



SLOVENSKI STANDARD

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Gradbeno apno – 2. del: Metode preskušanja

Building lime - Part 2: Test methods

Baukalk - Teil 2: Prüfverfahren

Chaux de construction - Partie 2: Méthodes d'essai

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ICS:

91.100.10 Cement. Mavec. Apno. Malta Cement. Gypsum. Lime.
Mortar

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 459-2

October 2001

ICS 91.100.10

Supersedes EN 459-2:1994

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 16 February 2001.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 51 "Cement and building of limes", the secretariat which is held by IBN.

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of an identical text or by endorsement, at the latest by April 2002, and conflicting national standards shall be withdrawn at the latest by April 2002.

This European Standard supersedes EN 459-2:1994.

Opposite to EN 459-2:1994 the following modifications are presented:

- editorial changes were made and minor mistakes corrected
- alternative method for particle size determination of hydrated limes (prEN 12485:1996)
- vibrating table from EN 196-1:1994 was over taken
- Amendments for the determination of strength, chemical analysis and sampling of buildings limes

European Standard EN 459 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 EN 459-2:2001. However, given the chemical and physical properties of building limes, test methods other than those used for cement and masonry cement are necessary and have been incorporated into this European Standard.

The precision data (repeatability and reproducibility) are not available in all CEN member countries at the time being. After implementation of the three parts of EN 459 CEN/TC 51 will collect the necessary information to provide the precision data for the next revision of EN 459-2.

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

Introduction

For the European Standard for building lime EN 459-2:2001 the objective 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 should be applied (Indications on drawings by "ISO 2768-m").

All measurements are in millimetres.

1 Scope

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This European Standard describes the test methods for all building limes covered by EN 459-1:2001.

This European Standard describes reference test methods and in some cases alternative test methods. In the event of a dispute, only the reference method is used.

If methods other than the following are used it is necessary to show that they give results equivalent to those given by the reference methods.

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 (including amendments).

EN 196-1:1994, *Methods of testing cement – Part 1: Determination of strength*.

EN 196-2, *Methods of testing cement – Part 2: Chemical analysis of cement*.

EN 196-3:1994, *Methods of testing cement – Part 3: Determination of setting time and soundness*.

EN 196-6:1989, *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*.

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EN 196-21¹⁾, *Methods of testing cement – Part 21: Determination of the chloride, carbon dioxide and alkali content of cement.*

EN 459-1:2001, *Building lime – Part 1: Definitions, specifications and conformity criteria.*

EN 932-1, *Test for general properties of aggregates – Part 1: Methods for sampling.*

EN ISO 3696, *Water for analytical laboratory use – Specification and test methods (ISO 3696:1997).*

EN 10003-1, *Metallic materials - Brinell hardness test – Part 1: Test method.*

EN 10088-2, *Stainless steels – Part 2: Technical delivery conditions for sheet/plate and strip for general purposes.*

EN 12485, *Chemicals used for treatment of water intended for human consumption – Calcium carbonate, high-calcium lime and half-burnt dolomite – Test methods.*

ISO 383, *Laboratory glassware – Interchangeable conical ground joints.*

ISO 2768-1, *General tolerances - Part 1: Tolerances for linear and angular dimensions without individual tolerance indications.*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ASTM C 230-90, *Standard specification for flow table for use in tests of hydraulic cement.*

3 Sampling**3.1 General**

Sampling shall be carried out as specified in 3.1 to 3.4 taking into account the need to minimize moisture and carbon dioxide absorption.

3.2 Sampling of powdered material

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

Before carrying out chemical analysis, the sample shall be reduced in mass by means of a sample divider and/or by quartering to produce a sub-sample of suitable mass for the intended determinations. Any coarse material shall be reduced in size to less than 0,3 mm.

3.3 Sampling of granular material

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

Before carrying out chemical analysis, the sample shall be crushed, ground and reduced in mass to produce a sub-sample finer than 0,3 mm and of suitable mass for the intended determinations.

3.4 Sampling lime putties

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

Where lime putties are sampled, the increments shall be blended thoroughly.

¹⁾ EN 196-21 is currently being incorporated into EN 196-2.

4 Chemical analysis

4.1 General requirements for testing

4.1.1 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 a 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).

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

4.1.2 Expression of masses, volumes, factors and results

Express masses in grams to the nearest 0,0001 g and volumes from burettes in millilitres to the nearest 0,05 ml.

Express the factors of solutions, given by 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 (laboratory), repeat the test and take the mean of the two closest test results.

The results of all individual tests shall be recorded.

4.1.3 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.

4.1.4 Reagents

All reagents shall be of an analytical grade appropriate for the method being used. The water used shall comply with the appropriate grade specified in EN ISO 3696.

4.1.5 Evaluation of test results

The chemical requirements for building limes are specified in Table 2 of EN 459-1:2001. For quicklime the specified values correspond to the finished product.

For all other types (hydrated lime, lime putty, hydraulic and natural hydraulic lime) 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 calcium and magnesium oxide (4.2), magnesium oxide (4.3), carbon dioxide (4.4), sulfate (4.6) and available lime (4.7) are for the products without subtraction of the free water and bound water content. To compare these values with Table 2 of EN 459-1:2001, 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 4.4 and the loss on ignition as described in 4.5. 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 percentage by mass of the sample using.

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W_T = loss on ignition (m/m %) – carbon dioxide content (m/m %)

Calculate the factor F from the following equation:

$$F = 100 / (100 - W_T)$$

4.2 Calcium and magnesium oxide (CaO + MgO)

Determine the (CaO + MgO) content using the method given in EN 196-2.

4.3 Magnesium oxide (MgO)

Determine the (MgO) content using the method given in EN 196-2.

4.4 Carbon dioxide (CO₂)**4.4.1 Reference method**

Determine the CO₂ content using the reference method given in EN 196-21.

4.4.2 Volumetric determination of carbon dioxide (alternative method)**4.4.2.1 Principle**

The CO₂ contained in the building lime in the form of carbonates is given off by reaction with hydrochloric acid and determined volumetrically.

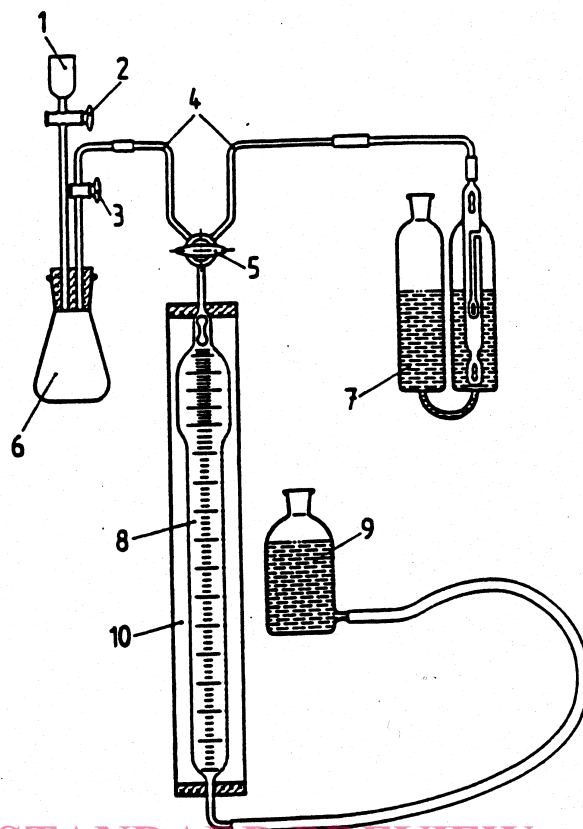
4.4.2.2 Apparatus

The apparatus is shown in Figure 1.

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Key

- 1 Dropping funnel
- 2 Stopcock 1
- 3 Stopcock 2
- 4 Capillary tubes
- 5 Three-way tap
- 6 Decomposition flask with a volume of 50 ml
- 7 Absorption vessel containing potassium hydroxide solution
- 8 Burette
- 9 Levelling vessel with sealing liquid
- 10 Jacketed tube filled with water

Figure 1 – Kleine apparatus for determination of carbon dioxide

EN 459-2:2001 (E)**4.4.2.3 Reagents**

a) Copper sulfate	CuSO_4 .
b) Concentrated hydrochloric acid	HCl ($\rho = 1,18$ to $1,19 \text{ g/cm}^3$).
c) Dilute hydrochloric acid	1 + 1 (1 part of concentrated hydrochloric acid and 1 part of water by volume).
d) Methyl red solution	Dissolve 0,2 g of methyl red in water and make up to 100 ml.
e) Sealing liquid	Add 20 g of sodium sulfate and 5 ml of concentrated sulfuric acid to water and make up to 100 ml and colour with a few drops of methyl red solution. The sealing liquid shall be saturated with CO_2 .
f) Potassium hydroxide solution	containing 50 % by mass of KOH.
g) Calcium carbonate	CaCO_3 dried to constant mass at $(250 \pm 5) ^\circ\text{C}$.

4.4.2.4 Procedure

The mass of the building lime sample used depends on the expected CO_2 content and shall be chosen approximately as follows:

2 g	for	$0 \% \leq \text{CO}_2 \leq 2 \%$;
1 g	for	$2 \% < \text{CO}_2 \leq 5 \%$;
0,5 g	for	$5 \% < \text{CO}_2 \leq 10 \%$;
0,3 g	for	$10 \% < \text{CO}_2 \leq 15 \%$;
0,2 g	for	$15 \% < \text{CO}_2 \leq 40 \%$;
0,1 g	for	$40 \% < \text{CO}_2 \leq 50 \%$.

