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Building lime - Part 2: Test methods

Baukalk - Teil 2: Prüfverfahren

Chaux de construction - Partie 2: Méthodes d’essai

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ICS:

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EUROPEAN STANDARD
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EN 459-2

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English Version

Building lime - Part 2: Test methods

Chaux de construction - Partie 2: Méthodes d'essai

Baukalk - Teil 2: Prüfverfahren

This European Standard was approved by CEN on 30 July 2010.

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Contents

Page

Foreword

This document (EN 459-2:2010) 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of an identical text or by endorsement, at the latest by March 2011, and conflicting national standards shall be withdrawn at the latest by March 2011.

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 459-2:2001.

The following modifications have been made from EN 459-2:2001:

- description of chemical procedures for CaO, MgO, CO_2 gravimetrically, SO_3 and loss on ignition;
- amendments for the determinations of free water and available lime;
- description of the methods for particle size determination;
- description of the determination of setting times;
- amendments for the determination of strength;
- editorial changes were made and minor mistakes corrected.

EN 459, *Building lime*, consists of the following parts:

- *Part 1: Definitions, specifications and conformity criteria*
- *Part 2: Test methods*
- *Part 3: Conformity evaluation*

The existing standards from the EN 196 series were used as a basis for the testing of physical and mechanical properties in EN 459-2. For the testing of chemical properties of building limes, test methods described in EN 12485 have been incorporated into this European Standard.

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

Introduction

The objective in this European Standard has been to adopt as many European standardized methods as possible and where this has not been possible to use other appropriate proven methods.

Unless otherwise stated, tolerance class m of ISO 2768-1:1989 should be applied (indications on drawings by "ISO 2768-m").

All dimensions are in millimetres.

1 Scope

This European Standard describes the test methods for all building limes covered by EN 459-1:2010.

This European Standard specifies the methods used for the chemical analyses and the determination of physical properties of building limes.

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.

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.

2 Normative references

The following referenced documents are indispensable for the application 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 196-1:2005, *Methods of testing cement — Part 1: Determination of strength*

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

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

EN 459-1:2010, *Building lime — Part 1: Definitions, specifications and conformity criteria*

EN 932-1, *Tests for general properties of aggregates — Part 1: Methods for sampling*

EN ISO 6506-1:2005, *Metallic materials — Brinell hardness test — Part 1: Test method (ISO 6506-1:2005)*

3 Sampling

3.1 General

Sampling shall be carried out as specified in 3.2 to 3.4 taking into account the need to minimize moisture and carbon dioxide absorption. Samples shall therefore be transported and stored in air-tight containers and all the handling shall be carried out as quickly as possible.

3.2 Sampling of powdered material

Sampling shall be carried out in accordance with EN 196-7.

3.3 Sampling of granular material

Sampling shall be carried out in accordance with EN 932-1.

3.4 Sampling of lime putty and milk of lime

The spot sample size shall be (10 ± 5) dm^3.

Where lime putty or milk of lime is sampled, the increments shall be blended thoroughly.

3.5 Preparation of the test portion

Before carrying out the analysis, the sample shall be reduced in mass by means of a sample divider and/or by quartering to produce a homogeneous test sample of suitable mass for the intended determinations. Lime putty and milk of lime shall be dried before the chemical analysis (see 5.4.4.2).

The sample preparation for the appropriate test is described in Table 1.

Table 1 — Sample preparation for the single tests

Test	Clause in this standard	Type and form of the building lime	Sample preparation
Chemical analysis	5	All types of building lime	The sample of granular material shall be crushed and ground. All tests shall be performed on materials of a grain size ≤ 0,2 mm
Grain size distribution by sieving	6.1 and 6.2	Quicklime	Material in the as-delivered state
Grain size distribution by air-jet sieving	6.2	Hydrated lime, hydrated dolomitic lime, lime with hydraulic properties	Material in the as-delivered state
Bulk density	6.3	All types of building lime	See 6.3.2
Soundness	6.4.2.1 and 6.4.2.2	Hydrated lime, lime with hydraulic properties	Material in the as-delivered state
	6.4.2.3	Hydraulic lime with an SO_3 content of more than 3 % and up to 7 %	Material in the as-delivered state
	6.4.3	Hydrated lime, lime putty und hydrated dolomitic lime	Material in the as-delivered state
	6.4.4	Quicklime, lime putty, dolomitic quicklime, hydrated dolomitic lime	See 6.4.4.3.1
Setting times	6.5	Lime with hydraulic properties	Material in the as-delivered state
Reactivity	6.6	Quicklime	The test shall be performed on materials of a grain size ≤ 0,2 mm. If 100 % of the material pass the 5 mm sieve the product can alternatively be tested in the as-delivered state
Yield	6.7	Quicklime	See 6.7.2
Mortar tests	6.8 to 6.10	Hydrated lime, hydrated dolomitic lime, lime with hydraulic properties	Material in the as-delivered state
Compressive strength	6.11	Lime with hydraulic properties	Material in the as-delivered state

4 General requirements for testing

4.1 Number of tests

Analysis of a building lime 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 standard.

Where the analysis is one of a series subject to statistical control, 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 2 (see also 4.3).

In the case of a dispute, the number of tests for determination of each chemical property shall be 2 (see also 4.3).

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 (see Annex B) are expressed as repeatability standard deviation(s) and reproducibility standard deviation(s) 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 0,001 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.

4.4 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.

4.5 Reagents

Use only reagents of analytical quality. References to water mean distilled or deionised water having an electrical conductivity $\leq 0,5$ mS/m.

Unless otherwise stated percent means percent by mass.

For the concentrated liquids used to make up the reagents in this standard the densities (ρ) are given in grams per millilitre at 20 °C. The degree of dilution is always given as a volumetric sum, for example: dilute hydrochloric acid 1 + 2 means that one volume of concentrated hydrochloric acid is to be mixed with two volumes of water.

The concentrations of reference and standard volumetric solutions are specified as amount-of-substance concentrations, c (mol/l).

4.6 Evaluation of test results

4.6.1 General

The chemical requirements for building limes are specified in EN 459-1:2010, Tables 2, 9, 16, 20 and 24.

4.6.2 Test results for quicklime

For quicklime the specified values correspond to the finished product.

4.6.3 Test results for all other types

For all other types (hydrated lime, lime putty, milk of lime, lime with hydraulic properties) the values are based on the product after subtraction of its free water and bound water content. The values obtained by application of procedures described in this European Standard for total calcium oxide and magnesium oxide (5.2), magnesium oxide (5.2), sulfate (5.3) and carbon dioxide (5.5 or 5.6) are for the products without subtraction of the free water and bound water content. To compare these values with EN 459-1:2010, Tables 2 or 9, they shall first be corrected by multiplication by factor F. Factor F shall be determined in the following way:

Determine the carbon dioxide content as described in 5.5 or 5.6 and the loss on ignition as described in 5.7. The loss on ignition is the sum of the free water, bound water and carbon dioxide, provided that the sample does not contain any highly volatile compounds or oxidizable constituents. Calculate the total (free + bound) water content W_T as a mass fraction in percent of the sample using:

$$W_T = \text{loss on ignition in \%} - \text{carbon dioxide content in percent} \qquad (1)$$

Calculate the factor F from the following equation:

$$F = 100/(100 - W_T) \qquad (2)$$

4.6.4 Test results for available lime

The values for available lime, obtained by the application of the procedure described in 5.8 correspond to either available CaO for quicklime or available $Ca(OH)_2$ for all other types (hydrated lime, lime putty, milk of lime, lime with hydraulic properties).

5 Chemical analysis

5.1 Extraction with hydrochloric acid

5.1.1 General

Extraction with hydrochloric acid is used to dissolve building lime in order to determine calcium oxide and magnesium oxide.

5.1.2 Principle

The sample is boiled with hydrochloric acid and the solution filtered. The pH value is adjusted to 6 to 7 to precipitate the iron (III) and aluminium oxides. After refiltering, the filtrate is transferred to a suitable volumetric flask.

5.1.3 Reagents

5.1.3.1 Hydrochloric acid, ρ (HCl) = 1,16 to 1,19 g/ml.

5.1.3.2 Hydrogen peroxide solution, c (H_2O_2) = 30 %.

5.1.3.3 Hydrogen peroxide solution, diluted, 1 + 9.

5.1.3.4 Ammonium hydroxide solution, c (NH_4OH) = 25 %.

5.1.3.5 Ammonium hydroxide solution, diluted, 1 + 9.

5.1.3.6 Ammonium chloride, NH_4Cl.

5.1.4 Apparatus

5.1.4.1 Ordinary laboratory equipment.

5.1.4.2 Hot plate.

5.1.4.3 Analytical balance accurate to 0,001 g.

5.1.4.4 Magnetic stirrer and magnetic rod, inert e.g. PTFE covered.

5.1.4.5 pH-meter with glass electrode, capable of measuring to an accuracy of 0,05.

