BS EN 459-2:2010

BSI Standards Publication

Building lime

Part 2: Test methods

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National foreword

This British Standard is the UK implementation of EN 459-2:2010. It supersedes BS EN 459-2:2001 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/516/11, Lime.

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

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

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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of an identical text or by endorsement, at the latest by March 2011, and conflicting national standards shall be withdrawn at the latest by March 2011.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 $(10 \pm 5)\ \mathrm{dm}^3$.

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 ≤ 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:

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

Calculate the factor F from the following equation:

$$F = 100/(100 - W_T) \tag{2}$$

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:

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

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:

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

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:

$$SO_3 = \frac{0{,}343 \times m_4 \times 100}{m_3} = \frac{34{,}3 \times m_4}{m_3} \qquad (6)$$

where

m_4 is the final mass of $BaSO_4$, in grams;

m_3 is the mass of the test portion, in grams.

5.4 Free water

5.4.1 General

The method is used to determine free water in building lime. In the case of a hydrated lime or a lime with hydraulic properties, free water means the moisture attached to the product, in the case of milk of lime or lime putty, it refers to the water content of the suspension.

5.4.2 Principle

When heating a sample of hydrated lime or lime with hydraulic properties to (105 ± 5) °C, or for milk of lime or lime putty to (150 ± 5) °C the free water is driven off. The loss of mass at this temperature is referred to as free water in the case of hydrated lime and lime with hydraulic properties, and as the content of water in the suspension for milk of lime or lime putty.

5.4.3 Apparatus

5.4.3.1 Ordinary laboratory equipment.

5.4.3.2 Drying oven, thermostatically controlled to maintain a temperature of (105 ± 5) °C or (150 ± 5) °C (alternative equipment 1).

5.4.3.3 Automated moisture balance, being capable to be controlled between (105 ± 5) °C or (150 ± 5) °C (alternative equipment 2).

5.4.3.4 Analytical balance, accurate to 0,001 g.

5.4.3.5 Desiccator, containing drying agent.

5.4.3.6 Unglazed porcelain crucible.

5.4.3.7 Pipette.

5.4.4 Procedure

5.4.4.1 Hydrated lime and lime with hydraulic properties

Weigh between (5 ± 0,1) g and (10 ± 0,1) g to the nearest 0,001 g (m_5) of the sample in the as-delivered state in a pre-weighed crucible. Dry the sample until constant mass using the oven (5.4.3.2) or the balance (5.4.3.3). When using the oven limit the time of drying to approximately 2 h. Cover the crucible after removing it from the oven to prevent carbon dioxide and water vapour in the atmosphere from being absorbed. Cool in the desiccator and reweigh.

5.4.4.2 Milk of lime and lime putty

Homogenize the suspension by shaking before taking the sample aliquot. Use a pipette to take approximately 20 g and weigh to the nearest of 0,0,001 g in a glass vessel. Dry the sample to constant mass using the oven (5.4.3.2) or the balance (5.4.3.3). Cover the crucible after removing it from the oven to prevent carbon dioxide and water vapour in the atmosphere from being absorbed. After cooling down to ambient temperature in the desiccator, determine the loss in mass.

5.4.5 Evaluation and expression of results

The free water content expressed as H_2O in mass fraction in percent is given by the following equation:

$$\mathrm{H_2O} = \frac{(m_5 - m_6)}{m_5} \times 100 \tag{7}$$

where

m_5 is the mass of the test portion before heating, in grams;

m_6 is the mass of the test portion after heating, in grams.

5.5 Gravimetric determination of carbon dioxide (CO_2) (reference method)

5.5.1 Principle

The sample is treated with phosphoric acid to decompose the carbonate present. The carbon dioxide liberated is entrained in a current of carbon dioxide-free gas or air through a series of absorption tubes. The first two remove hydrogen sulfide and water and the following absorb carbon dioxide. Two absorption tubes, each containing a granular absorbent for carbon dioxide and anhydrous magnesium perchlorate to retain the water formed during the absorption reaction are weighed to determine the mass of carbon dioxide released.

5.5.2 Reagents

5.5.2.1 Sulfuric acid, H_2SO_4, concentrated, > 98 %.

5.5.2.2 Phosphoric acid, $\rho (H_3PO_4)$ = 1,71 g/ml to 1,75 g/ml.

5.5.2.3 Copper (II) sulfate ($CuSO_4 \cdot 5H_2O$).

5.5.2.4 Saturated water solution of copper (II) sulphate.

5.5.2.5 Absorbent for hydrogen sulphide.

Place a weighed quantity of dried pumice stone with a grain size between 1,2 mm and 2,4 mm into a flat dish and cover with a volume of saturated copper sulfate solution (5.5.2.4) so the mass of the copper sulfate solution is approximately half of that of the pumice stone. Evaporate the mixture to dryness, while stirring frequently with a glass rod. Dry the contents of the dish for at least 5 h in an oven at a temperature of (150 ± 5) °C. Allow the solid mixture to cool in a desiccator and store in an airtight bottle.

5.5.2.6 Absorbent for water.

Anhydrous magnesium perchlorate ($Mg(ClO_4)_2$) with a particle size between 0,6 mm and 1,2 mm.

5.5.2.7 Absorbent for carbon dioxide.

Synthetic silicates with a particle size between 0,6 mm to 1,2 mm impregnated with sodium hydroxide (NaOH).

NOTE This absorbent can be obtained ready for use.

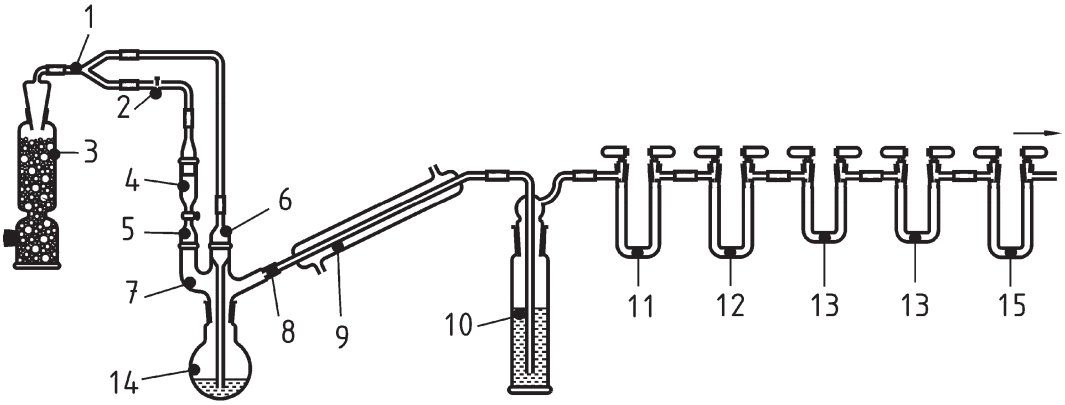
5.5.3 Apparatus

5.5.3.1 Analytical balance accurate to 0,001 g.

5.5.3.2 Apparatus for the determination of the carbon dioxide (reference method).

Typical apparatus is shown in Figure 1 which can be fitted with either a cylindrical pressure container, a small electrical compressor or a suitable suction pump which will ensure an even flow of gas or air. The gas (air or nitrogen) entering the apparatus has previously had its carbon dioxide removed by first being passed through an absorbent tube or tower containing the carbon dioxide absorbent (5.5.2.7). The apparatus consists of a 100 ml distillation flask (14) fitted with a three neck adaptor. Neck (5) is connected to a dropping funnel (4), neck (6) to a connecting tube and neck (8) to a water cooled condenser. The funnel onto (5) and the connecting tube onto (6) are joined together by means of a Y-piece (1), so that the carbon dioxide-free air can flow either through the connecting tube or the funnel by means of a Mohr clip (2).

After the condenser (9), the gas is passed through concentrated sulfuric acid (5.5.2.1) (10), then through absorption tubes containing the absorbent for hydrogen sulfide (5.5.2.5) (11) and for water (5.5.2.6) (12) and subsequently through two absorption tubes (13) which can be weighed and which are three-quarters filled with the absorbent for carbon dioxide (5.5.2.7) and a quarter with the absorbent for water (5.5.2.6). The absorbent for carbon dioxide (5.5.2.7) is placed upstream of the absorbent for water (5.5.2.6) with respect to the gas flow. Absorption tubes (13) are followed by an additional absorption tube (15), which also contains the absorbent for carbon dioxide and water, which is fitted in order to protect second absorption tube (13) against penetration by carbon dioxide and water from the air.



Key

1 Y-piece
2 Mohr clip
3 absorption tower containing carbon dioxide absorbent (5.5.2.7)
4 dropping funnel
5 dropping funnel connector
6 connecting tube connector
7 three-armed still head
8 condensor connector
9 condensor
10 wash bottle with concentrated sulfuric acid (5.5.2.1)
11 absorption tube with absorbent for hydrogen sulfide (5.5.2.5)
12 absorption tube with absorbent for water (5.5.2.6)
13 absorption tubes with absorbents for carbon dioxide (5.5.2.7) and water (5.5.2.6)
14 100 ml distillation flask
15 absorption tube with absorbents for carbon dioxide (5.5.2.7) and water (5.5.2.6)

Figure 1 — Typical apparatus for the determination of carbon dioxide (reference method)

The absorption tubes (13) which are to be weighed may have, for example, the following approximate sizes.

a) external distance between branches: 45 mm;

b) internal diameter: 20 mm;

c) distance between the lower part of the tube and the upper part of the ground section: 75 mm;

d) tube wall thickness: 1,5 mm.

5.5.4 Procedure

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

a) 2 g for 0 % $\leq CO_2 \leq$ 2 %;

b) 1 g for 2 % $< CO_2 \leq$ 5 %;

c) 0,5 g for 5 % $< CO_2 \leq$ 10 %;

d) 0,3 g for 10 % $< CO_2 \leq$ 15 %;

e) 0,2 g for 15 % $< CO_2 \leq$ 40 %;

f) 0,1 g for 40 % $< CO_2 \leq$ 50 %.

Weigh out the sample to 0,001 g (m_7) and place it in a dry 100 ml distillation flask. Connect the flask to the apparatus as shown in Figure 1, but without the two absorption tubes (13). Pass a current of carbon dioxide-free gas through the apparatus for 15 min at approximately three bubbles per second (bubble counter) via the connecting tube onto (6) (branch onto (5), Mohr clip closed). Release the Mohr clip and remove the gas supply from the funnel (4). Add 30 ml concentrated phosphoric acid (5.5.2.2) into the dropping funnel and reconnect the gas supply to fill the funnel (4).

Condition the closed absorption tubes (13) for 15 min in the balance case in order to achieve temperature equilibrium. Then weigh each tube separately. Shut off the flow of gas and attach the tubes to the apparatus as shown in Figure 1.

NOTE Care should be taken when handling the tubes to avoid affecting their weight, causing damage or sustaining injury. It is advisable to wear protective gloves when carrying out this operation.

Reopen the gas flow. After 10 min close absorption tubes (13), remove them, place them in the balance case for 15 min and then weigh them separately. Repeat the passage of gas, removal and weighing of absorption tubes (13) for as long as is required for the results of two successive weighings of a tube not to differ by more than 0,000 5 g.

If the change in mass of the absorption tubes (13) remains greater than 0,000 5 g, renew the absorbents in tubes (11) and (12).

Attach the weighed absorption tubes (13) to the apparatus, as shown in Figure 1. Open the funnel tap and allow the phosphoric acid (5.5.2.2) to drop into the distillation flask (14). After the reaction has ceased, heat the contents of the flask to boiling and boil gently for 5 min. Maintain the gas flow through the apparatus until the flask has cooled to room temperature.

Close absorption tubes (13), remove them and place them in the balance case for 15 min and then weigh them separately. The increase in mass of each tube is used for the calculation of the carbon dioxide content.

The carbon dioxide is practically completely absorbed by first tube (13). If the increase in mass of second tube (13) exceeds 0,000 5 g, renew the absorbent in first tube (13) and start the test again.

5.5.5 Evaluation and expression of results

Calculate the carbon dioxide content as CO_2 in mass fraction in percent from the following equation:

$$\mathrm{CO}_2 = \frac{(m_8 + m_9)}{m_7} \times 100 \quad (8)$$

where

m_7 is the mass of the test portion, in grams;

m_8 is the increase in mass of first tube (13) after absorption, in grams;

m_9 is the increase in mass of second tube (13) after absorption, in grams.

5.6 Volumetric determination of carbon dioxide (CO_2) (alternative method)

5.6.1 Principle

The CO_2 contained in the building lime in the form of carbonates is given off by reaction with hydrochloric acid and determined volumetrically.

5.6.2 Reagents

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

5.6.2.2 Hydrochloric acid, diluted, 1 + 1.

5.6.2.3 Sulfuric acid, H_2SO_4, concentrated, > 98 %.

5.6.2.4 Copper sulfate, $CuSO_4.5H_2O$.

5.6.2.5 Methyl red.

5.6.2.6 Methyl red solution.

Dissolve 0,2 g of methyl red (5.6.2.5) in water and make up to 100 ml

5.6.2.7 Sodium sulfate, Na_2SO_4

5.6.2.8 Sealing liquid.

Add 20 g of sodium sulfate (5.6.2.7) and 5 ml of sulfuric acid (5.6.2.3) to water, make up to 100 ml and colour with a few drops of methyl red solution. The sealing liquid shall be saturated with CO_2.

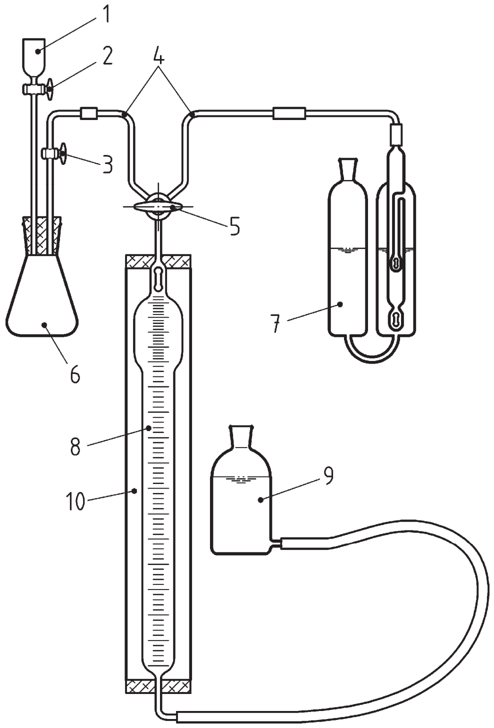
5.6.2.9 Potassium hydroxide solution, c (KOH) = 50 %.

