



BSI Standards Publication

Building lime

Part 2: Test methods

National foreword

This British Standard is the UK implementation of EN 459-2:2021. It supersedes BS EN 459-2:2010, which is withdrawn.

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

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

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European foreword

This document (EN 459-2:2021) has been prepared by Technical Committee CEN/TC 51 “Cement and building limes”, the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2022, and conflicting national standards shall be withdrawn at the latest by January 2022.

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

This document supersedes EN 459-2:2010.

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

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

EN 459, *Building lime*, is currently composed of the following parts:

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

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

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

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

Introduction

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

Unless otherwise stated, tolerance class m of ISO 2768-1 apply (Indications on drawings by "ISO 2768-1").

All dimensions are in millimetres.

1 Scope

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

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

This document specifies the reference methods and, in certain cases, an alternative method which can be considered to be equivalent. In the case of a dispute, only the reference methods are used.

Any other methods may be used provided they are calibrated, either against the reference methods or against internationally accepted reference materials, in order to demonstrate their equivalence.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 196-1:2016, *Methods of testing cement - Part 1: Determination of strength*

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

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

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

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

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

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

4 Sampling

4.1 General

Sampling shall be carried out as specified in 4.2 to 4.4 taking into account the need to minimize moisture and carbon dioxide absorption. Samples shall therefore be transported and stored in air-tight containers and all the handling shall be carried out as quickly as possible.

4.2 Sampling of powdered material

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

4.3 Sampling of granular material

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

4.4 Sampling of lime putty and milk of lime

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

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

4.5 Preparation of the test portion

Before carrying out the analysis, the sample shall be reduced in mass by means of a sample divider and/or by any homogenization process (quartering, mixing, etc.) to produce a representative homogeneous test sample of suitable mass for the intended determinations.

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

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

Table 1 — Sample preparation for the single tests

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

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

5 General requirements for testing

5.1 Methods of chemical analysis

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

Table 2 — Methods of chemical analysis

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

5.2 Number of tests

Analysis of a building lime may require the determination of a number of its chemical properties. For each determination one or more tests shall be carried out in which the number of measurements to be taken shall be as specified in the relevant clause of this document.

Where the analysis is one of a series subject to statistical control, determination of each chemical property by a single test shall be the minimum required.

Where the analysis is not part of a series subject to statistical control, the number of tests for determination of each chemical property shall be two (see also 5.3).

5.3 Repeatability and reproducibility

Repeatability — Precision under repeatability conditions where independent test results are obtained with the same method on identical test items (material) in the same laboratory by the same operator using the same equipment within short intervals of time.

Reproducibility — Precision under reproducibility conditions where test results are obtained with the same method on identical test items (material) in different laboratories with different operators using different equipment.

Repeatability and reproducibility in this document (see Annex B) are expressed as repeatability standard deviation(s) and reproducibility standard deviation(s) in e.g. absolute percent, grams, etc., according to the property tested.

5.4 Expression of masses, volumes, factors and results

Express masses in grams to the nearest 0,001 g and volumes from burettes in millilitres to the nearest 0,05 ml.

Express the factors of solutions, given by the mean of three measurements, to three decimal places.

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

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

If the test is in duplicate, express the result as a mean value. If the two test results differ by more than twice the standard deviation of repeatability, repeat the test and take the mean of the two closest test results.

The results of all individual tests shall be recorded.

5.5 Blank determinations

Carry out a blank determination without a sample, where relevant, following the same procedure and using the same amounts of reagents. Correct the results obtained for the analytical determination accordingly.

5.6 Reagents

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

Unless otherwise stated percent means percent by mass.

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

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

5.7 Evaluation of test results

5.7.1 General

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

5.7.2 Test results for quicklime

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

5.7.3 Test results for all other types

For all other types (hydrated lime, lime putty, milk of lime, lime with hydraulic properties) the values are based on the product after subtraction of its free water and bound water content. The values obtained by application of procedures described in this document for total calcium oxide and magnesium oxide (6.3), magnesium oxide (6.3), sulfate (6.4) and carbon dioxide (6.6 or 6.7) are for the products without subtraction of the free water and bound water content. To compare these values with EN 459-1 (see clauses for calcium lime or dolomitic lime), they shall first be corrected by multiplication by factor F . Factor F shall be determined in the following way:

