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Cement – Provning – Del 4: Kvantitativ bestämning av ingående beståndsdelar

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



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TECHNICAL REPORT
RAPPORT TECHNIQUE
TECHNISCHER BERICHT

CEN/TR 196-4

September 2007

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English Version

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

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

Prüfverfahren für Zement - Teil 4: Quantitative Bestimmung
der Bestandteile

This Technical Report was approved by CEN on 14 April 2007. It has been drawn up by the Technical Committee CEN/TC 51.

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Foreword

This document (CEN/TR 196-4:2007) has been prepared by Technical Committee CEN/TC 51 "Cement and building limes", the secretariat of which is held by NBN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes ENV 196-4:1993.

This European Technical Report was drawn up by Technical Committee CEN/TC 51 "*Cement and building limes*" the Secretariat of which is held by NBN. It is based on a revision of the European Pre-standard ENV 196-4 of July 1993.

The main aim of this document is to quantitatively verify the compositions (analysis of the constituents) of all the cements included in EN 197-1:2000 (Cements – Part1: composition, specifications and conformity criteria for common cements) as set out in Table 1 "The 27 products in the family of common cements".

Further to this objective, original methods of analysis were devised, firstly, for cements with 3 constituents and then a reference method for cements with more constituents. Following the progress of work on EN 197-1, cements with blastfurnace slag, siliceous fly ash and natural pozzolans have been successively studied. As a result the first draft of ENV 196-4 was published in December 1989, followed by the ENV 196-4 in July 1993.

The main aim of the revision of the Pre-standard 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 had not been included in the 1989 draft ENV 196-4). This entailed revising the analytical procedure and the calculation of the constituents.

The opportunity was taken at the same time to unify the presentation of the different methods, reference and alternative, endeavoring to standardize the notational symbols to eliminate all ambiguities in the interpretation of the formulae for calculations.

Table 1 of ENV 197-1:1992 introduced further new constituent materials. One of them, silica fume, could be routinely determined by the reference method, while calcareous fly ash and burnt shale, being composites of several minerals, react partially like other constituents capable of being determined by the reference method. Where these materials are constituents it has proved not to be possible to determine the mass composition of the cement but only to obtain an overall bulk analysis.

Almost all of the cements manufactured in Europe can be correctly characterized and quantified by the reference method. However, for cements containing burnt shale (CEM II/A-T and B-T) or calcareous fly ash (CEM II/A-W and B-W) it would be necessary to undertake further research in order to obtain an acceptable reference method.

For cements having constituents that can be analyzed by the current reference method as defined in section 1 "Scope" the method will be adequate. Where other constituents are known, or suspected, to be included it will be necessary to develop additional methods for the quantitative determination of those particular constituents.

The European Standard on the methods of testing cement comprises the following Parts:

EN 196-1 *Methods of testing cement — Part 1: Determination of strength*

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EN 196-2 *Methods of testing cement — Part 2: Chemical analysis of cement*

EN 196-3 *Methods of testing cement — Part 3: Determination of setting times and soundness*

EN 196-5 *Methods of testing cement — Part 5: Pozzolanicity test for pozzolanic cement*

EN 196-6 *Methods of testing cement — Part 6: Determination of fineness*

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

EN 196-8 *Methods of testing cement — Part 8: Heat of hydration — Solution method*

EN 196-9 *Methods of testing cement — Part 9: Heat of hydration — Semi-adiabatic method.*

NOTE A previous Part, EN 196- 21: Methods of testing cement — Part 21: Determination of the chloride, carbon dioxide and alkali content of cement, has been revised and incorporated into EN 196-2

1 Scope

This European Technical Report describes procedures for determining the contents of most of the constituents of the cements that fall within the scope of EN 197-1.

In principle, the method described in Clause 6 applies to all cements, whatever the number and nature of their constituents, but in practice is limited to the cements identified in Table 1.

The method in clause 6 should be considered to be the method of choice and is based on a sequential selective dissolution of the cement's constituents, generally of an unknown number, where they are not available separately for analysis at the same time as the cement.

The method of choice enables the quantitative determination (by mass) of: Portland cement clinker, blastfurnace slag, siliceous fly ash, natural pozzolans, limestone, silica fume and set regulators in cements of the types identified in Table 1. Table 1 is derived from Table 1 of EN 197-1.

Table 1 — Common cement types specified in EN 197-1

Type of cement	Designation	Notation	Excluding ⁽¹⁾
CEM I	Portland cement	I	
CEM II	Portland-slag cement (with blastfurnace slag)	II/A-S II/B-S	
	Portland-silica fume cement	II/A-D	
	Portland pozzolana cement (with natural pozzolana)	II/A-P II/B-P	
	Portland-fly ash cement (with siliceous fly ash)	II/A-V II/B-V	
	Portland-limestone cement	II/A-L or LL* II/B-L or LL*	
	Portland-composite cement	II/A-M II/B-M	limestone Burnt shale
CEM III	Blastfurnace cement	III/A III/B III/C	
CEM IV	Pozzolanic cement	IV/A IV/B	
CEM V	Composite cement	V/A V/B	

*The method is incapable of distinguishing between limestones of type L and LL

NOTE 1 Where cements contain calcareous fly ash, burnt shale and/or constituents that partly contain mineral phases, similar to those of clinker, further investigation into the characteristics of those constituents will be necessary before the method can be applied.

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The method of choice has limitations, as indicated earlier, and cannot be considered to be a means by which clinker content can simply be determined in isolation from any other constituent. Clinker content is determined 'by difference' and other constituents contain, in part, mineral phases similar to those present in clinker and can cause interferences that lead to difficulties in interpretation of the results.

Where apparently anomalous results are obtained, it is recommended that further investigations are undertaken in accordance with the procedure given in Section 6.2.5.4.

Any other method with the same objectives, and intended for use where the constituents are unavailable for separate analysis, can be considered to be an alternative to the method of choice when it is shown that, with appropriate statistical validity, 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) in order to be able to calculate the clinker content by difference;
- the cement contains only three constituents, i.e. a set regulator, clinker and one of the following three: slag, pozzolana or siliceous fly ash. Some of the methods in clause 7 are variations on the method of choice whereas others are based on physical separation of constituents and different analytical principles.

NOTE 2 This European Technical Report adopts the following use of terms for major constituents:

- 'Portland cement clinker' as defined in EN 197-1 is referred to as 'clinker';
- 'granulated blastfurnace slag' as defined in EN 197-1 is referred to as 'slag';
- 'natural pozzolans' as defined in EN 197-1 is referred to as 'pozzolans';
- 'siliceous fly ash' as defined in EN 197-1 is referred to as 'fly ash';
- 'calcium sulfate' as defined in EN 197-1 is referred to as 'set regulator'.

2 Normative references

This European Technical Report 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 Technical Report only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 196-2, *Methods of testing cement — Part 2: Chemical analysis of cements*

EN 196-7, *Methods of testing cement — Part 7: Methods of taking and preparing samples of cement*

EN 197-1, *Cement — Part 1: Composition, specifications and conformity criteria for common cements*

ISO 3534, *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 method of choice 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 method of choice 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: 2005, are as follows:

Analyte	Standard deviation for repeatability (s_r in % by mass)
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 weighing is less than 0,0005 g.