5.6.2.10 Calcium carbonate, $CaCO_3$, dried to constant mass at (200 ± 10) °C.

5.6.3 Apparatus

5.6.3.1 Analytical balance, accurate to 0,001 g.

5.6.3.2 Apparatus for the determination of the carbon dioxide (alternative method).



Key

1	dropping funnel with a volume of 100 ml	7	absorption vessel with a volume of 100 ml containing potassium hydroxide solution (5.6.2.9) fitted with trap
2	stopcock 1	8	burette with a volume of 100 ml
3	stopcock 2	9	levelling vessel with a volume of 500 ml with sealing liquid (5.6.2.8)
4	capillary tubes	10	jacketed tube filled with water
5	three-way tap		
6	decomposition flask with a volume of 50 ml		

Figure 2 — Typical apparatus for determination of carbon dioxide (alternative method)

5.6.4 Procedure

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

a) 2 g for 0 % $\leq CO_2 \leq$ 2 %;

b) 1 g for 2 % $< CO_2 \leq$ 5 %;

c) 0,5 g for 5 % $< CO_2 \leq$ 10 %;

d) 0,3 g for 10 % $< CO_2 \leq$ 15 %;

e) 0,2 g for 15 % $< CO_2 \leq$ 40 %;

f) 0,1 g for 40 % $< CO_2 \leq$ 50 %.

Weigh out the sample accurately to 0,001 g (m_{10}) into the decomposition flask, add a spatula-tip of copper sulfate (5.6.2.4) 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 (5.6.2.8) up to the three-way tap by raising the levelling bottle. Now close stopcock 1 and fill the funnel with dilute hydrochloric acid (5.6.2.2). Add dilute hydrochloric acid (5.6.2.2) 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 (5.6.2.2) 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_7.

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 (5.6.2.9) 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_8.

The difference in volume ($V_7 - V_8$) corresponds to the carbon dioxide content of the sample.

5.6.5 Calibration of the apparatus

Weigh 0,1 g of calcium carbonate (5.6.2.10) to an accuracy of 0,001 g into the decomposition flask. Carry out the determination as described in 5.6.4.

The volume difference ($V_9 - V_{10}$) 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 (9):

$$F_1 = \frac{82{,}96 \times T}{(V_9 - V_{10}) \times P} \tag{9}$$

The meanings of the symbols correspond to those given in 5.6.6.

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

5.6.6 Evaluation and expression of results

Calculate the carbon dioxide content as CO_2 in mass fraction in percent from the following equation:

$$CO_2 = 0{,}053 \quad F_1 \frac{(V_7 - V_8) \cdot P}{T \cdot m_{10}} \tag{10}$$

where

F_1 is the correction factor in accordance with 5.6.5;

V_7 is the volume of the gas before absorption, in millilitres;

V_8 is the volume of the gas after absorption, in millilitres;

P is the corrected barometer reading, in pascals × 100;

T is the measurement temperature, in kelvins;

m_{10} 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 (10) is simplified to:

$$CO_2 = \frac{4{,}397(V_7 - V_8)\ \text{of measurement}}{(V_9 - V_{10})\ \text{of calibration} \cdot m_{l0}} \tag{11}$$

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

5.7 Loss on ignition

5.7.1 General

The method is used to determine the loss on ignition in building lime.

5.7.2 Principle

The loss on ignition of the materials concerned is determined at (1 050 ± 25) °C.

5.7.3 Apparatus

5.7.3.1 Ordinary laboratory equipment.

5.7.3.2 Electric furnace, capable of being maintained at (1 050 ± 25) °C, with a thermoelectric temperature indicator.

5.7.3.3 Analytical balance, accurate to 0,001 g.

5.7.3.4 Desiccator, containing drying agent.

5.7.3.5 Unglazed porcelain or platinum crucible.

5.7.4 Procedure

5.7.4.1 Hydrated lime and lime with hydraulic properties

Weigh (5 ± 0,1) g to the nearest of 0,001 g of the sample (m_{11}) in the as-delivered state in a pre-weighed crucible. Heat the sample in the furnace at (1 050 ± 25) °C for 2 h. Cover the crucible after removing it from the furnace to prevent carbon dioxide and water vapour in the atmosphere from being absorbed. Cool to room temperature in the desiccator and weigh (m_{12}).

5.7.4.2 Milk of lime and lime putty

The water content of the milk of lime shall be determined as specified in 5.4.4.2. After drying the sample this way, the determination shall be carried out as specified in 5.7.4.1.

5.7.5 Evaluation and expression of results

The loss on ignition expressed as LoI in mass fraction in percent is given by the following equation:

$$LoI = \frac{(m_{11} - m_{12})}{m_{11}} \cdot 100 \tag{12}$$

where

m_{11} is the mass of the sample before ignition at (1 050 ± 25) °C, in grams;

m_{12} is the mass of the sample after ignition at (1 050 ± 25) °C, in grams.

5.8 Available lime

5.8.1 General

The method serves to determine the available lime content:

- in calcium lime in the form of quicklime and hydrated lime;
- in calcium lime in the form of milk of lime and lime putty (in dry substance after drying as described in 5.4.4.2); and
- in all types of lime with hydraulic properties.

The suspended samples of lime with hydraulic properties shall be filtered for the titration.

This method designates those constituents which enter into the reaction under the conditions of this specified method. The interpretation of results obtained by the following method shall be determined in consideration of this limiting definition.

5.8.2 Principle

The sample is slaked and dispersed with water. To avoid agglomeration of calcium oxide on slaking which can lead to incomplete suspension of a quicklime sample such limes shall be heated. The lime is solubilized by reaction with sugar to form calcium sucrate which is then determined by titration against hydrochloric acid using phenolphthalein as the indicator.

5.8.3 Reagents

5.8.3.1 Hydrochloric acid, standard volumetric solution, c (HCl) = 1 mol/l.

5.8.3.2 Sodium hydroxide solution, c (NaOH) = 0,1 mol/l.

5.8.3.3 Phenolphthalein indicator solution.

Dissolve 0,5 g of phenolphthalein in 50 ml of ethanol (5.8.3.4) and dilute to 100 ml with water.

5.8.3.4 Ethanol, ρ = 0,79 g/ml.

5.8.3.5 Water, freshly boiled to remove CO_2 and cooled.

5.8.3.6 Sucrose, refined sugar, commercially available.

5.8.3.7 Sucrose solution.

Prepare a 40 % solution (w/v) using refined sugar and CO_2-free water (5.8.3.5) in a large beaker and stir until dissolved. Add several drops of phenolphthalein indicator solution. Add NaOH solution (5.8.3.2) dropwise with stirring until a faint pink colour persists. Stock solution of sugar may be made for convenience; however, it shall not be stored for more than two days. As an alternative the acidity of each lot of sugar can be determined, and a correction applied to the titration.

5.8.4 Apparatus

5.8.4.1 Ordinary laboratory equipment.

5.8.4.2 Analytical balance, accurate to 0,001 g.

5.8.4.3 Heatable magnetic stirrer, with magnetic rod.

5.8.4.4 Cold-water bath.

5.8.4.5 Buchner funnel.

5.8.4.6 Burette, 50 ml.

5.8.5 Procedure

5.8.5.1 For quicklime

Weigh (1 ± 0,1) g of quicklime (m_{13}) to the nearest of 0,001 g and transfer it immediately to a 500 ml Erlenmeyer flask containing about 100 ml of water. Cover the flask with a watch glass and bring the contents to the boil while stirring on the heatable magnetic stirrer. After boiling for 5 min, spray the wall of the conical flask with approximately 50 ml of water. Remove from the hot plate, stopper the flask loosely, and place in a cold-water bath to cool to room temperature.

5.8.5.2 For all other products

Weigh (1,3 ± 0,1) g of hydrated lime, milk of lime, lime putty or lime with hydraulic properties (m_{14}) to the nearest of 0,001 g and transfer it immediately to a 500-ml Erlenmeyer flask containing about 150 ml of water.

5.8.6 Sugar extraction

Add 50 ml of the neutralized sugar solution (or, alternatively, 20 g of pure sugar and additionally 40 ml of water) to the flask containing the sample. Stopper the flask and shake the contents for (10 ± 2) min to react.

5.8.7 Determination for calcium lime

Remove stopper, add four to five drops of phenolphthalein indicator solution and wash down the stopper and sides of the flask with water.

Then slowly add hydrochloric acid dropwise (at a rate of 12 ml/min) from a 50-ml burette until the solution just becomes colourless. Now stir for 60 s without adding further hydrochloric acid and then titrate dropwise (at a rate of 4 ml/min) to decolouration, ignoring any red colouration of the solution which may return after some seconds.

5.8.8 Determination for lime with hydraulic properties

Filter the suspension using a Buchner funnel through two fine filter papers (mean pore diameter of approximately 2 µm, ash content < 0,01 %). Wash the flask and the filter with water. Titrate the filtrate with the hydrochloric acid using phenolphthalein as indicator.

5.8.9 Evaluation and expression of results

The content of available lime, expressed as available CaO of quicklime or available $Ca(OH)_2$ of all other products, and expressed in mass fraction in percent is given by one of the following equations:

for quicklime: available CaO = $$\frac{28{,}04 \times V \times 100}{1\,000 \times m_{13}} = \frac{2{,}804 \times V}{m_{13}} \qquad (13)$$

for all other products: available $Ca(OH)_2$ = $$\frac{37{,}05 \times V \times 100}{1\,000 \times m_{14}} = \frac{3{,}705 \times V}{m_{14}} \qquad (14)$$

where

V is the volume of hydrochloric acid used, in millilitres;

m_{13} is the mass of the sample of CaO product, in grams;

m_{14} is the mass of the sample of $Ca(OH)_2$ product, in grams.

6 Physical tests

6.1 Particle size by dry sieving

6.1.1 General

This method is used for the determination of the particle size distribution. It applies to quicklime according to EN 459-1:2010, Tables 5 and 12.

6.1.2 Principle

The test consists of dividing up and separating, by means of a series of sieves, a material into several particle size classifications of decreasing sizes. The aperture sizes and the number of sieves are selected in accordance with the requirements in Tables 5 and 12 of EN 459-1:2010.

The mass of the particles retained on the various sieves is related to the initial mass of the material. The cumulative percentages passing each sieve are reported in numerical form (see Annex A).

6.1.3 Apparatus

6.1.3.1 **Test sieves,** aperture sizes 10,0 mm, 5 mm and 2,0 mm.

6.1.3.2 **Tightly fitting pan and lid,** for the sieves.

6.1.3.3 **Trays or other suitable containers,** of sufficient size to contain the test portion.

6.1.3.4 **Balance,** accurate to 0,001 g.

6.1.3.5 **Sieving machine** (optional).

6.1.3.6 **Sample divider.**

6.1.4 Preparation of test portions

Samples shall be reduced by means of a sample divider (6.1.3.6). The mass of each test portion shall be recorded.

6.1.5 Procedure

Pour the sample into the sieving column. The column comprises a number of sieves fitted together and arranged, from top to bottom, in order of decreasing aperture sizes with the pan and lid.

Shake the column, manually or mechanically, then remove the sieves one by one, commencing with the largest aperture size opening and shake each sieve manually ensuring no material is lost by using, for example, a pan and lid.

Transfer all the material which passes each sieve onto the next sieve in the column before continuing the operation with that sieve.

Weigh the retained material for the sieve with the largest aperture size and record its mass as R_1. Carry out the same operation for each of the sieves immediately below and record the mass retained as R_2 or R_3.

Weigh the screened material remaining in the pan and record its mass as P. This material is sieved following the air-jet sieving method in 6.2.

6.1.6 Evaluation and expression of results

Record the various masses on a test data sheet, an example of which is given in Annex A.

Calculate the mass retained on each sieve as a $m\,(P_\mathrm{i})$ in mass fraction in percent of the original mass M_1 using the following equation:

$$m\,(P_\mathrm{i}) = \frac{R_\mathrm{i} \times 100}{M_1} \qquad (15)$$

where

M_1 is the mass of the test portion, in grams;

R_i is the mass of the residue retained on the test sieves, in grams.

Calculate the cumulative percentage of the original mass retained on each sieve down to the 0,09 mm sieve inclusive.

Calculate the cumulative percentage of the original mass passing each sieve down to the 0,09 mm sieve inclusive.

6.2 Particle size by air-jet sieving

6.2.1 General

The method determines the retention on sieving of particles which pass a 2,0 mm test sieve. The method can be used to determine the particle size of the agglomerates of very fine particles. This method uses test sieves with aperture sizes of 0,2 mm and 0,09 mm.

This method applies to hydrated lime, all types of lime with hydraulic properties and quicklime according to EN 459-1:2010, Tables 5,6, 12, 13, 18, 22 and 26.

6.2.2 Apparatus

6.2.2.1 Air-jet sieving apparatus, of the general form shown in Figure 3.

The apparatus shall be set to give a pressure difference of 2 kPa to 2,5 kPa across the sieves.

6.2.2.2 Test sieves, 200 mm diameter, aperture sizes 0,2 mm and 0,09 mm.

NOTE The effective operation of some makes of air-jet apparatus can require non-standard sieve frames and additional gaskets. This is permissible, provided the sieving medium and general method of construction comply with the requirements of this standard.

6.2.2.3 Trays or other suitable containers, of sufficient size to contain the test portion.

6.2.2.4 Balance, accurate to 0,001 g.

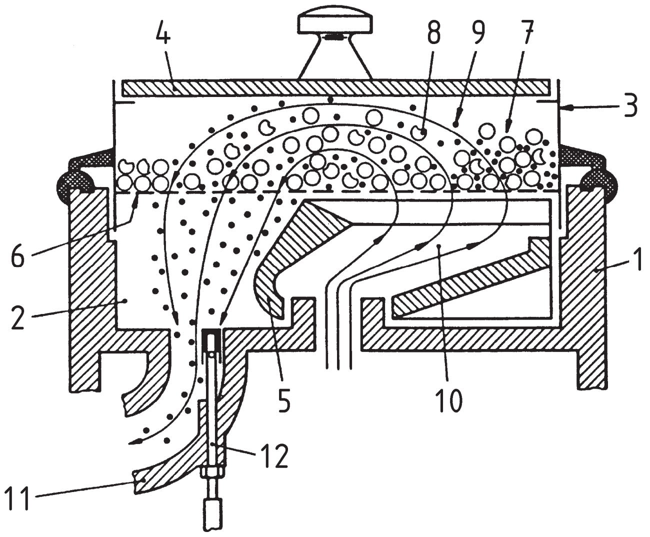
6.2.2.5 Soft brush.

6.2.2.6 Ultrasonic cleaning bath, for cleaning the mesh of the sieves.

6.2.2.7 Mallet, if there is a tendency for material to adhere to the lid of the apparatus.

A rubber or plastic tipped mallet is preferred.

6.2.2.8 Drying oven (optional), thermostatically controlled to maintain a temperature of (105 ± 5) °C.



Key

1	housing
2	dish
3	sieve drum
4	lid
5	slit-nozzle
6	sieve
7	test sample
8	oversize material
9	undersize material
10	air jet
11	air discharge
12	pressure gauge socket, with dust hood

Figure 3 — Typical apparatus for air-jet sieving

6.2.3 Procedure

Weigh an appropriate amount of the building lime sample to the nearest 0,001 g (m_{15}). Fit the test sieve with the aperture size 0,09 mm into the apparatus and transfer all of the test portion onto the sieve mesh. Take care not to lose any of the test portion.

Fit the lid and switch on the apparatus. Check that the vacuum created is above the minimum value stated in the manufacturer's instructions, and that the slit nozzle is rotating properly. If material adheres to the lid of the apparatus, gently tap the centre of the lid with the mallet. If the material agglomerates under the action of the air-jet, interrupt the sieving process, and break up the agglomerates with the soft brush taking care not to push particles through the sieve.

After (5 ± 0,2) min, switch off the apparatus and carefully remove the sieve. Transfer the material retained on the sieve into a tray or other suitable container. Carefully clean the mesh of the sieve over the tray using a soft brush. Determine the mass of the residue, including the material brushed from the sieve mesh, and record the mass to the nearest 0,001 g.

Refit the sieve into the apparatus and transfer all of the residue back to the sieve mesh. Repeat the weighing and sieving stages until the sieving end-point has been achieved, and record the end-point mass to the nearest 0,001 g. The sieving end-point is defined as being when not more than 0,2 % of the mass of the original test portion passes through the sieve in 1 min.

Fit the 0,20 mm test sieve into the apparatus, and repeat the weighing and sieving stages until the mass of the residue confirms that the sieving end-point has been reached. Record the end-point mass to the nearest 0,001 g.

6.2.4 Evaluation and expression of results

The mass retained on each sieve expressed as $m\,(P_{0{,}20 \text{ or } 0{,}09})$ in mass fraction in percent is given by the following equation:

$$m\,(P_{0{,}20} \text{ or }_{0{,}09}) = \frac{m_{16} \times 100}{m_{15}} \qquad (16)$$

where

m_{15} is the mass of the dry substance in the test portion, in grams;

m_{16} is the mass of the residue retained on the test sieves, in grams.

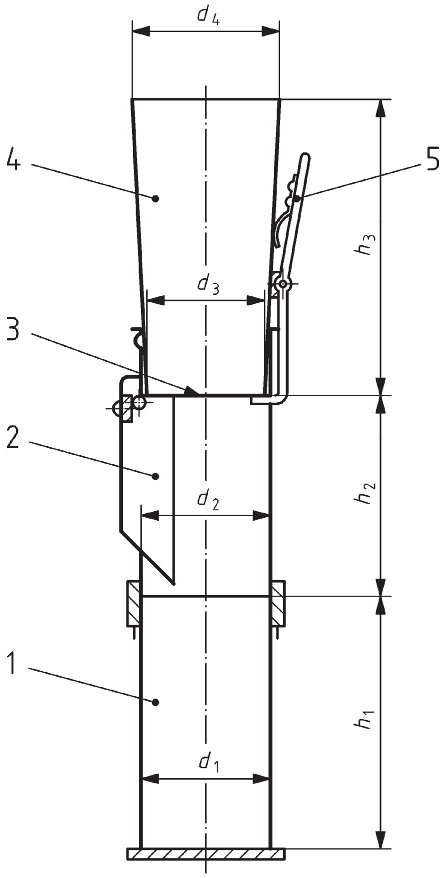
6.3 Bulk density

6.3.1 Apparatus

The apparatus by which the bulk density is to be determined (see Table 2 and Figure 4) shall consist of a cylindrical one litre vessel (1), a connecting piece (2) with a closure flap (3), and a hopper (4) with the sprung closure lever (5) used for holding and releasing the closure flap. By operating the lever, the closure flap opens and allows the sieved material contained in the hopper to fall into the one litre vessel.

Table 2 — Dimensions of density apparatus

Part No	Part name	Dimension
1	1 l vessel	
	Internal diameter Internal height	d_1: (87 ± 1) mm h_1: corresponding to filled contents of 1 000 cm^3, limit of error = ± 5 cm^3
2	Connecting piece	
	Internal diameter Height	d_2: (87 ± 1) mm h_2: (135 ± 1) mm
4	Hopper	
	Internal diameter, bottom Internal diameter, top Height	d_3: (79 ± 1) mm d_4: (99 ± 1) mm h_3: (199 ± 1) mm



Key

1 cylindrical 1 l vessel, see Table 2 for the dimensions of h_1 and d_1
2 connecting piece, see Table 2 for the dimensions of h_2 and d_2
3 closure flap
4 hopper, see Table 2 for the dimensions of h_3 and d_3
5 closure lever

Figure 4 — Typical apparatus for the determination of bulk density

6.3.2 Procedure

Pour a sufficient mass of the building lime sample into the hopper of the density apparatus with a shovel so that the powder above the rim assumes its natural angle of repose. Now open the closure flap by operating the closure lever on the hopper. After a waiting time of 2 min, remove the emptied hopper, skim off the amount of lime powder which has run into the vessel and projects above the top edge with a ruler and determine the mass of the contents of the vessel.

Carry out the test three times, in each case with a new sample of lime powder, and if the resulting values deviate from one another by more than 10 g, repeat it twice. Take the average of the three values which deviate from one another least as the bulk density of the lime powder. Report the bulk density in kilograms per cubic decimetre.

6.4 Soundness

6.4.1 General

Owing to the variation in chemical and physical characteristics of building limes it is not possible to determine the soundness of building limes by one method for all types of lime. For this reason soundness has to be determined as follows:

— hydrated calcium lime and all types of lime with hydraulic properties	6.4.2.1 (Reference method) 6.4.2.2 (Alternative method)
— hydraulic lime with an SO_3 content of more than 3 % up to 7 %	6.4.2.3
— hydrated calcium lime, lime putty and hydrated dolomitic lime which include grains larger than 0,2 mm	6.4.3
— quicklime, lime putty, dolomitic lime and hydrated dolomitic lime	6.4.4

6.4.2 For hydrated calcium lime and all types of lime with hydraulic properties

6.4.2.1 Reference method

6.4.2.1.1 Principle

If the slaking of lime is incomplete, the steam hydration of the calcium oxide (CaO) present in a sample pressed into a disc-shaped specimen leads to expansion that is measured by the variation of its disc diameter.

6.4.2.1.2 Apparatus

6.4.2.1.2.1 Moulds, as shown in Figure 5.

The piston shall have a milled cross groove on its end that forms the surface of the specimen.

6.4.2.1.2.2 Press, capable of applying a 2 kN force to the piston of the mould.

The force should be accurate to the nearest 0,5 kN. Alternatively, the pressure from the piston (1) on the specimen in the mould (Figure 5) may be controlled using a pressure gauge.

6.4.2.1.2.3 Length measuring device, capable of measuring to the nearest 0,1 mm.

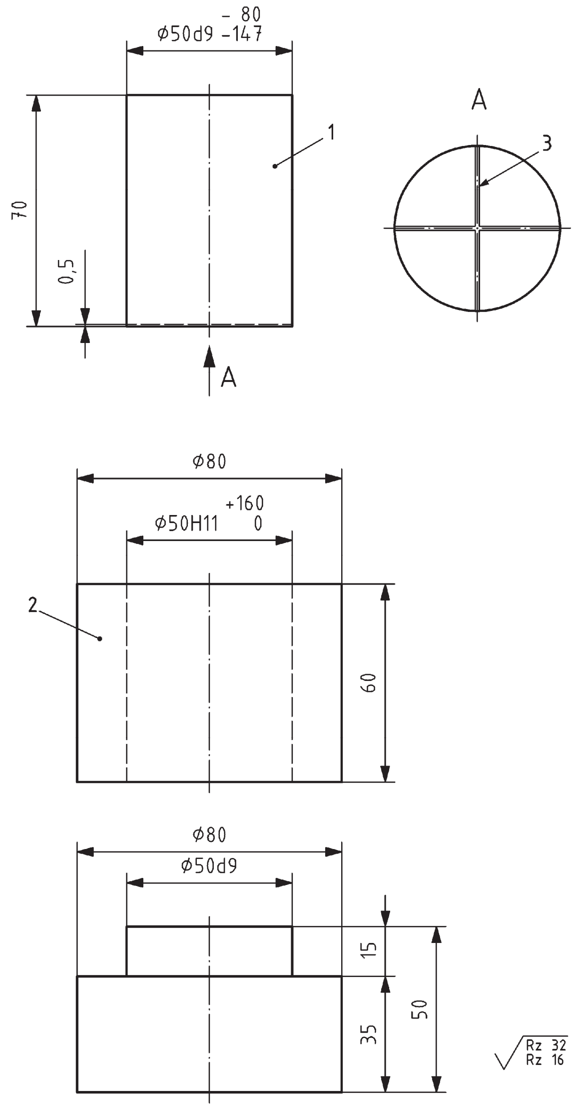
6.4.2.1.2.4 Steam cabinet, for the steam treatment of the specimens.

The simplest assembly comprises a cylindrical tank inside which a perforated disc (e.g. stainless wire gauze, sieve) enables the specimens to be kept at approximately 50 mm above the boiling water. The lid shall be designed so that condensed water does not drip onto the specimens.

6.4.2.1.2.5 Heating source, which enables the water to be kept at boiling point, such as an electric hot plate fitted with a regulator or an adjustable gas burner.

6.4.2.1.2.6 Balance, for weighing to approximately 0,001 g.

Dimensions in millimetres



Tolerances in accordance with ISO 2768-m.

Key

1 piston
2 mould
3 0,5-cross groove

Figure 5 — Typical specimen mould

6.4.2.1.3 Procedure

Weigh (25 ± 0,1) g of the building lime to be tested, then add sufficient water such that the specimen can be handled after demoulding (usually 5 g but less than 2,5 g for lime with hydraulic properties) and mix carefully. If the balance allows it, it is best to work directly in the specimen mould but, if this is not possible, work in an intermediate container and transfer the mixture to the specimen mould in as complete a state as possible. Then place the mould with the piston inserted under the press.

Press up to 2 kN force and hold this pressure for approximately 5 s and then demould. The specimen obtained is generally within the region of 10 mm thick with a diameter of approximately 50 mm. Measure two diameters to each other to approximately 0,1 mm, e.g. on the cross-diameter. Calculate D_i as the average of these two measurements. Place the specimen in the steam cabinet and keep it for 90 min in the steam atmosphere produced by the boiling water. Remove the specimen, leave to cool below 40 °C, and calculate D_e as the average of the two measurements after steaming.

6.4.2.1.4 Expression of results

The value of expansion, expressed in millimetres to the nearest 0,1 mm, is given by the difference ($D_e - D_i$).

6.4.2.2 Alternative method

6.4.2.2.1 General

Carry out the test for soundness in accordance with EN 196-3 except that for lime with hydraulic properties of the class 5 the moulded specimens shall be precured for (48 ± 0,5) h at a minimum of 90 % relative humidity and (20 ± 1) °C.

The following modifications shall apply for CL 70, CL 80, CL 90, and lime with hydraulic properties of classes 2 and 3,5.

6.4.2.2.2 Apparatus

Use a steam cabinet in place of the water bath and humidity cabinet described in EN 196-3.

6.4.2.2.3 Procedure

Prepare three test specimens by carefully hand mixing 75 g of the dry hydrated lime sample with 20 ml of water. Immediately fill each mould completely but loosely with the prepared mix and lightly tamp the sample to avoid the inclusion of air. Repeat this procedure until the mix is level with the top of the mould.

Where this procedure is not appropriate to fill the mould according to EN 196-3, the amount of water may be altered and the quantity used reported.

Measure the distance (A) separating the ends of the indicator points to the nearest 1 mm. Transfer the moulds immediately to the steam cabinet which should be boiling vigorously.

Subject the moulds to the continuous action of steam at atmospheric pressure for a period of (180 ± 10) min. At the end of this time remove the moulds, allow to cool to room temperature and measure the distance (B) separating the ends of the indicator points.

6.4.2.2.4 Expression of results

For each specimen record the measurements A and B and calculate the differences ($B - A$) in millimetres to the nearest 1 mm.

If individual results differ by more than 2 mm repeat the test.

6.4.2.3 For hydraulic lime with an SO_3 content of more than 3 % and up to 7 % (cold water test)

6.4.2.3.1 Preparation of specimens

Work 200 g of hydraulic lime thoroughly by kneading to a stiff paste with about 45 g to 90 g of water for 3 min. The amount of water added is correct if the paste spreads slowly only when the glass plate is shaken several times.

Make two cakes from the paste by placing the two halves of the paste as lumps onto the middle of two lightly oiled (releasing agent), flat glass plates (mirror glass) and shaking the plates gently until cakes 50 mm to

70 mm in diameter and about 10 mm thick are formed. After spreading, the cakes shall not be worked with a knife or trowel.

The two cakes are intended for the duplicate determination.

6.4.2.3.2 Procedure

Immediately after preparation, place the two cakes in a moist air storage cabinet with a relative humidity of not less than 90 % and leave them undisturbed therein to harden. About 24 h after their preparation, detach the cakes carefully from the glass plate and place them in water at 18 °C to 21 °C. Observe them for a further 27 days. If warping or gaping edge cracks appear, by themselves or together with netted cracks, this indicates "blowing", i.e. the cake splits with gradual loosening of the cohesion originally obtained, which can lead to complete disintegration (see Figure 8).

The curvature of the bottom surface of the cake may have a camber of not more than 2 mm.

The blowing phenomenon is frequently found on the cakes after only three days; nevertheless observation for up to 28 days suffices to recognize blowing reliably.

The cake may be taken out of the water for observation for not more than 30 min, since otherwise radial shrinkage cracks can readily form on the edges (Figure 7).

6.4.3 For hydrated calcium lime, lime putty and hydrated dolomitic lime which include grains larger than 0,2 mm

6.4.3.1 Principle

Large particles of overburnt quicklime can cause popping and pitting when used for plastering. In this test hydrated calcium lime is added to a non-retarded plaster of Paris and, after the plaster has set, it is subjected to the action of steam. The steam hydrates any unreacted calcium and/or magnesium oxide present causing expansion and thus eruption of the plaster surface.