5.1.5 Procedure

Weigh (1 ± 0,1) g of the sample to the nearest 0,001 g (m_1), transfer it to a 250 ml beaker, moisten with 10 ml of water, then gradually add 30 ml of hydrochloric acid (5.1.3.1). Make the solution up to about 100 ml with water then boil it for 10 min. After boiling, filter the solution immediately through a fluted filter paper (particle retention size 2,5 µm) into a 400 ml beaker and wash the residue well with hot water.

Add about 4 g of ammonium chloride (5.1.3.6) and a few drops of hydrogen peroxide (5.1.3.3) to the solution, then dilute with about 150 ml of water and heat to boiling. During boiling, add ammonium hydroxide solution (5.1.3.4) to adjust the pH value to 6 to 7 and precipitate aluminium hydroxides and iron hydroxides and the soluble silicic acid.

Continue boiling for 3 min and, after the precipitate has settled, filter the solution immediately through a fluted filter paper (particle retention size 2,5 µm) into a 500 ml volumetric flask. Wash the filter residue three times with ammonium hydroxide solution (5.1.3.5) and three times with water. After the solution has cooled to room temperature, make the solution up to the mark with water and shake the contents of the flask thoroughly. This solution (V_1) is ready for further chemical analyses.

5.2 Calcium oxide (CaO) and magnesium oxide (MgO)

5.2.1 General

The method is suitable for determining calcium oxide and magnesium oxide.

5.2.2 Principle

Calcium oxide is first determined in an aliquot of the test solution by complexation titration with EDTA solution at a pH value of 13, the colour change of a calcium-specific indicator being used to indicate the end point. During the titration, the EDTA first reacts with the free calcium ions and then with the calcium ions bonded to the indicator, causing the colour of the latter to change sharply from wine red to blue.

In the same way, the total calcium oxide and magnesium oxide (Σ CaO + MgO) is determined at a pH value of 10 by titration with EDTA using Eriochrome Black T as indicator. During the titration, the EDTA first reacts with the free calcium and magnesium ions and then with those bonded to the indicator, causing the colour of the latter to change from red to blue. The difference in the amount-of-substance concentrations, c (CaO + MgO) and c (CaO), gives the magnesium content, c (MgO).

5.2.3 Reagents

5.2.3.1 Hydrochloric acid, ρ (HCl) = 1,16 g/ml to 1,19 g/ml.

5.2.3.2 Hydrochloric acid, diluted, 1 + 1.

5.2.3.3 Triethanolamine, c [$N(C_2H_5OH)_3$] = 99 %.

5.2.3.4 Triethanolamine solution, diluted, 1 + 1.

5.2.3.5 Sodium hydroxide solution, c (NaOH) = 4 mol/l.

5.2.3.6 Ammonium hydroxide solution, c (NH_4OH) = 25 %.

5.2.3.7 Ammonium chloride, NH_4Cl.

5.2.3.8 Ethylenediaminetetra-acetic acid disodium salt dihydrate (EDTA), ($C_{10}H_{14}N_2Na_2O_8 \cdot 2\ H_2O$), dried to constant mass at 80 °C before weighing.

5.2.3.9 Calcium carbonate, c ($CaCO_3$) = 99,9 % (dried at (200 ± 10) °C).

5.2.3.10 Buffer solution.

Make 70 g of ammonium chloride (5.2.3.7) and 570 ml of ammonium hydroxide solution (5.2.3.6) up to the mark with water in a 1 000 ml volumetric flask.

5.2.3.11 EDTA solution, c (EDTA) = 0,04 mol/l.

a) Preparation:

Dissolve 14,89 g of EDTA (5.2.3.8) in water and making up to 1 000 ml in a volumetric flask.

b) Standardization:

Pipette 50 ml of the calcium ion reference solution (5.2.3.12) into a 400 ml beaker and dilute with 100 ml of water. Adjust the pH value of the solution to (12,5 ± 0,5) with sodium hydroxide solution (5.2.3.5) using a pH meter. Add 0,1 g of calconcarboxylic indicator (5.2.3.13) and titrate with the EDTA solution being standardized until the colour changes to blue.

