

ICS 91.100.01

English Version

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

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

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

This Technical Specification (CEN/TS) was approved by CEN on 9 March 2018 for provisional application.

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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.

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 has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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:—<sup>1</sup>, *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/>

<sup>1</sup> 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.