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Metode preskušanja cementa - 2. del: Kemijska analiza cementa

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

Prüfverfahren für Zement - Teil 2: Chemische Analyse von Zement

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

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

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

Prüfverfahren für Zement - Teil 2: Chemische Analyse
von Zement

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 51.

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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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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European foreword

This document (prEN 196-2:2024) has been prepared by Technical Committee CEN/TC 51 “Cement and building limes”, the secretariat of which is held by NBN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 196-2:2013.

prEN 196-2:2024 includes the following significant technical changes with respect to EN 196-2:2013:

- redefinition of the introduction;
- introduction of concept;
- determination of reactive silica;
- introduction of the concept of determination of Total Carbonate Content instead of determination of CO₂;
- determination of chloride by potentiometric titration as alternative method;
- determination of Total Carbonate Content by gas volumetric method as alternative method;
- determination of Total Carbonate Content by infrared detection system (method A and B) as alternative method;
- determination of SO₃ by inductively coupled plasma optical emission spectroscopy as alternative method.

This European Standard series, under the general title *Methods of testing cement*, comprises 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;*
- *Part 11: Heat of hydration — Isothermal conduction calorimetry method.*

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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.

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Introduction

In this document, the elemental chemical analysis of a clinker or cement, as of any natural or artificial mineral material (volcanic pozzolan, blast furnace slag, fly ash, etc.) is expressed in a conventional manner in the form of the weight proportions of the common oxides corresponding to the most frequent and stable degree of oxidation (Si: SiO₂; Al: Al₂O₃, etc.). This expression of the chemical analysis does not indicate how these elements are combined into minerals such as silicates, oxides, carbonates, sulphates or sulphides.

This means that the presence of SiO₂ in a chemical analysis sheet does not necessarily implies the presence of crystalline silica e.g. quartz or cristobalite, but most likely silicates. Moreover, even if Quartz is detected in the material, this does not necessarily imply an inhalation hazard if the particle size is larger than the inhalable fraction. Similarly, the presence of TiO₂ in a chemical analysis report does not necessarily imply the presence of the mineralogical phase(s) corresponding to this formula (Rutile, Anatase, Brookite), although the presence of the mineralogical phase(s) does not necessarily imply an inhalation hazard if the particle size is larger than the inhalable fraction. In the same way, determination of Total Carbonate Content (TCC) can be done by dosing CO₂ induced by acid attack, which does not mean, in any case, that the material contains CO₂.

The only way to identify mineralogical phases in a powdered mineral material is by X-ray diffraction, which is the only analytical technique that is sensitive to the crystalline character of minerals. For an accurate quantification of inhalable fractions, it might be necessary to perform a quantitative particle size selection (e.g. by aerosolization technique) in order to eliminate coarse fractions that could reduce the quantitative character of the X-ray Diffraction analysis.

Standardization to 100 made it possible to know if no constituent element had been left out. Hence the expression "percentage by weight of oxides" in the chemical analysis.

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prEN 196-2:2024 (E)**1 Scope**

This document 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 SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, SO₃, K₂O, Na₂O, TiO₂, P₂O₅, Mn₂O₃, SrO, Cl and Br. 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.

An alternative performance-based method using inductively coupled plasma optical emission spectroscopy (ICP-OES) is described for SO₃.

When correctly calibrated according to the specified procedures and reference materials, XRF and ICP-OES provides methods equivalent to the reference methods but has not been validated for use yet as a reference procedure for conformity and dispute purposes. They can be applied to other relevant elements when adequate calibrations have been established.

Any other methods can 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 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 196-7, *Methods of testing cement — Part 7: Methods of taking and preparing samples of cement*

EN 13656, *Soil, treated biowaste, sludge and waste — Digestion with a hydrochloric (HCl), nitric (HNO₃) and tetrafluoroboric (HBF₄) or hydrofluoric (HF) acid mixture for subsequent determination of elements*

ISO 385, *Laboratory glassware — Burettes*

ISO 835, *Laboratory glassware — Graduated pipettes*

ISO 33401, *Reference materials — Contents of certificates, labels and accompanying documentation*

ISO Guide 30, *Reference materials — Selected terms and definitions*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology 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

analyte

element to be determined

3.2

calibration solution

solution used (at least two) to calibrate the instrument, prepared from stock solutions by adding acids, buffer, reference element and salts as needed

3.3

stock solution

solution with accurately known analyte concentration(s), prepared from pure chemicals. Certified commercial standard solutions can be used

Note 1 to entry: Stock solutions are reference materials within the meaning of ISO Guide 30.

3.4

blank test solution

solution prepared in the same way as the test sample solution but omitting the test portion

4 General requirements for testing

4.1 Number of tests

Analysis of a cement can 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 4.3 and 6.8).

In the case of a dispute, the number of tests for determination of each chemical property shall be two (see also 4.3 and 6.8).

4.2 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_r) and reproducibility standard deviation (S_R) in e.g. absolute percent, grams, etc., according to the property tested.

