SLOVENSKI STANDARD
SIST EN 14016-2:2004

01-april-2004

Veziva za magnezitne estrihe ï Kaustiļ ni magnezit in magnezijev klorid - 2. del: Preskusne metode

Binders for magnesite screeds - Caustic magnesia and magnesium chloride - Part 2: Test methods

Bindemittel für Magnesiaestriche - Kaustische Magnesia und Magnesiumchlorid - Teil 2: Prüfverfahren

Liants pour chapes a base de magnésie - Magnésie caustique et chlorure de magnésium - Partie 2: Méthodes d'essai

Ta slovenski standard je istoveten z: EN 14016-2:2004

ICS:

91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

SIST EN 14016-2:2004 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14016-2

January 2004

ICS 91.100.50

English version

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Bindemittel für Magnesiaestriche - Kaustische Magnesia und Magnesiumchlorid - Teil 2: Prüfverfahren

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COMITÉ EUROPÉEN DE NORMALISATION
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Ref. No. EN 14016-2:2004: E

Contents

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of an identical text or by endorsement, at the latest by July 2004, and conflicting national standards shall be withdrawn at the latest by July 2004.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No existing European Standard is superseded by this standard.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. "ρ" 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

— Balance accurate to 0,000 1 g;

— Platinum or porcelain crucible;

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

— Desiccator with drying agent.

3.2.2.2 Reagents

— Silver nitrate, $AgNO_3$;

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

— 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:

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

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

— Platinum crucible;

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

— Desiccator with drying agent.

3.2.3.2 Reagents

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

— Hydrochloric acid, dilute 1 + 11;

— Barium chloride – 2 – hydrate $BaCl_2 \cdot 2H_2O$;

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

— Silver nitrate $AgNO_3$;

— Nitric acid HNO_3;

— 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:

$$\text{Sulfate content } SO_{4total}^{2-} = \frac{8{,}232 \times m_{A1} x100}{m_E} \text{ in \%}$$

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

$$\text{Sulfate content } SO_{4total}^{2-} = \frac{4{,}116 \times m_{A2} \times 100}{m_E} \text{ in \%}$$

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

— Sodium hydroxide NaOH;

— 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;

— Murexide: purpuric acid, ammonium salt;

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

— Sodium chloride NaCl;

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

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

— 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:

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

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:

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

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

- Ammonium chloride NH_4Cl;
- Ammonia solution (ρ = 0,91 g/cm^3) NH_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, sodiumsalt;
- Eriochrome black T indicator: grind1 g of eriochrome black T with 100 g of sodium chloride;
- EDTA: ethylenediamine tetraacetic acid, disodium salt, dihydrate;
- EDTA solution 0,1 mol/l (a commercially available ready-to-use solution may be used);
- Sodium sulfide $Na_2S \cdot nH_2O$;
- Triethanolmine $N(CH_2CH_2OH)_3$ (ρ = 1,12 g/cm^3);
- 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:

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

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:

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

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.

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

then sulfate is present as magnesium sulfate and calcium sulfate.

$$\text{Sulfate content} \quad SO_{4\,MgSO_4}^{2-} = SO_{4\,MgSO_{total}}^{2-} - \frac{3{,}3967 \times Ca_{total}^{2+}}{1{,}4172} \text{ in \%}$$

$$\text{Magnesium sulfate } MgSO_4 = 1{,}253 \cdot SO_{4\,MgSO_4}^{2-} \text{ in \%}$$

$$\text{Calcium sulfate } CaSO_4 = 3{,}3967 \cdot Ca_{total}^{2+} \text{ in \%}$$

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

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

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: