



BSI Standards Publication

Winter service equipment and products — De-icing agents

Part 2: Calcium chloride and Magnesium chloride — Requirements and test methods

National foreword

This British Standard is the UK implementation of EN 16811-2:2016.

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Partie 2 : Chlorure de calcium et chlorure de
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Winterdienstausrüstung - Enteisungsmittel - Teil 2:
Calcium- und Magnesiumchlorid - Anforderungen und
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Contents		Page
European foreword.....		4
Introduction		5
1	Scope	6
2	Normative references	6
3	Terms and definitions	7
4	Requirements for calcium chloride and magnesium chloride	7
4.1	Chemical requirements.....	7
4.1.1	Calcium chloride.....	7
4.1.2	Magnesium chloride.....	8
4.2	Sieve analysis.....	8
4.2.1	Calcium chloride.....	8
4.2.2	Magnesium chloride.....	9
4.3	General requirements	9
4.4	Packaging and marking	10
4.5	Material safety data sheet and product description.....	11
5	Requirements for solutions of calcium chloride and magnesium chloride.....	11
5.1	Chemical Requirements.....	11
5.1.1	Calcium chloride.....	11
5.1.2	Magnesium chloride.....	11
5.2	General requirements	12
5.3	Marking, material safety data sheet and product description	13
6	Requirements for blends of calcium chloride and magnesium chloride with sodium chloride and/or potassium chloride.....	13
6.1	General.....	13
6.2	Chemical requirements.....	13
6.2.1	General.....	13
6.2.2	Solid blends	13
6.2.3	Liquid blends	13
6.3	Sieve analysis.....	14
6.4	General Requirements	14
6.5	Packaging, marking, material safety data sheet and product description.....	14
7	Sampling.....	14
8	Test methods	15
8.1	General.....	15
8.2	Calcium chloride.....	15
8.3	Magnesium chloride.....	15
8.4	Other chlorides	15
8.4.1	Other chlorides (magnesium chloride, potassium chloride, sodium chloride) in calcium chloride	15
8.4.2	Other chlorides (calcium chloride, potassium chloride, sodium chloride) in magnesium chloride.....	15
8.5	Sodium chloride.....	16
8.6	Potassium chloride.....	16

8.7	Sulfate	16
8.8	Water insoluble matter	16
8.9	Sieve analysis	16
8.10	Heavy metals	16
8.11	Hydrocarbons	16
8.12	Total organic carbon (TOC).....	16
8.13	pH	17
8.14	Bulk density.....	17
8.15	Density.....	17
Annex A (normative) Product descriptions		18
A.1	Product description for calcium chloride.....	18
A.2	Product description for magnesium chloride	20
A.3	Product description for blends of calcium chloride and magnesium chloride with sodium chloride and/or potassium chloride.....	23
Annex B (normative) Sampling.....		26
B.1	Solid form	26
B.1.1	Package shipments.....	26
B.1.2	Bulk shipments.....	26
B.2	Liquid form	26
B.3	Labelling and distribution of samples	26
B.4	Sampling report	27
Annex C (normative) Analytical methods — Determination of calcium chloride by complexometry.....		28
C.1	Scope	28
C.2	Principle.....	28
C.3	Reagents.....	28
C.4	Apparatus	29
C.5	Procedure	29
C.5.1	Solid samples.....	29
C.5.2	Liquid samples.....	29
C.5.3	Titration.....	29
C.6	Calculation.....	30
Bibliography		31

European foreword

This document (EN 16811-2:2016) has been prepared by Technical Committee CEN/TC 337 “Road operation equipment and products”, 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 February 2017, and conflicting national standards shall be withdrawn at the latest by February 2017.

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

EN 16811, *Winter service equipment and products — De-icing agents*, is currently composed with the following parts:

- *Part 1: Sodium chloride — Requirements and test methods*;
- *Part 2: Calcium chloride and Magnesium chloride — Requirements and test methods*;
- *Part 3: Other solid and liquid de-icing agents — Requirements and test methods* [CEN/TS].

