

SLOVENSKI PREDSTANDARD

SIST ENV 196-4

prva izdaja
september 1995

Metode preskušanja cementa - 4. del: Kvantitativno določanje sestavin (prevzet predstandard ENV 196-4:1993 z metodo platnice)

Methods of testing cement - Part 4: Quantitative determination of constituents

Méthodes d'essais des constituants - Partie 4: Détermination quantitative des constituants

Prüfverfahren für Zement - Teil 4: Quantitative Bestimmung der Bestandteile

Deskriptorji: cement, določanje, sestavine, žindre, cementni klinker, pucolani, pepeli, preskusi

ICS 91.100.10

Referenčna številka
SIST ENV 196-4:1995 (en)

Nadaljevanje na straneh od II do III in 1 do 39

UVOD

Predstandard SIST ENV 196-4 (en), Metode preskušanja cementa - 4. del: Kvantitativno določanje sestavin, prva izdaja, 1995, ima status slovenskega predstandarda in je z metodo platnice prevzet evropski predstandard ENV 196-4, Methods of testing cement - Part 4: Quantitative determination of constituents, 1993, v angleškem jeziku.

NACIONALNI PREGOVOR

Evropski predstandard ENV 196-4:1993 je pripravil tehnični odbor Evropske organizacije za standardizacijo CEN/TC 51 Cement in apno.

Odločitev za prevzem tega predstandarda po metodi platnice je sprejela delovna skupina USM/TC CAA/WG 1 Cement, potrdil pa tehnični odbor USM/TC CAA Cement, apno in vlaknatocementni izdelki.

Ta slovenski predstandard je dne 1995-09-01 odobril direktor USM.

SLOVENSKI STANDARD SIST EN 196 ZA PRESKUŠANJE CEMENTA OBSEGA NASLEDNJE DELE:

SIST EN 196-1:1995 (en)	Metode preskušanja cementa - 1. del: Določanje trdnosti
SIST EN 196-2:1995 (en)	Metode preskušanja cementa - 2. del: Kemijska analiza cementa
SIST EN 196-3:1995 (en)	Metode preskušanja cementa - 3. del: Določanje časa vezanja in prostorninske obstojnosti
SIST ENV 196-4:1995 (en)	Metode preskušanja cementa - 4. del: Kvantitativno določanje sestavin
SIST EN 196-5:1995 (en)	Metode preskušanja cementa - 5. del: Določanje pucolanske aktivnosti za pucolanske cemente
SIST EN 196-6:1995 (en)	Metode preskušanja cementa - 6. del: Določanje finosti
SIST EN 196-7:1995 (en)	Metode preskušanja cementa - 7. del: Metode odvzemanja in priprave vzorcev cementa
SIST EN 196-21:1995 (en)	Metode preskušanja cementa - 21. del: Določanje količine kloridov, ogljikovega dioksida in alkalij v cementu

OSNOVA ZA IZDAJO PREDSTANDARDA

- Prevzem predstandarda ENV 196-4:1993

OPOMBI

- Povsod, kjer se v besedilu predstandarda uporablja izraz "evropski predstandard", v SIST ENV 196-4:1995 to pomeni "slovenski predstandard".
- Uvod in nacionalni predgovor nista sestavni del predstandarda.

VSEBINA	stran
1 Obseg in področje uporabe	4
2 Zveze z drugimi standardi	4
3 Splošne zahteve pri preskušanju.....	5
3.1 Število preskusov.....	5
3.2 Določanje stalne mase	6
3.3 Podajanje količin in rezultatov	6
3.4 Ponovljivost in reproducirnost.....	6
4 Priprava vzorca cementa	7
5 Reagenti.....	7
6 Določanje količine sestavin v cementih.....	7
6.1 Splošno.....	7
6.2 Postopek selektivnega raztapljanja	8
7 Določanje količine sestavin v cementih s tremi sestavinami.....	17
7.1 Splošno.....	17
7.2 Določanje količine žindre visokih peči.....	17
7.3 Določanje količine elektrofiltrskega pepela.....	29
7.4 Določanje količine pucolana	31

iTech STANDARD PREVIEW
(standards.itteh.ai)
SIST ENV 196-4:1995
<https://standards.itteh.ai/catalog/standards/sist/989f64d6-6dc3-487f-9d53-3d7c3e36cb51/sist-env-196-4-1995>

