SLOVENSKI STANDARD
SIST-TS CEN/TS 17197:2019+AC:2019

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Nadomešča:

SIST-TS CEN/TS 17197:2019

Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - Analiza anorganskih snovi po razklopu in v izlužkih - Analiza z optično emisijsko spektrometrijo z induktivno sklopljeno plazmo (ICP/OES) (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 - Optical Emission Spectrometry (ICP-OES)

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

Produits de construction - Évaluation des émissions de substances dangereuses - Analyse des substances inorganiques dans les digestats et les éluats - Partie 1: Analyse par plasma inductif couplé – Spectrométrie d'émission optique (ICP OES)

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91.100.01	Gradbeni materiali na splošno	Construction materials in general

SIST-TS CEN/TS 17197:2019+AC:2019 **en,fr,de**

TECHNICAL SPECIFICATION
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CEN/TS 17197:2018+AC

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ICS 91.100.01

Supersedes CEN/TS 17197:2018

English Version

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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.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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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 17197:2018+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 17197:2018.

This document includes the corrigendum 1 which corrects two values in D.4.8 and D.4.9.

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.

Two similar documents have been developed for drinking water, surface water and waste water and different types of waste respectively, see Annex B.

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 document has been adopted from the work carried out in the context of CEN/TC 400 (project HORIZONTAL) and is very similar to EN 16170:2016, *Sludge, treated biowaste and soil - Determination of elements using inductively coupled plasma optical emission spectrometry (ICP-OES)* [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 16220:2011, *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 – Optical Emission Spectrometry (ICP-OES). It refers to the following 44 elements: Aluminium (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), bismuth (Bi), boron (B), cadmium (Cd), calcium (Ca), cerium (Ce), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), lanthanum (La), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), neodymium (Nd), nickel (Ni), phosphorus (P), potassium (K), praseodymium (Pr), samarium (Sm), scandium (Sc), selenium (Se), silicon (Si), silver (Ag), sodium (Na), strontium (Sr), sulphur (S), tellurium (Te), thallium (Tl), thorium (Th), tin (Sn), titanium (Ti), tungsten (W), uranium (U), vanadium (V), zinc (Zn), and zirconium (Zr).

For the determination of low levels of As, Se and Sb, hydride generation may be applied. This method is described in Annex D.

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 [1].

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

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*

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*

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)*

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:

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

- 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
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]

3.6
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]

3.7

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]

3.8

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

ICP	Inductively coupled plasma
ICS	Interference check solution
IDL	Instrumental detection limit
IEC	Inter-element correction
MDL	Method detection limit (limit of detection)
MS	Mass spectrometry
OES	Optical emission spectrometry

5 Principle

This method describes multi-elemental determinations by ICP-OES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Additional interferences and matrix effects shall be recognized and appropriate corrections made; tests for their presence are described.

Alternatively, users may choose multivariate calibration methods (e.g. Multicomponent Spectral Fitting). In this case, point selections for background correction are superfluous since whole spectral regions are processed.

For the determination of low levels of As, Se and Sb, hydride generation may be applied. This method is described in Annex D.

6 Interferences

Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

Background emission and *stray light* can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement shall be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analysed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. The interferences are listed in Table A.1.

Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they shall be reduced by diluting the sample, by using an internal standard or a high solids nebulizer. They can also be minimized by matrix matching particularly by matching the acid concentration.

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (radio-frequency power, observation position, gas flow rate and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition in the uptake tubing or to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The occurrence memory effects depend on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element shall be estimated prior to analysis during method development.

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, $c(HNO_3)$ = 16 mol/l, $\rho \sim$ 1,4 kg/l.

NOTE Nitric acid is available both as ρ(HNO3) = 1,40 kg/l (w(HNO3) = 650 g/kg) and ρ(HNO3) = 1,42 kg/l (w(HNO3) = 690 g/kg). Both are suitable for use in this method provided they have a minimal content of the analytes of interest.

7.4 Hydrochloric acid, $c(HCl)$ = 12 mol/l, $\rho \sim$ 1,18 kg/l.

7.5 Single-element standard stock solutions

Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cu, Fe, Hg, K, La, Li, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, Pr, (total) S, Si, Sb, Sc, Se, Sm, Sn, Sr, Te ,Ti, Th, Tl, U, V, W, Zn, Zr, c = 1 000 mg/l each.

Both single-element stock solutions and multi-element stock solutions with adequate specification stating the acid used and the preparation technique are commercially available. Single element solutions can be made from high purity metals.

These solutions are considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered.

7.6 Multi-element standard stock solutions

7.6.1 General

Depending on the scope, different multi-element calibration solutions may be necessary. In general, when combining multi-element calibration solutions, their chemical compatibility and the possible hydrolysis of the components shall be regarded. Care shall be taken to prevent chemical reactions (e.g. precipitation).

NOTE In multi element standards precipitation of Ag, Ba or Pb can occur; Ag is only stable in high hydrochloric acid concentrations or nitric acid.

The multi-element calibration solutions are considered to be stable for several months, if stored in the dark. This does not apply to multi-element calibration solutions that are prone to hydrolysis, in particular solutions of Bi, Mo, Sn, Sb, Te, W, Hf and Zr.

7.6.2 Multi-element standard stock solution A at the mg/l level may contain the following elements:

Ag, Al, As, B, Ba, Be, Bi, Cd, Ce, Co, Cr, Cs, Cu, La, Li, Mn, Ni, Pb, Rb, Sr, Th, Tl, U, V, Se, Zn.

Use nitric acid for stabilization of standard solution A.

7.6.3 Multi-element standard stock solution B at the mg/l level may contain the following elements:

Mo, Sb, Sn, W, Zr