The concentration of the EDTA solution is given by the following equation:

$$c = \frac{50 \times m_2}{100{,}09 \times 0{,}04 \times V_2} = \frac{12{,}489 \times m_2}{V_2} \qquad (3)$$

where

m_2 is the initial mass of calcium carbonate taken to prepare the calcium ion reference solution, in grams;

V_2 is the volume of the EDTA solution used in the titration, in millilitres.

5.2.3.12 Calcium ion reference solution, c (Ca^{2+}) = 0,01 mol/l.

Transfer (1 ± 0,002) g of calcium carbonate (5.2.3.9) (m_2) and about 100 ml of water to a 400 ml beaker, cover the beaker with a watch glass and carefully add about 10 ml of hydrochloric acid (5.2.3.2). After the calcium carbonate has dissolved completely, remove the carbon dioxide by boiling, then allow the solution to cool and make it up to 1 000 ml in a volumetric flask.

5.2.3.13 Calconcarboxylic acid indicator.

Grind 0,2 g of calconcarboxylic acid intensively with 20 g of anhydrous sodium sulfate in a mortar.

5.2.3.14 Eriochrome Black T indicator.

Grind 1 g of Eriochrome Black T intensively with 100 g of sodium chloride in a mortar.

5.2.4 Apparatus

5.2.4.1 Ordinary laboratory equipment.

5.2.4.2 Magnetic stirrer with magnetic rod.

5.2.4.3 pH-meter with glass electrode, capable of measuring to an accuracy of 0,05.

5.2.5 Procedure

5.2.5.1 Determination of CaO

To determine the calcium oxide content, transfer 25 ml (V_3) of the solution prepared in accordance with 5.1.5 to a 400 ml beaker, dilute the solution with about 150 ml of water and add 5 ml of triethanolamine solution (5.2.3.4).

Adjust the pH value of this solution to (12,5 ± 0,5) with sodium hydroxide solution (5.2.3.5) using a pH-meter. Add 0,1 g of calconcarboxylic acid indicator (5.2.3.13) and titrate with the EDTA solution while stirring constantly with a magnetic stirrer until the colour changes from wine red to blue, record the volume (V_4) of the EDTA solution added. During titration the pH-value shall not fall below 12,0.

5.2.5.2 Determination of total CaO and MgO content

Transfer about 150 ml of water and 25 ml (V_5) of the solution prepared in accordance with 5.1.5 to a 400 ml beaker. Add 5 ml of triethanolamine solution (5.2.3.4) and adjust the pH value of the solution to (10,5 ± 0,5) with buffer solution (5.2.3.10) using a pH-meter. Then add about 90 % of the volume of EDTA solution used during the calcium oxide titration (as described in 5.2.5.1) and, after adding 0,1 g of Eriochrome Black T indicator (5.2.3.14), titrate the solution until the colour changes from wine red to blue. Record the total volume (V_6) of EDTA solution added.

5.2.6 Evaluation and expression of results

The calcium oxide content of the sample expressed as CaO in mass fraction in percent is given by the following equation:

$$CaO = \frac{V_1 \times 0{,}04 \times 56{,}08 \times c \times V_4 \times 100}{V_3 \times 1\,000 \times m_1} \tag{4}$$

where

V_1 is the volume of the digestion solution (5.1.5), in millilitres;

V_3 is the volume of the aliquot of the digestion solution V_1 taken for titration as described in 5.2.5.1, in millilitres;

V_4 is the volume of EDTA solution used for the CaO determination as described in 5.2.5.1, in millilitres;

c is the concentration of the EDTA solution, as determined in 5.2.3.11;

m_1 is the mass, in grams, of the test portion used in 5.1.5.

