**SLOVENSKI STANDARD**

# SIST-TS CEN/TS 17200:2019+AC:2019

**01-februar-2019**

**Nadomešča:**

**SIST-TS CEN/TS 17200:2019**

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## Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - Analiza anorganskih snovi po razklopu in v izlužkih - Analiza z masno spektrometrijo z induktivno sklopljeno plazmo (ICP/MS) (vključno s popravkom AC)

Construction products: Assessment of release of dangerous substances - Analysis of inorganic substances in digests and eluates - Analysis by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)

Bauprodukte - Beurteilung der Freisetzung von gefährlichen Stoffen - Analyse von anorganischen Stoffen in Aufschlusslösungen und Eluaten - Analyse mit induktiv gekoppeltem Plasma - Massenspektrometrie (ICP-MS)

Produits de construction - Évaluation des émissions de substances dangereuses - Analyse des substances inorganiques dans les digestats et les éluats - Analyse par spectrométrie de masse avec plasma à couplage inductif

**Ta slovenski standard je istoveten z:** **CEN/TS 17200:2018+AC:2018**

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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-TS CEN/TS 17200:2019+AC:2019** **en,fr,de**

TECHNICAL SPECIFICATION

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**CEN/TS 17200:2018+AC**

December 2018

ICS 91.100.01

Supersedes CEN/TS 17200:2018

English Version

# Construction products: Assessment of release of dangerous substances - Analysis of inorganic substances in digests and eluates - Analysis by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS)

Produits de construction: Évaluation des émissions de substances dangereuses - Analyse des substances inorganiques dans les digestats et les éluats - Analyse par spectrométrie de masse avec plasma à couplage inductif

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This Technical Specification (CEN/TS) was approved by CEN on 2 April 2018 for provisional application and includes Corrigendum issued by CEN on 19 December 2018.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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

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# Contents

# European foreword

This document (CEN/TS 17200:2018+AC: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 supersedes CEN/TS 17200:2018.

This document includes the corrigendum 1 which corrects a value in 6.3.

The start and finish of text introduced or altered by corrigendum is indicated in the text by tags AC⟩ ⟨AC

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

A similar document has been developed for drinking water, surface water and waste water and different types of waste respectively, 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 and eluate analysis in construction products (CEN/TR 16045; [1]) it was concluded that multi element analysis methods have preference over methods developed for single elements or small groups of elements. This implies that for inorganic substances ICP methods are preferred for the analysis of extracts obtained from digestion or eluates obtained from leaching.

This standard has been adopted from the work carried out in the context of CEN/TC 400 (project HORIZONTAL) and is very similar to EN 16171, *Sludge, treated biowaste and soil - Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS)* [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, *Construction products: Assessment of release of dangerous substances – Complement to sampling* [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.

# 1 Scope

This Technical Specification specifies the method for the determination of major, minor and trace elements in aqua regia and nitric acid digests and in eluates of construction products by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS). It refers to 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).

NOTE 1 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 [1].

The working range depends on the matrix and the interferences encountered.

NOTE 2 The limit of detection of most elements will be affected by their natural abundance, ionization behaviour, on abundance of isotope(s) free from isobaric interferences and by contamination (e.g. handling and airborne). Handling contaminations are in many cases more important than airborne ones.

The limit of detection will be higher in cases where the determination is likely to be interfered (see Clause 4) or in case of memory effects (see e.g. EN ISO 17294-1:2006, 8.2).

The method in this Technical Specification is applicable to construction products and validated for the product types listed in Annex B.

# 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.

CEN/TS 16637-2, *Construction products — Assessment of release of dangerous substances — Part 2: Horizontal dynamic surface leaching test*

CEN/TS 16637-3, *Construction products — Assessment of release of dangerous substances — Part 3: Horizontal up-flow percolation test*

CEN/TS 17196, *Construction products: Assessment of release of dangerous substances — Digestion by aqua regia for subsequent analysis of the major, minor and trace elements*

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

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025)*

EN ISO 17294-1:2006, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines (ISO 17294-1:2004)*

# 3 Terms and definitions

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

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

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

**3.1**
**digest**
solution resulting from acid digestion of a sample

[SOURCE: EN 16687:2015, 3.2.8]

**3.2**
**eluate**
solution obtained from a leaching test

[SOURCE: EN 16687:2015, 4.2.7]

**3.3**
**analyte**
**determinand**
element, ion or substance to be determined by an analytical method

[SOURCE: EN 16687:2015, 4.1.11]

**3.4**
**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]

**3.5**
**laboratory sample**
sample or subsample(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]

### 3.6
**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]

### 3.7
**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]

### 3.8
**instrument detection limit**
**IDL**

smallest analyte concentration that can be detected with a defined statistical probability using a contaminant free instrument and a blank calibration solution