Weigh out the sample accurately to 0,1 mg into the decomposition flask, add a spatula-tip of copper sulfate to bind any hydrogen sulfide formed and suspend in a little water.

Connect the flask to the apparatus with the aid of the double-bored stopper. A funnel and the feed tube to the measuring burette pass through the stopper. Open the stopcocks in these two lines. Bring the three-way tap to the position such that it connects the flask and measuring burette with one another. Fill the burette with sealing liquid up to the three-way tap by raising the levelling bottle. Now close stopcock 1 and fill the funnel with dilute hydrochloric acid (1 + 1). Add dilute hydrochloric acid (1 + 1) to the flask through the dropping funnel until the flask is half full. Stopcock 1 being closed, a little acid remains as sealing liquid in the funnel.

Allow the mixture to react for a few minutes in the cold and then heat it to the boiling point and boil for about another 3 min. Fill the flask completely with dilute hydrochloric acid (1 + 1) up to stopcock 2 with the aid of the dropping funnel in order to transfer the remaining gas mixture into the burette. Take care that no dilute hydrochloric acid also flows over. Close the burette with the three-way tap. After about 5 min, bring the sealing liquid in the burette and in the levelling bottle to the same level and read off the gas volume V_1 .

Turn the three-way tap to connect the measuring burette with the absorption vessel and wash out the air/ CO_2 mixture collected. For this, raise the levelling bottle so that all the gas is forced through the potassium hydroxide solution in the absorption vessel. The CO_2 is thereby absorbed. Repeat the absorption operation about seven or eight times until, finally, the measuring burette contains only the residual gas. Close the three-way tap, bring the sealing liquid in the burette and in the levelling vessel to the same level and read off the volume V_2 .

The difference in volume ($V_1 - V_2$) corresponds to the carbon dioxide content of the sample.

4.4.2.5 Evaluation

Calculate the carbon dioxide content as the content by mass of CO_2 , in percent from the equation:

$$C = 0,053 F_1 \frac{(V_1 - V_2) \times P}{T \times m_1} \quad (1)$$

where

C	is the carbon dioxide content by mass, in percent;
F_1	is the correction factor in accordance with 4.4.2.6;
V_1	is the volume of the gas before absorption, in millilitres;
V_2	is the volume of the gas after absorption, in millilitres;
P	is the corrected barometer reading, in pascals $\times 100$;
T	is the measurement temperature, in kelvins;
m_1	is the mass of the sample, in grams.

If the calibration and determination are carried out directly after one another, the temperature and atmospheric pressure need not be taken into consideration. For this case, equation (1) is simplified to:

$$C = \frac{4,397 (V_1 - V_2) \text{ of measurement}}{(V_3 - V_4) \text{ of calibration} \times m_1} \quad (2)$$

if 0,1 g of CaCO_3 is weighed out for the calibration.

Using this method the value calculated is expressed as a percentage of the "as delivered" product (refer to 4.1).

4.4.2.6 Calibration of the apparatus

Weigh 0,1 g of calcium carbonate, dried to constant mass at $(250 \pm 5)^\circ\text{C}$, to an accuracy of 0,5 mg into the decomposition flask. Carry out the determination as described in 4.4.2.4.

The volume difference $(V_3 - V_4)$ corresponds to the carbon dioxide content of the calibration material. Calculate the correction factor F_1 of the absorption apparatus from the following relationship, obtained by rearranging equation (1):

$$F_1 = \frac{82,96 \times T}{(V_3 - V_4) \times P} \quad (3)$$

The meanings of the symbols correspond to those given in 4.4.2.5.

The factor shall be in the range 1,00 to 1,04, otherwise the apparatus is to be checked for tightness and proper functioning and the calibration repeated.

4.5 Loss on ignition

Determine the loss on ignition using the method given in EN 196-2.

NOTE An alternative desiccant may be used, if it is proved to be as effective as magnesium perchlorate.

4.6 Sulfate (SO_3)

Determine the sulfate content using the method given in EN 196-2.

4.7 Available lime

4.7.1 General

Use the method given in 4.7.2 for air lime and the method given in 4.7.3 for hydraulic lime.

4.7.2 Determination of available lime content of air limes

4.7.2.1 Principle

The available (unbound) lime (calcium oxide and calcium hydroxide) is dissolved in a solution of sucrose and titrated with hydrochloric acid, phenolphthalein being used as an indicator.

To ensure complete dissolution of highly reactive limes ($t_u < 2$ min, according to 5.10.5) which are prone to form large particles of calcium hydroxide when slaked, those limes should be wetted with 1 ml of methanol before being added to the water.

