BS EN 196-2:2013

BSI Standards Publication

Method of testing cement

Part 2: Chemical analysis of cement

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National foreword

This British Standard is the UK implementation of EN 196-2:2013. It supersedes BS EN 196-2:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/516/12, Sampling and testing.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

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Method of testing cement - Part 2: Chemical analysis of cement

Méthodes d'essais des ciments - Partie 2: Analyse chimique des ciments

Prüfverfarhen für Zement - Teil 2: Chemische Analyse von Zement

This European Standard was approved by CEN on 5 April 2013.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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Foreword

This document (EN 196-2:2013) has been prepared by Technical Committee CEN/TC 51 “Cement and building limes”, the secretariat of which is held by NBN.

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of an identical text or by endorsement, at the latest by December 2013, and conflicting national standards shall be withdrawn at the latest by December 2013.

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 EN 196-2:2005.

This edition adds, to the previous version EN 196-2:2005, provisions for the use of X-ray fluorescence (XRF) analysis as an alternative method. In relation to correctly calibrating the method, specified procedures, reference materials and performance criteria are included in order to attain and maintain suitable accuracy and precision for equivalence. The method has not been validated for use yet as a reference procedure for conformity or dispute purposes.

This European Standard on the methods of testing cement is comprised of the following parts:

— *Part 1: Determination of strength*

— *Part 2: Chemical analysis of cement*

— *Part 3: Determination of setting times and soundness*

— *Part 5: Pozzolanicity test for pozzolanic cement*

— *Part 6: Determination of fineness*

— *Part 7: Methods of taking and preparing samples of cement*

— *Part 8: Heat of hydration — Solution method*

— *Part 9: Heat of hydration — Semi-adiabatic method*

— *Part 10: Determination of the water-soluble chromium (VI) content of cement*

NOTE Another document, CEN/TR 196-4 *Methods of testing cement — Part 4: Quantitative determination of constituents*, has been published as a CEN Technical Report.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies the methods for the chemical analysis of cement.

This document describes the reference methods and, in certain cases, an alternative method which can be considered to be equivalent. In the case of a dispute, only the reference methods are used.

An alternative performance-based method using X-ray fluorescence (XRF) is described for SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, K_2O, Na_2O, TiO_2, P_2O_5, Mn_2O_3, SrO, Cl and Br. When correctly calibrated according to the specified procedures and reference materials, it provides a method equivalent to the reference methods but has not been validated for use yet as a reference procedure for conformity and dispute purposes. It can be applied to other relevant elements when adequate calibrations have been established. This method is based on beads of fused sample and analytical validation using certified reference materials, together with performance criteria. A method based on pressed pellets of un-fused sample can be considered as equivalent, providing that the analytical performance satisfies the same criteria.

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

This document 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. Standard specifications state which methods are to be used.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 385, *Laboratory glassware — Burettes*

ISO 835, *Laboratory glassware — Graduated pipettes*

ISO Guide 30, *Terms and definitions used in connection with reference materials*

ISO Guide 31, *Reference materials — Contents of certificates and labels*

3 General requirements for testing

3.1 Number of tests

Analysis of a cement may require the determination of a number of its chemical properties. 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 document.

Where the analysis is one of a series subject to statistical control, the determination of each chemical property 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 property shall be two (see also 3.3 and 5.8).

In the case of a dispute, the number of tests for determination of each chemical property shall be two (see also 3.3).

3.2 Repeatability and reproducibility

Repeatability: Precision under repeatability conditions where independent test results are obtained with the same method on identical test items (material) in the same laboratory by the same operator using the same equipment within short intervals of time.

Reproducibility: Precision under reproducibility conditions where test results are obtained with the same method on identical test items (material) in different laboratories with different operators using different equipment.

Repeatability and reproducibility in this document are expressed as repeatability standard deviation(s) and reproducibility standard deviation(s) in e.g. absolute percent, grams, etc., according to the property tested.

3.3 Expression of masses, volumes, factors and results

Express masses in grams to the nearest 0,000 1 g and volumes from burettes in millilitres to the nearest 0,05 ml.

Express the factors of solutions, given by the mean of three measurements, to three decimal places.

Express the results, where a single test result has been obtained, as a percentage generally to two decimal places.

Express the results, where two test results have been obtained, as the mean of the results, as a percentage generally to two decimal places.

If the two test results differ by more than twice the standard deviation of repeatability, repeat the test and take the mean of the two closest test results.

The results of all individual tests shall be recorded.

4 Analysis by wet chemistry

4.1 General

4.1.1 Ignitions

Carry out ignitions as follows.

Place the filter paper and its contents into a crucible which has been previously ignited and tared. Dry it, then incinerate slowly in an oxidising atmosphere in order to avoid immediate flaming, while ensuring complete combustion. Ignite the crucible and its contents at the stated temperature then allow to cool to the laboratory temperature in a desiccator. Weigh the crucible and its contents.

4.1.2 Determination of constant mass

Determine constant mass by making successive 15 min ignitions followed each time by cooling and then weighing. Constant mass is reached when the difference between two successive weighings is less than 0,000 5 g.

4.1.3 Check for absence of chloride ions (silver nitrate test)

After generally five to six washes of a precipitate, rinse the base of the filter stem with a few drops of water. Wash the filter paper and its contents with several millilitres of water and collect this in a test tube. Add several drops of silver nitrate solution (4.2.44). Check the absence of turbidity or precipitate in the solution. If present, continue washing while carrying out periodic checks until the silver nitrate test is negative.

4.1.4 Blank determinations

Carry out a blank determination without a sample, where relevant, following the same procedure and using the same amounts of reagents. Correct the results obtained for the analytical determination accordingly.

4.1.5 Preparation of a test sample of cement

Before chemical analysis, treat the laboratory sample, taken in accordance with EN 196-7, as follows to obtain a homogeneous test sample.

Take approximately 100 g of the laboratory sample by means of a sample divider or by quartering. Sieve this portion on a 150 µm or 125 µm sieve until the residue remains constant. Remove metallic iron from the material retained on the sieve by means of a magnet (see Note 1). Then grind the iron-free fraction of the retained material so that it completely passes the 150 µm or 125 µm sieve. Transfer the sample to a clean dry container with an airtight closure and shake vigorously to mix it thoroughly.

Carry out all operations as quickly as possible to ensure that the test sample is exposed to ambient air only for the minimum time.

NOTE 1 Where the analysis is one of a series subject to statistical control and the level of the metallic iron content has been shown to be insignificant in relation to the chemical properties to be determined then it is not necessary to remove metallic iron.

NOTE 2 Where the sample is to be used for XRF analysis and it contains quartz, it might be necessary to grind the sample to pass a 90 µm sieve in order to obtain a satisfactory fusion (see 5.6). The time and temperature required to obtain a satisfactory fusion is affected by the fineness of the sample.

NOTE 3 Where the sample is to be used for XRF analysis using pressed pellets, accuracy can be improved by grinding the sample more finely.

4.2 Reagents

4.2.1 General

Use only reagents of analytical quality. References to water mean distilled or de-ionised water having an electrical conductivity ≤ 0,5 mS/m.

Unless otherwise stated, percent means percent by mass.

Unless otherwise stated, the concentrated liquid reagents used in this document have the following densities (ρ) (in g/cm^3 at 20 °C):

hydrochloric acid	1,18 to 1,19	acetic acid	1,05 to 1,06
nitric acid	1,40 to 1,42	phosphoric acid	1,71 to 1,75
perchloric acid	1,60 to 1,67	ammonium hydroxide	0,88 to 0,91

The degree of dilution is always given as a volumetric sum, for example: dilute hydrochloric acid 1 + 2 means that 1 volume of concentrated hydrochloric acid is to be mixed with 2 volumes of water.

4.2.2 Concentrated hydrochloric acid (HCl)

4.2.3 Dilute hydrochloric acid (1 + 1)

4.2.4 Dilute hydrochloric acid (1 + 2)