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**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 digestats et les éluats - Analyse par spectrométrie d'émission optique avec plasma à couplage inductif (ICP-OES)

**Ta slovenski standard je istoveten z: prEN 17197**

**ICS:**

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91.100.01	Gradbeni materiali na splošno	Construction materials in general

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## 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)

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This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 351.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

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

<b>Contents</b>	<b>Page</b>
European foreword.....	4
Introduction .....	5
1 Scope.....	6
2 Normative references.....	6
3 Terms and definitions .....	6
4 Abbreviations .....	8
5 Principle .....	8
6 Interferences .....	9
7 Reagents .....	9
8 Apparatus.....	11
9 Procedure.....	12
9.1 Test sample .....	12
9.2 Test portion.....	12
9.3 Instrument set up.....	12
9.3.1 General requirements .....	12
9.3.2 Inter-element correction.....	13
9.3.3 Internal standard .....	13
9.3.4 Instrument performance check.....	13
9.4 Calibration .....	13
9.4.1 Linear calibration function.....	13
9.4.2 Nonlinear calibration function.....	14
9.4.3 Standard addition calibration.....	14
9.5 Sample measurement.....	14
10 Calculation.....	15
10.1 Calculation for digests of construction products.....	15
10.2 Calculation for eluates of construction products .....	15
11 Expression of results.....	15
12 Performance characteristics.....	15
12.1 General.....	15
12.2 Blank.....	15
12.3 Calibration check .....	15
12.4 Interference .....	15
12.5 Recovery .....	16
12.6 Indicative values for MDL.....	16
12.7 Precision.....	16
13 Test report.....	17
Annex A (informative) Wavelengths, spectral interferences and estimated method detection limits.....	18
Annex B (informative) Indicative values for MDL .....	22

<b>Annex C (informative) Precision data for analysis of eluates and digests from construction products .....</b>	<b>23</b>
<b>C.1 Precision data for analysis of eluates from construction products.....</b>	<b>23</b>
<b>C.2 Precision data for analysis of digests from construction products.....</b>	<b>30</b>
<b>Annex D (informative) Inter element correction (IEC).....</b>	<b>37</b>
<b>Annex E (normative) Determination of arsenic, antimony and selenium using hydride generation ICP-OES.....</b>	<b>39</b>
<b>E.1 Scope .....</b>	<b>39</b>
<b>E.2 Principle.....</b>	<b>39</b>
<b>E.3 Apparatus.....</b>	<b>39</b>
<b>E.4 Reagents.....</b>	<b>40</b>
<b>E.5 Procedure.....</b>	<b>41</b>
<b>E.6 Calculation .....</b>	<b>42</b>
<b>Bibliography .....</b>	<b>43</b>

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**prEN 17197:2022 (E)****European foreword**

This document (prEN 17197:2022) 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 document is currently submitted to the CEN Enquiry.

This document will supersede CEN/TS 17197:2018+AC:2018.

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

- updated normative references and bibliography;
- addition of performance data for construction products in Clause 12 and in Annex C;
- removal of performance data for soil, sludge and biowaste.

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

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## 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 [2].

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 16220:2011 [3] 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 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 can be applied. This method is described in Annex E.

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.

## 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.

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

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

EN 16687:2015, *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*

prEN 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 16687:2015 and the following apply.

ISO and IEC maintain terminological 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****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]

**prEN 17197:2022 (E)****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 ( $3 \times S_r$ ) 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 ( $3 \times S_r$ ) 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 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
MDL	Method detection limit (limit of detection)
MSF	Multicomponent spectral fitting
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 E.

## 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 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.

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

*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 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 Reagents

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.

**prEN 17197:2022 (E)**

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

**7.4 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,  $\rho = 1\ 000 \text{ mg/l}$  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 shall be considered.

**7.5 Multi-element standard stock solutions.**

**7.5.1 General**

Depending on the scope, different multi-element calibration solutions can 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.

**7.5.2 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.

**7.5.3 Multi-element standard stock solution B** at the mg/l level may contain the following elements: Mo, Sb, Sn, W, Zr.

Use hydrochloric acid (7.3) for stabilization of multi-element standard stock solution B. Other elements of interest may be added to the standard stock solution, provided that the resulting multi-element solution is stable.

**7.5.4 Multi-element standard stock solution C** at the mg/l level may contain the following elements: Ca, Mg, Na, K, P, S.

Use nitric acid (7.2) for stabilization of multi-element standard stock solution C.

**7.6 Multi-element calibration solutions.**

Prepare in one or more steps calibration solutions at the highest concentration of interest. If more concentration levels are needed, prepare those similarly in an equidistant concentration range.

Add acids (7.2 and/or 7.3) to match the acid concentration of samples closely.

If traceability of the values is not established check the validity by comparison with a (traceable) independent standard.

Check the stability of the calibration solutions.

### 7.7 Internal standard solution.

The use of an internal standard can be a suitable method to correct for non-spectral interferences. The approach involves the addition of a known amount of a substance to the sample and calibration solutions. The ratio of responses of the analyte and the internal standard are measured in the sample and calibration solution. The observation for the internal standard is used to relate the analyte signal to the analyte concentration.

The choice of elements for an internal standard solution depends on the analytical problem. The solution of this/these internal standard(s) shall cover the mass range of interest. The concentrations of the selected elements (used as internal standard) shall be negligibly low in the digests of samples. The elements Sc, Y, In and Rh have been found suitable for this purpose.

Generally, a suitable concentration of the internal standard in samples and calibration solutions is 1 mg/l to 10 mg/l.

### 7.8 Calibration blank.

Prepare the calibration blank by acidifying water (7.1) to the same concentrations of the acids found in the calibration solutions, digests and eluates.

### 7.9 Method blank.

The method blank shall contain all of the reagents in the same volumes as used in the processing of the samples. The method blank shall be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

### 7.10 Calibration check solution.

Prepare the calibration check solution, purchased from a different supplier, by acidifying water (7.1) to the same concentration of the acids used in the calibration solutions, using the same standards used for calibration at an upper concentration level.

### 7.11 Interference check solution (ICS).

If interference cannot be excluded (see Table A.1) prepare the interference check solution to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. The choice of the concentration and interfering element are matrix dependent.

Avoid two or more interferences for an analyte in the same interference check solution. Spike the sample with the analytes of interest, particularly those with known interferences at 0,5 mg/l to 1 mg/l. In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

## 8 Apparatus

### 8.1 Inductively coupled argon plasma emission spectrometer.

- computer-controlled emission spectrometer with background correction;
- radio-frequency generator;