SLOVENSKI STANDARD
SIST EN 16174:2013

01-februar-2013

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

Sludge, treated biowaste and soil - Digestion of aqua regia soluble fractions of elements

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

Boue, biodéchet traité et sol - Digestion des éléments solubles dans l'eau régale

Ta slovenski standard je istoveten z: **EN 16174:2012**

ICS:

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

SIST EN 16174:2013 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16174

August 2012

ICS 13.030.01; 13.080.10

English Version

Sludge, treated biowaste and soil - Digestion of aqua regia soluble fractions of elements

Boues, biodéchets traités et sols - Digestion des éléments solubles dans l'eau régale

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

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 16174:2012) 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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

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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 *aqua regia* digestion of sludge, treated biowaste and soil prior to analysis. It is known that the digestion of environmental samples with *aqua regia* will not necessarily lead to a complete element breakdown, and that 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 [19] 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 Industrial sludge Sludge from electronic industry Ink waste sludge Sewage sludge
Biowaste (Method A)	Compost Composted sludge
Soil	Agricultural 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 two methods for digestion of sludge, treated biowaste and soil by the use of *aqua regia* as digestion solution.

This European Standard is applicable for the following elements:

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), rubidium (Rb), rhenium (Re), rhodium (Rh), ruthenium (Ru), samarium (Sm), scandium (Sc), selenium (Se), silicon (Si), silver (Ag), sodium (Na), strontium (Sr), sulphur (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).

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

Digestion with *aqua regia* 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 15936, *Sludge, treated biowaste, soil and waste — Determination of total organic carbon (TOC) by dry combustion*

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 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
aqua regia
digestion solution obtained by mixing one volume of concentrated nitric acid and three volumes of concentrated hydrochloric acid

4 Principle

A test portion is digested with *aqua regia* according to one of the following heating procedures:

— Method A: boiling under reflux for 2 h, followed by filtration and adjusting the volume in a volumetric flask;

— Method B: microwave digestion at (175 ± 5) °C for (10 ± 1) min in a closed vessel followed by filtration and adjusting the volume in a volumetric flask.

5 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.

High acid and solute concentrations in the digest may cause interferences at determination.

Contamination shall be avoided. Glass containing e.g. B, Na, K, Al can contaminate samples.

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

Some elements of interest can be lost due to precipitation with ions present in the digest solution, e.g. low soluble chlorides, fluorides and sulfates.

6 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.

6.1 Water, quality 2 according to EN ISO 3696 or better.

6.2 Hydrochloric acid, c(HCl) = 12 mol/l; ρ = 1,18 kg/l.

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

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

Dilute 35 ml nitric acid (6.3) to 1 l with water (6.1).

6.5 Antifoaming agent, e.g. n-dodecane ($C_{12}H_{26}$) or Octanol ($C_8H_{18}O$) are suitable.

7 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.

7.1 Apparatus used for Method A

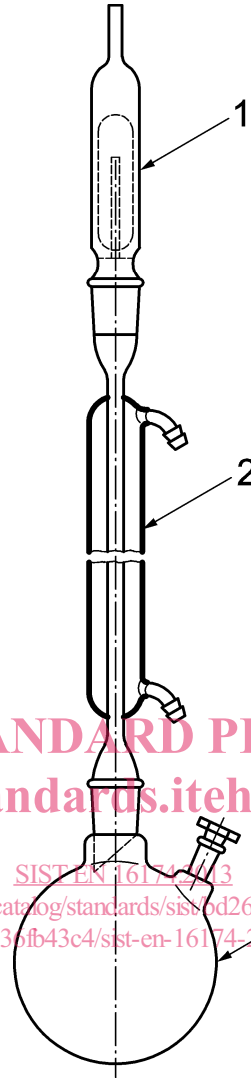
7.1.1 Digestion vessel, see Figure 1, temperature- and pressure-resistant and capable of containing the mixture of sample and digest solution, for example a glass flask of 250 ml. The inner wall of the vessel shall be inert and shall not release substances to the digest in excess of the purity requirements of the subsequent analysis.

NOTE 1 Quartz vessels can be used instead of glass vessels.

NOTE 2 It may be necessary to periodically clean the reaction vessels with a suitable surfactant to remove persistent deposits.

7.1.2 Reflux condenser adaptable to the digestion vessel (7.1.1).

7.1.3 Absorption vessel, volatile species trap, in an open digestion system capable of trapping one or more volatile measurement species, adaptable to the reflux condenser (7.1.2).



Key

1 absorption vessel
2 reflux condenser
3 digestion vessel

Figure 1 — Digestion vessel (7.1.1), reflux condenser (7.1.2) and absorption vessel (7.1.3), assembled

7.1.4 Heating device, for example a heating mantle, thermostatic controlled, or an aluminium block thermostat.