6.4.3.2 Apparatus

6.4.3.2.1 Large sheet of non-absorptive material for mixing (a glass plate 500 mm × 750 mm has been found to be suitable).

6.4.3.2.2 Three brass ring moulds, 100 mm internal diameter, 5 mm deep of an annular thickness of at least 5 mm, and having an internal taper of about 5°, each with a non-porous base plate about 120 mm × 120 mm.

6.4.3.2.3 Two broad palette knives.

6.4.3.2.4 Drying oven, controlled at (40 ± 5) °C.

6.4.3.2.5 Steam cabinet, in which soundness specimens can be submitted to the action of steam at atmospheric pressure.

The lid shall be designed so that condensed water does not drip onto the specimens.

6.4.3.3 Material

Plaster of Paris, containing more than 90 % by mass of calcium sulfate hemihydrate ($CaSO_4.1/2\ H_2O$) and passing the 0,2 mm sieve.

In addition when gauged with 50 % of its own mass of water at a temperature of (20 ± 2) °C, it shall show an initial setting time, when tested according to 6.5, of not less than 4 min and not greater than 15 min.

6.4.3.4 Procedure

Weigh approximately 250 g of the sample to an accuracy of 1 g and mix in sufficient water using the two palette knives to produce a lime putty, at a temperature of (20 ± 2) °C, on the large non-absorptive sheet. Gather the mix into a compact mass, cover it to avoid loss of water and allow to stand for (120 ± 10) min.

Grease the three ring moulds and base plates with petroleum jelly.

When 2 h have elapsed, mix the lime putty using the palette knives until a stiff plastic mass is obtained. If it is essential, a little more water may be added. Spread out the putty, then sprinkle 35 g of the plaster of Paris evenly over the surface of the putty and mix rapidly with the palette knives for (120 ± 5) s.

Using a palette knife, form three flat pats by pressing the gauged material, in small quantities, into a ring mould resting with its wider diameter downwards on a non-porous base plate. Ensure that no air is entrapped. Using the palette knife with its blade nearly vertical, cut off the excess putty and then, with the blade nearly horizontal, smooth off level with the top of the mould using not more than twelve strokes. Do not exceed 5 min total time, from the addition of the plaster of Paris, for the completion of the pats.

Leave the three pats to set for 1 h. Place the pats on their base plates with or without the ring moulds, in the drying oven at a temperature of (40 ± 5) °C for not less than 12 h. (It is usually convenient to dry the pats overnight.) Place the pats, still on their base plate, horizontally in the steam cabinet, in which the water shall be boiling vigorously as indicated by a constant egress of steam. Subject the three pats to continuous action of saturated steam at atmospheric pressure for (180 ± 10) min, taking precautions to prevent condensed water dripping on the pats.

If any pat shows shrinkage cracks after drying in the oven at a temperature of (40 ± 5) °C, then discard it and make a new pat. Continue to test the other pat(s) while preparing the new pat(s).

Remove the pats from the steam cabinet, allow them to cool and examine them in good light for disintegration, popping or pitting.

NOTE Drips of water can cause "water blistering" which should not be confused with popping or pitting.

6.4.3.5 Test report

Report as either free from, or as showing, disintegration, popping or pitting.

6.4.4 For quicklime, lime putty, dolomitic lime and dolomitic hydrated lime

6.4.4.1 Principle

Cakes are made by pouring pastes of slaked lime or lime putty upon absorbent porous plates. After 5 min the cakes, placed upon new dry plates, are put into a hot cabinet and after this treatment the cakes are inspected visually for expansion cracks.

6.4.4.2 Apparatus

6.4.4.2.1 **Absorbent plates,** approximately 100 mm × 100 mm, of hydrated calcium silicate, as used for thermal insulation, density approximately 1,0 g/cm^3, thickness approximately 25 mm, suction time up to 10 min.

NOTE Other absorbent materials leading to comparable water content may be used as alternatives.

6.4.4.2.2 **Hot cabinet**, controlled at (105 ± 5) °C.

6.4.4.3 Preparation of specimens

6.4.4.3.1 Sieve residue

If individual particles are found on the 0,2 mm wire sieve medium (see 6.2) when testing particle fineness, the procedure shall be as follows:

a) Embed all the particles found in the middle of a cake, so that they are completely covered.

b) Soak particles in quicklime first in accordance with the working instructions of the manufacturer and then introduce them into the middle of the cake.

c) Prepare the cake from the same lime specimen in which the residues have been found.

6.4.4.3.2 Quicklime and dolomitic lime

Slake about 200 g of quicklime or dolomitic lime with water at (20 ± 2) °C to give a paste and soak for the length of time required by the manufacturer in the working instructions. Then make two cakes of about 50 mm to about 70 mm in diameter and about 10 mm in thickness at the centre from this paste.

6.4.4.3.3 Lime putty

In the case of lime putty, make two cakes directly from the existing putty.

6.4.4.3.4 Dolomitic hydrated lime

For testing immediately workable dolomitic hydrated lime, mix about 100 g of lime powder to a paste with water at (20 ± 2) °C so that two cakes of about 50 mm to about 70 mm in diameter and about 10 mm in thickness at the centre can be poured, each onto a dry filter plate (see 6.4.4.2).

6.4.4.4 Procedure

After a sufficient resting time (approximately 5 min), remove the cakes and transfer them to two other dry absorbent plates and put them into the hot cabinet. Keep them in this cabinet at (105 ± 5) °C for 4 h.

6.4.4.5 Evaluation

The building lime shall be regarded as passing the test if the cakes are firm after the treatment described in 6.4.4.4 and show no expansion cracks. Shrinkage cracks are of no significance for the evaluation. In this context, see Figures 6 to 8.

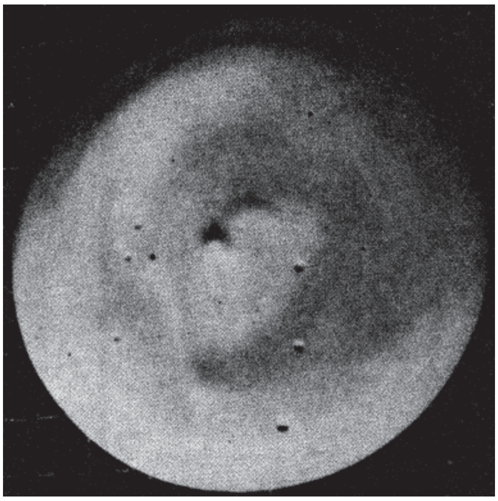


Figure 6 — "Test passed"

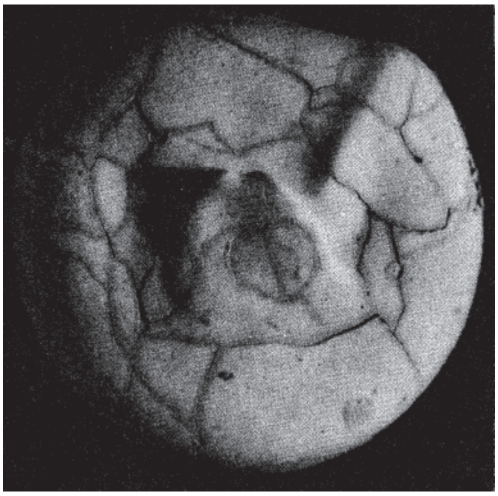


Figure 7 — Specimen with shrinkage cracks ("Test passed")

Figure 8 — Specimen with expansion cracks ("Test failed")

6.5 Setting times

6.5.1 Principle

The setting time is determined by observing the penetration of a needle into building lime paste of standard consistence until it reaches a specified value.

Lime paste of standard consistence has a specified resistance to penetration by a standard plunger. The water required for such a paste is determined by trial penetrations of pastes with different water contents.

6.5.2 Laboratory, apparatus and materials

6.5.2.1 Laboratory

The laboratory in which specimens are prepared and tested shall be maintained at a temperature of (20 ± 2) °C and a relative humidity of not less than 50 %.

The temperature and relative humidity of the air in the laboratory and the temperature of the water in the storage containers shall be recorded at least once per day during working hours.

Building lime, water and apparatus used to make and test specimens shall be at a temperature of (20 ± 2) °C.

Where temperature ranges are given, the target temperature at which the controls are set shall be the middle value of the range.

6.5.2.2 Apparatus and materials

6.5.2.2.1 **Balance,** capable of weighing to the nearest ± 1 g.

6.5.2.2.2 **Graduated cylinder or burette,** capable of dispensing to an accuracy of ± 1 ml.

6.5.2.2.3 **Mixer,** conforming to EN 196-1.

6.5.2.2.4 **Water,** distilled or deionized water shall be used for making, storing and boiling specimens.

6.5.2.2.5 Building lime, water and apparatus used to make and test specimens shall be at a temperature of (20 ± 2) °C.

NOTE Other water may be used provided that it can be shown to give the same test results.

6.5.3 Standard consistence test

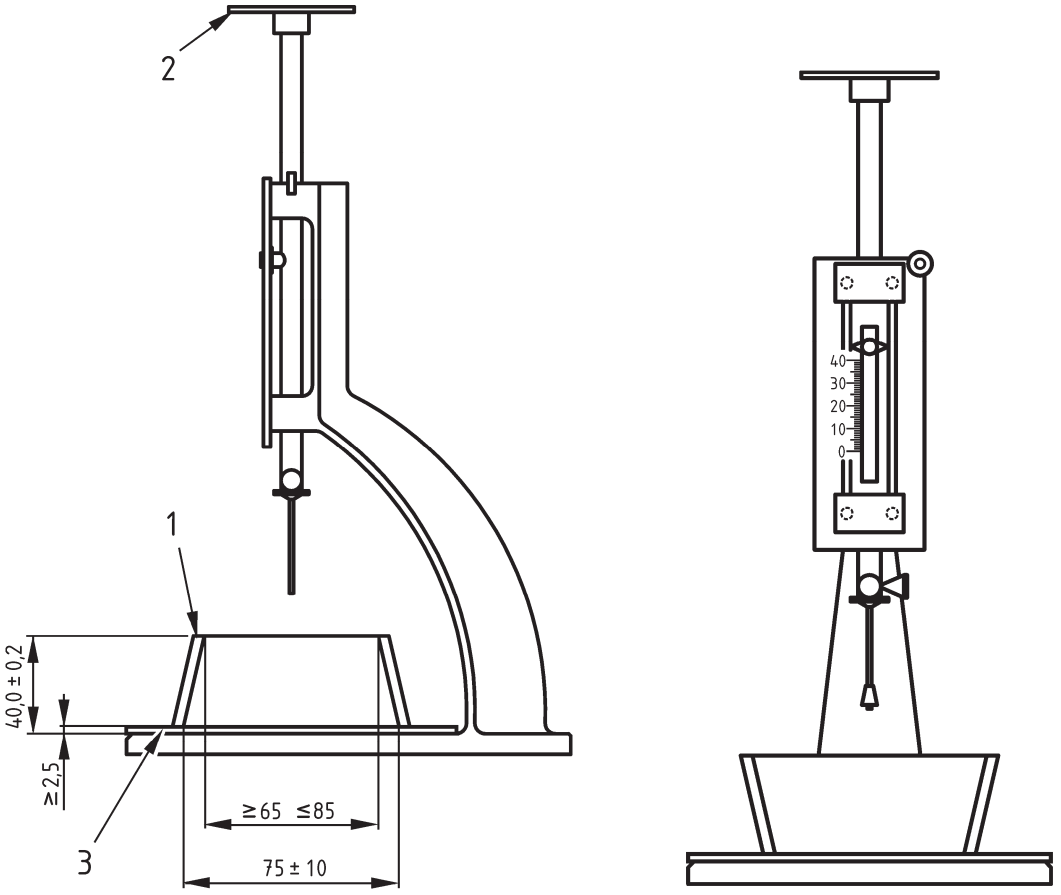
6.5.3.1 Apparatus

Use the Vicat apparatus as shown in Figures 9a) and 9b) with the plunger shown in Figure 9c). The plunger (Figure 9c)) shall be of non-corrodible metal in the form of a right cylinder of at least 45 mm effective length and of (10,00 ± 0,05) mm diameter. The total mass of moving parts shall be (300 ± 1) g. Their movement shall be truly vertical and without appreciable friction, and their axis shall coincide with that of the plunger.

The Vicat mould (see Figure 9a)) to contain the building lime paste under test shall be of hard rubber, plastics or brass. It shall be of cylindrical or preferably truncated conical form (40,0 ± 0,2) mm deep and shall have an internal diameter of (75 ± 10) mm. It shall be adequately rigid and shall be provided with a base-plate larger than the mould and at least 2,5 mm thick, constructed of impermeable material resistant to attack by the building lime paste, e.g. plane glass.

NOTE Moulds of other metal may be used provided that they are of the specified depth and that their use has been calibrated against the specified mould.

Dimensions in millimetres



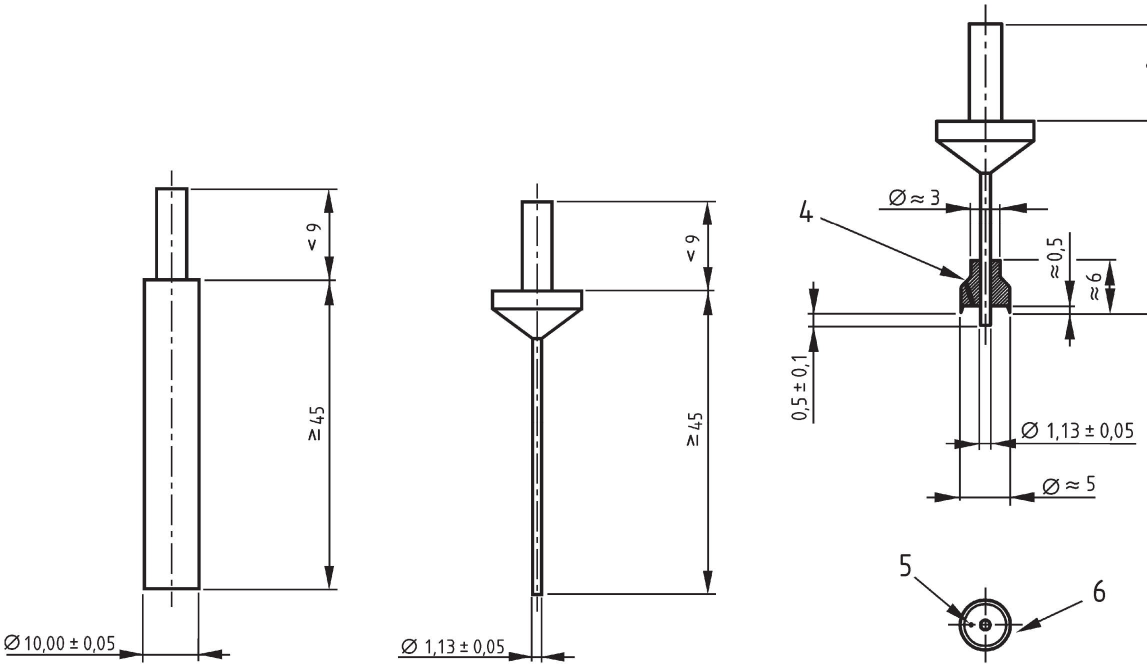
a) Side view with mould in upright position for initial setting time determination

b) Front view with mould inverted for final setting time determination

Key

1 mould
2 correcting weights
3 base plate
4 air vent (Ø ≈ 1,5 mm)
5 air vent
6 view from below needle with attachment for determining final setting time

Figure 9 — Typical manual Vicat apparatus for determination of standard consistence and setting time of building lime



c) Plunger for standard consistency **d) Needle for initial set** **e) Needle for and attachment for final set**

Key

1 mould
2 correcting weights
3 base plate
4 air vent (Ø ≈ 1,5 mm)
5 air vent
6 view from below needle with attachment for determining final setting time

Figure 9 *(continued)* **— Typical manual Vicat apparatus for determination of standard consistence and setting time of building lime**

NOTE Only one correcting weight is required for each Vicat apparatus if the mass of the plunger and the needles with and without the attachment is always the same e.g. (9,0 ± 0,5) g.

