BRITISH STANDARD

BS EN 14016-2:2004

Binders for magnesite screeds — Caustic magnesia and magnesium chloride —

Part 2: Test methods

The European Standard EN 14016-2:2004 has the status of a
British Standard

ICS 91.100.50

British Standards

National foreword

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The UK participation in its preparation was entrusted by Technical Committee B/507, Paving units, kerbs, screeds and in situ floorings, to Subcommittee B/507/6, Screeds and in situ floorings, which has the responsibility to:

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- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 14016-2:2004) has been prepared by Technical Committee CEN/TC 303 “Floor screeds and in-situ floorings in buildings”, the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by July 2004, and conflicting national standards shall be withdrawn at the latest by July 2004.

This document belongs to a series of standards on screed materials and floor screeds in buildings.

No existing European Standard is superseded by this standard.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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard applies to caustic magnesia and magnesium chloride used for the manufacture of magnesite screed material and magnesite floor screeds as specified in EN 13813 and describes test methods by means of which the fulfilment of the requirements in EN 14016-1 can be checked.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

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

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

EN 13813, *Screed materials and floor screeds - Screed material - Properties and requirements.*

EN 13892-1, *Methods of test for screed materials - Part 1: Sampling, making and curing specimens for test.*

EN 13892-2, *Methods of test for screed materials - Part 2: Determination of flexural and compressive strength.*

EN 14016-1, *Binders for magnesite screeds - Caustic magnesia and magnesiumchloride - Part 1: Definitions, requirements.*

ISO 3310-1, *Test sieves - Technical requirements and testing - Part 1: Test sieves of metal wire cloth.*

3 Magnesium chloride test

3.1 Sampling

3.1.1 General

For the chemical analysis, take a representative sample of the following minimum quantities from the batch to be tested.

- 2 000 g from an aqueous solution or

- 1 000 g from solid salt.

3.1.2 Aqueous solution

For sampling, thoroughly stir the whole quantity in the vessel. If salts have crystallised out through storage at low temperatures, bring them into solution again by gentle warming and stirring. If the solution has precipitated salts, do not take the sample just from the clear solution or from parts of the solution that contain solid salt.

If the vessel has been partially emptied before sampling and contains precipitated salt, it is not possible to obtain a representative sample.

3.1.3 Solid salt

Samples shall only be taken from the inside of the blocks or of the salt mass (melted, ground in flakes or granules, crystallised). In order to obtain correct representative samples, take several individual samples depending on quantity and type of packaging and combine them to make a cumulative sample. As magnesium chloride attracts a considerable amount of moisture from the air, sampling, size reduction and mixing of the individual samples shall be carried out as rapidly as possible. Store the final samples in tightly stoppered bottles.

3.2 Chemical analysis

3.2.1 General

This subclause describes reference methods for determining the requirements specified EN 14016-1. If other test methods are used, it shall be demonstrated that the results obtained with them are equivalent to the results of the reference methods. In cases of dispute, the reference method shall be used.

Only analytically pure reagents shall be used. Unless otherwise stated, % means percentage by mass. The term “water” shall be understood to mean distilled water or water of the same degree of purity. “ ” always designates the density of a liquid at 20 °C. Dilutions are given as a volume sum, e.g. dilute hydrochloric acid 1 + 19 means that 1 part per volume of concentrated hydrochloric acid shall be mixed with 19 parts per volume of water.

3.2.2 Determination of water-insoluble constituents

3.2.2.1 Apparatus

¾ Balance accurate to 0,000 1 g;

¾ Platinum or porcelain crucible;

¾ Electric oven, adjustable to (1 000 25) °C;

¾ Desiccator with drying agent.

3.2.2.2 Reagents

¾ Silver nitrate, $AgNO_3$;

¾ Nitric acid, concentrated, HNO_3 (= 1,40 g/cm^3 to 1,42 g/cm^3);

¾ Silver nitrate solution.

Dissolve 0,5 g of silver nitrate in 100 ml of water and add a few drops of nitric acid.

3.2.2.3 Procedure and evaluation

Add 200 ml of water to 100 g of aqueous magnesium chloride solution or to 50 g of solid magnesium chloride, weighed to the nearest 0,000 1 g, in a 400 ml beaker (tall form). After 15 min of intensive stirring, filter through a filter paper (medium fast: 140 s filtration time using the Herzberg test system) into a 500 ml volumetric flask. Wash the filtration residue with hot water free from chloride (check with silver nitrate solution). The filtrate is used to determine the content of the water-soluble constituents and the pH value. Dry the filter paper and the contents in a red-hot crucible of known mass and reduce to ash. Then heat the crucible for 15 min at (1 000 25) °C. Allow the crucible and its contents to cool down to room temperature in the desiccator and weigh the residue to the nearest 0,000 1 g. Calculate the water-insoluble constituents as a percentage by mass WC using the following equation:

$$WC = \frac{m_A \quad 100}{m_E} \text{ in \%}$$

where

m_A is the final mass in g;

m_E is the initial mass of aqueous magnesium chloride solution or solid magnesium chloride in g.

Make up the filtrate in the 500 ml volumetric flask containing the water-soluble constituents with water to the mark.

3.2.3 Sulfate content SO_4^{2-}

3.2.3.1 Apparatus

¾ Platinum crucible;

¾ Electric oven, adjustable to (800   25) °C;

¾ Desiccator with drying agent.

3.2.3.2 Reagents

¾ Hydrochloric acid, concentrated, HCl (= 1,18 g/cm^3 to 1,19 g/cm^3;

¾ Hydrochloric acid, dilute 1 + 11;

¾ Barium chloride – 2 – hydrate $BaCl_2 \times 2H_2O$;

¾ Barium chloride solution: Dissolve 120 g of barium chloride in water to make up 1 000 ml;

¾ Silver nitrate $AgNO_3$;

¾ Nitric acid HNO_3;

¾ Silver nitrate solution: Dissolve 0,5 g of silver nitrate in 100 ml of water and add a few drops of nitric acid.

3.2.3.3 Procedure and evaluation

If aqueous magnesium chloride solution is the material to be tested for determination of the sulfate content, pipette 25 ml of the solution prepared as described in 3.2.2 into a 400 ml beaker. Use 50 ml of the solution as specified in 3.2.2 for the determination if the material to be tested is solid magnesium chloride. Dilute the solution with water to approximately 250 ml and adjust the pH value to 2,0 to 2,5 with dilute hydrochloric acid. Depending on the barium sulfate content to be expected, add 3 ml to 5 ml of hot barium chloride solution in drops (1 ml of barium chloride solution to 100 mg of barium sulfate) to the boiling solution whilst stirring vigorously. Boil for a further 15 min to ensure good precipitation. Allow to stand overnight and filter the precipitate through a filter paper (slow: 1 500 s filtration time according to the Herzberg test system) and wash chloride free with boiling water (check with silver nitrate solution). Transfer the filter paper and the contents to a red-hot crucible of known mass and reduce to ash at full air flow. Then, heat at (800   25) °C to constant mass (0,000 5 g). Allow the crucible and its contents to cool down to room temperature in the desiccator and then finally weigh the residue to the nearest 0,000 1 g. If the material to be tested is aqueous magnesium chloride solution, calculate the percentage by mass of sulfate content using the following equation:

$$\text{Sulfate content } SO^{2}_{4total} = \frac{8{,}232 \quad m_{A1} x100}{m_E} \text{ in \%}$$

If the material to be tested is solid magnesium chloride, calculate the percentage by mass of sulfate content using the following equation:

$$\text{Sulfate content } SO^{2}_{4total} = \frac{4{,}116 \quad m_{A2} \quad 100}{m_E} \text{ in \%}$$

where

m_{A1} is the final mass of barium sulfate in the aqueous magnesium chloride solution as the material to be tested in grams;

m_{A2} is the final mass of barium sulfate in the solid magnesium chloride as the material to be tested in grams;

m_E is the initial mass of aqueous magnesium chloride solution or solid magnesium chloride specified in 3.2.2 in grams.

3.2.4 Calcium content Ca^{2+}

3.2.4.1 Reagents

¾ Sodium hydroxide NaOH;

¾ Sodium hydroxide solution approximately 1 mol/l: dissolve 40 g of sodium hydroxide in water and make up to 1 000 ml of solution; keep the solution in a polyethylene bottle;

¾ Murexide: purpuric acid, ammonium salt;

¾ Naphtol green B: 1-nitroso-2-naphthol-6-sulfonic acid, iron-sodium salt;

¾ Sodium chloride NaCl;

¾ Murexide mixed indicator: Grind 0,1 g of murexide and 0,3 g of naphthol green B with 30 g of sodium chloride;

¾ EDTA: ethylenediamine tetraacetic acid, disodium salt, dihydrate;

¾ EDTA solution 0,01 mol/l (a commercially available ready-to-use solution may be used).

3.2.4.2 Procedure and evaluation

To analyse aqueous magnesium chloride solution and solid magnesium chloride, pipette 50 ml and 100 ml respectively of the solution prepared as described in 3.2.2 into a 500 ml volumetric flask and make up to the mark with water. Pipette 100 ml of this solution into a 250 ml beaker (tall form). Adjust the pH value of the solution to at least 13 (check) using sodium hydroxide solution. After adding approximately 0,1 g of murexide mixed indicator, titrate the solution on a white base against a white background (approximately 10 cm away) with 0,01 mol/l of EDTA solution stirring continuously (e.g. using a magnetic stirrer). The end point is indicated by a change in colour from red to violet.

Determine the blank value in parallel.

Other common indicators may also be used, e.g. calconcarboxylic acid (colour change: wine red to blue). Traces of iron, manganese, zinc and copper interfere with the titration. Depending on the type and quantity of the interfering elements, add 10 drops of 10 % sodium sulfide solution or triethanolamine solution 1 + 4 to the solution before titration.

If the material to be tested is magnesium chloride solution, calculate the calcium content as the percentage by mass of Ca^{2+} using the following equation:

$$\text{Calcium content } Ca^{2+}_{total} = \frac{0{,}02004 \quad (V_1 \quad B) \quad 100}{m_E} \text{ in \%}$$

If the material to be tested is solid magnesium chloride, calculate the calcium content as the percentage by mass of Ca^{2+} using the following equation:

$$\text{Calcium content } Ca^{2+}_{total} = \frac{0{,}01002 \quad (V_2 \quad B) \quad 100}{m_E} \text{ in \%}$$

where

V_1 is the volume of 0,01 mol/l EDTA solution used when determining the aqueous magnesium chloride solution, in millilitres;

V_2 is the volume of 0,01 mol/l EDTA solution used when determining the solid magnesium chloride, in millilitres;

B is the blank value in millilitres;

m_E is the initial mass of aqueous magnesium chloride solution or solid magnesium chloride specified in 3.2.2 in grams.

3.2.5 Magnesium content Mg^{2+}

3.2.5.1 Reagents

¾ Ammonium chloride NH_4Cl;

¾ Ammonia solution (= 0,91 g/cm^3) NH_3;

¾ Ammonium chloride buffer: Dissolve 70 g of ammonium chloride and 570 ml of ammonia solution with water and make up to 1 000 ml;

¾ Eriochrome black T: 2-hydroxy-1-(1’-hydroxy-2-azonaphthyl)-6-nitronaphthalene-4-sulfonic acid, sodiumsalt;

¾ Eriochrome black T indicator: grind1 g of eriochrome black T with 100 g of sodium chloride;

¾ EDTA: ethylenediamine tetraacetic acid, disodium salt, dihydrate;

¾ EDTA solution 0,1 mol/l (a commercially available ready-to-use solution may be used);

¾ Sodium sulfide Na_2S ×nH_2O;

¾ Triethanolmine $N(CH_2CH_2OH)_3$ (= 1,12 g/cm^3);

¾ Triethanolmine solution 1 + 4.

3.2.5.2 Procedure and evaluation

Pipette 10 ml of the solution prepared as specified in 3.2.4 (500 ml volumetric flask) into a 250 ml beaker (tall form) and dilute to approximately 100 ml. Adjust the pH value of the solution with 10 ml of ammonium chloride buffer. After adding approximately 0,1 g of eriochrome black T indicator, titrate the solution on a white base against a white background (approximately 10 cm away) with EDTA solution, 0,1 mol/l, stirring continuously (e.g. using a magnetic stirrer). The end point is indicated by a change in colour from red to blue.

Determine the blank value in parallel.

Other common indicators may also be used. Traces of iron, manganese, zinc and copper interfere with the titration. Depending on the type and quantity of the interfering elements, add 10 drops of 10 % sodium sulfide solution or triethanolamine solution 1 + 4 to the solution before titration.

Other common indicators may also be used.

The percentage of magnesium determined Mg $_{total}$ comprises the magnesium bound to chloride and sulfate. The result also contains the calcium content of the corresponding water-soluble salts also determined during titration.