Determine the carbon dioxide content as described in 6.6 or 6.7 and the loss on ignition as described in 6.8. The loss on ignition is the sum of the free water, bound water and carbon dioxide, provided that the sample does not contain any highly volatile compounds or oxidizable constituents. Calculate the total (free + bound) water content W_T as a mass fraction in % of the sample using:

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

Calculate the factor F from the following formula:

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

5.7.4 Test results for available lime

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

6 Chemical analysis

6.1 Extraction with hydrochloric acid (Reference method)

6.1.1 General

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

6.1.2 Principle

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

6.1.3 Reagents

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

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

6.1.3.3 Hydrogen peroxide solution; diluted, 1 + 9.

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

6.1.3.5 Ammonium hydroxide solution, diluted, 1 + 9.

6.1.3.6 Ammonium chloride, NH_4Cl .

6.1.4 Apparatus

- 6.1.4.1 Ordinary laboratory equipment.
- 6.1.4.2 Hot plate.
- 6.1.4.3 Analytical balance accurate to 1 mg.
- 6.1.4.4 Magnetic stirrer and magnetic rod, inert e.g. PTFE covered.
- 6.1.4.5 pH-meter with glass electrode, capable of measuring to an accuracy of 0,05.
- 6.1.4.6 Fluted filter paper with particle retention size 2,5 microns.

6.1.5 Procedure

Weigh $(1 \pm 0,1)$ g of the sample to the nearest 1 mg (m_1), transfer it to a 250 ml beaker, moisten with 10 ml of water, then gradually add 30 ml of hydrochloric acid (6.1.3.1). Make the solution up to about 100 ml with water then boil it for 10 min. After boiling, filter the solution immediately through a filter paper (6.1.4.6) into a 400 ml beaker and wash the residue well with hot water.

Add about 4 g of ammonium chloride (6.1.3.6) and a few drops of hydrogen peroxide (6.1.3.3) to the solution, then dilute with about 150 ml of water and heat to boiling. During boiling, add ammonium hydroxide solution (6.1.3.4) 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 filter paper (6.1.4.6) into a 500 ml volumetric flask. Wash the filter residue three times with ammonium hydroxide solution (6.1.3.5) 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.

6.2 Fusion with lithium tetraborate (Alternative method)

6.2.1 General

Fusion with lithium tetraborate is used to dissolve building lime in order to determine appropriate parameters (see Table 2).

6.2.2 Principle

After fusing the sparingly soluble oxide constituents of the sample with lithium tetraborate, the fusion cake is dissolved in hydrochloric acid and transferred to a suitable volumetric flask.

6.2.3 Reagents

- 6.2.3.1 Lithium tetraborate, $\text{Li}_2\text{B}_4\text{O}_7$.
- 6.2.3.2 Hydrochloric acid, $\rho = 1,16$ g/ml.
- 6.2.3.3 Hydrochloric acid, diluted, (1 + 5).

6.2.4 Apparatus

6.2.4.1 Ordinary laboratory apparatus and the following:

6.2.4.2 Platinum-gold crucible.

6.2.4.3 Hot plate.

6.2.5 Procedure

Weigh $(0,25 \pm 0,02)$ g to the nearest 0,1 mg (m_1) of the sample into a platinum-gold crucible and add 1,5 g of lithium tetraborate (6.2.3.1). Roast at $(1\,000 \pm 25)$ °C until the melt is clear. Remove from the heat, cover the crucible with a watch glass and stand the crucible in water for a few seconds, remove and allow it to stand until it has cooled completely.

Transfer the cold melt to a beaker using approximately 50 ml of hydrochloric acid (6.2.3.3) and dissolve while stirring and heating at 100 °C. Then transfer the solution to a 250 ml volumetric flask and, after cooling to room temperature make up to the mark with water. This solution (V_1) is ready for further chemical analyses.

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

6.3.1 General

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

6.3.2 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 ($\Sigma \text{CaO} + \text{MgO}$) 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, $c(\text{CaO} + \text{MgO})$ and $c(\text{CaO})$, gives the magnesium content, $c(\text{MgO})$.

The end points can also be determined using potentiometric titration.

6.3.3 Reagents

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

6.3.3.2 Hydrochloric acid, diluted, 1 + 1.

6.3.3.3 Triethanolamine, $c[\text{N}(\text{C}_2\text{H}_5\text{OH})_3] = 99\%$.

6.3.3.4 Triethanolamine solution, diluted, 1 + 1.

6.3.3.5 Sodium hydroxide solution, $c(\text{NaOH}) = 4$ mol/l.

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

6.3.3.7 Ammonium chloride, NH_4Cl .

6.3.3.8 Ethylenediaminetetra-acetic acid disodium salt dihydrate (EDTA), ($\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2 \text{H}_2\text{O}$), dried to constant mass at 80 °C before weighing.

6.3.3.9 Calcium carbonate, $c(\text{CaCO}_3) = 99,9 \%$ (dried at $(200 \pm 10) ^\circ\text{C}$).

6.3.3.10 Buffer solution, make 70 g of ammonium chloride (6.3.3.7) and 570 ml of ammonium hydroxide solution (6.3.3.6) up to the mark with water in a 1 000 ml volumetric flask.

6.3.3.11 EDTA solution, $c(\text{EDTA}) = 0,04 \text{ mol/l}$.

Preparation:

Dissolve 14,89 g of EDTA (6.3.3.8) in water and making up to 1 000 ml in a volumetric flask.

Standardization:

Pipette 50 ml of the calcium ion reference solution (6.3.3.12) into a 400 ml beaker and dilute with 100 ml of water. Adjust the pH value of the solution to $(12,5 \pm 0,5)$ with sodium hydroxide solution (6.3.3.5) using a pH meter. Add 0,1 g of calconcarboxylic indicator (6.3.3.13) and titrate with the EDTA solution being standardized until the colour changes to blue.

The concentration factor c of the EDTA solution is given by the following formula:

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

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.

6.3.3.12 Calcium ion reference solution, $c(\text{Ca}^{2+}) = 0,01 \text{ mol/l}$: transfer $(1 \pm 0,002) \text{ g}$ of calcium carbonate (6.3.3.9) (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 (6.3.3.2). 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 with water in a volumetric flask.

6.3.3.13 Calconcarboxylic acid indicator: grind 0,2 g of calconcarboxylic acid intensively with 20 g of anhydrous sodium sulfate in a mortar.

6.3.3.14 Eriochrome Black T indicator: grind 1 g of Eriochrome Black T intensively with 100 g of sodium chloride in a mortar.

6.3.4 Apparatus

6.3.4.1 Ordinary laboratory equipment.

6.3.4.2 Magnetic stirrer with magnetic rod.

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

6.3.5 Procedure

6.3.5.1 Determination of CaO

To determine the calcium oxide content, transfer 25 ml (V_3) of the solution prepared in accordance with 6.1.5 or 6.2.5 to a 400 ml beaker, dilute the solution with about 150 ml of water and add 5 ml of triethanolamine solution (6.3.3.4).

Adjust the pH value of this solution to $(12,5 \pm 0,5)$ with sodium hydroxide solution (6.3.3.5) using a pH meter. Add 0,1 g of calconcarboxylic acid indicator (6.3.3.13) 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 $(12,5 \pm 0,5)$.

6.3.5.2 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 6.1.5 or 6.2.5 to a 400 ml beaker. Add 5 ml of triethanolamine solution (6.3.3.4) and adjust the pH value of the solution to $(10,5 \pm 0,5)$ with buffer solution (6.3.3.10) using a pH-meter. Then add about 90 % of the volume of EDTA solution used during the calcium oxide titration (as described in 6.3.5.1) and, after adding 0,1 g of Eriochrome Black T indicator (6.3.3.14), titrate the solution until the colour changes from wine red to blue. Record the total volume (V_6) of EDTA solution added.

6.3.6 Evaluation and expression of results

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

$$w_{\text{CaO}} = \frac{V_1 \cdot 0,04 \cdot 56,08 \cdot c \cdot V_4 \cdot 100}{V_3 \cdot 1000 \cdot m_1} \quad (4)$$

where

- V_1 is the volume of the digestion solution (6.1.5 or 6.2.5), in millilitres;
- V_3 is the volume of the aliquot of the digestion solution V_1 taken for titration as described in 6.3.5.1, in millilitres;
- V_4 is the volume of EDTA solution used for the CaO determination as described in 6.3.5.1, in millilitres;
- c is the concentration factor of the EDTA solution, as determined in 6.3.3.11;
- m_1 is the mass, in grams, of the test portion used in 6.1.5 or 6.2.5.

