SLOVENSKI STANDARD
SIST-TS CEN/TS 17196:2019

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Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - Razklop z zlatotopko za analizo anorganskih snovi

Construction products - Assessment of release of dangerous substances - Digestion by aqua regia for subsequent analysis of inorganic substances

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Königswasser-Aufschluss zur anschließenden Analyse von anorganischen Stoffen

Produits de construction - Evaluation de l'émission de substances dangereuses - Digestion par l'eau régale pour une analyse ultérieure de substances inorganiques

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TECHNICAL SPECIFICATION

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English Version

Construction products: Assessment of release of dangerous substances - Digestion by aqua regia for subsequent analysis of inorganic substances

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

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European foreword

This document (CEN/TS 17196:2018) has been prepared by Technical Committee CEN/TC 351 “Construction Products: Assessment of release of dangerous substances”, the secretariat of which is held by NEN.

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A similar standard has been developed for soil, sludge and biowaste, see Annex A.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to announce this Technical Specification: 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, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Following an extended evaluation of available methods for content analysis in construction products (CEN/TR 16045) it was concluded that multi matrix and multi-element digestion methods have preference over methods developed for single matrices or small groups of matrices. This implies that for inorganic substances aqua regia digestion is preferred for the digestion of construction products for content analysis.

This document has been adopted from the work carried out in the context of CEN/TC 292 and CEN/TC 400 and is very similar to EN 13657 *Characterization of waste – Digestion for subsequent determination of aqua regia soluble portion of elements* [1] and EN 16174 *Sludge, treated biowaste and soil – Digestion of aqua regia soluble fractions of elements* [2].

This Technical Specification is part of a modular horizontal approach which was adopted in CEN/TC 351. 'Horizontal' means that the methods can be used for a wide range of materials and products with certain properties. 'Modular' means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole chain of measurement (from sampling to analyses). Beneficial features of this approach are that modules can be replaced by better ones without jeopardizing the standard chain and duplication of work of in different Technical Committees for Products can be avoided as far as possible.

The modules that relate to the standards developed in CEN/TC 351 are specified in CEN/TR 16220 [3], which distinguishes between the modules. This Technical Specification belongs to the analytical step.

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain construction product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the entire test procedure.

WARNING — Persons using this Technical Specification should be familiar with usual laboratory practice. The reagents used in this Technical Specification 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 should 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 should 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 Technical Specification be carried out by suitably trained staff.

1 Scope

This Technical Specification specifies methods for obtaining the aqua regia digestible content of construction products. Solutions produced by this method are for analysis by inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled spectrometry (ICP-OES) for the following 67 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).

Solutions produced by the methods are suitable for analysis by cold vapour atomic absorption or fluorescent spectrometry (CV-AAS, CV-AFS), for mercury (Hg).

The method in this Technical Specification is applicable to construction products.

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.

NOTE Construction products include e.g. mineral-based products (S); bituminous products (B); metals (M); wood-based products (W); plastics and rubbers (P); sealants and adhesives (A); paints and coatings (C), see also CEN/TR 16045.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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 17087:—[1], *Construction products: Assessment of release of dangerous substances — Preparation of test portions from the laboratory sample for testing of release and analysis of content*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

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

NOTE The terms and definitions, where relevant, were taken from EN 16687:2015.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/

[1] Under preparation. Stage at the time of publication: prEN 17087:2017.

- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
analyte
determinand
element, ion or substance to be determined by an analytical method

[SOURCE: EN 16687:2015, 4.1.11]

3.2
aqua regia
digestion solution obtained by mixing one volume of concentrated nitric acid and three volumes of concentrated hydrochloric acid

3.3
digestion
mineralization of the organic matter of a sample and dissolution of its mineral part (as completely as possible) when reacted with a reagent mixture

Note 1 to entry: Usually done with a strong, concentrated acid like aqua regia or nitric acid to dissolve inorganic substances for chemical analysis.

[SOURCE: CEN/TR 16045:2010, 2.2.2]

3.4
digestion vessel
flask where the test portion and the acid solution are mixed together and the digestion is carried out

3.5
digest
solution resulting from acid digestion of a sample

[SOURCE: CEN/TR 16045:2010, 2.2.1]

3.6
dry matter
mass fraction of a sample excluding water expressed as mass fraction calculated by determination of dry residue or water content

[SOURCE: EN 15934:2012, 3.3]

3.7
microwave unit
microwave digestion system (oven and associated equipment)

3.8
sample
portion of material selected from a larger quantity of material

Note 1 to entry: The manner of selection of the sample should be prescribed in a sampling plan.