If the material to be tested is magnesium chloride solution, calculate the total magnesium content as the percentage by mass of Mg $_{total}$ using the following equation:

$$\text{Magnesium content Mg}_{\text{total}} = \frac{1{,}2152 \quad (V_3 \quad B) \quad 100}{m_E} \text{ in \%}$$

If the material to be tested is solid magnesium chloride, calculate the total magnesium content as the percentage by mass of Mg $_{total}$ using the following equation:

$$\text{Magnesium content Mg}_{\text{total}} = \frac{0{,}6076 \quad (V_4 \quad B) \quad 100}{m_E} \text{ in \%}$$

where

V_3 is the volume of 0,1 mol/l EDTA solution used when determining the aqueous magnesium chloride solution, in ml;

V_4 is the volume of 0,1 mol/l EDTA solution used when determining the solid magnesium chloride solution, in ml;

B is the blank value in ml;

m_E is the initial mass of aqueous magnesium chloride solution or solid magnesium chloride specified in 3.2.2 in g.

3.2.6 Magnesium sulfate $MgSO_4$, calcium sulfate $CaSO_4$, calcium chloride $CaCl_2$ and magnesium chloride $MgCl_2$

If the total sulfate content is greater than the sulfate content bonded as calcium sulfate i.e.

$$SO_{4\,total}^{\;2} > \frac{3{,}3967 \quad Ca^{2+}{}_{total}}{1{,}4172} \text{ in \%}$$

then sulfate is present as magnesium sulfate and calcium sulfate.

$$\text{Sulfate content} \quad SO^{2}_{4MgSO_4} = SO^{2}_{4MgSO_{total}} \quad \frac{3{,}3967 \quad Ca^{2+}_{total}}{1{,}4172} \text{ in \%}$$

$$\text{Magnesium sulfate } MgSO_4 = 1{,}253 \times SO^{2}_{4MgSO_4} \text{ in \%}$$

$$\text{Calcium sulfate } CaSO_4 = 3{,}3967 \times Ca^{2+}_{total} \text{ in \%}$$

Calculate the sulfate-bonded magnesium content as a percentage by mass of MG_{MgSO_4} using the following equation:

$$\text{Sulfate-bonded magnesium} \quad Mg_{MgSO_4} = 0{,}2019 \quad MgSO_4 \text{, in \%}$$

Calculate the magnesium chloride content from the total magnesium content and the sulfate-bonded magnesium content as the difference between the two percentages by mass in accordance with the following equation:

Magnesium chloride $MgCl_2$ = $3{,}9173 \times (Mg_{total} \quad Mg_{MgSO_4})$ in %

If the total sulfate content is less than the sulfate content bonded as calcium sulfate, i.e.

$$SO^{2}_{4_{total}} < \frac{3{,}397 \quad Ca^{2+}_{total}}{1{,}4172}$$

there is no magnesium sulfate and the calcium is present in the form of calcium sulfate and calcium chloride

Calcium sulfate $CaSO_4$ = $1{,}4172 \times SO^{2}_{4_{total}}$ in %;

Calcium chloride $CaCl_2$ = $(Ca^{2+}_{total} \quad 0{,}2944 \times CaSO_4) \times 2{,}769$ in %;

Magnesium chloride $MgCl_2$ = $3{,}9173 \times (Mg_{total} \quad 0{,}6064 \times Ca^{2+}_{total})$ in %.

3.2.7 Alkali chlorides (sodium chloride and potassium chloride)

3.2.7.1 Apparatus

Atomic emission spectrometry shall be used as the reference method for determining sodium chloride and potassium chloride. It is possible to use atomic absorption spectrometry (AAS) and inductively coupled plasma (ICP) as an alternative method.

For the atomic absorption spectrometry, a flame photometer shall be used by means of which it is possible to measure the intensities of the sodium line at 589,3 nm and of the potassium line at 768,2 nm.

3.2.7.2 Reagents

¾ Sodium chloride NaCl, dried at 105 °C to constant mass[1] ;

¾ Sodium chloride stock solution: Dissolve 1,0 g of sodium chloride in water and make up to 1 000 ml; keep the solution in a polyethylene bottle;

¾ Potassium chloride KCl, dried at 105 °C to constant mass[1] ;

¾ Potassium chloride stock solution: Dissolve 1,0 g of potassium chloride in water and make up to 1 000 ml; keep the solution in a polyethylene bottle;

¾ Calcium sulfate dihydrate $CaSO_4 \times 2H_2O$, precipitated;

¾ Calcium sulfate dihydrate stock solution: Dissolve 0,2530 g of calcium sulfate dihydrate in water and make up to 1 000 ml; keep the solution in a polyethylene bottle;

¾ Magnesium chloride hexahydrate $MgCl_2 \times 6H_2O$.

3.2.7.3 Determination of the calibration curves for sodium chloride

Introduce 5 ml of the potassium chloride stock solution, 5 ml of the calcium sulfate stock solution and 1 g of magnesium chloride into each of five 250 ml volumetric flasks. Then, from a calibrated burette, add the following quantities of the sodium chloride stock solution to the individual flasks:

---

1) Constant mass is reached if two successive weighings 24 h apart, differ by less than 0,1 %.

Volumetric flask	1	2	3	4	5
NaCl stock solution in ml	0	5	10	15	20

Then fill the volumetric flasks to the mark with water. For an initial mass of 100 g of $MgCl_2$ solution or 50 g of solid $MgCl_2$ and after taking 20 ml of the solution described in 3.2.2 and dilution as described in 3.2.7.5, the measured values of volumetric flasks 1 to 5 correspond to the following percentages by mass of sodium chloride:

Volumetric flask	1	2	3	4	5
NaCl in %	0,00	0,50	1,00	1,50	2,00

If a photometer of sufficient consistency is used, it is only occasionally necessary to record the calibration curve in its entirety. However, in each analysis, check the measured values of volumetric flasks 1 to 5. Sufficient consistency is given if, after having reset the zero point with water, the same calibration curve is obtained using the same calibration solutions.