4.7.2.2 Reagents

- | | |
|------------------------------|---|
| a) Water | |
| b) Sucrose | Refined sugar, commercially available |
| c) Methanol | CH_3OH |
| d) Hydrochloric acid | 1 mol/l (standard solution) |
| e) Phenolphthalein indicator | Dissolve 0,5 g of phenolphthalein in 50 ml of ethanol and dilute to 100 ml with water |

4.7.2.3 Procedure

Place 50 ml of water in a 300 ml conical flask. Transfer $(1 \pm 0,1)$ g of lime sample weighed to an accuracy of 1 mg to the flask and swirl to suspend. Add 10 g of sucrose, stopper the flask and keep the suspension in motion for 12 min to 15 min (magnetic stirrer, vibrating table or similar apparatus). Then filter through two fine filter papers. Wash the flask with six 25 ml portions of cold water and titrate the filtrate or an aliquot part with the hydrochloric acid using phenolphthalein as indicator.

EN 459-2:2001 (E)**4.7.2.4** Expression of results

Calculate the available lime content as CaO from the equation:

$$K_a = \frac{2,804 V_5 \times C_1}{m_2} \quad (4)$$

where

- K_a is the available lime content by mass, in percent;
- V_5 is the volume of hydrochloric acid used, in millilitres;
- C_1 is the factor of hydrochloric acid 1 mol/l;
- m_2 is the mass of the sample, in grams.

Using this method the value calculated is expressed as a percentage of the "as delivered" product (refer to 4.1).

4.7.3 Determination of available lime content of hydraulic lime**4.7.3.1** Principle

The available (unbound) lime (calcium oxide and calcium hydroxide) is extracted by means of ethylacetoacetate and the extract titrated with hydrochloric acid, bromophenol blue being used as an indicator.

4.7.3.2 Reagents

- | | |
|-----------------------------------|---|
| a) Ethylacetoacetate | $\text{CH}_3\text{COCH}_2\text{COOC}_2\text{H}_5$, for synthesis |
| b) 2-Propanol | $(\text{CH}_3)_2\text{CHOH}$ |
| c) Concentrated hydrochloric acid | HCl, see 4.4.2.3 b) |
| d) Hydrochloric acid | 1 mol/l (standard solution) |
| e) Dilute hydrochloric acid | 0,1 mol/l (standard solution) |
| f) Sodium hydroxide on support | NaOH, for elementary analysis |
| g) Ethanol | $\text{C}_2\text{H}_5\text{OH}$ |
| h) Bromophenol blue indicator | Tetrabromophenol sulphonphthalein |
| i) Bromophenol blue solution | Dissolve 0,1 g bromophenol blue in ethanol and make up the solution to 100 ml with the same |
| j) Isobutanol | $(\text{CH}_3)_2\text{CHCH}_2\text{OH}$ |

4.7.3.3 Procedure

Place 12 ml ethylacetoacetate and 80 ml Isobutanol in a 250 ml Erlenmeyer flask with a joint conforming to ISO 383, nominal diameter of the joint 29,2 mm, large 32 mm. Add approximately 1 g of the sample, dried out of contact with the air (vacuum-drying oven) at $(105 \pm 5)^\circ\text{C}$, ground to analytical fineness and weighed to the nearest 0,1 mg, to the mixture and shake. Bring the mixture to the boil while stirring (e. g. with a magnetic stirrer) and then reflux. The lower end of the reflux condenser is fitted with a joint conforming to ISO 383, nominal diameter of the joint 29,2 mm, large 32 mm, while a tube containing sodium hydroxide on a support is connected to its upper end and provides protection against carbon dioxide penetrating from the air.

Stop the extraction after 15 min. Cool the extract to below 50°C and siphon it off into a 250 ml suction bottle through a glass filter funnel. Insert a filter paper for slow filtration speed (particle retention size $2,0\ \mu\text{m}$ to $2,2\ \mu\text{m}$) and a filter paper for a medium filtration speed (particle retention size $6,5\ \mu\text{m}$) into the filter funnel. (Do not cut the filter paper by hand). Moisten the filters with the ethylacetoacetate prior to filtration. If the filtrate is cloudy, carry out a second filtration with the same filter combination. Wash the flask and residue with a maximum of 50 ml 2-propanol. It is not necessary to transfer the last remnants of the residue to the filter funnel.

Add 3 to 4 drops of bromophenol blue solution to the filtrate and titrate with hydrochloric acid until the colour changes to yellow. The concentration of hydrochloric acid is dependent on the probable available lime content. For HL 2 and/or NHL 2 titrate with the 1 mol/l hydrochloric acid and for HL 3,5, HL 5, NHL 3,5 and NHL 5 titrate with the 0,1 mol/l hydrochloric acid.

4.7.3.4 Expression of results

Calculate the available lime content in HL 2 and/or NHL 2 from the equation: