quartz, silicates, titanium dioxide, alumina and other oxides may not be dissolved. The bound elements are considered non-mobile in the environment and are excluded from most aquatic pollution transport mechanisms.

High acid and solute concentrations in the digest that cause interferences shall be properly addressed during determination.