3.2.7.4 Determination of the calibration curves for potassium chloride

Introduce 5 ml of the sodium chloride stock solution, 5 ml of the calcium sulfate stock solution and 1 g of magnesium chloride into each of five 250 ml volumetric flasks. Then, from a calibrated burette, add the following quantities of the potassium chloride stock solutions to the individual flasks:

Volumetric flask	1	2	3	4	5
KCl stock solution in ml	0,00	0,50	1,00	1,50	2,00

Then fill the volumetric flasks to the mark with water. For an initial mass of 100 g of $MgCl_2$ solution or 50,0000 g of solid $MgCl_2$ and after taking 20 ml of the solution described in 3.2.2 and dilution as described in 3.2.7.6, the measured values of volumetric flasks 1 to 5 correspond to the following percentages by mass of potassium chloride:

Volumetric flask	1	2	3	4	5
KCl in %	0,00	0,50	1,00	1,50	2,00

If a photometer of sufficient consistency is used, it is only occasionally necessary to record the calibration curve in its entirety. However, in each analysis, check the measured values of flasks 1 to 5. Sufficient consistency is given if, after having reset the zero point with water, the same calibration curve is obtained using the same calibration solutions.

3.2.7.5 Procedure and evaluation for sodium chloride

To determine the sodium chloride content, if the material to be tested is aqueous magnesium chloride solution, use a solution obtained by making up 20 ml of the solution described in 3.2.2 to 1 000 ml. To determine the sodium chloride content if the material to be tested is solid magnesium chloride, use a solution obtained by making up 20 ml of the described solution to 500 ml. Measure each solution in the flame photometer. The reading gives the sodium chloride content on the basis of the calibration curve.

3.2.7.6 Procedure and evaluation for potassium chloride

Carry out the flame photometric determination on the solution prepared as described above. The reading gives the potassium chloride content on the basis of the calibration curve.

3.2.8 Determination of the pH value

Determine the pH value of the solution described in 3.2.2 containing the water-soluble constituents electrometrically or using indicators, e.g. in the form of indicator paper whose transition intervals are in the pH range between 6,0 to 10,0.

4 Caustic magnesia test

4.1 Sampling and preparation of the sample

Take a representative sample of approximately 5 kg from each batch to be tested, place it immediately in a sealable air-tight vessel and mark it such that it cannot be mistaken.

Before testing, pass the caustic magnesia through a test sieve with a mesh size of 1,25 mm as specified in ISO 3310. If the caustic magnesia contains solid residue, this shall be recorded.

4.2 Chemical analysis

4.2.1 General

The principles described in 3.2 apply. Take a specimen of approximately 200 g from the representative sample as described in 4.1.

4.2.2 Hydrochloric acid insoluble residue

4.2.2.1 Apparatus

¾ balance accurate to 0,000 1 g;

¾ heating bath, adjustable to (105 5) °C;

¾ acid-resistantheating cabinet, adjustable to (120 5) °C;

¾ electric oven, adjustable to (1 000 25) °C;

¾ platinum or porcelain crucible;

¾ desiccator with drying agent.

4.2.2.2 Reagents

¾ hydrochloric acid, concentrated HCl (= 1,18 g/cm^3 to 1,19 g/cm^3);

¾ hydrochloric acid, diluted 1 + 1;

¾ hydrochloric acid, diluted 1 + 19;

¾ nitric acid, concentrated HNO_3 (= 1,40 g/cm^3 to 1,42 g/cm^3).

4.2.2.3 Procedure and evaluation

Weigh out 10 g to the nearest 0,000 1 g of the sample dried to a constant mass (0,000 5 g) at (105 5) °C, place it in a 500 ml beaker (tall form) and mix with approximately 100 ml of water to form a suspension. After adding a few drops of concentrated nitric acid, add 120 ml of dilute hydrochloric acid 1 + 1 and cover the beaker with a watch glass. Then boil the mixture for 5 min, evaporate it on a heating bath at (105 5) °C and heat the residue obtained for approximately 1 h in a heating cabinet at (120 5) °C. After cooling down to room temperature, add approximately 120 ml of the dilute hydrochloric acid 1 + 19 to the residue and heat it to just below boiling point.

Then, filter through a filter paper (medium fast: 140 s filtration time according to the Herzberg test system) into a 400 ml beaker, wash the precipitate firstly 5 times with hot dilute hydrochloric acid 1 + 19 and then twice with hot water. Use the filtrate to determine the sesquioxide content R_2O_3 (Al_2O_3 + Fe_2O_3). Transfer the filter and contents to a red hot crucible of known mass and carefully dry and reduce to ash. Then heat for 1 h at (1 000   25) °C. Allow the crucible and contents to cool down in the desiccator to room temperature and weigh the solid residue to the nearest 0,000 1 g.

Hydrochloric-acid-insoluble residue = $\frac{m_A \quad 100}{m_E}$ in %

Where

m_A is the final mass in g;

m_E is the initial mass in g.

4.2.3 Sesquioxides $R_2O_3(Al_2O_3 + Fe_2O_3)$

4.2.3.1 Apparatus

¾ electric oven adjustable to (1 000   25) °C;

¾ desiccator with drying agent;

¾ platinum or porcelain crucible.

4.2.3.2 Reagents

¾ ammonium chloride NH_4Cl;

¾ ammonia solution (= 0,91 g/cm^3) NH_3;

¾ ammonia-ammonium chloride solution: Dissolve 20 g of ammonium chloride in water in a 1 000 ml volumetric flask, add ammonia solution until the methyl red changes to yellow and make up to the mark with water;

¾ hydrochloric acid, concentrated HCl (= 1,18 g/cm^3 to 1,19 g/cm^3);

¾ hydrochloric acid, diluted 1+4;

¾ nitric acid, concentrated HNO_3 (= 1,40 g/cm^3 to 1,42 g/cm^3).

4.2.3.3 Procedure and evaluation

Add approximately 10 g of ammonium chloride and a few drops of concentrated nitric acid to the filtrate described in 4.2.2.3 and heat to boiling. During the boiling process, add drops of ammonia solution until the pH value is   6. Keep the solution boiling for 3 min (take care to ensure the solution remains ammonia alkaline), let the precipitate settle, filter hot through a filter paper (medium fast, 140 s filtering time in accordance with the Herzberg test system) into a 1 000 ml volumetric flask and wash twice with ammonia-ammonium chloride solution (wash off the filter paper from the top). Separate the precipitate from the filter paper with hot dilute hydrochloric acid 1 + 4, then with approximately 100 ml of water and return to the same beaker. Repeat the precipitation as described above adding ammonium chloride and a few drops of concentrated nitric acid. Then filter through the same filter paper (medium fast, 140 s filtering time in accordance with the Herzberg test system) to the main filtrate in the 1 000 ml volumetric flask and wash out the precipitate 5 times with ammonia-ammonium chloride solution. The combined filtrates in the volumetric flask are used to determine the calcium oxide CaO and the magnesium oxide MgO contents. Fill the volumetric flask up to the mark (at 20 °C) with water. Transfer the filter and precipitate to a red hot crucible of known mass, dry it carefully, reduce to ash and heat for 1 h in air at (1 000   25) °C. Allow the crucible and contents to cool down in the desiccator to room temperature and weigh the solid residue to the nearest 0,000 1 g.