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

Introduction

De-icing agents are important for the winter maintenance of roads. They can prevent and, in addition to it, eliminate slippery conditions.

The standard describes the requirements for de-icing agents and their testing methods. The requirements are different for using, storage and type of distribution (spreading and spraying). These need different properties.

The aim of this standard is an easy description of the product specifications for tenders and other purchasing procedures.

1 Scope

This European Standard specifies the essential requirements of calcium chloride and magnesium chloride for spreading on roads for winter maintenance and includes tests of these requirements. The requirements are specified for calcium chloride and magnesium chloride in solid form and in water solution, which is delivered to the customer.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

EN 1235, *Solid fertilizers — Test sieving (ISO 8397:1988 modified)*

EN 1236, *Fertilizers — Determination of bulk density (loose) (ISO 3944:1992 modified)*

EN 1484, *Water analysis — Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*

EN 14016-2, *Binders for magnesite screeds — Caustic magnesia and magnesium chloride — Part 2: Test methods*

EN 15144, *Winter maintenance equipment — Terminology — Terms for winter maintenance*

EN 16198, *Fertilizers — Determination of magnesium by complexometry*

EN 16811-1, *Winter service equipment and products — De-icing agents — Part 1: Sodium chloride — Requirements and test methods*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

EN ISO 9377-2, *Water quality — Determination of hydrocarbon oil index — Part 2: Method using solvent extraction and gas chromatography (ISO 9377-2)*

EN ISO 10523, *Water quality — Determination of pH (ISO 10523)*

EN ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885)*

EN ISO 12846, *Water quality — Determination of mercury — Method using atomic absorption spectrometry (AAS) with and without enrichment (ISO 12846)*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 649-2, *Laboratory glassware — Density hydrometers for general purposes — Part 2: Test methods and use*

ISO 758, *Liquid chemical products for industrial use — Determination of density at 20 degrees C*

ISO 2479, *Sodium chloride for industrial use — Determination of matter insoluble in water or in acid and preparation of principal solutions for other determinations*

ISO 2480, *Sodium chloride for industrial use — Determination of sulphate content — Barium sulphate gravimetric method*

ISO 2591-1, *Test sieving — Part 1: Methods using test sieves of woven wire cloth and perforated metal plate*

ISO 6227, *Chemical products for industrial use — General method for determination of chloride ions — Potentiometric method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15144 and the following apply.

3.1

calcium chloride

CaCl_2

substance which is produced from calcium carbonate and hydrochloric acid, as by-product of the synthetic soda ash process, and as by-product of magnesium oxide production

Note 1 to entry: It can also result from flue gas cleaning (e.g. waste incinerators). It is available in varying concentrations, as flakes with crystal water (calcium chloride dihydrate), and as anhydrous solid like pellets, prills or granules. For the application in winter maintenance the calcium chloride is also used as water solutions in varying concentrations.

3.2

magnesium chloride

MgCl_2

substance which is produced from natural underground deposits, from salt lakes, from natural brines, from sea water and as by-product of potassium chloride and potassium sulphate production

Note 1 to entry: It is available as flakes, prills, or pellets with crystal water (magnesium chloride hexahydrate). For the application in winter maintenance the magnesium chloride is also used as water solutions in varying concentrations.

3.3

multi-chloride blends

solid or liquid blends, respectively multi-chloride brines of calcium chloride and magnesium chloride with sodium and/or potassium chloride

Note 1 to entry: Such blends are produced by mixing or as by-product from potash production.

4 Requirements for calcium chloride and magnesium chloride

4.1 Chemical requirements

4.1.1 Calcium chloride

The product shall conform to the requirements specified in Table 1.

Table 1 — Chemical requirements for calcium chloride

Parameter	Limit Weight %	
	Flakes	Pellets, prills, granules
Calcium chloride	Min. 75	Min. 94
Other chlorides (magnesium chloride, potassium chloride, sodium chloride)	Max. 5	
Sulfate	Max. 0,5	
Water insoluble matter	Max. 0,5	
NOTE 1	Other chlorides and sulfate are expressed as percentage in the anhydrous product.	
NOTE 2	Other chlorides expressed as NaCl.	

4.1.2 Magnesium chloride

The product shall conform to the requirements specified in Table 2.

Table 2 — Chemical requirements for magnesium chloride

Parameter	Limit Weight %	
	Flakes, prills, pellets	
Magnesium chloride	Min. 46,5	
Other chlorides (calcium chloride, potassium chloride, sodium chloride)	Max. 5	
Sulfate	Max. 1,5	
Water insoluble matter	Max. 0,5	
NOTE 1	Other chlorides and sulfate are expressed as percentage in the anhydrous product.	
NOTE 2	Other chlorides expressed as NaCl.	

4.2 Sieve analysis

4.2.1 Calcium chloride

The product shall conform to the requirements specified in Table 3.

Table 3 — Sieve analysis for calcium chloride

Sieve size	Weight % passing test sieve	
	Flakes	Pellets, prills, granules
20 mm	100	-
10 mm	Min. 95	100
5 mm	80 - 100	80 - 100
2 mm	Max. 40	0 - 20
0,8 mm	0 - 10	0 - 5

4.2.2 Magnesium chloride

The product shall conform to the requirements specified in Table 4.

Table 4 — Sieve analysis for magnesium chloride

Sieve size	Weight % passing test sieve	
	Flakes	Pellets, prills
20 mm	100	-
10 mm	Min. 95	100
5 mm	Max. 90	80 - 100
2 mm	Max. 25	0 - 20
0,8 mm	0 - 10	0 - 5

4.3 General requirements

The product shall conform to the requirements specified in Table 5.

Table 5 — General requirements for calcium chloride and magnesium chloride

Parameter	Limit	Unit
pH of 10 weight % solution	between 5 and 11	
Al (Aluminium)	≤ 50	mg/kg
As (Arsenic)	≤ 2,5	mg/kg
Cd (Cadmium)	≤ 2	mg/kg
Co (Cobalt)	≤ 2	mg/kg
Cr (Chromium)	≤ 5	mg/kg
Cu (Copper)	≤ 5	mg/kg
Hg (Mercury)	≤ 0,5	mg/kg
Ni (Nickel)	≤ 5	mg/kg
Pb (Lead)	≤ 5	mg/kg
Zn (Zinc)	≤ 20	mg/kg
Hydrocarbons	≤ 100	mg/kg
NOTE 1 The limits for Al (Aluminium) and hydrocarbons are foreseen for products which are not of natural origin. There is no need to determine these parameters in natural based CaCl ₂ resp. MgCl ₂ .		
NOTE 2 The parameters are stated as mg/kg of anhydrous product.		

The product shall arrive at purchaser's delivery point in a free-flowing and usable condition.

For products from flue gas cleaning (e.g. waste incinerators, etc.) the supplier shall supply the content of total organic carbon (TOC) for the evaluation of the environmental impact.

4.4 Packaging and marking

The product shall be packaged and transported in a manner that protects the product from moisture.

The following information shall be marked on the packaging or on associated documents:

- a) the name and address of the producer or supplier;
- b) the name of the product ("Calcium chloride", "Magnesium chloride");
- c) the quality of the product ("Flakes", etc.);
- d) the percentage of calcium chloride resp. magnesium chloride;
- e) the net weight;
- f) the number of this European Standard.

NOTE The marking of the product will be in accordance with Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 [10].

All information shall be given in the languages of the countries of destination.

4.5 Material safety data sheet and product description

A material safety data sheet of the product is prepared. At time of delivery latest the material safety data sheet is made available for the customer.

NOTE This material safety data sheet will be prepared in accordance with the provisions of REACH [11].

