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Building lime - Part 2: Test methods

Baukalk - Teil 2: Prüfverfahren

Chaux de Construction - Partie 2: Méthodes d'essais

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Chaux de Construction - Partie 2: Méthodes d'essais

Baukalk - Teil 2: Prüfverfahren

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European foreword

This document (EN 459-2:2021) 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of an identical text or by endorsement, at the latest by January 2022, and conflicting national standards shall be withdrawn at the latest by January 2022.

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

This document supersedes EN 459-2:2010.

In comparison with EN 459-2:2010, the following modifications have been made:

- description of fusion with lithium tetraborate;
- a new Clause 5.1 included;
- amendments for the determinations of CaO and MgO;
- change of the order of methods for determination of CO₂;
- Phenolphthalein as indicator replaced by thymolphthalein;
- Amendments for the determination of available lime;
- amendments of the methods for particle size determination;
- amendment for the description of bulk density;
- amendments for the determination of reactivity;
- amendments for the Table B.1 and Table B.2 in Annex B;
- editorial changes were made and minor mistakes corrected.

EN 459, *Building lime*, is currently composed 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 document.

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Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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Introduction

The objective in this document 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 apply (Indications on drawings by “ISO 2768-1”).

All dimensions are in millimetres.

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1 Scope

This document specifies the test methods for all building limes covered by EN 459-1. They can also be applied to other lime materials, the standards for which call up these methods.

This document specifies in Table 2 the methods used for the chemical analyses and the determination of physical properties of building limes.

This document specifies 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 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 196-1:2016, *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, *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, *Metallic materials - Brinell hardness test - Part 1: Test method (ISO 6506-1)*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Sampling

4.1 General

Sampling shall be carried out as specified in 4.2 to 4.4 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.

4.2 Sampling of powdered material

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

4.3 Sampling of granular material

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

4.4 Sampling of lime putty and milk of lime

The spot sample size shall be $(10 \pm 5) \text{ dm}^3$.

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

4.5 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 any homogenization process (quartering, mixing, etc.) to produce a representative homogeneous test sample of suitable mass for the intended determinations.

Lime putty and milk of lime shall be dried before the chemical analysis (see 6.5.4.2).

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

Table 1 — Sample preparation for the single tests

Test	Clause in this document	Type and form of the building lime	Sample preparation
Chemical analysis	6	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 $\leq 0,2 \text{ mm}$
Grain size distribution by sieving	7.1 and 7.2	Quicklime	Material in the as-delivered state
Grain size distribution by air-jet sieving	7.2	Hydrated lime, hydrated dolomitic lime, lime with hydraulic properties	Material in the as-delivered state
Bulk density	7.3	All types of lime with hydraulic properties	See 7.3.2
Soundness	7.4.2.1 and 7.4.2.2	Hydrated lime, lime with hydraulic properties	Material in the as-delivered state
	7.4.2.3	Hydraulic lime with an SO_3 content of more than 3 % and up to 7 %	Material in the as-delivered state
	7.4.3	Hydrated lime, lime putty und hydrated dolomitic lime	Material in the as-delivered state
	7.4.4	Quicklime, lime putty, dolomitic quicklime, hydrated dolomitic lime	See 7.4.4.3.1
Setting times	7.5	Lime with hydraulic properties	Material in the as-delivered state

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Test	Clause in this document	Type and form of the building lime	Sample preparation
Reactivity	7.6	Quicklime, dolomitic quicklime	<p>Method 1:</p> <p>The test shall be performed in the as delivered state.</p> <p>If material contains particles coarser than 5 mm, the fraction above 5 mm shall be crushed to get 100 % of the material ≤ 5 mm.</p> <p>Method 2:</p> <p>The test shall be performed on materials of a grain size of ≥ 95 % passing 0,2 mm sieve.</p> <p>The method used for the test shall be declared in the test report.</p>
Yield	7.7	Quicklime	See 7.7.2
Mortar tests	7.8 to 7.10	Hydrated lime, hydrated dolomitic lime, lime with hydraulic properties	Material in the as-delivered state
Compressive strength	7.11	Lime with hydraulic properties	Material in the as-delivered state

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5 General requirements for testing

5.1 Methods of chemical analysis

The methods to be used for the chemical analysis of building lime products and the principle of each method are listed in Table 2.

Table 2 — Methods of chemical analysis

Determination	Method	Principle
Calcium oxide	6.3	Complexometric titration XRF melting tablet XRF powder tablet
Magnesium oxide	6.3	Complexometric titration XRF melting tablet XRF powder tablet ICP-OES AAS-Flame technique
Sulfate, expressed as SO ₃	6.4	Gravimetry XRF melting tablet XRF powder tablet ICP-OES Combustion at 1 300 °C minimum temperature and IR detection
Free water	6.5	Gravimetry, drying oven Gravimetry, moisture balance
Carbon dioxide	6.6 6.7	Volumetry Gravimetry Combustion at 900 °C minimum temperature and IR detection
Loss on ignition at 1 050 °C	6.8	Gravimetry
Available lime	6.9	Acidimetric titration

5.2 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 document.

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 two (see also 5.3).

EN 459-2:2021 (E)**5.3 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.

5.4 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 generally to one decimal place, except for results below 1.

If the result is below 1, then express the results, generally to two decimal places.

If the test is in duplicate, express the result as a mean value. 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.

5.5 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.

5.6 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 document, 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 1 volume of concentrated hydrochloric acid is to be mixed with 2 volumes of water.

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

5.7 Evaluation of test results**5.7.1 General**

The chemical requirements for building limes are specified in the relevant tables in EN 459-1.

5.7.2 Test results for quicklime

For quicklime the specified values correspond to the as-delivered product.

5.7.3 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 document for total calcium oxide and magnesium oxide (6.3), magnesium oxide (6.3), sulfate (6.4) and carbon dioxide (6.6 or 6.7) are for the products without subtraction of the free water and bound water content. To compare these values with EN 459-1 (see clauses for calcium lime or dolomitic lime), 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 6.6 or 6.7 and the loss on ignition as described in 6.8. 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 % of the sample using:

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

Calculate the factor F from the following formula:

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

5.7.4 Test results for available lime

The values for available lime, obtained by the application of the procedure described in 6.9 correspond to either available CaO for quicklime or available $\text{Ca}(\text{OH})_2$ for all other types (hydrated lime, lime putty, milk of lime, lime with hydraulic properties).

6 Chemical analysis

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6.1 Extraction with hydrochloric acid (Reference method)

6.1.1 General

Extraction with hydrochloric acid is used to dissolve building lime in order to determine appropriate parameters (see Table 2).

6.1.2 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.

6.1.3 Reagents

6.1.3.1 Hydrochloric acid, $\rho(\text{HCl}) = 1,16 \text{ g/ml}$ to $1,19 \text{ g/ml}$.

6.1.3.2 Hydrogen peroxide solution, $c(\text{H}_2\text{O}_2) = 30 \%$.

6.1.3.3 Hydrogen peroxide solution; diluted, 1 + 9.

6.1.3.4 Ammonium hydroxide solution, $c(\text{NH}_4\text{OH}) = 25 \%$.

6.1.3.5 Ammonium hydroxide solution, diluted, 1 + 9.

6.1.3.6 Ammonium chloride, NH_4Cl .