BS EN 12485:2026

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Chemicals used for treatment of water intended for human consumption — Calcium carbonate, high-calcium lime, half burnt dolomite, magnesium oxide, calcium magnesium carbonate and dolomitic lime — Test methods



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The UK participation in its preparation was entrusted to Technical Committee B/504/10.

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

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Chemicals used for treatment of water intended for human consumption - Calcium carbonate, high-calcium lime, half-burnt dolomite, magnesium oxide, calcium magnesium carbonate and dolomitic lime - Test methods

Produits chimiques utilisés pour le traitement de l'eau destinée à la consommation humaine - Carbonate de calcium, chaux et dolomie semi calcinée - Méthodes d'analyse

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Calciumcarbonat, Weißkalk, halbgebrannter Dolomit, Magnesiumoxid, Calciummagnesiumcarbonat und Dolomitkalk - Prüfverfahren

This European Standard was approved by CEN on 13 March 2026.

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European foreword

This document (EN 12485:2026) has been prepared by Technical Committee CEN/TC 164 “Water supply”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by October 2026, and conflicting national standards shall be withdrawn at the latest by October 2026.

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 12485:2017.

Annexes A and B are informative. For further information on methodology see [1] to [6].

Significant technical differences between this edition and EN 12485:2017 are as follows:

a) Table 1 – XRF methods are added;

b) changed for requirements of water quality;

c) loss on ignition 550 °C changed to 500 °C;

d) unglazed porcelain or platinum crucible changed in glazed porcelain or platinum crucible.

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.

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1 Scope

This document specifies the methods used for the chemical analyses and the determination of physical properties of calcium carbonate, high-calcium lime, half-burnt dolomite, magnesium oxide, calcium magnesium carbonate and dolomitic lime used to treat water for human consumption.

This document specifies the reference methods and, in certain cases, an alternative method which can be considered to be equivalent.

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 459-2, *Building lime - Part 2: Test methods*

EN 1017:2025, *Chemicals used for treatment of water intended for human consumption - Half-burnt dolomite*

EN 1018:2021, *Chemicals used for treatment of water intended for human consumption - Calcium carbonate*

EN 12518:2014, *Chemicals used for treatment of water intended for human consumption - High-calcium lime*

EN 16003:2011, *Chemicals used for treatment of water intended for human consumption - Calcium magnesium carbonate*

EN 16004:2011, *Chemicals used for treatment of water intended for human consumption - Magnesium oxide*

EN 16409:2013, *Chemicals used for treatment of water intended for human consumption - Dolomitic lime*

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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4 General requirements

4.1 Number of determinations

Two analyses shall be carried out to determine the various constituents (see Clause 6 to Clause 9; see also 4.6).

4.2 Methods for analysis

The methods to be used for the analysis of half-burnt dolomite, calcium carbonate, high calcium lime, magnesium oxide, calcium magnesium carbonate and dolomitic lime and the principle of each method are listed in Table 1. Schematic diagrams of the analyses are given in Annex A (Figures A.1 to A.7).

NOTE 1 Any other methods can be used provided they are calibrated, either against the reference methods or against internationally accepted reference materials, in order to demonstrate their equivalence.

NOTE 2 ICP-OES is a reference analytical instrument, but others can be used, as far they demonstrate equivalence.

The requirement values for free MgO and free $Mg(OH)_2$ in half-burnt dolomite shall be expressed as free MgO in accordance with EN 1017. The same requirement is related to free CaO. The requirement value for MgO in magnesium oxide shall be expressed as MgO in dry substance in accordance with EN 16004. Therefore, the analysis of half-burnt dolomite and magnesium oxide shall be performed on a sample which is bound-water free. Before starting the chemical analysis, the loss on ignition at 500 °C shall be determined as described in 7.2. The analysis itself shall be performed on the material obtained after the determination of the loss on ignition.

The requirement values for calcium carbonate, hydrated lime and calcium magnesium carbonate shall be expressed in dry substance in accordance with EN 1018, EN 12518 and EN 16003. Therefore, the analysis of these products shall be performed on a sample which has been dried in accordance with the procedure described in 7.1.

For the determination of the water-soluble content of high-calcium lime, the method in 7.5 is considered as the reference method. The sugar method described in 7.6 can be used as an alternative method if it is demonstrated that the results obtained with this method are equivalent to those of the reference method.

The requirement values for CaO and MgO in dolomitic lime shall be expressed in accordance with EN 16409. The requirement value for CaO and MgO in calcium magnesium dioxide shall be expressed as CaO and MgO in bound-water free substance and for CaO and MgO in calcium magnesium dihydroxide oxide shall be expressed as CaO and MgO in dry and bound-water free substance. Therefore, the analysis of calcium magnesium dihydroxide oxide shall be performed on a sample which has been dried in accordance with the procedure described in 7.1.

Table 1 — Methods for analysis

Determination	Method	Principle	Standard
Screen oversize	5.1	Air-jet sieving	EN 12518, EN 16409
	5.2	Wet sieving	EN 12518
Free water	7.1	Gravimetry	EN 1018, EN 12518, EN 16003, EN 16409
Loss on ignition at 500 °C	7.2	Gravimetry	EN 1017, EN 12518, EN 16004, EN 16409
Carbon dioxide	7.3	Gravimetry	EN 1017, EN 12518, EN 16004, EN 16409

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Determination	Method	Principle	Standard
Residue insoluble in hydrochloric acid	7.4	Gravimetry	EN 1018, EN 16003
Water-soluble calcium oxide or calcium hydroxide	7.5	Acidimetric titration	EN 12518
Sugar-soluble calcium oxide or calcium hydroxide	7.6	Acidimetric titration	EN 12518
Water-insoluble constituents	7.7	Gravimetry	EN 12518
Free calcium oxide	7.8	Extraction, acidimetric titration	EN 1017, EN 16004
Calcium oxide and magnesium oxide	7.9	Complexometric titration XRF melting tablet XRF powder tablet	EN 1017, EN 1018, EN 16003, EN 16004, EN 16409
Sulfate	7.10	Gravimetry	EN 1017
Solubility index	7.11	Conductivity	EN 12518
Magnesium	8.1 or 8.2	AAS (flame) ICP-OES XRF melting tablet XRF powder tablet	EN 1017, EN 1018, EN 16003, EN 16409, EN 16004
Silicium,Aluminium,Iron	8.1 or 8.2	AAS (flame) ICP-OES XRF melting tablet XRF powder tablet	EN 1017, EN 12518, EN 16003, EN 16004, EN 16409
Manganese	8.2	ICP-OES XRF melting tablet XRF powder tablet	EN 12518, EN 16409
Sulfur	8.2	ICP-OES XRF melting tablet XRF powder tablet	EN 1017
Lead, Cadmium, Chromium, Nickel	9.1 or 9.2	AAS (flameless) or ICP-OES	EN 1017, EN 1018, EN 12518, EN 16003, EN 16004, EN 16409
Arsenic, Antimony, Selenium	9.3	AAS (hydride)	EN 1017, EN 1018, EN 12518, EN 16003, EN 16004, EN 16409
Mercury	9.4	AAS (cold-vapour technique)	EN 1017, EN 1018, EN 12518, EN 16003, EN 16004, EN 16409

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4.3 Sample preparation

The general recommendations specified in ISO 3165 shall be observed when sampling. Sampling shall be performed in accordance with EN 459-2. For products less than 6 mm grain size, the size of the sample shall be 1 l.

Before performing the chemical analyses, the size of the sample shall be reduced by using a sample divider and/or by dividing it into four parts in order to obtain a suitable subsample. The coarse-grain material in this sample shall be reduced to a size of less than 0,2 mm before performing the chemical analysis.

When sampling milk of lime, the material from which the sample is to be taken shall be thoroughly mixed with an electrically driven stirrer of adequate power. The milk of lime shall be dried before being analysed chemically as described in 7.1.

Since the subsamples under examination are altered by the absorption of moisture and carbon dioxide, their exposure to air shall be minimized. They shall therefore be transported and stored in air-tight containers and all the handling shall be carried out as quickly as possible.

4.4 Reagents

All reagents shall be of a recognized analytical grade appropriate for the method being used.

Three types of water quality are used (essential requirements given below):

— specific electrical conductivity < 0,5 mS/m (at 25 °C), for most laboratory wet chemistry work

— specific electrical conductivity < 0,2 mS/m (at 25 °C), suitable for sensible analytical work

— specific electrical conductivity < 0,01 mS/m (at 25 °C), suitable for most stringent analytical requirements

In all cases the concentration of the element(s) to be determined shall be low enough not to influence the results of the analysis. This can be verified by analysing the blank solution.

The concentrated liquids used for the reagents in this standard have the following densities (ρ) (in grams per millilitre at 20 °C):

—	hydrochloric acid	1,16 to 1,19;
—	nitric acid	1,40 to 1,42;
—	ammonium hydroxide solution	0,88 to 0,91;
—	triethanolamine	1,12.

Dilutions are specified as the sum of the volumes. Thus, (1 + 2) dilute hydrochloric acid means 1 part by volume of concentrated hydrochloric acid mixed with 2 parts by volume of water.

The concentrations of reference and standard volumetric solutions are specified as amount-of-substance concentrations, c (mol/l), while those of stock and standard solutions are specified as concentrations by mass, ρ (g/l or mg/l).

Suitable element solutions for atomic absorption spectroscopy (AAS) and for optical emission spectroscopy by inductively coupled plasma (ICP-OES) are commercially available and can be used as stock solutions. They shall be prepared for the purposes of analysis in accordance with the manufacturer's instructions. The appropriate element standard solutions are prepared in accordance with the instructions given in this document.

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This document makes no stipulations relating to the shelf life of stock, standard and reference solutions. In the case of stock solutions having an element concentration of 1 g/l, the manufacturer generally specifies a shelf life of one year. The calibration solutions should be checked regularly.

4.5 Glassware

Glass containers and pipettes shall be cleaned with hot dilute nitric acid immediately before use and then rinse with water. If determining trace elements rinse with water of a specific conductivity < 0,2 mS/m (at 25 °C).

4.6 Expression of results

The analytical results for the major and minor constituents shall be reported as mass fraction in %, while those for trace elements shall be reported as mass fraction in milligrams per kilogram, as the mean of two determinations. In general, analytical values shall be reported to three significant figures.

EXAMPLE:

— (CaO) = 91,2 %;

— (SiO_2) = 3,70 %;

— (MnO_2) = 0,15 %;

— (Cr) = 0,32 mg/kg;

— (Hg) = 0,05 mg/kg.

If the results of a duplicate determination differ from one another by more than twice the repeatability standard deviation, the determination shall be repeated. The result shall then be deemed to be the mean of the two results with the lowest difference.

To assess whether the analytical results meet the requirements laid down in EN 12518 for high-calcium lime, in EN 1018 for calcium carbonate, in EN 1017 for half-burnt dolomite, in EN 16003 for calcium magnesium carbonate, in EN 16004 for magnesium oxide and in EN 16409 for dolomitic lime, the results have to be converted to the form in which the requirements are stated in the above standards.

Subclauses of this standard are given in Table 2 and contain the relevant calculation methods.

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Table 2 — Methods for calculation of the results

Requirements from standard	Parameter	Required value for	Calculation described in
EN 1017	CaO_{free}, CaO CO_2 MgO	$CaCO_3$ $MgCO_3$ MgO_{free}	7.12.2.3
EN 1018	CaO MgO	$CaCO_3$ $MgCO_3$	7.12.2.1
EN 12518	CO_2	$CaCO_3$	7.12.2.4
EN 16004	MgO, loss on ignition at (1 050 ± 25) °C and (500 ± 25) °C	(CO_2 and H_2O)	7.12.2.5
EN 16003	MgO	$CaMg(CO_3)_2$ $MgCO_3$	7.12.2.2
EN 16409	CaO, MgO	CaO + MgO	7.12.2.6

4.7 Repeatability and reproducibility limits

The repeatability and reproducibility limits were determined in an interlaboratory test (see Annex B) which was carried out in accordance with ISO 5725-2 and in which 28 laboratories took part.

4.8 Test report

The report shall contain the following information:

a) a complete identification of the sample;

b) a reference to this European Standard (including its year of publication);

c) the method used

d) results obtained and the method of expression used (see 4.6)

e) sample pretreatment, e.g. method of digestion (see Clause 6);

f) any deviation from this standard and an indication of any circumstances which can have affected the results.

5 Determination of screen oversize of high-calcium lime

5.1 Air-jet sieving method

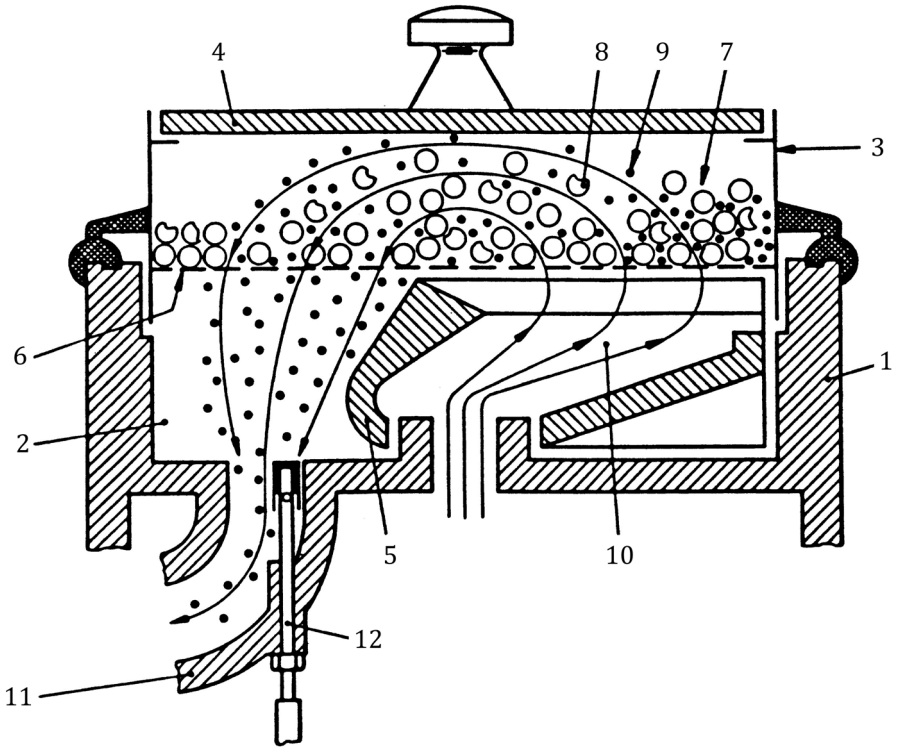
5.1.1 General

The method is used to determine the retention on sieving using airjet sieving apparatus.

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5.1.2 Apparatus

5.1.2.1 **Air-jet sieving apparatus,** of the general form shown in Figure 1. The apparatus shall be set to give a pressure difference of 2 kPa to 2,5 kPa across the sieves.



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 1 — Air-jet sieving apparatus

5.1.2.2 **Test sieves**, 200 mm diameter, aperture sizes 0,60 mm and 0,09 mm.

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 document.

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

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5.1.2.4 **Balance,** accurate to 0,1 mg.

5.1.2.5 **Soft brush.**

5.1.2.6 **Ultrasonic cleaning bath** for cleaning the mesh of the sieves.

5.1.2.7 **Mallet**, if there is a tendency for material to adhere to the lid of the apparatus. A rubber or plastics tipped mallet is preferred.

5.1.3 Procedure

Weigh an appropriate amount (between 10 g and 50 g pending on the type of material) of the dry sample to the nearest 1 mg (m). 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.

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 1 mg.

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 1 mg. 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 0,60 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 1 mg.

5.1.4 Expression of results

The mass retained on each sieve expressed as m ($P_{0,60}$ or $_{0,09}$) in mass fraction in %, is given by the following formula:

$$m\left(P_{0,60\ or\ 0,09}\right) = \frac{m_1 \times 100}{m} \quad (1)$$

where

m is the mass of the sample, in grams;

m_1 is the mass of the residue retained on the test sieves, in grams.

5.2 Wet sieving method

5.2.1 General

The method is exclusively used for the determination of the retentions on sieving of milk of lime as specified in EN 12518.

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5.2.2 Apparatus

5.2.2.1 **Balance**, accurate to 0,1 mg.

5.2.2.2 **500 ml wide-mouthed bottle, or other suitable vessel** which can be sealed with a stopper.

5.2.2.3 **200 mm diameter 0,60 mm and 0,09 mm aperture size test sieves.**

5.2.2.4 **5 mm bore rubber or plastics tubing.**

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

5.2.2.6 **Two sintered glass filter crucibles, of porosity 2** (as specified in ISO 4793:1980) (maximum porosity diameter 40 µm to 100 µm) with filter flask and adapter funnel for crucibles.

5.2.2.7 **Desiccator.**

5.2.2.8 **Wash bottle.**

5.2.2.9 **Filtration apparatus.**

5.2.3 Procedure

Superimpose the 0,60 mm mesh test sieve on the 0,09 mm mesh test sieve and wet the sieves with water. Place a volume of milk of lime containing (50 ± 0,5) g of dry substance in a 500 ml wide-mouthed bottle. Replace the stopper and shake the bottle for about 30 s. Remove the stopper immediately and pour the contents of the bottle onto the sieves. Wash any residue remaining in the bottle or on the stopper with a jet of water from the 5 mm bore tubing and at a pressure equal to a head of 1,2 m (approximately 10 kPa) on to the sieves. Then use the jet to wash the residue on the 0,60 mm mesh test sieve for (2 ± 0,1) min. Do not rub the residue through the sieve and take care not to flood the lower sieve during the operation. At the end of this period, wash the residue to one side of the sieve.

Detach the 0,60 mm mesh test sieve and, using a wash bottle, transfer the residue from the sieve through a glass funnel into one of the tared, sintered glass filter crucibles connected to the filtration apparatus. When all the residue has been washed into the filter, and the water extracted, remove the filter containing the residue and dry it at (105 ± 5) °C for 1 h. Cool the dried filter in a desiccator and reweigh.

Wash the residue on the 0,09 mm mesh test sieve for (5 ± 0,2) min. Transfer the residue to a tared filter crucible, dry at (105 ± 5) °C until constant mass is reached, cool and reweigh as described above.

5.2.4 Expression of results

The mass retained on each sieve expressed as m ($P_{0,60\ \text{or}\ 0,09}$) in mass fraction in % is given by the following formula:

$$m\left(P_{0,60\ or\ 0,09}\right) = \frac{m_1 \times 100}{m} \qquad (2)$$

where

m is the mass of the dry substance in the test portion, in grams;

m_1 is the mass of the residue retained on the test sieves, in grams.

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6 Preparation of test solutions

6.1 Fusion with lithium tetraborate

6.1.1 General

Fusion with lithium tetraborate is used to dissolve:

— high-calcium lime as specified in EN 12518 in order to determine silicon, aluminium, iron and manganese;

— half-burnt dolomite as specified in EN 1017 in order to determine calcium, magnesium, silicon, aluminium and iron;

— calcium carbonate as specified in EN 1018 in order to determine calcium and magnesium;

— magnesium oxide as specified in EN 16004 in order to determine magnesium, silicon, aluminium and iron;

— calcium magnesium carbonate as specified in EN 16003 in order to determine calcium and magnesium;

— dolomitic lime as specified in EN 16409 in order to determine calcium, magnesium, silicon, aluminium, iron and manganese.

6.1.2 Principle

After fusing the sparingly soluble oxide constituents of the sample with lithium tetraborate, the fusion cake is dissolved in hydrochloric acid and the solution made up to 250 ml in a volumetric flask. Calcium, magnesium, silicon, aluminium, iron and manganese are determined in aliquot parts of this solution by flame-AAS (8.1) or ICP-OES (8.2).

6.1.3 Reagents

6.1.3.1 Lithium tetraborate, $Li_2B_4O_7$.

6.1.3.2 Hydrochloric acid, ρ = 1,16 g/ml.

6.1.3.3 Hydrochloric acid, diluted, (1 + 5).

6.1.4 Apparatus

Ordinary laboratory apparatus and the following:

6.1.4.1 Platinum-gold crucible.

6.1.4.2 Hot plate.

6.1.5 Procedure

Weigh (0,25 ± 0,02) g to the nearest 0,1 mg of the sample into a platinum-gold crucible and add 1,5 g of lithium tetraborate (6.1.3.1). Roast at (1 000 ± 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 (6.1.3.3) and dissolve while stirring and heating at 100 °C. Then transfer the solution to a 250 ml volumetric flask with

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water (4.4) and, after cooling to room temperature, add each 10 ml of the appropriate reagent solutions (8.1.2.6 and 8.1.2.7 for the AAS measurement) or 1 ml of scandium solution (8.2.2.2, internal standard solution for the ICP measurement) and make up to the mark with water (digestion solution (I) for AAS and (II) for ICP).

6.2 Extraction with hydrochloric acid

6.2.1 General

Extraction with hydrochloric acid is used to dissolve samples exclusively in order to determine calcium and magnesium.

6.2.2 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 hydroxides. After refiltering, the filtrate is transferred to a suitable volumetric flask.

6.2.3 Reagents

6.2.3.1 **Hydrochloric acid**, ρ = 1,16 g/ml.

6.2.3.2 **Hydrogen peroxide solution**, c (H_2O_2) = 30 % (m/m).

6.2.3.3 **Hydrogen peroxide solution**, diluted, 1 + 9.

6.2.3.4 **Ammonium hydroxide solution**, c (NH_3) = 25 % (m/m).

6.2.3.5 **Ammonium hydroxide solution**, diluted, 1 + 9.

6.2.3.6 **Ammonium chloride**, NH_4Cl.

6.2.4 Apparatus

Ordinary laboratory apparatus and the following:

6.2.4.1 **Hot plate.**

6.2.4.2 **Magnetic stirrer and magnetic rod.**

6.2.4.3 **pH-meter with glass electrode.**

6.2.5 Procedure

Weigh (1 ± 0,1) g of the sample to the nearest 1 mg, transfer it to a 250 ml beaker, moisten with 10 ml of water, then gradually add 30 ml of hydrochloric acid (6.2.3.1). 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 water.

Add about 4 g of ammonium chloride (6.2.3.6) and a few drops of hydrogen peroxide (6.2.3.3) to the solution, then dilute with about 150 ml of water and heat to boiling. During boiling, add ammonium hydroxide solution dropwise (6.2.3.4) to adjust the pH value to 6 to 7 and precipitate aluminium hydroxides and iron hydroxides and the 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 (6.2.3.5) and three times with water. After the solution

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has cooled to room temperature, make the solution up to the mark with water and shake the contents of the flask thoroughly (digestion solution III).

6.3 Microwave digestion with nitric acid

6.3.1 General

The purpose of this method of digestion is to release trace elements from the accompanying matrix and determine the content of the various elements.

6.3.2 Principle

The sample is weighed into a perfluoroalkoxyethylene (PFA) beaker and nitric acid is added. After the mixture has been placed in a microwave apparatus, digestion is carried out. Any undissolved component is removed by filtering and the supernatant is used to determine the elements concerned.

6.3.3 Reagents

6.3.3.1 **Nitric acid**, ρ = 1,40 g/ml.

6.3.4 Apparatus

Ordinary laboratory apparatus and the following:

6.3.4.1 **Microwave digestion apparatus**, equipped with PFA digestion vessels and a programmable pressure and temperature control unit. Performance ≥ 600 W.

6.3.4.2 50 ml **polypropylene pressure vessel** with screw lid.

6.3.4.3 **Filtering apparatus.**

6.3.5 Procedure

The mass of the test sample depends on the test method. For AAS, the recommended mass is (0,5 ± 0,05) g and for ICP-OES (3 ± 0,05) g, weighed to the nearest 0,1 mg. In the first case, moisten the sample with 4 ml of water and carefully add 3 ml of nitric acid (6.3.3.1), in the second case moisten with 5 ml of water and add 10 ml of nitric acid (6.3.3.1).

Seal the digestion vessel and place in the microwave apparatus. Before programming the apparatus, the heating conditions shall be determined, and a temperature and pressure versus time plot shall be prepared.

Subject the test sample to a pressure of 500 kPa to 700 kPa and to a temperature of 90 °C for 10 min during a heating-up phase. Then subject the test sample to a pressure of 1 000 kPa and a temperature between 120 °C and 150 °C for 15 min to 20 min. Allow the digestion vessel to cool to room temperature. Open the digestion vessel, introduce the solution into a 100 ml volumetric flask rinse and make up to the mark with water. Filter and transfer the solution (digestion solution IV) to a polypropylene vessel in order to avoid adsorption on the glass.

6.4 Pressurized digestion with nitric acid

6.4.1 General

The purpose of this method of digestion is to release trace elements from the accompanying matrix and determine the content of the various elements.

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6.4.2 Principle

The sample is weighed out into a polytetrafluorethylen (PTFE) beaker and nitric acid is added. After the mixture has been placed in a pressure shell, digestion is carried out at 130 °C. Any undissolved component is removed by filtering and the supernatant is used to determine the elements concerned.

6.4.3 Reagent

6.4.3.1 **Nitric acid**, ρ (HNO_3) = 1,40 g/ml.

6.4.4 Apparatus

Ordinary laboratory apparatus and the following:

6.4.4.1 **Pressure digestion system**, consisting of a PTFE digestion vessel, pressure shell and a heater.

6.4.4.2 50 ml **polypropylene pressure vessel** with screw lid.

6.4.4.3 **Filtering apparatus.**

6.4.5 Procedure

Weigh (0,5 ± 0,05) g to the nearest of 0,1 mg of the sample into the digestion vessel, moisten the sample with 2 ml of water and add carefully 5 ml of nitric acid (6.4.3.1). Seal the pressure vessel, place it in a cold drying oven and heat to 130 °C. After about 15 min, remove the pressure vessel from the drying oven and cool it to room temperature. Open the pressurized digestion system, introduce the solution into a 100 ml volumetric flask, rinse and make up to the mark with water. Filter and transfer the solution (digestion solution V) to a polypropylene vessel in order to avoid adsorption on the glass.