The magnesium oxide content of the sample expressed as MgO in mass fraction in percent is given by the following equation:

$$\text{MgO} = \frac{V_1 \times 0{,}04 \times 40{,}311 \times c \times (V_6 - V_4) \times 100}{V_5 \times 1\,000 \times m_1} \qquad (5)$$

where

V_1 is the volume of the digestion solution (5.1.5), in millilitres;

V_5 is the volume of the aliquot of the digestion solution V_1 taken for titration as described in 5.2.5.2, in millilitres;

V_4 is the volume of EDTA solution used for the CaO determination as described in 5.2.5.1, in millilitres;

V_6 is the volume of EDTA solution used for the determination of the total CaO and MgO as described in 5.2.5.2, in millilitres;

c is the concentration of the EDTA solution, as determined in 5.2.3.11;

m_1 is the mass, in grams, of the test portion used in 5.1.5.

5.3 Sulfate (expressed as SO_3)

5.3.1 General

The method is used to determine the sulfate content of building lime.

5.3.2 Principle

The sulfate compounds in the sample are dissolved in hydrochloric acid and the pH value is adjusted to 1 to 1,5 to prevent precipitation of the oxides of iron and aluminium. The sulfate content is determined gravimetrically by boiling the solution and precipitating the sparingly soluble barium sulfate with a barium chloride solution.

5.3.3 Reagents

5.3.3.1 Hydrochloric acid, ρ (HCl) = 1,16 g/ml to 1,19 g/ml.

5.3.3.2 Hydrochloric acid, diluted, 1 + 1.

5.3.3.3 Hydrochloric acid, diluted, 1 + 50.

5.3.3.4 Nitric acid, ρ (HNO_3) = 1,40 g/ml to 1,42 g/ml.

5.3.3.5 Ammonium hydroxide solution, c (NH_4OH) = 25 %.

5.3.3.6 Ammonium hydroxide solution, diluted, 1 + 16.

5.3.3.7 Barium chloride, $BaCl_2 \cdot 2H_2O$.

5.3.3.8 Barium chloride solution.

Dissolve 120 g of barium chloride in water and make up to 1 000 ml with water in a volumetric flask.

5.3.3.9 Silver nitrate, $AgNO_3$.

5.3.3.10 Silver nitrate solution.

Dissolve 5 g of silver nitrate (5.3.3.9) in water, add 10 ml of nitric acid (5.3.3.4) and making up to 1 000 ml with water in a volumetric flask.

5.3.4 Apparatus

5.3.4.1 Ordinary laboratory equipment.

5.3.4.2 Analytical balance, accurate to 0,001 g.

5.3.4.3 Hot plate or sand bath.

5.3.4.4 Muffle furnace.

5.3.5 Procedure

Weigh out (1 ± 0,1) g to the nearest 0,001 g of the sample (m_3) into a 250 ml beaker, add 90 ml of cold water and then add 10 ml of hydrochloric acid (5.3.3.1) while stirring vigorously. Carefully heat the solution in a fume cupboard on a hot plate or in a sand bath to just below boiling point and leave at this temperature for 15 min. Filter through a fine filter paper (mean pore diameter of approximately 2 µm, ash content < 0,01 %) into a 400 ml beaker and wash the residue several times with small portions of hot diluted hydrochloric acid (5.3.3.3).

Dilute the filtrate to about 250 ml with water and, if necessary, adjust the pH value to 1 with hydrochloric acid (5.3.3.2) or ammonium hydroxide solution (5.3.3.6). Boil the solution for 5 min and then add 10 ml of hot barium chloride solution (5.3.3.8) dropwise to the still boiling solution while stirring continuously. Allow the solution to boil for a further 15 min to enable the precipitate to form satisfactorily. Leave the precipitation vessel on a hot plate at 60 °C overnight, ensuring that the solution will not evaporate down.

Filter the precipitate through a fine filter paper (mean pore diameter of approximately 2 µm, ash content < 0,01 %) and wash the residue until chloride-free with boiling water, using silver nitrate solution (5.3.3.10) to test the filtrate (see below). After washing the precipitate, rinse the stem of the funnel with a few drops of water. Wash the filter paper and contents with a few millilitres of water, collect the wash water in a test tube and add a few drops of silver nitrate solution. If no cloudiness or precipitate appears in the solution, chloride is absent. If this is not the case, repeat the washing process until the silver nitrate test is negative.

Transfer the filter paper and residue to a preweighed platinum crucible and incinerate to constant mass at (925 ± 25) °C in a muffle furnace. An incineration time of 15 min will generally be sufficient to achieve constant mass. Record the mass (m_4).

5.3.6 Evaluation and expression of results

The sulfate content expressed as SO_3 in mass fraction in percent is given by the following equation: