SLOVENSKI STANDARD
oSIST prEN 17201:2022

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Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - Vsebnost anorganskih snovi - Metode za analizo po razklopu z zlatotopko

Construction products: Assessment of release of dangerous substances - Content of inorganic substances - Methods for analysis of aqua regia digests

Bauprodukte: Bewertung der Freisetzung von gefährlichen Stoffen - Gehalt an anorganischen Stoffen - Verfahren zur Analyse von Königswasseraufschlusslösungen

Produits de construction - Évaluation du relargage de substances dangereuses - Teneur en substances inorganiques - Méthodes d'analyse de digestats obtenus par digestion à l'eau régale

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91.100.01	Gradbeni materiali na splošno	Construction materials in general

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English Version

Construction products: Assessment of release of dangerous substances - Content of inorganic substances - Methods for analysis of aqua regia digests

Produits de construction - Évaluation du relargage de substances dangereuses - Teneur en substances inorganiques - Méthodes d'analyse de digestats obtenus par digestion à l'eau régale

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

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European foreword

This document (prEN 17201:2022) 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 17201:2018+AC:2018.

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

Introduction

Following an extended evaluation of available methods for content analysis in construction products (CEN/TR 16045, [1]) it was concluded that eluate analysis methods are very similar to analytical methods used to determine content after digestion of a solid matrix.

This document has a similar structure as prEN 17195.

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. Similar standards have been developed for content determination in different types of matrices, see Annex A.

The modules that relate to the standards developed in CEN/TC 351 are specified in CEN/TR 16220 [2], 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.

1 Scope

This document specifies analytical methods for the determination of major, minor and trace elements in aqua regia digests of construction products. 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).

The methods in this document are applicable to construction products.

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 selection of analytical methods to be applied is based on the required sensitivity of the method, which is provided for all combinations of substance and analytical procedure.

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 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*

prEN 17197, *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)*

prEN 17200, *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)*

EN ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples (ISO 5667-3)*

EN ISO 12846:2012, *Water quality — Determination of mercury — Method using atomic absorption spectrometry (AAS) with and without enrichment (ISO 12846:2012)*

EN ISO 15586:2003, *Water quality — Determination of trace elements using atomic absorption spectrometry with graphite furnace (ISO 15586:2003)*

EN ISO 17852:2008, *Water quality — Determination of mercury — Method using atomic fluorescence spectrometry (ISO 17852:2006)*

ISO 17378-1:2014, *Water quality — Determination of arsenic and antimony — Part 1: Method using hydride generation atomic fluorescence spectrometry (HG-AFS)*

ISO 17378-2:2014, *Water quality — Determination of arsenic and antimony — Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)*

ISO/TS 17379-1:2013, *Water quality — Determination of selenium — Part 1: Method using hydride generation atomic fluorescence spectrometry (HG-AFS)*

ISO/TS 17379-2:2013, *Water quality — Determination of selenium — Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)*

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
digestion
mineralisation 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: EN 16687:2015, 3.2.9]

3.3
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, mixing, diluting, etc. 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.

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]

3.4
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 × *Sr*) 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]

3.5
sample
portion of material selected from a larger quantity of material

Note 1 to entry: The manner of selection of the sample should be described 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]

3.6
test portion
analytical portion
amount of the test sample taken for testing/analysis purposes, 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, after any further treatment (e.g. dilution) results the test sample and the amount taken for the analytical procedure the test portion.

[SOURCE: EN 16687:2015, 3.2.3]

3.7
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]

4 Abbreviations

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

CV-AAS	Cold vapour atomic absorption spectrometry
CV-AFS	Cold vapour atomic fluorescence spectrometry
GF-AAS	Graphite furnace atomic absorption spectrometry
HG-AAS	Hydride generation atomic absorption spectrometry
ICP	Inductively coupled plasma
MDL	Method detection limit (limit of detection)
MS	Mass spectrometry
OES	Optical emission spectrometry

5 Sample pre-treatment

The digestion of the construction products is executed according to prEN 17196.

The laboratory sample as obtained from sampling as specified in product specific requirements complemented with requirements as set out in CEN/TR 16220 [2], shall be prepared for digestion according to the specifications provided in the aqua regia digestion method prEN 17196.

If necessary apply sample pre-treatment as specified in EN 17087.

Split the digest in an adequate number of test portions for different chemical analyses and preserve them according to the requirements in EN ISO 5667-3.

6 Selection of suitable analytical test method

6.1 Table of test methods

Select the appropriate standardized test method listed in Table 1 to analyse the digest resulting from the aqua regia digestion according to the concentration range of the parameter of interest, regulatory requirements, the expected interferences and the precision needed.

For analytical quality control purposes ISO/TS 13530 and EN ISO/IEC 17025 should be considered.

6.2 General validation information

A selection of the test methods listed in Table 1 has been validated in robustness validation [4, 5] and in an interlaboratory trial for a limited number of construction product matrices [6]. Their suitability for other construction product digests shall be checked in the laboratory performing the analysis.

It is pointed out that the standardized test methods listed in Table 1 have primarily been developed for the analysis of water samples. Most of them were validated by CEN/TC 223, CEN/TC 292 and CEN/TC 308 in interlaboratory trials for a limited number of digests from various matrices. The validation data on these other matrices obtained in the evaluation of the analytical performance of laboratories are referenced in the bibliography.

If the methods referred to in Table 1 are found to be inappropriate by reason of, for example, detection limits, repeatability or interferences, other methods validated for water analysis may be used. Their suitability for construction product eluates shall be checked in the laboratory performing the analysis. The reason for the deviation shall be stated in the test report.

Indicative values for MDL are listed in Table B.1 in Annex B. The values for MDL as listed in Annex B are indicative values in the sense that they are not to be used as sole guideline to select between methods to be applied. An actual MDL will depend on the equipment, matrix properties, interferences, and laboratory experience. Detection limits also depend on the amount of sample processed. If based on better precision for the listed methods another MDL is chosen than the MDL that would result from applying the tabulated values, this shall be mentioned in the test report.

Table 1 — Substances and test methods for digests

Parameter	Test method	Method type
Major, minor and trace elements		
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, Sb, Sc, Se, Si, Sm, Sn, Sr, Te, Th, Ti, Tl, U, V, W, Zn and Zr	prEN 17197	ICP-OES
Ag, Al, As, Au, Ba, Be, Bi, B, Cd, Ce, Ca, Cr, Co, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Hg, Ho, In, Ir, K, La, Li, Lu, Mg, Mo, Mn, Na, Nd, Ni, P, Pb, Pd, Pr, Pt, Rb, Re, Rh, Ru, Total S, Sb, Sc, Se, Si, Sm, Sn, Sr, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Yb, Zn and Zr	prEN 17200	ICP-MS
More sensitive methods	**Test method**	**Method type**
As, Sb	ISO 17378-1:2014	HG-AFS
As, Sb	ISO 17378-2:2014	HG-AAS
Cd	EN ISO 15586:2003	GF-AAS
Hg	EN ISO 12846:2012	CV-AAS
	EN ISO 17852:2008	CV-AFS
Sb, Se	EN ISO 15586:2003	GF-AAS
Se	ISO/TS 17379-1:2013	HG-AFS
Se	ISO/TS 17379-2:2013	HG-AAS

6.3 Content in mg/kg

In Annex B the analytical sensitivity in terms of MDL is given in µg/l. To convert this value to an indicative value for MDL on content (mg of the substance under consideration per kg of dry solid) a multiplication factor of 500 is used, which is based on the sample mass used and the dilutions of the digest made before measurement. So for example a MDL of 0,1 µg/l means a MDL value of 0,05 mg/kg on content.

The conversion to a concentration in mg of the substance under consideration per kg of mass of the construction product under investigation (concentration in the dry matter) shall be done in line with the specifications from the microwave digestion method that has been used.