In tendering processes the offers shall include a product description which is dated not longer than 12 months before the date of the offer. The supplier shall supply the bulk density (loose) for information purposes (see A.1 and A.2).

All information shall be given in the languages of the countries of destination.

5 Requirements for solutions of calcium chloride and magnesium chloride

5.1 Chemical Requirements

5.1.1 Calcium chloride

The product shall conform to the requirements specified in Table 6.

Table 6 — Chemical requirements for calcium chloride solutions

Parameter	Limit Weight %
Calcium chloride	16 - 36
Other chlorides (magnesium chloride, potassium chloride, sodium chloride)	Max. 5
Sulfate	Max. 0,5
Water insoluble matter	Max. 0,2 ^a
The concentrations of liquid calcium chloride shall be specified by the purchaser. Typical concentrations vary from 16 weight % to 36 weight %.	
NOTE 1 Other chlorides and sulfate are expressed as percentage in the anhydrous product.	
NOTE 2 Other chlorides expressed as NaCl.	
^a 10 l of calcium chloride solution shall pass a test sieve (size 0,5 mm) without water insoluble residues after washing with water on the sieve.	

5.1.2 Magnesium chloride

The product shall conform to the requirements specified in Table 7.

Table 7 — Chemical requirements for magnesium chloride solutions

Parameter	Limit Weight %
Magnesium chloride	16 - 33
Other chlorides (calcium chloride, potassium chloride, sodium chloride)	Max. 5
Sulfate	Max. 1,0
Water insoluble matter	Max. 0,1 ^a
The concentration of liquid MgCl ₂ shall be specified by the purchaser. Typical concentrations vary from 16 weight % to 33 weight %.	
NOTE 1	Other chlorides are expressed as percentage in the liquid product.
NOTE 2	Other chlorides expressed as NaCl.
NOTE 3	Sulfate measured in a 10 weight % MgCl ₂ solution and expressed as SO ₄ .
^a 10 l of magnesium chloride solution shall pass a test sieve (size 0,5 mm) without water insoluble residues after washing with water on the sieve.	

5.2 General requirements

The product shall conform to the requirements specified in Table 8.

Table 8 — General requirements for calcium chloride and magnesium chloride solutions

Parameter	Limit	Unit
pH of 10 weight % solution	between 5 and 11	
Al (Aluminium)	≤ 50	mg/kg
As (Arsenic)	≤ 2,5	mg/kg
Cd (Cadmium)	≤ 2	mg/kg
Co (Cobalt)	≤ 2	mg/kg
Cr (Chromium)	≤ 5	mg/kg
Cu (Copper)	≤ 5	mg/kg
Hg (Mercury)	≤ 0,5	mg/kg
Ni (Nickel)	≤ 5	mg/kg
Pb (Lead)	≤ 5	mg/kg
Zn (Zinc)	≤ 20	mg/kg
Hydrocarbons	≤ 100	mg/kg
NOTE 1	The limits for Al (Aluminium) and hydrocarbons are foreseen for products which are not of natural origin. There is no need to determine these parameters in natural based CaCl ₂ resp. MgCl ₂ .	
NOTE 2	The parameters are stated as mg/kg of anhydrous product.	

For products from flue gas cleaning (e.g. waste incinerators, etc.) the supplier shall supply the content of total organic carbon (TOC) for the evaluation of the environmental impact.

5.3 Marking, material safety data sheet and product description

The product shall be transported in a manner that protects the product from moisture.

The following information shall be marked on associated documents:

- a) the name and address of the producer or supplier;
- b) the name of the product (“Calcium chloride”, “Magnesium chloride”);
- c) the concentration of calcium chloride resp. magnesium chloride;
- d) the net weight;
- e) the number of this European Standard.

NOTE The marking of the product will be in accordance with Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 [10].

A material safety data sheet of the product is prepared in accordance with the provisions of REACH [11]. At time of delivery latest the material safety data sheet is made available for the customer.

