SLOVENSKI STANDARD
SIST EN 16173:2013

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Blato, obdelani biološki odpadki in tla - Razklop v dušikovi kislini topnih frakcij elementov

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

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

Boue, biodéchet traité et sol - Digestion des éléments solubles dans l'acide nitrique

Ta slovenski standard je istoveten z: **EN 16173:2012**

ICS:

13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge
13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

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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

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Contents

Page

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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.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

Ensure that the complete test portion is brought into contact with the acid mixture in the digestion vessel.

There is a potential for vigorous reaction, especially with samples containing volatile or easily oxidizable species. When digesting a matrix of this type, use no more than 0,1 g sample to begin with. If a vigorous reaction occurs upon addition of nitric acid, allow the sample to pre-digest in the uncapped digestion vessel until the reaction ceases. Heat may be added in this step for safety considerations (for example, the rapid release of carbon dioxide from carbonates, easily oxidizable organic matter). Once the initial reaction has ceased, the sample may continue through the digestion procedure.

If the digested solution is filtrated, take care that the filtration procedure does not introduce contamination.

5 Reagents

Use only acids and reagents of recognized analytical grade to avoid high blank values for subsequent analytical measurements. Use a test blank solution throughout the procedure applying all steps with the same amount of acids, but without a sample.

5.1 Water, grade 2 as specified in EN ISO 3696 or better.

The water for preparation of reagents shall meet the requirements of the subsequent analysis. Verify the purity by performing a blank test.

5.2 Nitric acid, $c(HNO_3)$ = 15 mol/l, ρ = 1,4 kg/l.

5.3 Nitric acid, $c(HNO_3)$ = 0,5 mol/l; ρ = 1,0 kg/l.

6 Apparatus

Usual laboratory apparatus. All glassware and plastics ware shall be adequately cleaned and stored in order to avoid any contamination.

Depending on the concentration of the element of interest, a particular caution to the cleaning of the vessels shall be taken.

6.1 Microwave digestion system, corrosion resistant and well ventilated. All electronics shall be protected against corrosion for safe operation.

Use a laboratory-grade microwave oven with temperature feedback control mechanisms.

The microwave digestion system should be able to control the temperature with an accuracy of ± 5 °C and automatically adjust the microwave field output power within 2 s of sensing. Temperature sensors shall be accurate to ± 2 °C, including the final reaction temperature of (175 ± 5) °C. Temperature feedback control provides the primary performance mechanism for the method. Due to the variability in sample matrix types and microwave digestion equipment (i.e. different vessel types and microwave designs), control of the temperature during digestion is important for reproducible microwave heating and comparable data.

The accuracy of the temperature measurement system should be periodically controlled at an elevated temperature according to the manufactures instructions. If the temperature deviates by more than 2 °C from the temperature measured by an external, calibrated temperature measurement system, the microwave temperature measurement system should be calibrated.

6.2 Rotating turntable, with a minimum speed of 3 min^{-1}.

6.3 Sample containers, plastics and glass containers are both suitable.

All containers shall be adequately acid cleaned and stored in order to avoid any contamination.

6.4 Digestion vessels of microwave transparent and reagent and temperature resistant materials, such as fluorocarbon (e.g. perfluoro alkoxyl alkane (PFA), modified polytetrafluoroethene (PTFE)) or quartz.

The vessels may contain layers of different microwave transparent materials for strength, durability and safety. The internal volume shall be at least 45 ml, and the vessel shall be capable of withstanding pressures of at least 3 000 kPa and capable of controlled pressure relief.

The inner wall of the vessel shall be inert and shall not release substances to the digest in excess of the purity requirements of the subsequent analysis. The vessel shall be suitable for the safe application in the temperature and pressure range applied.

Energy regulation of the microwave digestion system shall be based on the temperature in the digestion solutions. Depending on the construction of the unit used temperature is measured indirectly in every vessel, outside the vessels with optical systems or only in one vessel.

All digestion vessels shall be adequately acid cleaned and stored in order to avoid any contamination.

NOTE Digestion vessels may be cleaned in e.g. 10 % nitric acid.

6.5 Filter paper, cellulose-based ashless type, hardened and resistant to nitric acid.

6.6 Filter funnel, glass, polypropene (PP) or other appropriate material.

6.7 Volumetric flask, usually of a nominal capacity of 50 ml or 100 ml.

6.8 Graduated pipettes or dispensers.

6.9 Analytical balance, with an accuracy of 0,1 mg or better.

7 Digestion procedure

Pretreat the test sample according to EN 16179, if not otherwise specified.

Carry out a reagent blank test digestion in parallel with the determination, using the same procedure and the same quantity of nitric acid as in the determination but omitting the test portion.

NOTE 1 The measurement of a blank is introduced to determine the contribution of the extracting solution, glassware and filter paper used to the measured value.

Weigh an amount of no more than 2 g of the test portion (typically 0,5 g to 1 g of dry sample) containing not more than 0,5 g of organic carbon with an accuracy of 0,001 g and transfer it into the digestion vessel (6.4). Add (10 ± 0,1) ml of concentrated nitric acid (5.2) to the digestion vessel with test portion in a fume hood (or fume exhausted enclosure). Swirl and allow the mixture to stand until any visible reaction has stopped.

In case of liquid sludge either refer to EN ISO 15587-2 or perform a suitable drying procedure.

Cap the digestion vessel according to the manufacturer's instructions. Weigh the digestion vessel before digestion. Place in all positions of the microwave carrousel (usually 6, 12, 16 or 40 positions) sample vessels. If a lower number of samples are available compared to the vessel positions, place vessels filled with same amount of nitric acid without sample. This is to ascertain same microwave energy absorption during each digestion procedure. This method is an operationally defined method, designed to achieve consistent digestion of samples by specific reaction conditions. The temperature of the digestion mixture in each vessel shall be raised with a heating rate of approximately 10 °C/min to 15 °C/min to (175 ± 5) °C and remain at (175 ± 5) °C for (10 ± 1) min. Cool down to room temperature.

WARNING — Too high a temperature increase may cause a vigorous, exothermic reaction in digestion solution with high pressure increase and blow off of security valve. Losses of analytes are possible.