

SLOVENSKI STANDARD SIST EN 17197:2024

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Nadomešča:

SIST-TS CEN/TS 17197:2019+AC: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)

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

Produits de construction - Évaluation de l'émission de substances dangereuses -Analyse des substances inorganiques dans les éluats et les digestats - Analyse par spectrométrie d'émission optique avec plasma à couplage inductif (ICP-OES)

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iTeh Standards (https://standards.iteh.ai) Document Preview

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English Version

Construction products: Assessment of release of dangerous substances - Analysis of inorganic substances in eluates and digests - Analysis by inductively coupled plasma optical emission spectrometry (ICP-OES)

Produits de construction : Évaluation de l'émission de substances dangereuses - Analyse des substances inorganiques dans les éluats et les digestats - Analyse par spectrométrie d'émission optique avec plasma à couplage inductif (ICP-OES) Bauprodukte: Bewertung der Freisetzung von gefährlichen Stoffen - Analyse von anorganischen Stoffen in Aufschlusslösungen und Eluaten - Analyse mit induktiv gekoppeltem Plasma - Optische Emissionsspektrometrie (ICP-OES)

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

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 17197:2023) 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 17197:2018+AC: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.

Introduction

Following an extended evaluation of available methods for content and eluate analysis in construction products (CEN/TR 16045) 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.

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 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.

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1 Scope

This document specifies the method for the determination of major, minor and trace elements in eluates and in *aqua regia* and nitric acid digests 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, Hg, Se and Sb, chemical vapour generation systems can be applied. This method is described in Annex E (normative).

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.

The method in this document is applicable to construction products and validated for the product types listed in Annex C (informative).

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

EN 16637-3, Construction products: Assessment of release of dangerous substances — Part 3: Horizontal up-flow percolation SISTEN 17197-2024

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

EN 17196, Construction products: Assessment of release of dangerous substances — Digestion by aqua regia for subsequent analysis of inorganic substances

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

agua 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

eluate

solution obtained from a leaching test

[SOURCE: EN 16687:2023, 3.3.2.8]

3.5

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

[SOURCE: EN 16687:2023, 3.3.1.13 – modified, Note 1 to entry removed]

Journal of Standards and Stand

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]

3.7

method detection limit

MDL

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

[SOURCE: EN 16687:2023, 3.3.1.12; modified – Note 1 to entry removed]

3.8

sample

portion of material selected from a larger quantity of material

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

3.9

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, Examples removed]

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 16687:2023, 3.2.2.2]

4 Abbreviations

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

ICP	Inductively coupled plasma
ICS	Interference check solution
IDL	Instrumental detection limit
IEC	Inter-element correction Change Standards
LOD	Limit of detection 11th Standards

LOD Limit of detection

MS Mass spectrometry S://Standards.iteh.ai)

MSF Multicomponent spectral fitting

OES Optical emission spectrometry

QC Quality control

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, Hg, Se and Sb, chemical vapour generation can be applied. This method is described in Annex E (normative).

6 Interferences

6.1 General

Several types of interference effects can contribute to inaccuracies in the determination of elements.

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.

Non-spectral interferences, usually called matrix effects, can have their origin in three different processes or locations: in the nebulization process, in the plasma, and in the interface and the lens area. These types of interferences also include blockage of the nebulizer, torch injector tube and sampling cone caused by high concentrations of dissolved matter or the nebulization of organic solvents.

When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. The interferences are listed in Annex A (informative).

Detailed information on spectral and non-spectral interferences is given in Clause 6 of EN ISO 11885:2009.

6.2 Spectral interferences

6.2.1 Background emission and stray light

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 can 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.

6.2.2 Spectral overlaps

Spectral overlaps can 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.

6.3 Non-spectral interferences

6.3.1 Physical interferences

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.

6.3.2 Chemical interferences

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.

6.3.3 Memory interferences

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 of 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 shall be recognized within an analytical run and suitable rinse times shall be used to reduce them. The rinse times necessary for a particular element shall be estimated prior to analysis during method development.

7 ht **Reagents** ds.iteh.ai/catalog/standards/sist/5b491382-cede-459f-b031-d43e89a166c1/sist-en-17197-2024

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 shall be negligible compared to the lowest concentration to be determined.

- **7.1 Water,** with a specific conductivity not higher than 0,2 mS/m at 25 °C.
- **7.2 Nitric acid,** molar concentration $c(\text{HNO}_3) = 16 \text{ mol/l}$, mass concentration $\rho \sim 1.4 \text{ kg/l}$.

NOTE Nitric acid is available both as $\rho(\text{HNO}_3) = 1,40 \text{ kg/l}$ ($w(\text{HNO}_3) = 650 \text{ g/kg}$) and $\rho(\text{HNO}_3) = 1,42 \text{ kg/l}$ ($w(\text{HNO}_3) = 690 \text{ g/kg}$). Both are suitable for use in this method provided they have a minimal content of the analytes of interest.

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