Note 2 to entry: The term “sample” is often accompanied by a prefix (e.g. laboratory sample, test sample) specifying the type of sample and/or the specific step in the sampling process to which the obtained material relates.

[SOURCE: EN 16687:2015, 3.1.5]

3.9
laboratory sample
sample or sub-sample(s) sent to or received by the laboratory

Note 1 to entry: When the laboratory sample is further prepared by subdividing, cutting, sawing, coring, mixing, drying, grinding, and curing or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test/ analysis or for the preparation of a test specimen.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

[SOURCE: EN 16687:2015, 3.2.1]

3.10
test sample
analytical sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

[SOURCE: EN 16687:2015, 3.2.2]

3.11
test portion
analytical portion
amount of the test sample taken for testing/ analysis, usually of known weight or volume

EXAMPLE 1 A bag of aggregates is delivered to the laboratory (the laboratory sample). For test purposes a certain amount of the aggregate is dried, the result is the test sample. Afterwards the column for a percolation test is filled with a test portion of dried aggregate

EXAMPLE 2 A piece of flooring is delivered to the laboratory (the laboratory sample). For the purpose of digestion a certain amount is size reduced, the result is the test sample. From the size-reduced test sample a test portion is taken to execute the digestion. If the digest is to be analysed afterwards e.g. by ICP-MS, the whole amount of the digest is the laboratory sample again (and without any further treatment also the test sample), the amount taken for the analytical procedure the test portion.

[SOURCE: EN 16687:2015, 3.2.3]

4 Symbols and abbreviations

For the purposes of this document, the following abbreviations apply.

CAS	Chemical Abstracts Service
CASRN	CAS Registry Number
CV-AAS	Cold vapour atomic absorption spectrometry
CV-AFS	Cold vapour atomic fluorescence spectrometry
ICP	Inductively coupled plasma
MS	Mass spectrometry
OES	Optical emission spectrometry

PFA	Perfluoroalkoxylalkane
PTFE	Polytetrafluoroethene
SRM	Standard reference material

5 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 if necessary and by 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 if necessary and adjusting the volume in a volumetric flask.

NOTE In the validation testing for other materials (sludge, compost, soil) no significant difference between the reflux and the microwave method was found. So there seems to be no need to prescribe a particular extraction method for construction products.

6 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 thoroughly mixed 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.

7 Reagents

Use only acids and reagents of recognized analytical grade to avoid high blank values for subsequent analytical measurements.

7.1 Water, quality 2 according to EN ISO 3696:1995 or better.

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

Other grade may be used provided it is ascertained that the reagent is of sufficient purity to permit its use without decreasing the accuracy of the subsequent analysis.

7.3 Nitric acid, c(HNO3) = 16 mol/l, ρ = 1,4 kg/l.

7.4 Nitric acid, c(HNO3) = 0,5 mol/l, ρ = 1,0 kg/l.

Dilute 35 ml nitric acid (7.3) to 1 l with water (7.1).

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

8 Apparatus

All glassware and plastic ware shall be adequately cleaned and stored in order to avoid any contamination.

Depending on the concentration of the element of interest, special attention shall be given to the cleaning of the vessels.

8.1 Apparatus used for Method A

8.1.1 Digestion vessel, 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.

8.1.2 Water cooled reflux condenser adaptable to the digestion vessel (8.1.1). The minimum length of the condensor is 340 mm, see EN 13657.

8.1.3 Absorption vessel, volatile species trap, in an open digestion system capable of trapping one or more volatile measurement species, adaptable to the reflux condenser (8.1.2).

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

8.2 Apparatus used for Method B

8.2.1 Digestion vessel, for pressurized microwave digestion, preferably of 100 ml volume, reagent-, temperature- and pressure-resistant and capable of containing the mixture of sample and digest solution. The vessel shall be suitable for the safe application in the temperature and pressure range applied, capable of withstanding pressures of at least 3 000 kPa.

Digestion vessels made of PFA, modified PTFE or quartz glass, and equipped with a safety pressure releasing system to avoid explosion of the vessel, shall be used. The inner wall of the vessel shall be inert and shall not release contaminations to the digest solution.

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

8.2.2 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.