In tendering processes the offers shall include a product description which is dated not longer than 12 months before the date of the offer. The supplier shall supply the density (20 °C) for information purposes (see A.1 and A.2).

All information shall be given in the languages of the countries of destination.

6 Requirements for blends of calcium chloride and magnesium chloride with sodium chloride and/or potassium chloride

6.1 General

For solid or liquid blends resp. multi-chloride brines of calcium chloride and magnesium chloride with sodium chloride and/or potassium chloride the following requirements shall apply.

6.2 Chemical requirements

6.2.1 General

The individual components shall fulfil the requirements for the individual components (see 4.1, 5.1 and EN 16811-1).

6.2.2 Solid blends

Calcium chloride resp. magnesium chloride shall exist in a concentration above 10 weight % of the total accumulation of chloride based salts in percent including calcium chloride, magnesium chloride, sodium chloride and potassium chloride. The concentration shall be calculated with the anhydrous forms of calcium chloride and magnesium chloride.

Solid blends shall not contain more than 1,5 weight % sulfate in the anhydrous product.

6.2.3 Liquid blends

The product shall contain no less than 16 weight % concentration of the total accumulation of chloride based salts in percent including calcium chloride, magnesium chloride, sodium chloride and potassium chloride. Calcium chloride resp. magnesium chloride shall exist in a concentration above 2,5 weight % of the total accumulated concentration.

Liquid blends with calcium chloride shall not contain more than 0,5 weight % sulfate (expressed in the anhydrous product). Liquid blends with magnesium chloride shall not contain more than 1,0 weight % (measured in a solution with a concentration of 10 weight %).

Liquid blends shall not contain more than 0,2 weight % water insoluble matter.

6.3 Sieve analysis

For solid blends the gradation of the individual components shall be in accordance with the requirements for the individual components (see 4.2 and EN 16811-1). Un-compacted blends shall not have particles with sizes above 20 mm, and shall not contain more than 10 weight % particles with a size between 0 mm and 0,8 mm.

For compacted products the gradation shall be in accordance with the requirements in 4.2.1.

6.4 General Requirements

The blends resp. multi-chloride brines shall not contain heavy metals in excess of the established concentration limits for calcium chloride and magnesium chloride.

For products from flue gas cleaning (e.g. waste incinerators, etc.) the supplier shall supply the content of total organic carbon (TOC) for information purposes.

The pH shall be between 5 and 11 (see 4.3 and 5.2).

6.5 Packaging, marking, material safety data sheet and product description

The blend in either solid or liquid form shall be packaged and transported in a manner that protects the product from moisture.

The following information shall be marked on the packaging or on associated documents:

- a) the name and address of the producer or supplier;
- b) the name of the product;
- c) the concentration of calcium chloride resp. magnesium chloride;
- d) the net weight;
- e) the number of this European Standard.

NOTE The marking of the product will be in accordance with Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 [10].

A material safety data sheet of the product is prepared in accordance with the provisions of REACH [11]. At time of delivery latest the material safety data sheet is made available for the customer.

In tendering processes the offers shall include a product description which is dated not longer than 12 months before the date of the offer. The supplier shall supply the bulk density (loose) of solid blends and the density (20 °C) of liquid blends for information purposes (see A.3).

All information shall be given in the languages of the countries of destination.

7 Sampling

The sampling of solid and liquid forms of calcium chloride, magnesium chloride and blends as well as multi-chloride brines shall be according the procedures described in Annex B.

8 Test methods

8.1 General

The test for compliance with the requirements shall be in accordance with the following methods or in accordance with validated methods:

8.2 Calcium chloride

The calcium chloride content shall be determined by titration with ethylenediaminetetraacetate (EDTA) solution in accordance with the analytical method given in Annex C.

NOTE Alternatively, the determination of content of calcium can be carried out by inductively coupled plasma optical emission spectrometry (ICP-OES), on the basis of EN ISO 11885. The CaCl_2 content results from the analysed calcium content by multiplication with 2,770.