4.3 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.

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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.

5 Analysis by wet chemistry

5.1 General

5.1.1 Ignitions

Carry out ignitions as follows.

Place the ashless 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.

5.1.2 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.

5.1.3 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 ashless filter paper and its contents with several millilitres of water and collect this in a test tube. Add several drops of silver nitrate solution (5.2.44). 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.

5.1.4 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.

5.1.5 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.

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.

Then grind the iron-free fraction of the retained material so that it completely passes the 150 μm or 125 μm sieve.

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 6.6). The time and temperature required to obtain a satisfactory fusion is affected by the fineness of the sample.

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

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.

5.2 Reagents

5.2.1 General

Use only reagents of analytical quality. References to water mean distilled or de-ionized water having an electrical conductivity $\leq 0,5$ 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/cm³ 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.

5.2.2 Concentrated hydrochloric acid (HCl)

5.2.3 Dilute hydrochloric acid (1 + 1)

5.2.4 Dilute hydrochloric acid (1 + 2)

5.2.5 Dilute hydrochloric acid (1 + 3)

5.2.6 Dilute hydrochloric acid (1 + 9)

5.2.7 Dilute hydrochloric acid (1 + 11)

5.2.8 Dilute hydrochloric acid (1 + 19)

5.2.9 Dilute hydrochloric acid (1 + 99)

5.2.10 Dilute hydrochloric acid of pH (1,60 \pm 0,05)

Prepare by adjusting the pH of two litres of water to (1,60 \pm 0,05) by adding five or six drops of concentrated hydrochloric acid (5.2.2). Control using the pH meter (5.3.18.1). Store the solution in a polyethylene container.

5.2.11 Concentrated hydrofluoric acid (>40 %) (HF)

5.2.12 Dilute hydrofluoric acid (1 + 3)

5.2.13 Concentrated nitric acid (HNO₃)

prEN 196-2:2024 (E)**5.2.14 Dilute nitric acid (1 + 2)****5.2.15 Dilute nitric acid (1 + 100)****5.2.16 Concentrated sulfuric acid (>98 %) (H₂SO₄)****5.2.17 Dilute sulfuric acid (1 + 1)****5.2.18 Dilute sulfuric acid (1 + 4)****5.2.19 Concentrated perchloric acid (HClO₄)****5.2.20 Concentrated phosphoric acid (H₃PO₄)****5.2.21 Dilute phosphoric acid (1 + 19)**

Store this solution in a polyethylene container.

5.2.22 Boric acid (H₃BO₃)**5.2.23 Concentrated acetic acid (CH₃COOH)****5.2.24 Amino-acetic acid (NH₂CH₂COOH)****5.2.25 Metallic chromium (Cr), in powder form****5.2.26 Concentrated ammonium hydroxide (NH₄OH)****5.2.27 Dilute ammonium hydroxide (1 + 1)****5.2.28 Dilute ammonium hydroxide (1 + 10)****5.2.29 Dilute ammonium hydroxide (1 + 16)****5.2.30 Sodium hydroxide (NaOH)****5.2.31 Sodium hydroxide solution (4 mol/l)**

Dissolve 160 g of sodium hydroxide (5.2.30) in water and make up to 1 000 ml. Store in a polyethylene container.

5.2.32 Sodium hydroxide solution (2 mol/l)

Dissolve 80 g of sodium hydroxide (5.2.30) in water and make up to 1 000 ml. Store in a polyethylene container.

5.2.33 Ammonium chloride (NH₄Cl)**5.2.34 Tin (II) chloride (SnCl₂·2H₂O)****5.2.35 Potassium iodate (KIO₃) dried to constant mass at (120 ± 5) °C****5.2.36 Potassium periodate (KIO₄)****5.2.37 Sodium peroxide (Na₂O₂) in powder form****5.2.38 Sodium chloride (NaCl) dried to constant mass at (110 ± 5) °C****5.2.39 Potassium chloride (KCl) dried to constant mass at (110 ± 5) °C**

5.2.40 Sodium carbonate (Na₂CO₃) dried to constant mass at (250 ± 10) °C**5.2.41 Mixture of sodium carbonate and sodium chloride**

Mix 7 g of sodium carbonate (5.2.40) with 1 g sodium chloride (NaCl) (5.2.38).

5.2.42 Barium chloride solution

Dissolve 120 g of barium chloride (BaCl₂·2H₂O) in water and make up to 1 000 ml.

5.2.43 Silver nitrate (AgNO₃) dried to constant mass at (150 ± 5) °C**5.2.44 Silver nitrate solution**

Dissolve 5 g of silver nitrate (AgNO₃) (5.2.43) in water, add 10 ml of concentrated nitric acid (HNO₃) (5.2.13) and make up to 1 000 ml with water.

5.2.45 Silver nitrate solution (0,05 mol/l)

Dissolve (8,494 0 ± 0,000 5) g of silver nitrate (AgNO₃) (5.2.43) in water in a 1 000 ml volumetric flask and make up to the mark. Store in a brown glass container and protect from the light.