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

$$w_{\text{MgO}} = \frac{V_1 \cdot 0,04 \cdot 40,311 \cdot c \cdot (V_6 - V_4) \cdot 100}{V_5 \cdot 1000 \cdot m_1} \quad (5)$$

where

- V_1 is the volume of the digestion solution (6.1.5 or 6.2.5), in millilitres;
- V_5 is the volume of the aliquot of the digestion solution V_1 taken for titration as described in 6.3.5.2, in millilitres;
- V_4 is the volume of EDTA solution used for the CaO determination as described in 6.3.5.1 in millilitres;
- V_6 is the volume of EDTA solution used for the determination of the total CaO and MgO as described in 6.3.5.2, in millilitres;
- c is the concentration factor of the EDTA solution, as determined in 6.3.3.11;
- m_1 is the mass, in grams, of the test portion used in 6.1.5 or 6.2.5.

6.4 Sulfate (expressed as SO_3)

6.4.1 General

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

6.4.2 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.

6.4.3 Reagents

- 6.4.3.1** Hydrochloric acid, ρ (HCl) = 1,16 g/ml to 1,19 g/ml.
- 6.4.3.2** Hydrochloric acid, diluted, 1 + 1.
- 6.4.3.3** Hydrochloric acid, diluted, 1 + 50.
- 6.4.3.4** Nitric acid, ρ (HNO_3) = 1,40 g/ml to 1,42 g/ml.
- 6.4.3.5** Ammonium hydroxide solution, $c(\text{NH}_4\text{OH}) = 25 \%$.
- 6.4.3.6** Ammonium hydroxide solution, diluted, 1 + 16.
- 6.4.3.7** Barium chloride, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$.
- 6.4.3.8** Barium chloride solution: dissolve 120 g of barium chloride in water and make up to 1 000 ml with water in a volumetric flask.
- 6.4.3.9** Silver nitrate, AgNO_3 .

6.4.3.10 Silver nitrate solution: dissolve 5 g of silver nitrate (6.4.3.9) in water, add 10 ml of nitric acid (6.4.3.4) and making up to 1 000 ml with water in a volumetric flask.

6.4.4 Apparatus

6.4.4.1 Ordinary laboratory equipment.

6.4.4.2 Analytical balance accurate to 1 mg.

6.4.4.3 Hot plate or sand bath.

6.4.4.4 Muffle furnace.

6.4.4.5 Filter paper with mean pore diameter of approximately 2 microns with ash content < 0,01 %.

6.4.5 Procedure

Weigh out $(1 \pm 0,1)$ g to the nearest 1 mg of the sample (m_3) into a 250 ml beaker, add 90 ml of cold water and then add 10 ml of hydrochloric acid (6.4.3.1) 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 filter paper (6.4.4.5) into a 400 ml beaker and wash the residue several times with small portions of hot diluted hydrochloric acid (6.4.3.3).

Dilute the filtrate to about 250 ml with water and, if necessary, adjust the pH value to 1 with hydrochloric acid (6.4.3.2) or ammonium hydroxide solution (6.4.3.6). Boil the solution for 5 min and then add 10 ml of hot barium chloride solution (6.4.3.8) 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 filter paper (6.4.4.5) and wash the residue until chloride-free with boiling water, using silver nitrate solution (6.4.3.10) 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 pre-weighed 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 (m_4).

6.4.6 Evaluation and expression of results

The sulfate content expressed as SO_3 in mass fraction in % is given by the following formula:

$$w\text{SO}_3 = \frac{0,343 \cdot m_4 \cdot 100}{m_3} = \frac{34,3 \cdot m_4}{m_3} \quad (6)$$

where

m_4 is the final mass of BaSO_4 , in grams;

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

6.5 Free water

6.5.1 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 of the product, in the case of milk of lime or lime putty, it refers to the water content of the suspension.

6.5.2 Principle

When heating a sample of hydrated lime or lime with hydraulic properties to $(105 \pm 5)^\circ\text{C}$, or for milk of lime or lime putty to $(150 \pm 5)^\circ\text{C}$ 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.

6.5.3 Apparatus

6.5.3.1 Ordinary laboratory equipment.

6.5.3.2 Drying oven, thermostatically controlled to maintain a temperature of $(105 \pm 5)^\circ\text{C}$ or $(150 \pm 5)^\circ\text{C}$ (alternative equipment 1).

6.5.3.3 Automated moisture balance, being capable to be controlled between $(105 \pm 5)^\circ\text{C}$ or $(150 \pm 5)^\circ\text{C}$ (alternative equipment 2).