8.3 Magnesium chloride

Calculate the percentage of MgCl_2 as follows:

$$\text{MgCl}_2 = (\text{Total magnesium content} - \text{magnesium from MgSO}_4) \times 3,917.$$

The total magnesium content shall be determined by titration with EDTA solution in accordance with the test methods given in EN 14016-2 or EN 16198.

NOTE Alternatively, the determination of the total content of magnesium can be carried out by inductively coupled plasma optical emission spectrometry (ICP-OES), on the basis of EN ISO 11885.

The magnesium from MgSO_4 results by multiplication of the SO_4 content (determined according to 8.7) with 0,253.

8.4 Other chlorides

8.4.1 Other chlorides (magnesium chloride, potassium chloride, sodium chloride) in calcium chloride

Calculate the percentage of other chlorides as follows:

$$\text{Other chlorides as NaCl} = (\text{Total chloride content} - \text{chloride from CaCl}_2) \times 1,649$$

The total chloride content in the product shall be determined by potentiometric titration in accordance with ISO 6227.

The chloride from CaCl_2 results by multiplication of the CaCl_2 content (determined according to 8.2) with 0,639.

8.4.2 Other chlorides (calcium chloride, potassium chloride, sodium chloride) in magnesium chloride

Calculate the percentage of other chlorides as follows:

$$\text{Other chlorides as NaCl} = (\text{Total chloride content} - \text{chloride from MgCl}_2) \times 1,649$$

The total chloride content in the product shall be determined by potentiometric titration in accordance with ISO 6227.

The chloride from MgCl_2 results by multiplication of the MgCl_2 content (determined according to 8.3) with 0,745.

8.5 Sodium chloride

The determination of content of sodium in blends can be carried out by inductively coupled plasma optical emission spectrometry (ICP-OES), on the basis of EN ISO 11885. The sodium chloride content results from the analysed sodium content by multiplication with 2,542.

NOTE Alternatively, the determination of the content of sodium can be carried out by atomic absorption spectrometry (AAS), on basis of EN ISO 6869.

8.6 Potassium chloride

The determination of content of potassium in blends can be carried out by inductively coupled plasma optical emission spectrometry (ICP-OES), on the basis of EN ISO 11885. The potassium chloride content results from the analysed potassium content by multiplication with 1,907.

NOTE Alternatively, the determination of the content of potassium can be carried out by atomic absorption spectrometry (AAS) in accordance with the test method given in EN 973.

8.7 Sulfate

The sulfate shall be determined based on the test method in ISO 2480 (gravimetric method).

NOTE Alternatively, the determination of content of sulfate can be carried out by inductively coupled plasma optical emission spectrometry (ICP-OES), on the basis of EN ISO 11885 or EN 15749. A further alternative test method for the determination of sulfate is the ionic chromatography method (IC), on basis of EN 15749.

8.8 Water insoluble matter

The matter insoluble in water shall be determined in accordance with the test method in ISO 2479. With solutions, start with filtration of the undiluted test sample (min. 350 ml).

8.9 Sieve analysis

The particle size distribution shall be determined by sieving in accordance with the test method in ISO 2591-1 (test sieves according to ISO 565) or in accordance with the test method in EN 1235.

8.10 Heavy metals

The content of heavy metals, except for mercury, shall be determined by inductively coupled plasma optical emission spectrometry (ICP-OES), on the basis of the test method in EN ISO 11885.

NOTE Alternatively, the determination of contents of heavy metals can be carried out by atomic absorption spectrometry (AAS), in accordance with the test method in EN ISO 15586.

The content of mercury shall be determined by cold vapour atomic absorption spectrometry, on the basis of the test method in EN ISO 12846.

8.11 Hydrocarbons

The content of hydrocarbons shall be determined in accordance with the test method in EN ISO 9377-2.

8.12 Total organic carbon (TOC)

The content of total organic carbon shall be determined on the basis of the test method in EN 1484 (modified: the presence of chloride requires an appropriate pre-treatment).

8.13 pH

The pH shall be determined in accordance with the test method in EN ISO 10523 (modified: determination in 10 weight % solution of the product resp. in diluted solutions of calcium chloride or magnesium chloride with a concentration of 10 weight %; dissolving and dilution with distilled water).

8.14 Bulk density

The bulk density of loose products shall be determined in accordance with the test method in EN 1236. On the basis of the same method the bulk density of products which contain more than 20 weight % of particles exceeding 5 mm in diameter shall be determined.

8.15 Density

The density of solutions at 20 °C shall be determined in accordance with the test method in ISO 758 or with the test method in ISO 649-2.

Annex A (normative)

Product descriptions

A.1 Product description for calcium chloride

Product

Trade name

Origin from flue gas cleaning (e.g. waste incinerators, etc.):

Yes

No

Supplier

.....

Product specification (results of supplier tests; requirements in brackets)

Flakes

Pellets, prills, granules

- **CaCl₂**:Weight % (Flakes min. 75, Pellets, prills, granules min. 94)
- **Other chlorides**:Weight % (max. 5)
- **Sulphate**:Weight % (max. 0,5)
- **Water insoluble matter**:Weight % (max. 0,5)
- **Sieve analysis**

Sieve size	Weight % passing test sieve	
	Flakes	Pellets, prills, granules
20 mm (100)	-
10 mm (min. 95) (100)
5 mm (80-100) (80-100)
2 mm (max. 40) (0-20)
0,8 mm (0 - 10) (0-5)

- **pH**:(5 - 11)
- **Bulk density (loose)**:kg/dm³

- **Heavy metals, etc.:**

Al (Aluminium)*	mg/kg (≤ 50)
As (Arsenic)	mg/kg ($\leq 2,5$)
Cd (Cadmium)	mg/kg (≤ 2)
Co (Cobalt)	mg/kg (≤ 2)
Cr (Chromium)	mg/kg (≤ 5)
Cu (Copper)	mg/kg (≤ 5)
Hg (Mercury)	mg/kg ($\leq 0,5$)
Ni (Nickel)	mg/kg (≤ 5)
Pb (Lead)	mg/kg (≤ 5)
Zn (Zinc)	mg/kg (≤ 20)
Hydrocarbons*	mg/kg (≤ 100)
TOC*	mg/kg

*data necessary if the product is not of natural origin

Product specification for liquid calcium chloride (results of supplier tests; requirements in brackets)

- **CaCl₂**:.....Weight % (specified concentration by purchaser)
- **Other chlorides**:.....Weight % (max. 5)
- **Sulphate**: Weight % (max. 0,5)
- **Water insoluble matter**:Weight % (max. 0,2)
- **pH**:(5 - 11)
- **Density (20 °C)**:.....kg/dm³
- **Heavy metals, etc.:**

Al (Aluminium)*	mg/kg (≤ 50)
As (Arsenic)	mg/kg ($\leq 2,5$)
Cd (Cadmium)	mg/kg (≤ 2)
Co (Cobalt)	mg/kg (≤ 2)
Cr (Chromium)	mg/kg (≤ 5)
Cu (Copper)	mg/kg (≤ 5)
Hg (Mercury)	mg/kg ($\leq 0,5$)
Ni (Nickel)	mg/kg (≤ 5)
Pb (Lead)	mg/kg (≤ 5)
Zn (Zinc)	mg/kg (≤ 20)
Hydrocarbons*	mg/kg (≤ 100)
TOC*	mg/kg

*data necessary if the product is not of natural origin

- Sieve analysis

Sieve size	Weight % passing test sieve	
	Flakes	Pellets, prills
20 mm (100)	-
10 mm(min. 95) (100)
5 mm (max. 90)(80 - 100)
2 mm (max. 25)(0 - 20)
0,8 mm(0 - 10)(0 - 5)

