



SLOVENSKI STANDARD SIST EN 17196:2024

01-april-2024

Nadomešča:

SIST-TS CEN/TS 17196:2019

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

Ta slovenski standard je istoveten z: **EN 17196:2023**

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ICS:

13.020.99	Drugi standardi v zvezi z varstvom okolja	Other standards related to environmental protection
91.100.01	Gradbeni materiali na splošno	Construction materials in general

SIST EN 17196:2024

en,fr,de

EUROPEAN STANDARD

EN 17196

NORME EUROPÉENNE

EUROPÄISCHE NORM

November 2023

ICS 91.100.01

Supersedes CEN/TS 17196:2018

English Version

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

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This European Standard was approved by CEN on 14 August 2023.

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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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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (EN 17196:2023) 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.

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of an identical text or by endorsement, at the latest by May 2024, and conflicting national standards shall be withdrawn at the latest by May 2024.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 17196:2018.

In comparison with the previous edition, the following technical modifications have been made:

- the addition of performance data and data from intercomparison validation;
- alignment of terms and definitions within the working groups of CEN/TC 351, i.e. through the revised version of EN 16687.

This document has been prepared under a Standardization Request given to CEN by the European Commission and the European Free Trade Association.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and the United Kingdom.

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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 and EN 16174.

This document 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, which distinguishes between the modules. This document 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 document should be familiar with usual laboratory practice. The reagents used in this document 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 can 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.

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.

The use of this document may involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate measures to ensure the safety and health of personnel prior to application of the document and to fulfil statutory and regulatory requirements for this purpose.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document 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), caesium (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 document is applicable to construction products.

Digestion with *aqua regia* will not necessarily accomplish total decomposition of the sample. The extracted analyte concentrations might 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, *Soil, waste, treated biowaste and sludge — Determination of total organic carbon (TOC) by dry combustion*, <https://standards.iteh.ai/catalog/standards/sist/40b765fb-4b13-4d23-a6c8-f6ff3bc35df6/sist-en-17196-2024>

EN 16687:2023, *Construction products: Assessment of release of dangerous substances — Terminology*

EN 17087, *Construction products: Assessment of release of dangerous substances — Preparation of test portions from the laboratory sample for testing of release and analysis of content*

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 16687:2023 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

analyte

determinant

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

[SOURCE: EN 16687:2023, 3.3.1.11]

3.2

aqua regia

solution obtained by mixing one volume of nitric acid and three volumes of hydrochloric acid

[SOURCE: EN 16687:2023, 3.2.2.10]

3.3

digest

solution resulting from acid digestion of a sample

[SOURCE: EN 16687:2023, 3.2.2.8]

3.4

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

[SOURCE: EN 16687:2023, 3.2.2.9 – modified, Note 1 to entry removed]

3.5

digestion vessel

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

[SOURCE: EN 16687:2023, 3.2.2.11]

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 16687:2023, 3.2.2.15]

3.7

laboratory sample

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

[SOURCE: EN 16687:2023, 3.2.2.1 – modified, Notes to entry removed]

3.8**microwave unit**

microwave digestion system (oven and associated equipment)

[SOURCE: EN 16687:2023, 3.2.2.12]

3.9**sample**

portion of material selected from a larger quantity of material

[SOURCE: EN 16687:2023, 3.2.1.5 – modified, Notes to entry removed]

3.10**test portion**

analytical portion

amount of the test sample taken for testing/analysis purposes, usually of known dimension, mass or volume

[SOURCE: EN 16687:2023, 3.2.2.3 – modified, Notes to entry removed]

3.11**test sample**

analytical sample

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

[SOURCE: EN 16687:2023, 3.2.2.2]

4 Abbreviations

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

CV-AAS Cold vapour atomic absorption spectrometry

CV-AFS Cold vapour atomic fluorescence spectrometry

ICP Inductively coupled plasma

LOD Limit of detection

MS Mass spectrometry

OES Optical emission spectrometry

PFA Perfluoroalkoxy alkanes

PTFE Polytetrafluoroethylene

QC Quality control

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;

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- Method B: microwave digestion at $(175 \pm 5)^\circ\text{C}$ 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 can 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 sulphates.

7 Reagents

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

7.1 Water, with a specific conductivity not higher than 0,2 mS/m at 25 °C.

7.2 Hydrochloric acid, molar concentration $c(\text{HCl}) = 12$ mol/l; mass concentration $\rho = 1,18$ 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, $c(\text{HNO}_3) = 16$ mol/l, $\rho = 1,4$ kg/l.

7.4 Nitric acid, $c(\text{HNO}_3) = 0,5$ mol/l, $\rho = 1,0$ kg/l.

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

7.5 Antifoaming agent, e.g. n-dodecane ($\text{C}_{12}\text{H}_{26}$) or octanol ($\text{C}_8\text{H}_{18}\text{O}$) 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.

Quartz vessels may be used instead of glass vessels.