6.5.3.2 Procedure

6.5.3.2.1 Mixing the building lime paste

Weigh, to the nearest 1 g, 500 g of building lime. Weigh a quantity of water, e.g. 125 g, in the mixer bowl or measure the water from the graduated cylinder or burette and place it into the mixer bowl.

Add the building lime carefully to the water in order to avoid loss of water or building lime. The time of addition shall be not less than 5 s nor more than 10 s. Note the time of completion of the addition as zero time from which later measurements of time shall be made. Start the mixer immediately and run at low speed for 90 s.

Stop the machine after 90 s for 30 s during which remove with a suitable scraper any paste adhering to the bowl outside the mixing zone and return it to the mix. Restart the machine and run at low speed for a further 90 s. The total mixer running time shall be 3 min.

NOTE Any other mixing method, whether by machine or hand, may be used provided that it can be shown to give the same test results as the specified method.

6.5.3.2.2 Filling the mould

Transfer the paste immediately to the mould, which has previously been placed on a lightly greased plane glass base-plate, and fill it to excess without undue compaction or vibration. Remove the excess by a gentle sawing motion with a straight-edged implement in such a way as to leave the paste filling the mould and having a smooth upper surface.

6.5.3.2.3 Penetration test

Calibrate the Vicat apparatus with the plunger (Figure 9c)), attached in advance of the test, by lowering the plunger to rest on the base-plate to be used and adjusting the pointer to read zero on the scale. Raise the plunger to the stand-by position. Immediately after levelling the paste, transfer the mould and base-plate to the Vicat apparatus and position it centrally under the plunger. Lower the plunger gently until it is in contact with the paste. Pause in that position for between 1 s and 2 s in order to avoid initial velocity or forced acceleration of the moving parts. Then release the moving parts quickly and allow the plunger to penetrate vertically into the centre of the paste. The release of the plunger shall occur 4 min ± 10 s after zero time. Read the scale when penetration has ceased or 30 s after the release of the plunger, whichever is the earlier.

Record the scale reading, which indicates the distance between the bottom face of the plunger and the base-plate, together with the water content of the paste expressed as a percentage by mass of the building lime. Clean the plunger immediately after each penetration.

Repeat the test with pastes containing different water contents until one is found to produce a distance between plunger and base-plate of (6 ± 2) mm. Record the water content of that paste to the nearest 0,5 % as the water for standard consistence.

6.5.4 Setting time test

6.5.4.1 Apparatus

6.5.4.1.1 Room or humidity cabinet, of adequate size and maintained at (20 ± 1) °C and a relative humidity of not less than 90 %.

6.5.4.1.2 Vicat apparatus for initial set.

Remove the plunger and replace it by the needle (Figure 9d)) which shall be of steel and in the form of a right cylinder of effective length of at least 45 mm and diameter (1,13 ± 0,05) mm. The total mass of moving parts shall be (300 ± 1) g. Their movement shall be truly vertical and without appreciable friction, and their axis shall coincide with that of the needle.

NOTE Automatic setting time machines are commercially available and may be used provided that they can be shown to give the same test results as the specified apparatus and procedure.

6.5.4.2 Determination of initial setting time

Calibrate the Vicat apparatus with the needle (Figure 9d)), attached in advance of the test, by lowering the needle to rest on the base-plate to be used and adjusting the pointer to read zero on the scale. Raise the needle to the stand-by position.

Fill a Vicat mould with paste of standard consistence and level it, in accordance with 6.5.3.2.1 and 6.5.3.2.2.

Place the filled mould and base-plate in the room or humidity cabinet specified in 6.5.4.1.1 and, after a suitable time, transfer to the Vicat apparatus and position under the needle. Lower the needle gently until it is in contact with the paste. Pause in that position for between 1 s and 2 s in order to avoid initial velocity or forced acceleration of the moving parts. Then release the moving parts quickly and allow the needle to penetrate vertically into the paste. Read the scale when penetration has ceased, or 30 s after the release of the needle, whichever is the earlier.

Record the scale reading, which indicates the distance between the end of the needle and the base-plate, together with the time from zero. Repeat the penetration test on the same specimen at conveniently spaced positions, not less than 10 mm from the rim of the mould or from each other, at conveniently spaced intervals of time, e.g. at 10 min intervals. Between penetration tests keep the specimen in a room or humidity cabinet as specified in 6.5.4.1.1. Clean the Vicat needle immediately after each penetration. Record the time measured from zero at which the distance between the needle and the base-plate is (6 ± 3) mm as the initial setting time of the building lime to the nearest 5 min. The required accuracy may be assured by reducing the time interval between penetration tests near the end-point and observing that successive results do not fluctuate excessively.

6.5.4.3 Determination of final setting time

Invert the filled mould used in 6.5.4.2 on its base-plate so that the tests for final set are made on the face of the specimen originally in contact with the base-plate. Fit the needle with a ring attachment (Figure 9e)) to facilitate accurate observation of small penetrations. Use the procedure described in 6.5.4.2. The intervals of time between penetration tests may be increased to e.g. 30 min.

Between penetration tests keep the specimen in a room or humidity cabinet as specified in 6.5.4.1.1. Clean the Vicat needle immediately after each penetration.

Record, to the nearest 15 min, the time measured from zero at which the needle first penetrates only 0,5 mm into the specimen as the final setting time of the building lime. This time is that at which the ring attachment first fails to mark the specimen and may be accurately established by reducing the time interval between tests near the end-point and observing that successive test results do not fluctuate excessively.

6.6 Reactivity

6.6.1 General

The quicklime sample prepared according to 3.5 shall be tested for reactivity on slaking by measuring the increase in temperature which occurs on reaction with water as a function of the reaction time (wet slaking curve). If 100 % of the material pass the 5 mm sieve the product can alternatively be tested in the as-delivered state.

6.6.2 Apparatus

The apparatus shall consist of the six parts shown in Figure 10:

1 Dewar vessel, 1 000 ml, internal diameter about 77 mm, internal height about 235 mm.
2 Stirrer motor, (300 ± 10) min^{-1} under load.
3 Stand and support.
4 Blade stirrer made of suitable plastics, diameter about 60 mm and thickness about 4 mm (see Figure 11).

NOTE Other forms of stirrers may be used if they lead to equal results for reactivity.

5 Plastics lid with segment which can be opened, feed opening and bore for the thermometer, to fit the Dewar vessel (see Figure 12).
6 Calibrated thermometer 0 °C to 100 °C with an accuracy of 0,5 °C and a high rate of response (adjustment time from 20 °C to 60 °C less than 10 s). The penetration depth shall be about 160 mm from a suitable hole on the top edge of the lid.

The following shall be used additionally:

- a recorder for the temperature measurement;
- weighing and feeding device, made of stainless steel (see Figure 13).

6.6.3 Determination of the water equivalent of the apparatus

Determine the water equivalent w by measuring the mixing temperature T_m of an amount of approximately 650 g in the Dewar vessel. Use the apparatus under the same conditions as for a reactivity test.

Fill (450 ± 1) g of water (m_c) of a temperature of (20 ± 0,1) °C (T_c) into the Dewar vessel. Then add (200 ± 1) g of water (m_h) of a temperature of (50 ± 0,1) °C (T_h). Run the stirrer at (300 ± 10) min^{-1}. Measure the temperature T_m to ± 0,1 °C after (5 ± 0,1) min.

$$w = \frac{m_h (T_h - T_m) - m_c (T_m - T_c)}{T_m - T_c} \times 4{,}19 \quad J / K \tag{17}$$

where

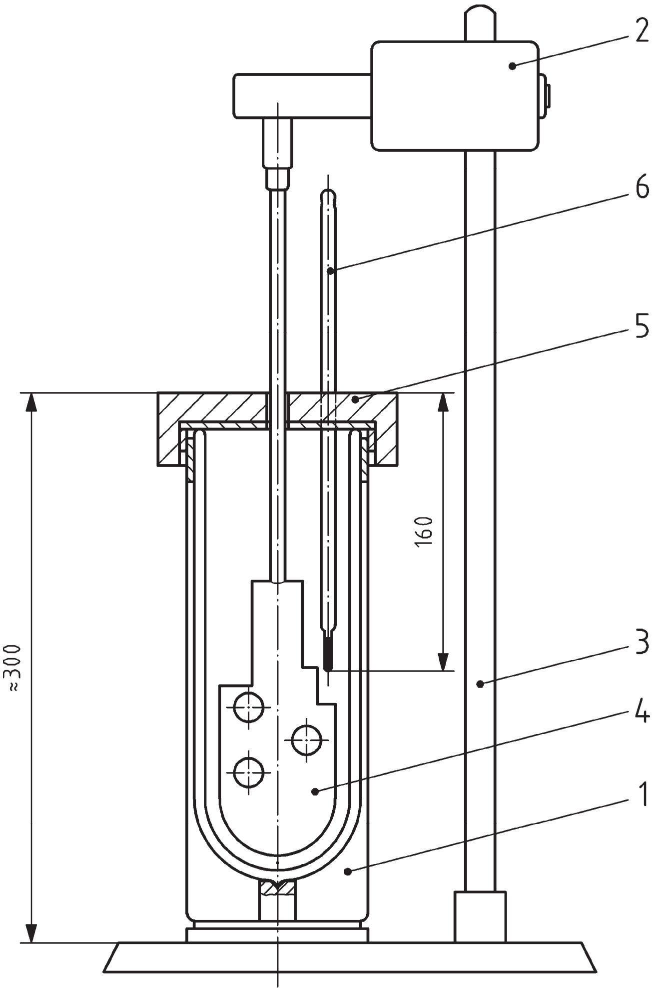
$T_{m,\,h,\,c}$ are temperatures, in degrees Celsius;

$m_{h,\,c}$ are masses, in grams;

w is the water equivalent value, in joules per kelvin.

The test apparatus shall have a water equivalent of 200 J/K to 300 J/K.

Dimensions in millimetres



Key

1 dewar vessel
2 stirrer motor
3 stand and support
4 blade stirrer
5 plastics lid with segment
6 calibrated thermometer

Figure 10 — Typical apparatus for testing reactivity

Dimensions in millimetres

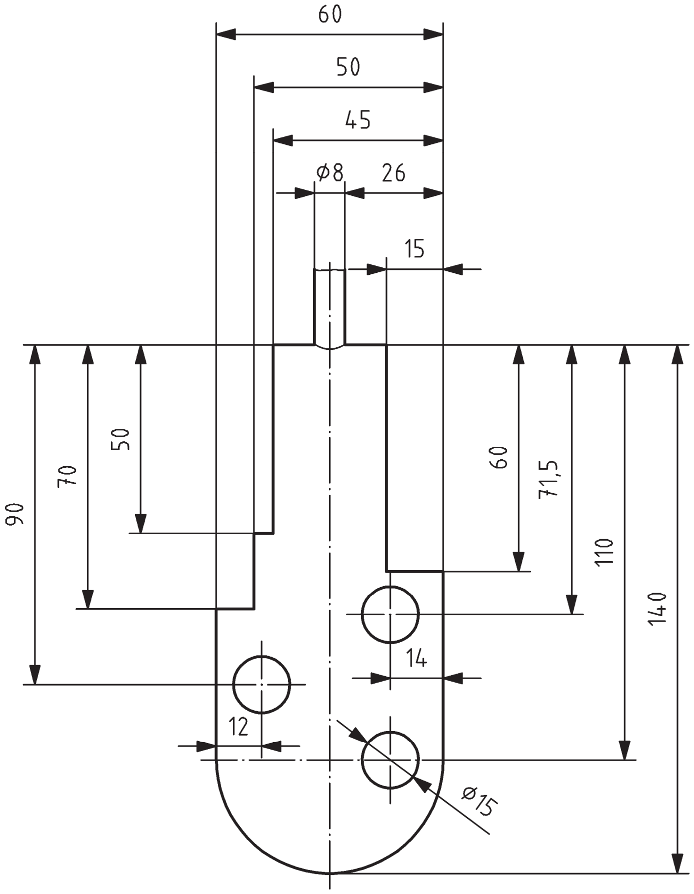
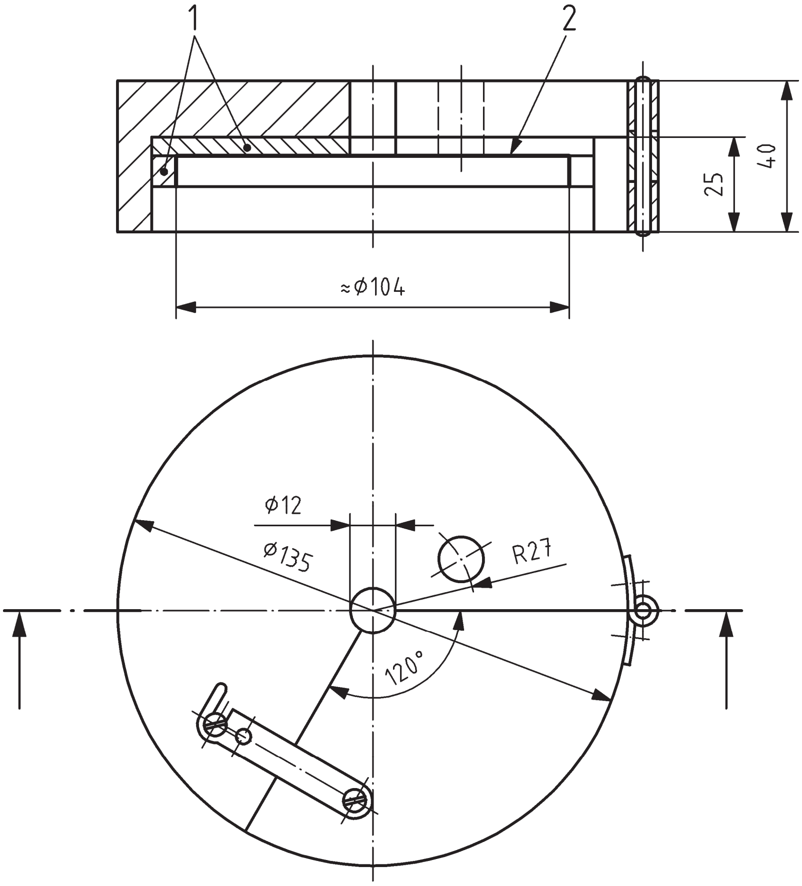


Figure 11 — Typical blade stirrer

Dimensions in millimetres



Key

1 insulating layer
2 polyethylene film

Figure 12 — Typical lid with a segment which can be opened and a hole for the thermometer

Dimensions in millimetres

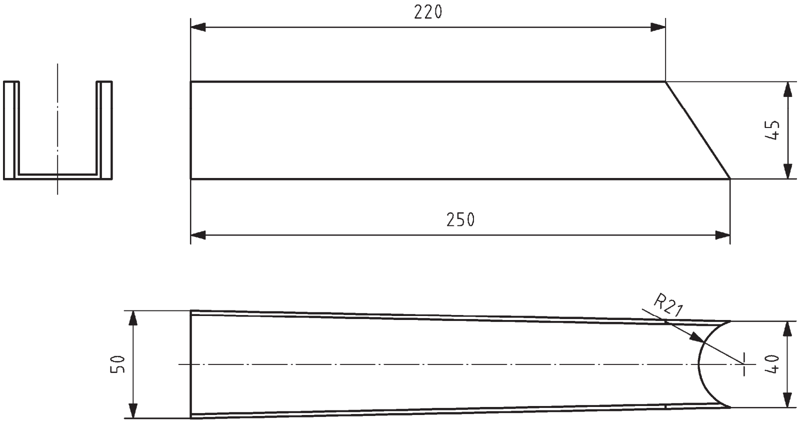


Figure 13 — Typical weighing and feeding vessel

6.6.4 Sample preparation

Test a sample of about 0,5 kg.

To ensure that the sample corresponds to the product to be tested, store it in a tightly closing container. Absorption of even small amounts of moisture influences the pattern of the wet slaking curve. Weigh out a quantity of (150 ± 0,5) g for the individual test.

6.6.5 Procedure

Fill the Dewar vessel with (600 ± 1) g of distilled water at about 20 °C (T_0), insert the thermometer and blade stirrer and monitor the temperature as the blade stirrer runs at (300 ± 10) min^{-1}; the temperature shall deviate from 20 °C by not more than ± 0,5 °C. The lime sample shall also have a temperature of about 20 °C.

With the stirrer running, introduce the weighed quantity of sample all at once into the water by means of the feeding vessel (Figure 13). This moment shall be considered to be the start of the test. Measure the slaking temperatures after half a minute, after 1 min and then at intervals of 1 min up to a time of 10 min, and thereafter only at intervals of 2 min.

With very reactive limes, measure the temperature at shorter intervals, since the reaction may be completed after a few minutes.

Ensure that the contents of the vessel are thoroughly mixed completely throughout the entire duration of the test. For limes which thicken severely, it may be necessary to increase the speed of the motor after a reaction temperature of about 60 °C has been reached.

6.6.6 Evaluation

6.6.6.1 Reference procedure

Plot the measured temperature values in degrees Celsius as a function of time in minutes (wet slaking curve, Figure 14).

The result is expressed as the time t necessary to reach the required temperature T. For instance, if the required temperature is 60 °C, t_{60} in minutes will be the result.

6.6.6.2 Alternative procedure

Plot the measured temperature values (degrees Celsius) as a function of time (minutes) to generate the wet slaking curve as shown in Figure 14. The slaking reaction of the lime is assumed to be 100 % complete at the time when the maximum temperature (T'_{max}) has been reached. By way of example, this point is shown in Figure 14. The reactivity of the lime shall be reported as the time (t_{u}) required for the reaction to be 80 % complete. The temperature T_{u} is defined as the temperature in degrees Celsius at which the reaction is 80 % complete and is calculated using the following equation:

$$T_{\text{u}} = (0{,}8 \times T'_{\text{max}}) + (0{,}2 \times T_0) \text{, in degrees Celsius} \qquad (18)$$

Having calculated T_{u} , the value of t_{u} shall be determined from the wet slaking curve as shown in Figure 14.

The fineness of the lime sample and the maximum temperature T_{max} shall also be reported. T_{max} is calculated in degrees Celsius from the observed maximum temperature (T'_{max}) corrected for the water equivalent using the following equation:

$$T_{\text{max}} = (1{,}1 \times T'_{\text{max}}) - 2 \text{, in degrees Celsius} \qquad (19)$$

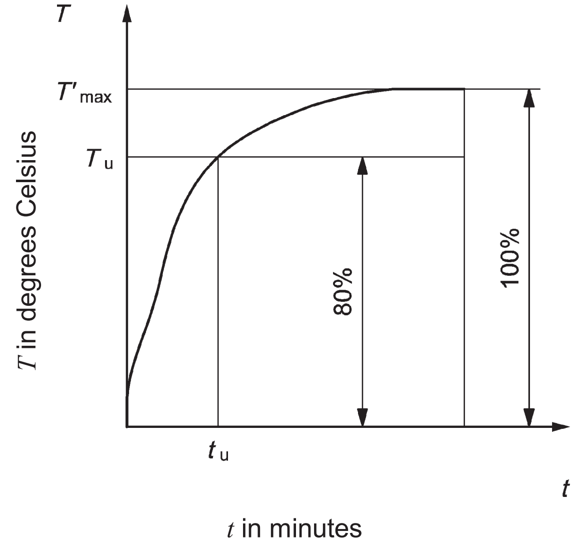


Figure 14 — Example of a wet slaking curve

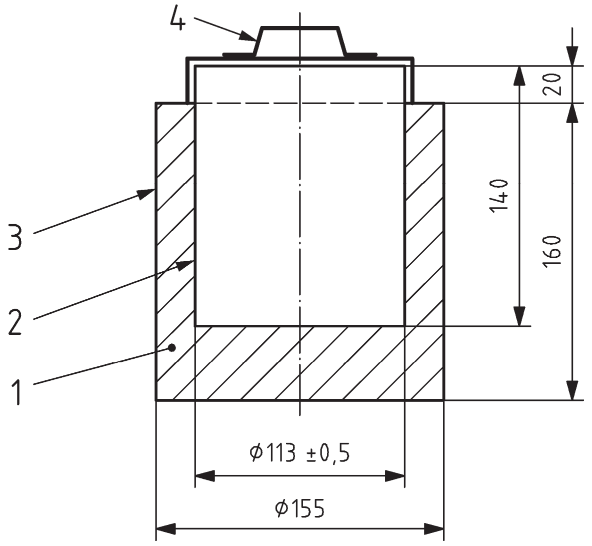
6.7 Yield

6.7.1 Slaking vessel

The slaking vessel in which the yield is determined (see Figure 15) shall be a double walled vessel made of stainless steel. The hollow space between the walls shall be filled with a thermal insulating material, e.g. mineral wool. The dimensions of the actual internal slaking cylinder shall be such that each 2 mm of depth is equivalent to a yield of 1 dm^3 per 10 kg of quicklime.

The vessel shall be closed with a lid.

Dimensions in millimetres



Key

1 thermal insulating material
2 inner cylinder
3 outer cylinder
4 lid

Figure 15 — Typical slaking vessel with lid

6.7.2 Procedure

First add (320 ± 1) ml of water at (20 ± 2) °C to the slaking vessel (Figure 15) and then pour in (200 ± 1) g of quicklime. Crush lump lime (e.g. to a particle size of 5 mm).

Stir the mixture gently until slaking starts, and then add the additional water required, according to the slaking properties of the lime (take special care for high reactive quicklime). Stir the putty further for a short time and close the slaking vessel with the lid. After being left to stand for about 24 h, remove the lid from the slaking vessel.

The water added during slaking has been chosen correctly if, 24 h after slaking, a small amount of water (up to a maximum of 40 ml) has settled on the surface of the lime putty.

Determine the yield when the lime putty has separated from the walls of the vessel. For this, measure the height of the lime putty. Use the mean value for four measurements for the evaluation. Each 2 mm in height of the putty corresponds to a yield of 1 dm^3 per 10 kg of quicklime.

6.8 Standard mortar by mass and water demand for values of flow and penetration

6.8.1 General

The measurement of the value of penetration, water retention and air content requires a standard mortar in accordance with EN 196-1, adjusted to a flow diameter as described in Table 3 and as measured on the flow table described in 6.8.2.1.2.

6.8.2 Composition and preparation of standard mortar

6.8.2.1 Laboratory and equipment

6.8.2.1.1 General

The laboratory, mixer, standard sand and water in accordance with EN 196-1.

6.8.2.1.2 Flow table

The supporting frame (1) for the flow table (see Figure 16) shall be made of welded or cast steel and shall be stable and free from any distortions. Figure 16 serves as basis for the dimensions of the profiles to be used. The width of each supporting arm of the frame (1) shall be at least 140 mm on the base and at least 20 mm longer than the diameter of the shaft (4) at the top (near the boss).

The steel cam (3) shall be located on the motor-driven axle (2) of the flow table. The steel axle shall rotate at a constant velocity (1 $rev \cdot s^{-1}$) and is operated via a flexible clutch.

The cam shall lift the shaft (4) and the plate (5) together by (10,0 ± 0,2) mm once per second. The jolting apparatus shall be switched off after 15 jolts.

The shaft (4) with a diameter of (22 ± 8) mm shall be made of stainless steel with a finely ground surface (maximum coarseness 0,010 mm). A shaft foot (12) made of heat treatable steel with a length of (12,0 ± 0,1) mm and a diameter of (10,0 ± 0,1) mm shall be attached to its lower end by means of an M8 thread. A tightened central bearing (6) which supports a rigid (4,0 ± 0,1) mm thick polished stainless steel plate (5) (maximum coarseness 0,003 2 mm) with a diameter of (300 ± 1) mm shall be fastened to the shaft by an M16 thread or by a secured fit (20,00 mm).

The top of the cam (3) and the surface of the attached foot (12) of the shaft (4) shall be hardened to a Brinell hardness of HBS 150 in accordance with EN ISO 6506-1:2005.

The rigidity of the plate (5) is increased:

a) by fastening the plate to the bearing (6), diameter (100 ± 0,2) mm, by means of five M5 screws arranged 72° apart and (37,5 ± 0,1) mm from the centre. In addition, the lower surface of the stainless steel plate shall be subjected to a uniform load by a turned metal ring, resistant to corrosion by mortar, with an outer diameter of (300 ± 1) mm, an inner diameter of (260 ± 0,1) mm and a height of 5 mm. The ring (7) shall be fastened securely to the stainless steel plate by six M5 countersunk screws, arranged 60° apart; or

b) alternatively, by means of three supporting arms radiating from the centre of the plate and arranged at an angle of 120° to each other, which connect the central bearing to the edge of the plate. The ends of the supporting arms form supporting surfaces which are horizontal and on a level with the central bearing. Each supporting surface shall be fastened to the plate (5) by an M5 countersunk screw.

The contact surfaces between the plate (5), the bearing (6) and either the ring (7) or the supporting parts mentioned in alternative b) shall be polished to a maximum coarseness of 0,003 2 mm.

A circle with a diameter of 100 mm and a depth of 0,2 mm shall be engraved at the centre of the upper surface of the stainless steel plate (5).

The stainless steel plate shall be horizontal in both the upper and lower shaft positions. The total weight of the shaft, bearing, stainless steel plate and the means of stabilization shall be between 4,2 kg and 4,5 kg. (Where additional weights are required for the alternative method b) for stabilization, they shall be of equal weight and attached to the lower surface of each supporting arm, being arranged symmetrically in relation to the centre of the plate.)

The shaft guides shall have a clearance of between 0,05 mm and 0,1 mm. No stop to prevent rotation shall be provided. Instead, the area of the cam and the part of the shaft in contact with it shall be designed with a degree of precision which ensures that the table rotates not more than 60° during the 15 jolts.

The face of the cam (3) is a smooth spiralled curve of increasing radius which touches the shaft (4) after each downward movement after rotating 120°. Contact between the cam and the shaft shall be ensured until each following downward movement.

The shaft shall pass through two finely ground guide bushes (maximum coarseness 0,010 0 mm) not less than 50 mm apart, one being positioned directly above the other.

Alternatively, the shaft may be placed in a vertical tube with a finely ground inner surface (maximum coarseness 0,010 0 mm). In this case, the design of the shaft shall be such that it comes into contact continuously with not more than two areas of the inside of the tube, these being not less than 50 mm apart.

In both cases, the area of the shaft in contact with the adjacent surface shall be between 25 cm^2 and 35 cm^2.

The shaft shall be able to fall unhindered and shall be kept clean. It shall be lubricated with a thin-bodied mineral oil resistant to resinification. Contact between the plate of the table and the boss (8) on the frame (1) shall be provided by means of a horizontal ring with a width of at least 5 mm.

There shall be neither fluid (oil, water) nor dust between the lower surface of the central bearing and the boss (8). This may be aided by increasing the diameter of the guide bushings by 3 mm over a length of 5 mm, starting at the boss (8).

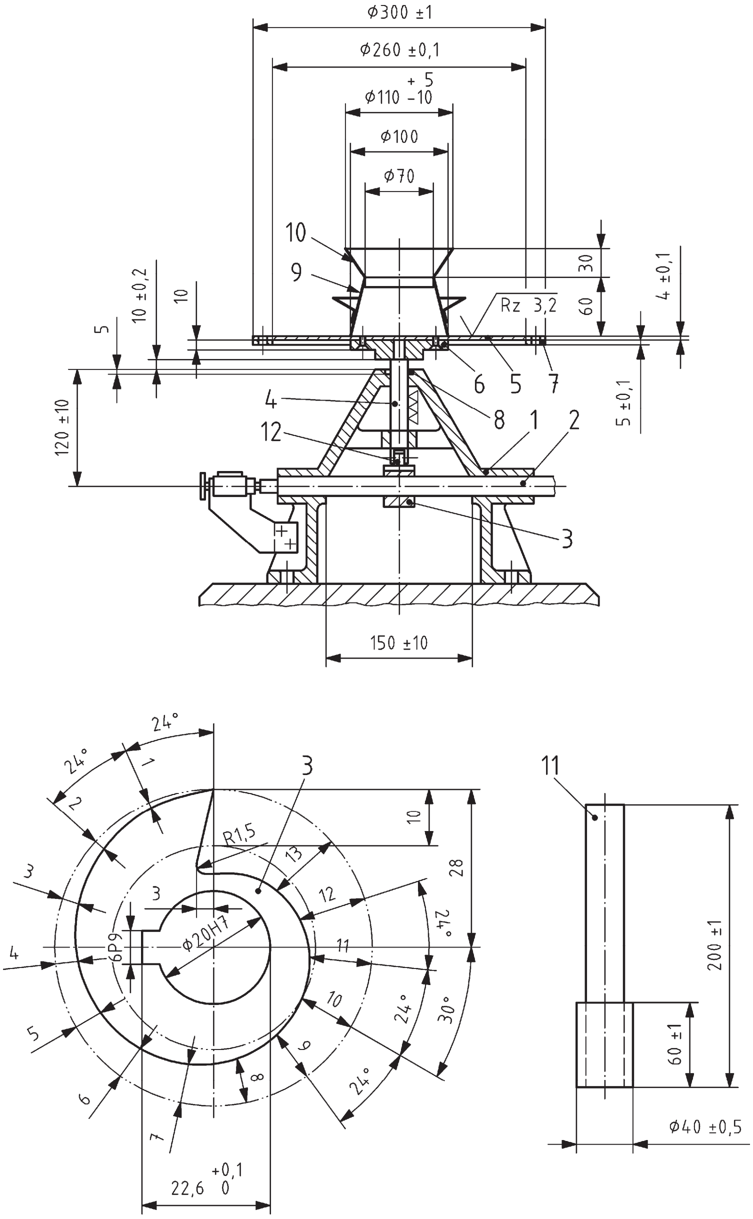
The mould (9) and hopper (10) shall be made of metal resistant to corrosion by mortar (stainless steel, brass). The lower and upper inner diameter and the height of the mould shall be adhered to within ± 0,5 mm. The minimum thickness of the mould shall be 2,0 mm in order to avoid denting.

The flow table shall be screwed securely to a horizontal, firm and non-plastic base without the use of levelling aids. For example, a concrete slab weighing not less than 50 kg is suitable.

All tolerances given above apply also to flow tables already in use.

The tamper (11) shall consist of a round rod made of an impermeable material with sheet metal protection and shall weigh (250 ± 15) g.

Dimensions in millimetres



Key

1	frame	7	ring
2	axle	8	boss
3	cam	9	mould
4	shaft	10	hopper
5	plate	11	tamper
6	bearing	12	shaft foot

Tolerances in accordance with ISO 2768-m.

Figure 16 — Typical flow table, cam and tamper

6.8.2.1.3 Apparatus for measuring value of penetration

The plunger apparatus shall correspond to that shown in Figure 17.

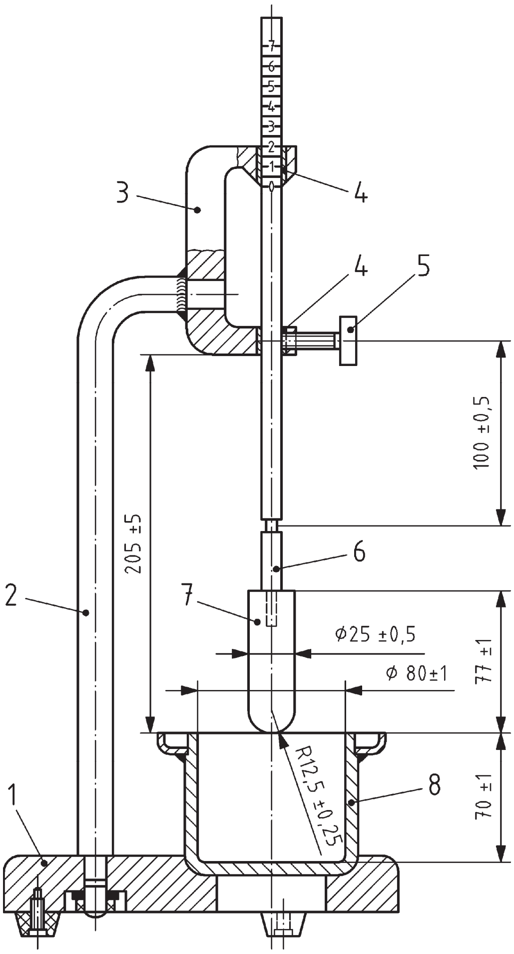
The total mass of the measuring rod and the plunger shall be (90 ± 2) g. The fixing screw shall enable the measuring rod to be set such that the distance between the lower end of the plunger and the upper surface of the mortar (height of fall) prior to commencing the test (initial position) is (100 ± 1) mm.

The container shall be set in the base plate such that no additional centering is required.

The plunger shall have a hemispherical lower end, be resistant to corrosion and not attacked by mortar.

The tamper shall correspond to that shown in Figure 16.

Dimension in millimetres



Key

1 base plate
2 support
3 holder
4 guide bushes
5 fixing screw
6 aluminium measuring rod (scale with 2 mm graduations)
7 plunger
8 container (with drip protection)

Figure 17 — Typical plunger apparatus for measuring the value of penetration

6.8.2.2 Mixing of mortar

Prepare the mortar by the procedure described in EN 196-1 except that the water content shall be that determined to give the consistence required as described in Table 3. The modifications for the mixing procedure shall apply as follows in Table 4.

If the required flow diameter (Table 3) is not achieved with the selected amount of water, determine the correct amount of water by using other mixtures with different quantities of water. Weigh the water to the nearest 1 g.

NOTE It is useful to draw a graph correlating the flow diameter with the quantity of water used. This facilitates the determination of the required quantity of water.

Table 3 — Values for flow diameter

Type of building lime	Flow diameter
hydrated lime, hydrated dolomitic lime	(165 ± 3) mm
NHL 2, FL 2	
NHL 3,5; FL 3,5	
NHL 5, FL 5, HL 2, HL 3,5, HL 5	(185 ± 3) mm

Table 4 — Mixing procedures for lime with hydraulic properties

Type of lime with hydraulic properties	Mixing procedure
All types of NHL, FL and HL	As in EN 196-1
for FL with organic additions (see EN 459-1:2010, Annex D) it is recommended to use this mixing procedure	a) place the water and binder into the bowl; b) start mixer at low speed (see Table 2 of EN 196-1:2005) whilst starting the timing of the mixing stages. After 30 s of mixing, add the sand steadily during the next 30 s. Continue the mixing for an additional 30 s at low speed; c) stop the mixer for 30 s, remove by means of a rubber or plastics scraper the mortar adhering to the wall and bottom part of bowl and place in the middle of the bowl; d) continue the mixing at low speed for 15 s.

6.8.2.3 Flow diameter

6.8.2.3.1 If the flow table has not been used during the hour prior to the test, jolt the empty table several times. There shall be neither fluid nor dirt between the bearing (6), and the boss (8), see Figure 16.

6.8.2.3.2 In order to determine the flow diameter, place the mould in the centre of the clean, dry surface of the flow table (see 6.8.2.1.2). Fill it with two layers of mortar during which operation holding the mould with the mounted hopper firmly onto the plate with one hand in such a way that its edge coincides with the circle engraved on the plate. Spread each layer of mortar by tamping it lightly ten times with the tamper (11 on Figure 16) so that the mould is filled uniformly.

Remove the hopper without delay and strike off excess mortar. Clean the flow table and remove any water in the proximity of the mould. After 10 s to 15 s have elapsed from the time the mortar has been struck off, lift the mould slowly and vertically from the plate and spread the mortar by jolting the plate 15 times by switching on the motor set at one jolt per second. Then measure the diameter of the pat with a calliper in two directions at right angles to one another. Report the mean value of these measurements to 1 mm as the flow.

6.8.3 Water demand for values of flow and penetration

The quantity of water in grams – water demand – required for a flow diameter (Table 3) using a mortar mix described in 6.8.2.2, shall be stated. In addition, the value of penetration, in millimetres, for the same mortar measured using the plunger apparatus described in 6.8.2.1.3 shall be stated.

For measuring the value of penetration, place the mortar in the container described in 6.8.2.1.3 in two layers within 3 min after the completion of the mixing operation. Spread each layer by tamping it ten times with the tamper.

Remove excess mortar slowly with a sawing motion using a steel ruler held at a slant and then smooth it in the same way. Avoid compaction of the mortar during this operation. Then place the mortar container in the base plate with the plunger in its initial position. Loosen the fixing screw to release the drop device. Read off the value of penetration in millimetres at the underside of the bevelled guide bush.

6.9 Water retention

6.9.1 Principle

The water retention of the fresh standard mortar prepared according to 6.8.2.2 is expressed as that percentage of water which remains in the mortar after a short suction time on a filter paper.

6.9.2 Apparatus

6.9.2.1 Filter paper plate, 190 mm × 190 mm × 2 mm.

More than one plate may be used to achieve the required depth of 2 mm.

6.9.2.2 Nonwoven tissue, 185 mm in diameter.

6.9.2.3 Conical plastics ring, 140 mm smaller and 150 mm larger inside diameter, 12 mm in height.

6.9.2.4 Balance, capable of weighing to the nearest 0,1 g.

6.9.2.5 Steel straightedge.

6.9.2.6 Two plastics plates, 200 mm × 200 mm × 5 mm.

6.9.3 Preparation of specimen material

Prepare the test mortar as described in 6.8.2.2. The apparatus, conditioning chamber and test room shall have a temperature of (20 ± 2) °C. The relative humidity in the conditioning chamber and/or test room shall be higher than 50 %.

6.9.4 Procedure

For determining the water retention, use a mortar of known water fraction (W_1) prepared as in 6.9.3.

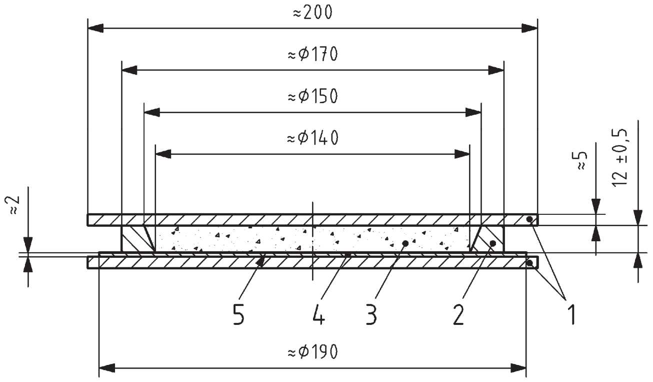
Use the test arrangement as shown in Figure 18. Weigh the dry filter plate together with one of the two plastics plates (m_{17}). Then place a piece of nonwoven tissue on the filter plate, the plastics ring, with its smaller opening downwards, on top of this, and weigh the assembly (m_{18}).

Commence testing 15 s after the mixing process has been completed, i.e. at the time when the mortar is poured into the plastics ring.

Place the mortar into the plastics ring as quickly and uniformly as possible and level it with the straightedge immediately. Following that, weigh the assembly again (m_{19}), cover it with the second plastics plate and leave it to stand for 5 min. Then invert the test arrangement and remove and weigh the lower plastics plate with the filter plate (m_{20}).

If the mass of water absorbed by the filter plate, $W_3 = m_{20} - m_{17}$, exceeds 10 g, repeat the test using two or more filter plates, selecting the number of filter plates used, n, so that the ratio $W_3/n \leq 10$ g.

Dimensions in millimetres



Key

1 plastics plate
2 plastics ring
3 test mortar
4 filter plate(s)
5 nonwoven tissue

Figure 18 — Typical test arrangement for determining the water retention of fresh mortar

6.9.5 Evaluation

Calculate the water retention (WR) on the basis of the following formulae.

Calculate the water fraction of the test mortar (W_1) from the equation:

$$W_1 = \frac{m_{21}}{m_{21} + m_{22}} \tag{20}$$

where

W_1 is the water fraction of the test mortar;

m_{21} is the total mass of water in fresh mortar, in grams;

m_{22} is the mass of dry mortar, in grams.

Calculate the water content of the mortar in the plastics ring (W_2) from the following equations:

$$W_2 = m_{23} \times W_1 \tag{21}$$

$$m_{23} = m_{19} - m_{18}$$

where

W_2 is the water content of the mortar in the plastics ring, in grams;

m_{23} is the mass of mortar in the plastics ring, in grams;

m_{18} is the mass of the plastics plate, filter plate, nonwoven tissue and plastics ring, in grams;

m_{19} is the mass of the plastics plate, filter plate, nonwoven tissue and plastics ring with mortar filling, in grams.

Calculate the mass of water absorbed by the filter plate (W_3) from the following equation:

$$W_3 = m_{20} - m_{17} \tag{22}$$

where

W_3 is the mass of water absorbed by the filter plate, in grams;

m_{17} is the mass of the dry filter plate and the plastics plate, in grams;

m_{20} is the mass of the soaked filter plate and the plastics plate, in grams.

Calculate the relative loss of water from the mortar (W_4) from the following equation:

$$W_4 = \frac{W_3}{W_2} \times 100 \tag{23}$$

where

W_4 is the relative loss of water from the mortar, in percent.

Calculate the water retention (WR) as a percentage from the following equation:

$$WR = 100 - W_4 \tag{24}$$

State the individual values and the mean of two individual values to the nearest 0,1 %.

6.10 Determination of air content

6.10.1 Apparatus

The air content of fresh mortar shall be measured by the pressure method using a preset test apparatus with a capacity of 1 dm^3 or 0,75 dm^3.

The test apparatus shall have a pressure chamber in which a defined pressure is produced. By opening an overflow valve a pressure balance is effected between the pressure chamber and the sample container (measuring vessel) which is filled with fresh mortar. The drop in pressure is a measure of the air content in the fresh mortar. The air content in the mortar shall be read from a calibration curve constructed according to 6.10.2.

6.10.2 Calibration of apparatus

6.10.2.1 General

The apparatus shall be calibrated at least every six months. A calibration curve has to be established between 0 % and 25 % air content with intervals of 5 %. Each corrected value shall be calculated from the mean value of three measurements according to the procedures described in 6.10.2.2 and 6.10.2.3.

6.10.2.2 Determination of the volume of the vessel

Determine the volume of the vessel, which during the test is filled with mortar, by determining the mass of clean water at (20 ± 2) °C, which the vessel can contain. Weigh the empty vessel and a glass cover plate. Fill the vessel completely and carefully cover it by sliding across a glass plate, leaving no air bubbles between the glass and the water. Weigh the full vessel and plate and calculate the mass of water by difference.

6.10.2.3 Calibration method

Fill the vessel with clean water at (20 ± 2) °C. Fix the upper section of the apparatus onto the edge of the vessel and fasten it securely. Fill the vessel with water by means of the filling tube until the water flows out of the tubes. Close both valves. Increase the pressure in the pressure chamber exactly to the starting mark. Equalize the pressure in the pressure chamber and vessel. Open the valve of the overflow tube (bent downwards) slightly.

NOTE The tube is fitted with an extension piece for this calibration procedure only.

The extension tube shall reach nearly to the bottom of the vessel. Drain off a sufficient quantity of water (10 ml corresponds to about 1 % of air). The exact percentage depends on the volume of the vessel measured as in 6.10.2.2 (by mass or volume to 0,1 cm^3). Read the air content indicated and continue the calibration with a newly built up pressure in the pressure chamber, equalize the pressure, open the valve in the overflow tube, etc.

6.10.3 Preparation of test mortar

Prepare the test mortar as described in 6.8.2.2. Carry out the air content determination not less than 5 min after mixing the test mortar. Carry out the test on two samples.

6.10.4 Procedure

Fill the measuring vessel with the mortar by means of a scoop up to about half height. To compact the mortar, raise the vessel about 30 mm on alternate sides and allow it to fall on a solid rigid substrate between ten times to 20 times. Then fill the measuring vessel with additional mortar to the top edge and compact it in the same manner.

The amount of mortar subsequently added shall be such that mortar projects above the edge. Then skim this off with a palette knife.

Attach the upper section of the test apparatus (gauge cylinder with fitting funnel) and fasten it securely.

Fill the remaining free volume in the vessel with water until all air above the specimen is displaced, which is indicated by overflow through the overflow tube. Then close the valves and pump air until the pressure in the upper part (pressure chamber) has reached the initial value. Correct too high a pressure with a special correction valve. With the pressure equalized, read the air content from the air content gauge calibration curve, using the indicated pressure.

6.10.5 Reporting of results

Report the air content as a percentage (mean value of two determinations) to the nearest 0,5 %.

6.11 Compressive strength

6.11.1 General

Determine the compressive strength in accordance with EN 196-1, "lime with hydraulic properties" being substituted where "cement" is mentioned. Prepare three specimens for each batch.

The modifications given in 6.11.2 shall apply.

6.11.2 Modifications to EN 196-1

6.11.2.1 Composition of mortar

See Table 5 for the water/binder ratios for the different building lime products.

Table 5 — Water/binder ratios

Type of product	Bulk density kg/dm^3	Water/binder ratio	Mass of water g
HL 5, NHL 5, FL 5	> 0,6	0,5	225 ± 2
HL 2, NHL 2, FL 2, HL 3,5; NHL 3,5; FL 3,5	> 0,6	0,55	248 ± 2
HL 2, NHL 2, FL 3,5; HL 3,5; NHL 3,5; NHL 5, FL 5	≤ 0,60	0,60	270 ± 2
FL 2	≤ 0,60	0,65	292,5 ± 2

6.11.2.2 Compaction of specimens

6.11.2.2.1 General

Compaction by means of the vibrating table shall be the reference method (6.11.2.2.2).

The method specified in EN 196-1 (jolting apparatus) is an alternative method.

Other alternative methods of compaction may be used provided that the absolute difference between the mean values of the 28 day compressive strength (based on at least ten test results) does not exceed 1,5 MPa in parallel tests with three different HL 5 and NHL 5.

6.11.2.2.2 Vibrating table

The vibrating table (see Figure 19) shall conform to the following requirements:

a) method of operation: electromagnetic vibrator with a nominally sinusoidal vibration;

b) electrical supply:
   1) voltage : 230/240 V;
   2) phase : single;
   3) current : maximum 6,3 A;
   4) frequency : nominal 50 Hz;

c) vibrating mass (including empty mould, hopper and clamp but excluding the vibrator): (35 ± 0,1) kg:

d) natural frequency of the vibrating mass: (53,00 ± 0,25) Hz;

e) vibrating plate The working surface shall be ground to give an even surface. Minimum size (width × depth) 400 mm × 300 mm consisting of either:

   1) a single layer of stainless steel, (minimum thickness 10 mm); or

   2) a double layer of metal (minimum thickness 20 mm), the top layer made of stainless steel with a minimum thickness of 1 mm being bonded permanently to the bottom layer by means of a frictional and inter-locking connection;

f) clamp for moulds Fixing attachment suitable for moulds

   40 mm × 40 mm × 160 mm including the mounted hopper;

g) mass of table ≥ 100 kg.

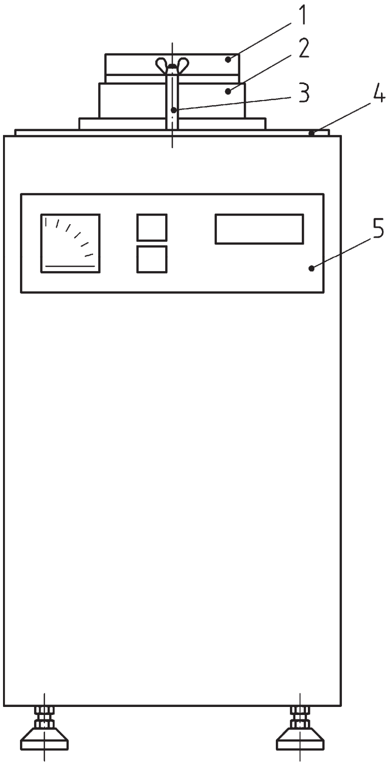
With the aid of the adjusting screws attached to its lower surface, the vibrating table shall be set up in such a way that the working surface of the oscillating plate deviates from the horizontal plane by not more than 1 mm/m.

The oscillating plate shall produce uniaxial vertical vibrations only. The peak-to-peak amplitude measured at the centre separating walls and the outer corners of the empty mould shall be (0,75 ± 0,10) mm. The amplitude shall be continuously displayed.

The table shall be equipped with guides or similar devices to hold the mould in the centre of the working surface.

It shall be possible to secure the mould and the hopper mounted on it firmly in position by means of a fixing attachment.

The duration of vibration shall be set to the nearest second with an automatic timer.



Key

1 hopper
2 mould
3 clamp
4 oscillating plate
5 control panel with display of amplitude, timer, amplitude setting and main switch

Figure 19 — Example of a vibrating table compaction apparatus (schematic representation)

6.11.2.2.3 Compaction procedure

Cast the specimens immediately after the preparation of the mortar.

Compact the mortar on the vibrating table.

The mould shall be mounted firmly on the vibrating table. After the vibrator has been switched on, fill the compartments of the mould with two layers of mortar within a maximum of 45 s, proceeding as follows.

Working from right to left, place the first layer of mortar into the compartments of the mould within 15 s so that the compartments are approximately half-full.

After an interval of 15 s, during which the vibrator remains switched on, place the second layer in the mould within the following 15 s, again working from right to left. Use the total amount of mortar specified in 6.11.2.1. The vibrator shall switch off automatically after (120 ± 1) s.

Lift the mould off the vibrating table as gently as possible and remove the hopper. Remove the excess mortar immediately afterwards using a straightedge (EN 196-1:2005, Figure 3) held almost vertically and moved slowly with a transverse sawing motion (EN 196-1:2005, Figure 2) once in each direction. Smooth the surface of the specimen using the same straightedge held almost level.

6.11.2.3 Storage of specimens after demoulding

6.11.2.3.1 For NHL 2, FL 2 and FL 3,5

Store the demoulded specimens in a large cabinet (conforming to 4.1 of EN 196-1:2005) maintained continuously at a temperature of (20 ± 1) °C and a relative humidity of not less than 60 %, observing the tolerances in respect of time specified in EN 196-1, until such time as the compressive strength test is carried out. No enrichment of atmospheric carbon dioxide level is permitted.

6.11.2.3.2 For all other types of lime with hydraulic properties

Store the demoulded specimens in a large cabinet (conforming to 4.1 of EN 196-1:2005) maintained continuously at a temperature of (20 ± 1) °C and a relative humidity of not less than 90 %, observing the tolerances in respect of time specified in EN 196-1, until such time as the compressive strength test is carried out.

6.11.2.4 Rate of increase in compressive load

Increase the compressive load at a rate of (400 ± 40) N/s for all types of lime with hydraulic properties.

Annex A
(informative)

Example for the calculation of the results on sieving

Sieve aperture size	R		R		Retention on sieving			Mass passing the sieve
					Single values m (P_i)		Cumulative values	Cumulative values
	g		g		g	%	%	%
10		0,0			0,0	0,0	0,0	100,0
5		32,9			32,9	22,5	22,5	77,5
2	130,5	97,6			97,6	66,9	89,4	10,6
0,2				11,0	11,0	7,5	96,9	3,1
0,09			11,6	0,6	0,6	0,5	97,4	2,6
pan	15,4		3,8		3,8	2,6	100,0	0,0
	145,9	130,5	15,4		145,9	100,0		

sample mass	9 087,0	g
dividing of sample	yes	
number of divisions	6	
mass of test sample	145,9	g

sample mass on 2 mm	145,9	g
sample mass on 5 mm	130,5	g
sample mass on 0,09 mm	15,4	g
sample mass on 0,2 mm	11,6	g

Annex B
(informative)

Precision data for the test methods

The repeatability and reproducibility limits were determined in an interlaboratory test which was carried out in accordance with ISO 5725-2.

Table B.1 — Precision data for the chemical test methods

Parameter		Product	L	N	$\overline{\overline{x}}$	s_r	s_R	r	R
CaO	%	CL90-Q	14	52	96,188	0,126	0,385	0,349	1,067
		CL90-S	15	57	74,187	0,148	0,341	0,409	0,946
		DL90-S	13	49	47,446	0,201	0,918	0,556	2,543
MgO		CL90-Q	15	56	1,078	0,076	0,113	0,21	0,312
		CL90-S	13	49	0,495	0,017	0,066	0,047	0,183
		DL90-S	13	49	32,786	0,267	0,678	0,74	1,878
CO_2		CL90-Q	14	54	0,423	0,017	0,059	0,048	0,164
		CL90-S	13	50	0,561	0,038	0,076	0,105	0,21
		DL90-S	12	46	0,855	0,035	0,145	0,097	0,4
free water		CL90-S	14	55	0,448	0,057	0,138	0,157	0,384
		DL90-S	13	52	0,465	0,044	0,158	0,122	0,437
loss on ignition		CL90-Q	9	36	1,228	0,012	0,066	0,034	0,182
		CL90-S	12	48	24,879	0,05	0,108	0,139	0,3
		DL90-S	13	52	17,101	0,061	0,15	0,17	0,415
available lime	%	CL 90-Q	17	64	94,46	0,19	0,46	0,53	1,28
		CL 90-S	17	68	92,38	0,29	0,70	0,82	1,96
		CL 80-Q	14	56	69,6	0,29	1,06	0,81	3,0
		CL 80-S	17	56	70,73	0,31	1,28	0,88	3,59
		FL A	17	64	52,97	0,45	1,49	1,25	4,18
		FL B	17	64	47,90	0,57	3,00	1,59	8,39
		NHL 3,5	17	64	32,12	0,41	3,18	1,16	8,90

L	number of laboratories evaluated	s_R	standard deviation of reproducibility
N	number of single analytical values without outliers	r	repeatability
$\overline{\overline{x}}$	total mean value	R	reproducibility
s_r	standard deviation of repeatability		

Table B.2 — Precision data for the physical test methods

Parameter		Product	L	N	$\bar{\bar{x}}$	s_r	s_R	r	R
compressive strength	MPa	NHL 2	10	28	4,1	0,24	0,71	0,67	1,97
		NHL 3,5	10	28	3,8	0,38	1,0	1,0	2,8
		FL A2	10	28	3,3	0,21	0,49	0,58	1,37
water demand for flow	ml	NHL 2	9	24	343,09	2,43	24,17	6,82	67,68
		NHL 3,5	9	22	324,33	2,54	14,70	7,11	41,15
		FL A2	8	21	380,91	3,72	25,99	10,42	72,77
flow value	mm	NHL 2	9	24	164,54	1,25	2,05	3,49	5,75
		NHL 3,5	10	27	184,73	1,42	1,78	3,98	4,99
		FL A2	9	24	165,63	1,76	1,54	4,94	4,30
penetration	mm	NHL 2	7	21	23,15	0,95	6,56	2,67	18,38
		NHL 3,5	7	21	28,76	2,00	10,32	5,60	28,90
		FL A2	7	21	20,22	1,59	7,21	4,45	20,17
air content	%	NHL 2	8	24	2,79	0,17	0,61	0,46	1,70
		NHL 3,5	8	24	2,80	0,24	0,94	0,68	2,63
		FL A2	8	24	7,10	0,36	0,85	1,00	2,37
initial setting time	h	NHL 2	9	24	5,61	0,63	2,01	1,76	5,64
		NHL 3,5	9	24	4,95	0,62	1,64	1,73	4,58
		FL A2	9	24	5,61	0,18	2,09	0,49	5,85
final setting time	h	NHL 3,5	8	21	11,02	0,63	3,95	1,78	11,06
reactivity t_{50}	min	CL 80-Q	15	56	10,2	0,38	0,7	1,06	2,0
reactivity t_{60}	min	CL 80-Q	15	56	21	0,84	2,03	2,34	5,7
reactivity t_{60}	min	CL 90-Q	15	56	9,9	0,36	0,58	1,0	1,64
air-jet sieving 0,2 mm	%	CL 80-Q	15	60	4,08	0,19	0,38	0,53	1,07
air-jet sieving 0,09 mm	%	CL 80-Q	15	60	15,4	0,27	0,74	0,77	2,06
air-jet sieving 0,09 mm	%	CL 90-Q	15	60	1,0	0,08	0,21	0,23	0,59

L	number of laboratories evaluated
N	number of single analytical values without outliers
$\bar{\bar{x}}$	total mean value
s_r	standard deviation of repeatability
s_R	standard deviation of reproducibility
r	repeatability
R	reproducibility

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