5.2.46 Sodium carbonate solution

Dissolve 50 g of anhydrous sodium carbonate (5.2.40) in water and make up to 1 000 ml.

5.2.47 Potassium hydroxide solution

Dissolve 250 g of potassium hydroxide (KOH) in water and make up to 1 000 ml. Store in a polyethylene container.

5.2.48 Ammoniacal zinc sulfate solution

Dissolve 50 g of zinc sulfate (ZnSO₄·7H₂O) in 150 ml water and add 350 ml of concentrated ammonium hydroxide (5.2.26). Leave to stand for at least 24 h and filter.

5.2.49 Lead acetate solution

Dissolve approximately 0,2 g of lead acetate (Pb(CH₃COO)₂·3H₂O) in water and make up to 100 ml.

5.2.50 Starch solution

To 1 g of starch (water soluble), add 1g of potassium iodide (KI), dissolve in water and make up to 100 ml. Use within two weeks.

5.2.51 Polyethylene oxide solution

Dissolve 0,25 g of polyethylene oxide (-CH₂-CH₂-O-)n of average molecular mass 200 000 to 600 000, in 100 ml water while stirring vigorously. Use within two weeks.

5.2.52 Boric acid solution, saturated

Dissolve approximately 50 g of boric acid (H₃BO₃) (5.2.22) in water and make up to 1 000 ml.

5.2.53 Citric acid solution

Dissolve 10 g of citric acid (C₆H₈O₇·H₂O) in water and make up to 100 ml.

5.2.54 Calcium carbonate (CaCO₃) dried to constant mass at (200 ± 10) °C (of purity > 99,9 %)**5.2.55 Ammonium molybdate solution**

prEN 196-2:2024 (E)**5.2.55.1 Ammonium molybdate: solution a)**

Dissolve 10 g of ammonium molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ in water and make up to 100 ml. Store the solution in a polyethylene flask. Use within one week.

5.2.55.2 Ammonium molybdate: solution b)

Dissolve 63 g of ammonium molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ in water and make up to 500 ml. Prepare 500 ml of sulphuric acid 1+4 (5.2.18) and add the 500 ml of the molybdate solution.

5.2.56 Copper sulfate solution

Dissolve 0,45 g of copper sulfate $(\text{CuSO}_4\cdot 5\text{H}_2\text{O})$ in water and make up to 50 ml in a volumetric flask.

5.2.57 Ammonium acetate solution

Dissolve 250 g of ammonium acetate $(\text{CH}_3\text{COONH}_4)$ in water and make up to 1 000 ml.

5.2.58 Triethanolamine $[\text{N}(\text{CH}_2\text{CH}_2\text{OH})_3]$, (>99 %) diluted to (1 + 4) solution**5.2.59 Reducing solution**

Dissolve 1 g of tin (II) chloride $(\text{SnCl}_2\cdot 2\text{H}_2\text{O})$ (5.2.34) in water to which has been added 1 ml of concentrated hydrochloric acid (5.2.2). Make up to 100 ml with water. Use within one day.

5.2.60 Buffer solution of pH 1,40

Dissolve $(7,505 \pm 0,001)$ g of amino-acetic acid (5.2.24) and $(5,850 \pm 0,001)$ g of sodium chloride (NaCl) (5.2.38) in water and make up to 1 000 ml. Dilute 300 ml of this solution to 1 000 ml with hydrochloric acid 1 + 99 (5.2.9).

5.2.61 Standard potassium iodate solution, approximately 0,016 6 mol/l

Weigh, to $\pm 0,000$ 5 g, $(3,6 \pm 0,1)$ g, of potassium iodate (KIO_3) (5.2.35) (m_1) and place in a 1 000 ml volumetric flask. Add 0,2 g of sodium hydroxide (5.2.30), 25 g of potassium iodide (KI), dissolve all the solids in freshly boiled and cooled water and make up to the mark using the same water.

Calculate the factor F of the potassium iodate solution from the following formula:

$$F = \frac{m_1}{35668} \quad (1)$$

where

m_1 is the mass of the portion of potassium iodate, in grams.

5.2.62 Sodium thiosulfate solution, approximately 0,1 mol/l**5.2.62.1 Preparation**

Dissolve $(24,82 \pm 0,01)$ g of sodium thiosulfate $(\text{Na}_2\text{S}_2\text{O}_3\cdot 5\text{H}_2\text{O})$ in water and make up to 1 000 ml. Before each test series, determine the factor f of this solution as described in 5.2.62.2.

5.2.62.2 Standardization**5.2.62.2.1 Standardization using potassium iodate solution**

This standardization is carried out preferably using the standard potassium iodate solution (5.2.61). For this standardization, pipette 20 ml of the standard potassium iodate solution (5.2.61) into a 500 ml conical flask and dilute with approximately 150 ml of water. Acidify with 25 ml of hydrochloric acid 1+1