7 Conventional methods of determining major and minor constituents

7.1 Determination of free water

7.1.1 General

Free water means the moisture attached to the product, in the case of milk of lime, it refers to the water content of the suspension. The method does not apply for high-calcium quicklime, half-burnt dolomite or calcium magnesium dioxide. The determination of the water content of milk of lime is required to calculate the water-soluble content of the suspended hydrated lime (see 7.5).

7.1.2 Principle

When heating a sample of hydrated high-calcium lime, calcium carbonate, calcium magnesium carbonate or calcium magnesium dihydroxide oxide to (105 ± 5) °C, or for milk of lime to (150 ± 5) °C the free water is driven off. The loss of mass at this temperature is referred to as free water.

7.1.3 Apparatus

Ordinary laboratory apparatus and the following:

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

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

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7.1.3.3 **Analytical balance** accurate to 0,1 mg.

7.1.3.4 **Desiccator** containing drying agent.

7.1.3.5 **Glazed porcelain or platinum crucible.**

7.1.3.6 **Pipette.**

7.1.4 Procedure

7.1.4.1 Hydrated high-calcium lime, calcium carbonate, calcium magnesium carbonate and calcium magnesium dihydroxide oxide

Weigh (5 ± 0,1) g to a nearest 1 mg of the sample in the as-delivered state in a pre-weighed crucible. Dry the sample until constant mass using the oven (7.1.3.1) or the balance (7.1.3.2). 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.

7.1.4.2 Milk of lime

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. Dry the sample to constant mass using the oven (7.1.3.1) or the balance (7.1.3.2). 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.

7.1.5 Expression of results

The free water content expressed as (H_2O) in mass fraction in % is given by the following formula:

$$\omega\left(H_2O\right) = \frac{\left(m_1 - m_2\right)}{m_1} \times 100 \qquad (3)$$

where

m_1 is the mass of the test portion before heating, in grams;

m_2 is the mass of the test portion after heating, in grams.

7.2 Loss on ignition at 500 °C

7.2.1 General

The method is used to determine the loss on ignition after drying as specified in 7.1.

7.2.2 Principle

The loss on ignition at 500 °C is basically due the water of hydration.

7.2.3 Apparatus

Ordinary laboratory apparatus and the following:

7.2.3.1 **Electric furnace** capable of being maintained at (500 ± 25) °C, with a thermoelectric temperature indicator.

7.2.3.2 **Analytical balance** accurate to 0,1 mg.

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7.2.3.3 **Desiccator** containing phosphorous pentoxide, P_2O_5.

7.2.3.4 **Glazed porcelain or platinum crucible.**

7.2.4 Procedure

Weigh (5 ± 0,1) g to the nearest of 1 mg of the sample (m_1) in a pre-weighed crucible. Heat the sample in the furnace at (500 ± 25) °C for 2 h and then cool it in a desiccator. Cover the crucible after removing it from the furnace to prevent carbon dioxide and water vapour in the atmosphere from being absorbed. Cool in the desiccator and weigh (m_2).

7.2.5 Expression of results

The loss on ignition at 500 °C expressed as LoI in mass fraction in % is given by the following formula:

$$\left(LoI\right) = \frac{\left(m_1 - m_2\right)}{m_1} \times 100 \tag{4}$$

where

m_1 is the mass of the sample before ignition at (500 ± 25) °C, in grams;

m_2 is the mass of the sample after ignition at (500 ± 25) °C, in grams.

7.3 Determination of carbon dioxide

7.3.1 General

The method is used to determine carbon dioxide in high-calcium lime as specified in EN 12518, in half-burnt dolomite as specified in EN 1017, in magnesium oxide as specified in EN 16004 and in dolomitic lime as specified in EN 16409.

7.3.2 Principle

The carbon dioxide content of the materials concerned is determined from the loss on ignition at 1 050 °C. The loss on ignition at 500 °C is basically due to free water and the water of hydration. The difference in mass between the loss on ignition at (1 050 ± 25) °C and the loss on ignition at (500 ± 25) °C is equal to the carbon dioxide content of the product.

7.3.3 Apparatus

Ordinary laboratory apparatus and the following:

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

7.3.3.2 **Analytical balance** accurate to 0,1 mg.