Sesquioxides R_2O_3 (Al_2O_3 + Fe_2O_3) = $\frac{m_A \quad 100}{m_E}$ in %

where

m_A is the final mass in g;

m_E is the initial mass in g.

4.2.4 Calcium oxide CaO

4.2.4.1 Reagents

¾ sodium hydroxide NaOH;

¾ sodium hydroxide solution approximately 4 mol/l: Dissolve 160 g of sodium hydroxide in water and make up to 1 000 ml of solution; keep the solution in a polyethylene bottle;

¾ murexide: purpuric acid, ammonium salt;

¾ naphthol green B: 1-nitroso-2-naphthol-6-sulfonic acid, iron-sodium salt;

¾ sodium chloride NaCl;

¾ murexide mixed indicator: grind 0,1 g of murexide and 0,3 g of naphthol green B with 30 g of sodium chloride;

¾ EDTA: ethylenediamine tetraacetic acid, disodium salt, dihydrate;

¾ EDTA solution, 0,01 mol/l: a commercially available ready-to-use solution may be used.

4.2.4.2 Procedure and evaluation

Pipette 20 ml of the filtrate as described in 4.2.3.3 from a 1 000 ml volumetric flask into a 500 ml beaker and dilute to approximately 200 ml with water. Adjust the pH value of the solution to at least 13 (check) using sodium hydroxide solution. The pH value shall not fall below this value during titration. After adding approximately 0,1 g of murexide mixed indicator, titrate the solution on a white base against a white background (approximately 10 cm away) with 0,01 mol/l EDTA solution stirring continuously (e.g. using a magnetic stirrer). The end point is indicated by a change in colour from violet to blue.

Determine the blank value in parallel.

Other common indicators may also be used, e.g. calconcarboxylic acid (colour change: wine red to blue). Traces of iron, manganese, zinc and copper interfere with the titration. Depending on the type and quantity of the interfering elements, add 10 drops of 10 % sodium sulfide solution or triethanolamine solution 1 + 4 to the solution before titration.

Calcium oxide CaO = $\frac{0{,}02804 \quad (V_1 \quad B_1) \quad 100}{m_E}$ in %

where

V_1 is the volume of 0,01 mol/l EDTA solution consumed in ml;

B_1 is the blank value in ml;

m_E is the initial mass in accordance with 4.2.2.3 in g.

4.2.5 Determination of the free CaO content

4.2.5.1 Reagents

- ethylene glycol $C_2H_6O_2$;
- ethanol C_2H_5OH;
- ammonia solution (= 0,91 g/cm^3), NH_3;
- EDTA solution, 0,01 mol/l.

4.2.5.2 Procedure and evaluation

Weigh out 1 g to the nearest 0,000 1 g of the caustic magnesia dried to a constant mass and place it in a 100 ml round bottom flask with 50 ml of ethylene glycol. Seal the flask with a rubber stopper and place in a water bath at 70 °C for 30 min. During this time, continuously stir the sample (e.g. magnetic stirrer). Then, filter the solution through a filter paper wetted with ethylene glycol (medium fast; 140 s filtration time in accordance with the Herzberg test system) by means of a Büchner funnel into a filter flask. Rinse the flask 3 times with 10 ml of ethanol. To precipitate the magnesium add ammonia solution to the filtrate so that the pH value of the solution reaches at least 12. The pH value shall not fall below this value during titration. After adding approximately 0,1 g of murexide mixed indicator, titrate the solution on a white base against a white background (approximately 10 cm away) with 0,01 mol/l EDTA solution stirring continuously (e.g. using a magnetic stirrer). The end point is indicated by a change in colour from red to violet.

Determine the blank value in parallel.

Other common indicators, e.g. calconcarboxylic acid (colour change: wine red to blue) may also be used.

$$\text{Calcium oxide CaO}_{\text{free}} = \frac{0{,}0005608 \quad (V_1 \quad B_1) \quad 100}{m_E} \text{ in \%}$$

where

V_1 is the volume of 0,01 mol/l EDTA solution consumed in ml;

B_1 is the blank value in ml;

m_E is the initial mass in accordance with 4.2.2.3 in g.

4.2.6 Magnesium oxide MgO

4.2.6.1 Reagents

- ammonium chloride NH_4Cl;
- ammonia solution NH_3 (= 0,91 g/cm^3);
- ammonium chloride buffer: dissolve 70 g of ammonium chloride and 570 ml of ammonia solution with water and make up to 1 000 ml;
- eriochrome black T: 2-hydroxy-1-(1'-hydroxy-2-azonaphthyl)-6-nitronaphthalene-4-sulfonic acid, sodium salt;
- eriochrome black T indicator: grind 1 g of eriochrome black T with 100 g of sodium chloride;
- EDTA solution 0,1 mol/l;
- sodium sulfide $Na_2S \times nH_2O$;

¾ triethanolamine solution 1 + 4.

4.2.6.2 Procedure and evaluation

Pipette 10 ml of the filtrate as specified in 4.2.3.3 from a 1 000 ml volumetric flask into a 600 ml beaker and dilute to approximately 200 ml. Adjust the pH value of the solution to 10 (check) with ammonium chloride buffer. After heating to approximately 60 °C and adding approximately 0,1 g of eriochrome black T indicator, titrate the solution on a white base against a white background (approximately 10 cm away) with 0,1 mol/l EDTA solution, stirring continuously (e.g. using a magnetic stirrer). The end point is indicated by a change in colour from red to blue. As the blue colour tends to fade, after adding approximately 10 ml of concentrated ammonia solution at approximately 60 °C with a few drops of complexone solution, titrate the solution to the end point, i.e. until the blue colour is permanent. Determine the blank value in parallel.