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST ENV 196-4:1995

<https://standards.iteh.ai/catalog/standards/sist/989f64d6-6dc3-487f-9d53-3d7c3e36cb51/sist-env-196-4-1995>

EUROPEAN PRESTANDARD

ENV 196-4:1993

PRÉNORME EUROPÉENNE

EUROPÄISCHE VORNORM

July 1993

UDC 666.94:691.54:620.1:539.215

Supersedes ENV 196-4:1989

Descriptors: Cement, determination, components, slags, cement clinker, pozzolans, ashes, tests

English version

**Methods of testing cement - Part 4: Quantitative
determination of constituents**

iTeh STANDARD PREVIEW
(standards.iteh.ai)

Méthodes d'essais des ciments - Partie 4:
Détermination quantitative des constituants

Prüfverfahren für Zement - Teil 4: Quantitative
Bestimmung der Bestandteile

[ST ENV 196-4:1995](https://standards.iteh.ai/catalog/standards/sist/989f64d6-6dc3-487f-9d53-3d7c3e36cb51/sist-env-196-4-1995)

<https://standards.iteh.ai/catalog/standards/sist/989f64d6-6dc3-487f-9d53-3d7c3e36cb51/sist-env-196-4-1995>

This European Prestandard (ENV) was approved by CEN on 1993-01-08 as a prospective standard for provisional application. The period of validity of this ENV 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 ENV can be converted into an European Standard (EN).

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

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Contents list		Page
	Foreword	3
1	Scope	4
2	Normative references	4
3	General requirements for testing	5
3.1	Number of tests	5
3.2	Determination of constant mass	6
3.3	Expression of masses and results	6
3.4	Repeatability and reproducibility	6
4	Preparation of a cement sample	7
5	Reagents	7
6	Determination of the contents of cement constituents	7
6.1	General	7
6.2	Selective dissolution method	8
6.2.1	Principle	8
6.2.2	Reagents	9
6.2.3	Apparatus	9
6.2.4	Procedure	10
6.2.5	Calculation of the contents of cement constituents	13
6.2.6	Repeatability and reproducibility	16
6.2.7	Qualitative determination of cement type	16
7	Determination of the contents of constituents for cements with three constituents	17
7.1	General	17
7.2	Determination of the slag content	17
7.2.1	Dense liquid separation method	17
7.2.2	Microscope method	24
7.3	Determination of the fly ash content	29
7.3.1	Selective dissolution method	29
7.4	Determination of the pozzolana content	31
7.4.1	Selective dissolution method	31
7.4.2	Dense liquid separation method	34

Foreword

This European Prestandard was drawn up by Technical Committee CEN/TC 51 "Cement and building lime" the Secretariat of which is held by IBN. It is a revision of the European Prestandard ENV 196-4 of December 1989.

The main aim of this revision was to adapt the reference method in such a way that it would be qualitative and quantitative whatever the constituent materials, including blastfurnace slag (which was not the case for ENV 196-4 : 1989). This entailed recasting the analytical procedure and the calculation of the contents.

The opportunity was taken at the same time to unify the presentation of the different methods, reference and alternative, and to endeavour to standardize better the formation of the symbolic notations so that all ambiguity in the interpretation of the formulae for the calculations was ruled out.

According to the Common CEN/CENELEC Rules, the following countries are bound to announce this European Prestandard :

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

The European Standard EN 196 on methods of testing cement consists of the following Parts:

- SIST ENV 196-4:1995
<https://standards.iteh.ai/>
<https://standards.iteh.ai/standards/sist/989f64d6-6dc3-487f-9d53-3d7c3e36cb51/sist-env-196-4-1995>
- Part 1 : Determination of strength
 - Part 2 : Chemical analysis of cement
 - Part 3 : Determination of setting time and soundness
 - Part 4 : Quantitative determination of constituents
 - Part 5 : Pozzolanicity test for pozzolanic cements
 - Part 6 : Determination of fineness
 - Part 7 : Methods of taking and preparing samples of cement
 - Part 21: Determination of the chloride, carbon dioxide and alkali content of cement

1 Scope

This European Prestandard lays down the procedures for determining the contents of the constituents of cements that fall within the scope of ENV 197-1.

The first method outlined in clause 6 applies to all cements, whatever the number and nature of their constituents. This is a selective dissolution method which is to be considered as the reference method where the various constituents, generally of an unknown number, are not available separately at the same time as the cement, which is usually the case.