7.3.3.3 **Desiccator** containing phosphorous pentoxide, P_2O_5.

7.3.3.4 **Glazed porcelain or platinum crucible.**

7.3.4 Procedure

After determining the mass (m_2) from the determination of the loss on ignition at (500 ± 25) °C (7.2.5), heat the sample at (1 050 ± 25) °C for 3 h. Cover the crucible after removing it from the furnace to prevent

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carbon dioxide and water vapour in the atmosphere from being absorbed. Cool in the desiccator and reweigh (m_3).

7.3.5 Expression of results

The carbon dioxide content expressed as CO_2 in mass fraction in % is given by the following formula:

$$c\left(CO_2\right) = \frac{\left(m_2 - m_3\right)}{m_1} \times 100 \qquad (5)$$

where

m_1 is the mass of the sample before ignition at (500 ± 25) °C (7.2.5), in grams;

m_2 is the mass of the sample before ignition at (1 050 ± 25) °C, in grams;

m_3 is the mass of the sample after ignition at (1 050 ± 25) °C, in grams.

7.4 Determination of residue insoluble in hydrochloric acid

7.4.1 General

The method is used to determine the residue which is insoluble in hydrochloric acid.

7.4.2 Principle

After the sample has been dissolved in hydrochloric acid and filtered, the filtration residue is washed, dried and incinerated to constant mass to determine the insoluble component gravimetrically.

7.4.3 Reagents

7.4.3.1 **Hydrochloric acid**, ρ = 1,16 g/ml.

7.4.3.2 **Hydrochloric acid**, diluted, 1 + 1.

7.4.3.3 **Hydrochloric acid**, diluted, 1 + 3.

7.4.4 Apparatus

Ordinary laboratory apparatus and the following:

7.4.4.1 **Magnetic stirrer with hot plate.**

7.4.4.2 **Glazed porcelain or platinum crucible.**

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

7.4.5 Procedure

Weigh (1 ± 0,1) g to the nearest of 1 mg of the sample into a 250 ml beaker. Suspend the sample in 5 ml to 10 ml of water and dissolve it by adding 25 ml of hydrochloric acid (7.4.3.2). After boiling this solution for 2 min, dilute it with 50 ml of water as soon as the dissolution is complete and immediately filter it through a filter (particle retention size 2,5 µm, ash < 0,01 %) into a 250 ml beaker. Use a rubber-tipped glass rod to ensure that all the insoluble residue is transferred to the filter. Wash the filtration residue twice with 10 ml of hot dilute hydrochloric acid (7.4.3.3) and then several times with small portions of hot water.

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Reduce the washed filter to ash in a roasted and pre-weighed platinum crucible. After incinerating to constant mass at (1 050 ± 25) °C, cool in a desiccator and weigh. An incineration time of 15 min will generally be sufficient to achieve constant mass.

7.4.6 Expression of results

The content of residue insoluble in hydrochloric acid expressed as (HCl_{insol}) in mass fraction in % is given by the following formula:

$$\left(HCl_{insol}\right) = \frac{m_1 \times 100}{m} \quad (6)$$

where

m_1 is the incineration residue, in grams;

m is the mass of the sample, in grams.

7.5 Determination of content of water soluble calcium oxide or calcium hydroxide (reference method)

7.5.1 General

The method serves to determine the water-soluble lime content (calcium oxide and calcium hydroxide) in high-calcium lime products as specified in EN 12518. It is also used to determine the water-soluble calcium hydroxide in milk of lime.

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.

7.5.2 Principle

The amount of water-soluble lime which can be titrated with hydrochloric acid after digesting the lime sample in water is determined. To avoid agglomeration of calcium oxide on slaking which can lead to incomplete suspension of a high-calcium lime sample such limes shall be heated. The indicator used and the low rate of titration ensure that the titration proceeds in the alkaline range.

7.5.3 Reagents

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

7.5.3.2 **Thymolphthalein indicator solution**: dissolve 0,5 g of thymolphthalein in 50 ml of ethanol and dilute to 100 ml with water.

7.5.3.3 **Ethanol**, ρ = 0,79 g/ml.

7.5.3.4 **Water of a specific conductivity < 0,5 mS/m (at 25 °C).**

7.5.4 Apparatus

Ordinary laboratory apparatus and the following:

7.5.4.1 **Analytical balance**, accurate to 0,1 mg.

7.5.4.2 **pH-meter** with glass electrode.

7.5.4.3 **Heatable magnetic stirrer** with magnetic rod.

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