Other common indicators, e.g. indicator buffer tablets (change in colour from red to green via an intermediate grey colour) may also be used. Traces of iron, manganese, zinc and copper interfere with titration. Depending on the type and quantity of the interfering elements, add 10 drops of 10 % sodium sulfide solution or triethanolamine solution 1 + 4 to the solution.

$$\text{Magnesium oxide MgO} = \frac{0{,}2016 \quad [2 \quad (V_2 \quad B_2) \quad 0{,}1 \quad (V_1 \quad B_1)] \quad 100}{m_E}$$

where

V_1 is the volume of the used 0,01 mol/l EDTA solution as specified in 4.2.4.2, in ml;

V_2 is the volume of the used 0,1 mol/l EDTA solution as specified in 4.2.6.2, in ml;

B_1 is the blank value as described in 4.2.4.2 in ml;

B_2 is the blank value as described in 4.2.6.2 in ml;

m_E is the initial mass in accordance with 4.2.2.3 in g.

4.2.7 Loss on ignition

4.2.7.1 Apparatus

¾ electric oven, adjustable to (1 000   25) °C;

¾ platinum or porcelain crucible;

¾ desiccator with drying agent.

4.2.7.2 Procedure and evaluation

Weigh out to the nearest 0,000 1 g 1 g of the sample, dried to a constant mass (0,000 5 g), and add to it at a temperature of (105   5) °C in a red-hot crucible of known mass heated beforehand in an electric oven at (1 000   25) °C (generally for about 2 h). Then, allow the crucible and its contents to cool down to room temperature in the desiccator and weigh the residue to the nearest 0,000 1 g.

$$\text{Loss on ignition} = \frac{(m_E \quad m_A) \quad 100}{m_E} \text{ in \%}$$

where

m_A is the final mass in g;

m_E is the initial mass in g.

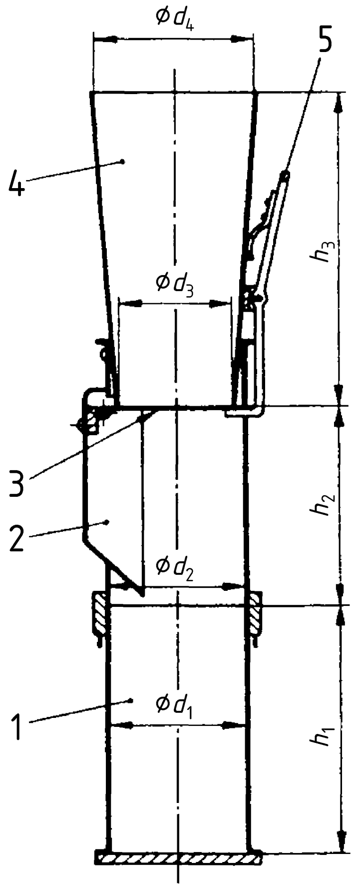
4.3 Bulk density

4.3.1 General

The bulk density is determined in principle in accordance with EN 459-2.

4.3.1.1 Apparatus

The apparatus by which the bulk density shall be determined (see Table 1 and Figure 1) 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.



Key

1 cylindrical one-litre vessel

2 connecting piece

3 closure flap

4 hopper

5 sprung closure lever

Figure 1 — Density apparatus

Table 1 — Dimensions of the density apparatus

Designation	**Dimensions**
One-litre vessel	
Internal diameter	d_1 = (87 + 1) mm
Internal height	h_1 = corresponding to filled contents of 1 000 cm^3 limit of error 5 cm^3
Connecting piece	
Internal diameter	d_2 = (87 1) mm
Height	h_2 = (135 1) mm
Hopper	
Internal diameter, bottom	d_3 = (79 1) mm
Internal diameter, top	d_4 = (99 1) mm
Height	h_3 = (199 1) mm

4.3.1.2 Procedure and evaluation

Pour sufficient of the caustic magnesia, prepared as described in 4.1, into the hopper of the density apparatus with a shovel (with a capacity of approximately 150 g) up to 1 h after sieving 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 powder which has run into the vessel and now projects above the top edge with a ruler and determine the mass of the contents of the vessel.

Carry out two tests; if the resulting values differ from one another by more than 10 g, a third test is necessary. If this value also differs by more than 10 g, record all 3 individual values. Record the average of the two values with the lowest difference from each other as the bulk density of the caustic magnesia in kg/m^3.

4.4 Fineness

4.4.1 Apparatus

Test sieve with a mesh size of 0,09 mm as specified in ISO 3310-1.

4.4.2 Procedure and evaluation

Generally, use wet sieving.

Weigh out 10,0 g of the representative sample as described in 4.1 on an analytical balance and place the caustic magnesia dried at 105 °C on the test sieve.

Distribute the material to be tested by directing a water jet on it backwards and forwards and continue until the passage through the sieve of the material to be tested does not perceptibly vary.

After drying at 105 °C, record the amount of residue in percent of the mass of the original material to be tested to the nearest 0,1 %.

Carry out two sievings. If the results deviate by more than 2 % from each other, carry out a third test. The definitive value is the average of the two values with the lowest difference from each other.

4.5 Setting time

4.5.1 General

In principle, setting time is determined in accordance with EN 196-3.

NOTE When determining the standard consistency and the final set, the penetration depth differs from that in EN 196-3.

4.5.2 Temperature and relative humidity

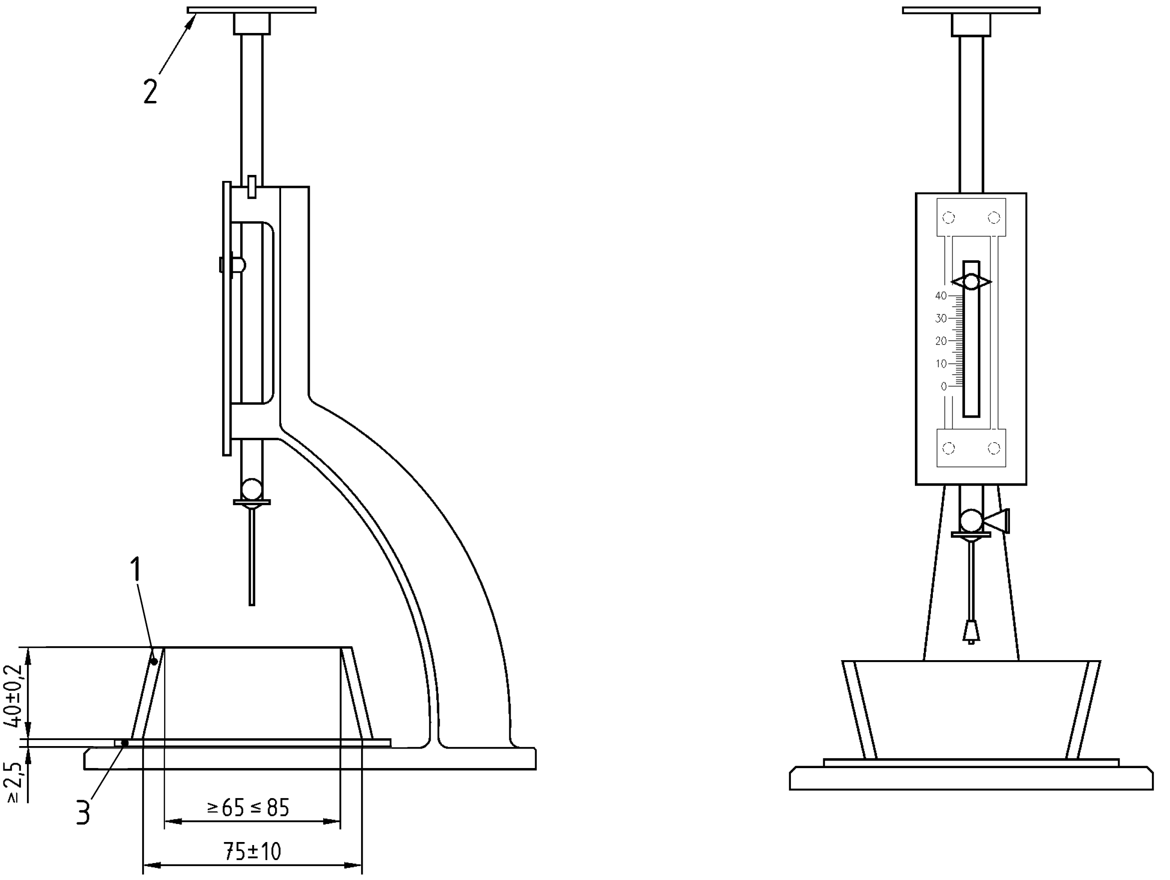
The air temperature in the test room and the storage room for the setting time sample shall be (20 2) °C and the relative humidity (65 5) %. The temperature of the materials and the apparatus shall also be (20 2) °C.

4.5.3 Apparatus

Use the Vicat apparatus (see Figures 2a) and 2b)) with the plunger (see Figure 2c)) made of non-corrodible metal in the form of a right cylinder of (50 1) mm effective length and (10,00 0,05) mm diameter having steel needles (see Figures 2d) and 2e)) of cylindrical form of (50 1) mm effective length (or (30 1) mm needle, (see Figure 2e)), and (1,13 0,05) mm diameter. The total mass of moving parts shall be (300 1) g. Their movement shall be fully vertical and without appreciable friction. Their axis shall coincide with that of the plunger.

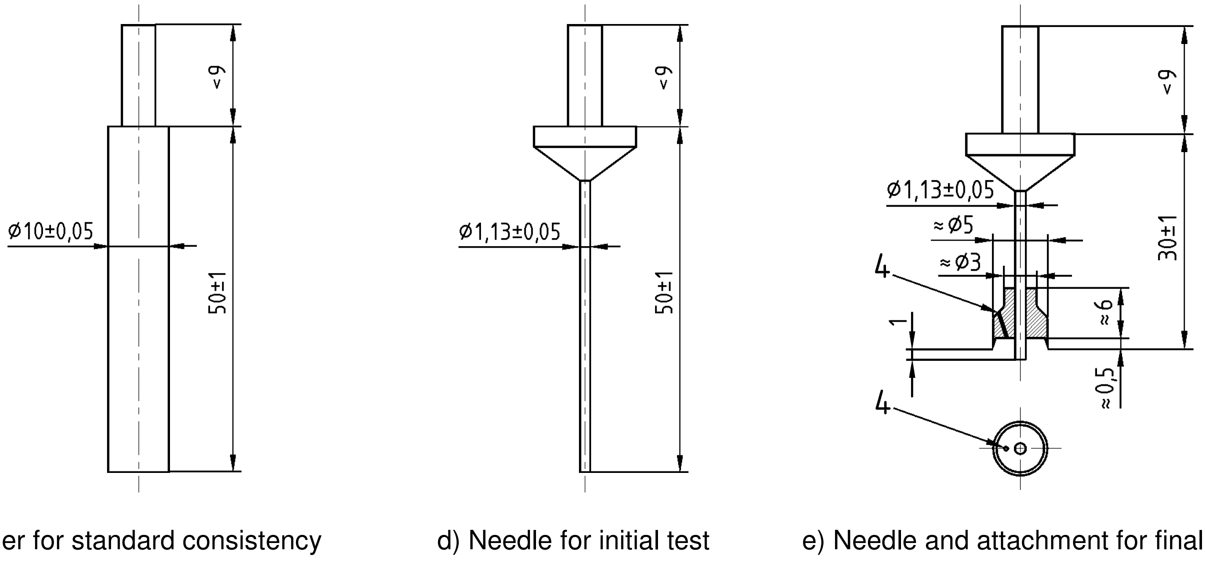
The Vicat mould (see Figure 2a)), in which the magnesia paste under test is contained, shall be of truncated conical form with a depth of (40,0 0,2) mm and have internal diameters at top between 65 mm and 85 mm and at bottom of (75 10) mm respectively. It shall be adequately rigid and shall be provided with a plane glass baseplate larger than the mould and at least 2,5 mm thick.

Dimensions in mm



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

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



c) Plunger for standard consistency

d) Needle for initial test

e) Needle and attachment for final test

Key

1	hard rubber mould
2	platform for correcting weights
3	basis plate
4	air vent (diameter 1,5 mm)

Figure 2 — Vicat apparatus for determination of the standard consistency and setting time

4.5.4 Magnesium chloride test solution

The magnesium chloride test solution shall have a density of 1,157 g/cm^3 at 20 °C and be prepared from magnesium chloride and distilled or demineralised water. If the solution is turbid, it shall be filtered before use.

4.5.5 Preparation of the magnesia paste of standard consistency

Use a magnesia paste (without fillers) for testing the setting characteristics.

Fill the mixer as specified in EN 13892-1 with a specific amount of the $MgCl_2$ test solution as described in 4.5.4 and 300 g of caustic magnesia from the representative sample as described in 4.1. Then, run the mixer at low speed for 30 s. Stop the mixer after 30 s for 15 s during which remove with a suitable scraper the magnesia paste adhering to the bowl outside the mixing zone and return it to the mix. Restart the mixer and run at low speed for a further 60 s. The total mixer running time shall be 90 s.

Then, transfer the paste to the conical hard rubber mould which has previously been placed on a lightly greased glass base-plate shaking it lightly to facilitate filling. Level the top of the magnesia paste flush with the edge of the mould.

Calibrate the Vicat apparatus with the plunger (see Figure 2c)) attached in advance of the test by lowering the plunger to rest on the base-plate underneath and adjusting the pointer to read zero on the scale. Raise the plunger to the stand-by position.

Immediately after levelling the magnesia 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 magnesia paste. Pause in that position for 1 s to 2 s 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 magnesia paste. The release of the plunger shall occur 4 min after the beginning of mixing. Read the scale when penetration has ceased or 30 s after release of the plunger, whichever is the earlier. Standard consistency has been attained when the plunger is (6 2) mm above the base-plate in the magnesia paste 30 s after it has been released.

If necessary, repeat the test with different amounts of solution until standard consistency has been attained.

Express the amount of magnesium chloride test solution added (in g) as a percentage by mass of the dry caustic magnesia. When the magnesia paste attains standard consistency, immediately remove the plunger and determine the setting time.

4.5.6 Initial and final setting time

Set the Vicat apparatus (Figure 2d)) with the needle attached in advance of the test by lowering the needle to rest on the glass base-plate and adjusting the pointer to read zero on the scale. Raise the needle to the stand-by position.

After a suitable time, transfer the filled mould to the Vicat apparatus and position under the needle. Lower the needle gently until it is in contact with the magnesia paste. Pause in that position for 1 s to 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 centre of the magnesia 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 glass base-plate, together with the time from the beginning of the mixing. Repeat the penetration test on the same magnesia 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. Clean the Vicat needle immediately after each penetration. Record the time measured from the start of mixing at which the distance between the needle and the glass base-plate is (4 1) mm as the initial setting time 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.

Invert the filled mould used for determining the initial setting time on the glass base-plate so that tests for final set are made on the face of the specimen originally in contact with the glass base-plate. For this, use the needles shown in Figures 2d) or 2e). Ensure that the total mass of the movable parts is (300 1) g.

The interval of time between penetration tests may be increased to e.g. 30 min. Record to the nearest 15 min the time from the beginning of mixing up to the moment at which the needle penetrates only 1,0 mm into the setting magnesia paste as the final setting time of the caustic magnesia. 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.

When the needle as shown in Figure 2e) is used, the final setting time is attained when the ring attachment of the needle first does not leave any mark on the specimen surface.

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

4.6 Flexural strength and compressive strength

4.6.1 General

Determine the flexural strength and compressive strength for each age class on 3 prisms of magnesia test mortar with the dimensions 4 cm x 4 cm x 16 cm.

4.6.2 Preparation of the magnesia test mortar and the test specimens

The magnesia test mortar consists of 1 part by weight of caustic magnesia as described in 4.1, 3 parts by weight of CEN standard sand as specified in EN 196-1 and a 26° Baumé $MgCl_2$ solution.

The prepared mixture shall always be sufficient for 3 specimens. The mixture shall consist of:

¾ 450 g of caustic magnesia;

¾ 1 350 g of CEN standard sand as specified in EN 196-1;

¾ 500 g of a 26° Baumé $MgCl_2$ solution.

Each mixture shall be prepared in accordance with EN 196-1 in the mechanical mixer described in EN 13892-1. When the mixer is ready for operation:

a) introduce the $MgCl_2$ solution into the pan and add the caustic magnesia;

b) immediately start the mixer at low speed; after 30 s, add the sand at a uniform rate over the next 30 s; then, switch the mixer to the higher speed and continue mixing for a further 30 s;

c) then, stop the mixer for 90 s; in the first 15 s, remove all the mortar adhering to the wall and bottom of the pan by means of a rubber scraper and return it to the centre of the pan;

d) continue mixing for 60 s at the higher speed.

The timetable for the various mixing stages shall be adhered to with a tolerance of 5 s. Determine the flow value as specified in EN 459-2, 5 min after the beginning of mixing.

A flat, horizontal glass plate and mould with hopper are required for determining the flow value. The mould and hopper shall be made of a metal non-corrodible by mortar. The internal diameter of the bottom (100 mm), the internal diameter of the top (70 mm) and the height of the mould (60 mm) shall be adhered to with a tolerance of 0,5 mm. The thickness of the mould shall be at least 2,0 mm such that dents can be avoided.

To determine the flow value, place the mould, the inner surface of which has been wetted by means of a damp cloth, in the centre of a clean, dry glass plate. Fill the mould with mortar. Then, remove the hopper immediately and level off any excess mortar. If necessary, clean the glass plate again. After approximately 10 s, carefully lift off the mould vertically.

Determine the flow value after termination of the flow process but not later than after 30 s. The flow value is the diameter of the mortar cake determined from two measurements taken vertically to each other.

Record the flow value.

Prepare and store the test specimens as specified in EN 13892-1.

4.6.3 Procedure and evaluation

Determine the flexural strength and compressive strength of the magnesia test mortar after 3 days and after 28 days as specified in EN 13892-2.

5 Test report

The test report shall include the following information:

a) number, title and year of publication of this European Standard;

b) place, date and time of sampling, sampling method and the name of the sampler;

c) designation of the caustic magnesia and the magnesium chloride, including type, origin and marking in accordance with the standards relevant to the product EN 13813 or EN 14016-1;

d) date and time of the test;

e) for the chemical analysis of the magnesium chloride, the contents of magnesium chloride, water-insoluble residue, sodium chloride, potassium chloride, calcium chloride, magnesium sulfate and calcium sulfate and the pH value;

f) for the chemical analysis of the caustic magnesia, the contents of magnesium oxide, hydrochloric- acid-insoluble residue, sesquioxides and calcium oxide and the loss on ignition;

g) for the bulk density of the caustic magnesia, the number of tests, the mean value and where appropriate the individual values;

h) for the fineness of the caustic magnesia, the number of tests, the mean value and where appropriate the individual values;

i) for the setting of the caustic magnesia, the necessary amount of $MgCl_2$ test solution, the initial and final setting times;

j) for the flexural strength and the compressive strength, the sampling method (with reference to EN 13892-1), the flow value of the test mortar, the age of the mortar under test, the total mass of each test piece and the results of the strength tests;

k) the requirements from EN 14016-1 for comparison of the results;

l) any deviations from this standard that may have influenced the test results.

Bibliography

EN 13318, *Screed materials and floor screeds – Definitions.*

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