



Methods of testing cement — Chemical analysis of cement — Part 2: Analysis by X-ray fluorescence

Méthodes d'essais des ciments — Analyse chimique des ciments —

Partie 2: Analyse par spectrométrie de fluorescence X

ICS 91.100.10

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Foreword

This document prEN 196-2.2 has been prepared by Technical Committee CEN/TC 51 “*Cement and Building Limes*”, the secretariat of which is held by IBN.

This document is currently submitted to the joint CEN/ISO enquiry.

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

- EN 196-1, *Methods of testing cement - Part 1: Determination of strength*
- 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 time and soundness*
- EN 196-5, *Methods of testing cement - Part 5: Pozzolanicity test for pozzolanic cements*
- 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*

Another document, ENV 196-4 *Methods of testing cement - Part 4: Quantitative determination of constituents*, has been drafted and will be published as a CEN Technical Report.

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of an identical text or by endorsement, at the latest by xxxx 200y, and conflicting national standards shall be withdrawn at the latest by xxxx 200y.

This European Standard incorporates the following technical principles based on comments received by the Secretariat:

- a) It provides an analytical method utilising x-ray fluorescence (XRF) for use as the alternative method for the analysis of cement. When correctly calibrated according to the specified procedures and reference materials it provides a method of suitable precision for conformity and information purposes. It may be considered for superseding EN 196-2 as the reference method for analysis for SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, SO₃, K₂O, Na₂O, Mn₂O₃ and Cl when sufficient experience is obtained.
- b) It introduces a reference method for TiO₂, P₂O₅, SrO and Br analysis.
- c) Traceability of the method relies upon reference materials and ‘pure’ chemicals so that the ultimate traceability to basic international chemical standards relies upon classical analytical methods that are outside of the scope of this European Standard.

Any other methods may be used as alternative methods, e.g. instrumental methods such as inductively coupled plasma – mass spectrometry and atomic absorption spectrometry, provided they are calibrated against the reference method or against internationally accepted reference materials, in order to demonstrate their equivalence.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard describes a performance based method for the chemical analysis of cement for SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, SO₃, K₂O, Na₂O, TiO₂, P₂O₅, Mn₂O₃, SrO, Cl and Br using x-ray fluorescence (XRF). It may be applied to other relevant elements when adequate calibrations have been established.

This European Standard describes an alternative method for analyses of cement for conformity and information purposes based on beads of fused sample and analytical validation using certified reference materials together with performance criteria.

A method based on pressed pellets of unfused sample can be considered to be equivalent providing that the analytical performance satisfies the same criteria.

NOTE 1. The use of fused beads will generally improve the accuracy of analysis for non-volatile elements since it eliminates variability arising from differences in mineralogical forms or oxidation states. Pressed pellets will generally improve the accuracy of analysis for volatile elements and might give adequate accuracy for the routine analysis of non-volatile elements.

NOTE 2. The presence of sulfide in a sample also leads to restrictions on the scope of the analysis that can be undertaken using the XRF technique based upon fused beads. In particular, sulfate (SO₃) cannot be determined directly from such a fused bead because of the contribution to the analysis from the unknown amount of sulfide. In addition, sulfide cannot be determined directly (or accurately, indirectly) because of the contribution of the unknown amount of sulfate to the analysis and from the possibility that some sulfide can be lost by volatilisation during fusion. Consequently, the method of EN 196-2, included as Annex D to this European Standard, is the reference method for determining the sulfate content of samples containing sulfide species.

Any other methods may be used provided they are calibrated, either against the reference method or against internationally accepted reference materials, in order to demonstrate their equivalence.

In the case of dispute, unless otherwise agreed by all parties, only the reference method in EN 196-2 is to be used.

This European Standard describes methods which apply principally to cements, but which can also be applied to their constituent materials. They can also be applied to other materials, the standards for which call up these methods.

European Standard specifications state which methods are to be used.

2 Normative references

This European Standard 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 Standard 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 cement*

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

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results*
- Part 1: General principles and definitions

3 Terms and definitions

For the purposes of this European Standard the following terms and definitions apply.

3.1 beads

glassy discs of fused sample for presentation to x-rays in the spectrometer.

3.2 pellets

compressed discs of finely ground sample for presentation to x-rays in the spectrometer.

3.3 calibration beads or pellets

beads or pellets used for establishing the calibration equation.

3.4 analysis beads or pellets

beads or pellets containing the sample to be analysed.

3.5 repeatability

the closeness of agreement between independent test results obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

3.6 accuracy

the closeness of agreement between a test result and the certified value for a reference material.

3.7 reproducibility

the closeness of agreement between independent test results obtained with the same method on identical test items in different laboratories with different operators using different equipment

4 General requirements for testing

4.1 Number of tests

Analysis of a cement may require the determination of a number of its chemical elements. For each determination one or more tests shall be carried out in which the number of measurements to be taken shall be as specified in the relevant clause of this standard.

Where the analysis is one of a series subject to statistical control, determination of each chemical element by a single test shall be the minimum required.

Where the analysis is not part of a series subject to statistical control, the number of tests for determination of each chemical element shall be two (see also **13**).

4.2 Accuracy and precision limits

4.2.1 Accuracy limit

The accuracy performance criterion in this European Standard is measured as a limit on the closeness of agreement between a test result and an accepted reference value for a certified reference material. The limits for accuracy, expressed in percent absolute, are set out in Table **2**; one set is appropriate to the performance expected to be achieved by 'expert' laboratories whereas the other is appropriate to a 'normal' laboratory.



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