Any other method with the same objectives can be considered as an alternative to the reference method when it is shown that, with valid statistical calculation, it gives equivalent results.

In individual cases where the laboratory has been formally advised that:

- the cement contains only two constituents, the method is greatly simplified because it is sufficient to determine the set regulator content (R) to know the clinker content;
- the cement contains only three constituents, i.e. a set regulator, clinker and one of the following three: slag, pozzolana or fly ash. One of the methods given in clause 7 can then be applied. All the methods in this clause are variations of the reference method described in clause 6.

NOTE: In the different methods that this standard comprises, the term "granulated blastfurnace slag" defined in 4.2 of ENV 197-1 : 1992 is designated by the term "slag".

2 Normative references

This European Prestandard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 196-2 : 1987 Methods of testing cement - Part 2: Chemical analysis of cements

EN 196-7 : 1989 Methods of testing cement - Part 7: Methods of taking and preparing samples of cement

EN 196-21 : 1989 Methods of testing cement - Part 21: Determination of the chloride, carbon dioxide and alkali contents of cement

ENV 197-1 : 1992 Cement: Composition, specifications and conformity criteria
Part 1: Common cements

ISO 3534 : 1977 Statistics - Vocabulary and symbols

3 General requirements for testing

3.1 Number of tests

To carry out the calculation for the contents of the cement constituents, two tests shall be made for each. The following analytes are determined:

- for the general method by selective dissolution (clause 6): sulfuric anhydride and carbon dioxide contents, residues after EDTA and nitric acid dissolution and sulfide contents in the cement and in the EDTA residue;

- for the methods of analysis for cements with three constituents (clause 7): sulfuric anhydride and carbon dioxide contents, loss on ignition, calcium oxide, magnesium oxide and manganese oxide contents, sulfide contents and insoluble residues. Depending on the method used, only some of these analytes can be determined.

If, for each analyte, the difference between the two values obtained is less than twice the standard deviation for repeatability for this analyte, the value to take for further calculations is the arithmetic mean of the two values. If the difference between the two values is greater than twice the standard deviation for repeatability, a third test shall be carried out and the value to be taken for further calculations shall be the arithmetic mean of the two closest values.

Hence, for the general method by selective dissolution, only one calculation will need to be carried out for the quantitative determination of constituents, in particular for clinker.

Likewise, for the methods of analysis for cements with three constituents, a single calculation will enable the content of hydraulic or pozzolanic constituent to be determined.

The standard deviations for repeatability of the various analytes to be considered, most of which can be found in EN 196-2: 1987, are as follows:

Analyte content	Standard deviation for repeatability s_r in percent
SO ₃	0,07
CO ₂	0,07
a (EDTA residue)	0,50
b (HNO ₃ residue)	0,11
S ²⁻	0,02
CaO	0,18
MgO	0,15
MnO	0,003
insoluble residue	0,10
loss on ignition	0,04

3.2 Determination of constant mass

Constant mass shall be determined by making successive 15 min ignitions, followed each time by cooling and then by weighing. Constant mass is reached when the difference between two successive weighings is less than 0,0005 g.

<https://standards.iteh.ai/catalog/standards/sist/989f64d6-6dc3-487f-9d53-3d7c3e36cb51/sist-env-196-4-1995>

3.3 Expression of masses and results

Express masses in grams to the nearest 0,0001 g.

Express the values for the analytes, given by the mean of two determinations (see 3.1) as well as the contents of constituents calculated in percentages, to one decimal place.

3.4 Repeatability and reproducibility

The standard deviation for repeatability gives the closeness of agreement between successive results obtained with the same method on identical material tested under the same conditions (same operator, same apparatus, same laboratory and short time interval)¹⁾.

The standard deviation for reproducibility gives the closeness of agreement between individual results obtained with the same method on identical material, but tested under different conditions (different operators, different apparatus, different laboratories and/or different times)¹⁾.

1) Definitions taken from ISO 3534 : 1977.

The standard deviations for repeatability and reproducibility are expressed in % and relate to clinker contents for the general method of determination of the constituents by selective dissolution and to hydraulic and pozzolanic contents for the methods of analysis of cement with three constituents.

4 Preparation of a cement sample

Prior to analysis, the laboratory sample obtained in accordance with the provisions of EN 196-7 shall be treated to obtain a test sample.

This final treatment of the sample differs according to the methods used and is specified at the start of each procedure (see 6.2.4.1, 7.2.1.4.1, 7.2.2.3.1, 7.3.1.4.1, 7.4.1.4.1 and 7.4.2.4.1).

5 Reagents

Use only reagents of analytical quality and distilled water or water of equal purity during the analysis.

Unless otherwise specified, "%" means "% by mass"

The density " ρ " of liquids is given at 20 °C. The densities of concentrated liquid reagents are expressed in g/cm³.

The degree of dilution is always given in the form of a volumetric sum, for example, nitric acid (1+2) means that 1 volume of concentrated nitric acid has to be mixed with 2 volumes of water.

6 Determination of the contents of cement constituents

6.1 General

This method applies to cements with several constituents, i.e.:

- clinker;
- hydraulic, pozzolanic or inert constituents;
- set regulator(s).

The constituents determined by this method are classified as follows:

- set regulator;
- clinker;
- slag;

- calcareous additions (for example calcareous filler, either as: raw meal, limestone, chalk, kiln dust);

- siliceous additions (for example, siliceous filler either as: flint, natural pozzolana, fly ash).

Their number is therefore five in principle, but the additions, mainly calcareous or siliceous, can contain one or more components of the same nature although it may not be possible to identify them separately.

Should there be a need to define the nature of the additions more precisely, the inspection body or, failing this, any independent body commissioned by the interested parties to carry out the analyses, will proceed with further enquiries with the manufacturer of the cement as required in 6.2.5.4.

The results obtained from the application of this method are quantitatively valid, whatever the relative importance of the constituents identified.

6.2 Selective dissolution method

6.2.1 Principle

Following preparation, one fraction of the cement sample is treated with an EDTA solution and another fraction is treated with dilute nitric acid (see table 1).

SIST ENV 196-4:1995
<https://standards.iteh.ai/canonical/standards/sist/989f64d6-6dc3-487f-9d53-3d7c3e36cb51/sist-env-196-4-1995>
 Table 1

Reagent	Dissolved	Not dissolved
EDTA solution	Set regulator(s) Clinker Carbonate containing filler(s)	Slag Pozzolana Fly ash Siliceous filler(s)
Dilute nitric acid	Set regulator(s) Clinker Slag Carbonate containing filler(s)	Pozzolana Fly ash Siliceous filler(s)

The results from these two selective dissolutions and the additional determinations of the sulfuric anhydride and carbon dioxide contents of the cement together with the sulfide contents of the cement and the residue from the dissolution in the EDTA solution enable the contents of the various constituents to be calculated.

6.2.2 Reagents

- a) Triethanolamine (TEA): $N(\text{CH}_2\text{CH}_2\text{OH})_3$
[$\rho = 1,12 \text{ g/cm}^3$]
- b) EDTA: dihydrate of the disodium salt of ethylene-diamine-tetracetic acid
 $\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$
- c) Diethylamine (DEA): $(\text{C}_2\text{H}_5)_2\text{NH}$
- d) Ethanol: $\text{C}_2\text{H}_5\text{OH}$ (ethyl alcohol)
- e) Concentrated nitric acid: HNO_3
[$\rho = (1,40 \text{ to } 1,42) \text{ g/cm}^3$]

6.2.3 Apparatus

- a) *Balance*, capable of weighing to the nearest 0,0001 g.
- b) *Drying oven*, controlled at $(105 \pm 5) \text{ }^\circ\text{C}$.
- c) *Apparatus*, to check the temperature at $(20 \pm 0,5) \text{ }^\circ\text{C}$.
- d) *Electrically controlled stirrer*, fitted with a glass propeller.
- e) *Glass microfibre filter papers*, with a porosity of the order of 1 μm to 2 μm and a maximum diameter of 9 cm that fits the funnel of the filtration system. The filter is resistant to alcohol and alkalis and can be dried to constant mass at $105 \text{ }^\circ\text{C}$.
- f) *Vacuum filtration system*, capable of being used with glass microfibre filter papers.
- g) *Desiccator*, containing anhydrous magnesium perchlorate.
- h) *Volumetric glassware*, of analytical precision, i.e. class A quality as defined in the ISO standards for laboratory glassware.
- i) *Ordinary glassware*, beakers, flasks, watch glasses, Petri dishes, etc.
- j) *Agate mortar*.
- k) *Sieve*, with a mesh aperture of 75 μm .