6.5.3.4 Analytical balance accurate to 1 mg.

6.5.3.5 Desiccator containing drying agent.

6.5.3.6 Unglazed porcelain crucible.

6.5.3.7 Pipette.

6.5.4 Procedure

6.5.4.1 Hydrated lime and lime with hydraulic properties

Weigh between $(5 \pm 0,1)$ g and $(10 \pm 0,1)$ g to the nearest 1 mg (m_5) of the sample in the as-delivered state in a pre-weighed crucible. Dry the sample until constant mass using the oven (6.5.3.2) or the balance (6.5.3.3). 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 (m_6).

6.5.4.2 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 0,1 mg in a glass vessel (m_5). Dry the sample to constant mass using the oven (6.5.3.2) or the balance (6.5.3.3). 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 dessicator, determine the loss in mass (m_6).

6.5.5 Evaluation and expression of results

The free water content expressed as H₂O in mass fraction in % is given by the following formula:

$$w_{\text{H}_2\text{O}} = \frac{(m_5 - m_6)}{m_5} \cdot 100 \quad (7)$$

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.

6.6 Volumetric determination of carbon dioxide (CO₂) (reference method)

6.6.1 Principle

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

6.6.2 Reagents

6.6.2.1 Hydrochloric acid, ρ (HCl) = 1,16 g/ml to 1,19 g/cm³.

6.6.2.2 Hydrochloric acid, diluted, 1 + 1.

6.6.2.3 Sulfuric acid, H₂SO₄, concentrated, > 98 %.

6.6.2.4 Copper sulfate, CuSO₄.

6.6.2.5 Methyl red.

6.6.2.6 Methyl red solution: dissolve 0,2 g of methyl red (6.6.2.5) in water and make up to 100 ml.

6.6.2.7 Sodium sulfate, Na₂SO₄.

6.6.2.8 Sealing liquid: add 20 g of sodium sulfate (6.6.2.7) and 5 ml of sulfuric acid (6.6.2.3) to water, make up to 100 ml and colour with a few drops of methyl red solution. The sealing liquid shall be saturated with CO₂.

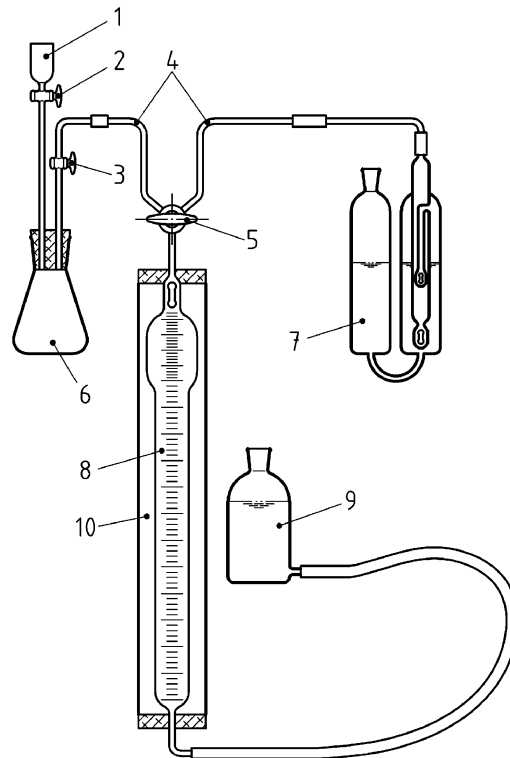
6.6.2.9 Potassium hydroxide solution, $c(\text{KOH}) = 50 \%$.

6.6.2.10 Calcium carbonate, $c(\text{CaCO}_3) = 99,9 \%$, dried to constant mass at $(200 \pm 10) ^\circ\text{C}$.

6.6.3 Apparatus

6.6.3.1 Analytical balance accurate to 1 mg.

6.6.3.2 Typical apparatus for the determination of the carbon dioxide (reference method) is shown in Figure 1.



Key

- 1 dropping funnel with a volume of 100 ml
- 2 stopcock 1
- 3 stopcock 2
- 4 capillary tubes
- 5 three-way tap
- 6 decomposition flask with a volume of 50 ml
- 7 absorption vessel with a volume of 100 ml containing potassium hydroxide solution (6.6.2.9) fitted with trap
- 8 burette with a volume of 100 ml
- 9 levelling vessel with a volume of 500 ml with sealing liquid (6.6.2.8)
- 10 jacketed tube filled with water

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

6.6.4 Procedure

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

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