Note 1 to entry: Usually determined by three times the repeatability standard deviation (3 × $S$r) calculated from multiple measurements (n > 8) of a solution within a single run

[SOURCE: EN 16687:2015, 4.1.13]

### 3.9
**limit of quantification**
**LOQ**

lowest value of an analyte (determinant) that can be determined with an acceptable level of accuracy and precision, generally determined as three times the limit of detection of the method

[SOURCE: EN 16687:2015, 4.1.14]

### 3.10
**method detection limit**
**MDL**

smallest analyte concentration that can be detected with a specified analytical method including sample preparation with a defined statistical probability

Note 1 to entry: Usually determined by three times the repeatability standard deviation (3 × $S$r) calculated from multiple measurements (n > 8) on different days and in different matrix solutions which contain a low analyte concentration.

[SOURCE: EN 16687:2015, 4.1.12]

# 4 Symbols and abbreviations

| | |
|---|---|
| FEP | Hexafluoroethene propene |
| HDPE | High-density polyethylene |
| ICP | Inductively coupled plasma |
| ICS | Interference check solution |
| IDL | Instrumental detection limit |
| IEC | Inter-element correction |
| LOQ | Limit of quantification |
| MDL | Method detection limit (limit of detection) |
| MS | Mass spectrometry |
| OES | Optical emission spectrometry |
| PFA | Perfluoroalkoxy alkane |
| PTFE | Polytetrafluorethylene |
| PVC | Polyvinylchloride |

# 5 Principle

This method describes the multi-elemental determination of analytes by ICP-MS in (diluted) nitric acid or aqua regia digests. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol is transported by argon gas into the plasma. The ions produced by high temperatures of the plasma are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer, sorted according to their mass-to-charge ratios and quantified with a detector (e.g. channel electron multiplier). Interferences shall be assessed and valid corrections applied. Interference correction shall include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

# 6 Interferences

## 6.1 General

Detailed information on spectral and non-spectral interferences is given in EN ISO 17294-1:2006, 6.1.

## 6.2 Spectral interferences

### 6.2.1 Isobaric elemental interferences

Isobaric elemental interferences are caused by isotopes of different elements of closely matched nominal mass-to-charge ratio and which cannot be separated due to an insufficient resolution of the mass spectrometer in use (e.g. $^{114}$Cd and $^{114}$Sn).

Element interferences from isobars may be corrected for taking into account the influence from the interfering element. The isotopes used for correction shall be free of interference if possible. Correction options are often included in the instrument software.

### 6.2.2 Isobaric molecular and doubly charged ion interferences

Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Examples include $^{40}\mathrm{Ar}^{35}\mathrm{Cl}^+$ and $^{40}\mathrm{Ca}^{35}\mathrm{Cl}^+$ ion on the $^{75}\mathrm{As}$ signal and $^{98}\mathrm{Mo}^{16}\mathrm{O}^+$ ions on the $^{114}\mathrm{Cd}^+$ signal. Natural isotope abundances are available from the literature. However, the most precise coefficients for an instrument will be determined from the ratio of the net isotope signals observed for a standard solution.

The accuracy of these types of equations is based upon the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary with operating conditions. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction shall be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent.

Other possibilities to correct for isobaric and doubly charged ion interferences are the use of an instrument with collision/reaction cell technology or high resolution ICP-MS.

The response of the analyte of interest shall be corrected for the contribution of isobaric molecular and doubly charged interferences if their impact can be higher than three times the instrumental detection limit or higher than half the lowest concentration to be reported.

### 6.3 Non spectral interferences

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the cones.

It is recommended to keep the level of total dissolved solids below 0,2 % (⟦AC⟩ 2,000 g/l ⟨AC⟧) to minimize deposition of solids in the sample introduction system of the plasma torch. An internal standard can be used to correct for physical interferences if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. Other possibilities to minimize non spectral interferences are matrix matching, particularly matching of the acid concentration, and standard addition.

When intolerable physical interferences are present in a sample, a significant suppression of the internal standard signals (to less than 30 % of the signals in the calibration solution) will be observed. Dilution of the sample (e.g. fivefold) will usually eliminate the problem.

## 7 Reagents

### 7.1 General

For the determination of elements at trace and ultra trace level, the reagents shall be of adequate purity. The concentration of the analyte or interfering substances in the reagents and the water should be negligible compared to the lowest concentration to be determined.

**7.2 Water**, complying with grade 1 as defined in EN ISO 3696:1995 for all sample preparations and dilutions.

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