- pH:(5 - 11)

- Bulk density (loose):kg/dm³

- Heavy metals, etc.:

Al (Aluminium)*	mg/kg (≤ 50)
As (Arsenic)	mg/kg ($\leq 2,5$)
Cd (Cadmium)	mg/kg (≤ 2)
Co (Cobalt)	mg/kg (≤ 2)
Cr (Chromium)	mg/kg (≤ 5)
Cu (Copper)	mg/kg (≤ 5)
Hg (Mercury)	mg/kg ($\leq 0,5$)
Ni (Nickel)	mg/kg (≤ 5)
Pb (Lead)	mg/kg (≤ 5)
Zn (Zinc)	mg/kg (≤ 20)
Hydrocarbons*	mg/kg (≤ 100)
TOC*	mg/kg

*data necessary if the product is not of natural origin

Product specification for liquid magnesium chloride (results of supplier tests; requirements in brackets)

- MgCl₂:.....Weight % (specified concentration by purchaser)

- Other chlorides:.....Weight % (max. 5)

- Sulphate:Weight % (max. 1,0)

- Water insoluble matter:Weight % (max. 0,1)

- pH:(5 - 11)

- **Density (20 °C):**.....kg/dm³

- **Heavy metals, etc.:**

Al (Aluminium)*	mg/kg (≤ 50)
As (Arsenic)	mg/kg (≤ 2,5)
Cd (Cadmium)	mg/kg (≤ 2)
Co (Cobalt)	mg/kg (≤ 2)
Cr (Chromium)	mg/kg (≤ 5)
Cu (Copper)	mg/kg (≤ 5)
Hg (Mercury)	mg/kg (≤ 0,5)
Ni (Nickel)	mg/kg (≤ 5)
Pb (Lead)	mg/kg (≤ 5)
Zn (Zinc)	mg/kg (≤ 20)
Hydrocarbons*	mg/kg (≤ 100)
TOC*	mg/kg

*data necessary if the product is not of natural origin

Other information (additives, etc.):

.....

.....
Date, signature and stamp of supplier

A.3 Product description for blends of calcium chloride and magnesium chloride with sodium chloride and/or potassium chloride

Product

Trade name

Origin from flue gas cleaning (e.g. waste incinerators, etc.):

Yes No

Supplier

Product specification for solid blends (results of supplier tests; requirements in brackets)

Compacted material Yes No

- **Composition:**

Calcium chloride (anhydrous):Weight %

Magnesium chloride (anhydrous):Weight %

Sodium chloride:Weight %

Potassium chloride:Weight %

Total chloride based salts:Weight %

- **Sulphate:**Weight % (max. 1,5)

- **Sieve analysis:**

Sieve size	Weight % passing test sieve		
	Un-compacted material	Compacted material	
		Flakes	Pellets, prills, granules
20 mm(100) (100)	-
10 mm (min. 95) (100)
5 mm(80-100)(80-100)
2 mm(max. 40)(0-20)
0,8 mm (0 -10) (0 - 10) (0-5)

- **pH:**(5 - 11)
- **Bulk density (loose):**kg/dm³
- **Heavy metals, etc.:**

Al (Aluminium)*	mg/kg (≤ 50)
As (Arsenic)	mg/kg (≤ 2,5)
Cd (Cadmium)	mg/kg (≤ 2)
Co (Cobalt)	mg/kg (≤ 2)
Cr (Chromium)	mg/kg (≤ 5)
Cu (Copper)	mg/kg (≤ 5)
Hg (Mercury)	mg/kg (≤ 0,5)
Ni (Nickel)	mg/kg (≤ 5)
Pb (Lead)	mg/kg (≤ 5)
Zn (Zinc)	mg/kg (≤ 20)
Hydrocarbons*	mg/kg (≤ 100)
TOC*	mg/kg

*data necessary if the product is not of natural origin

Product specification for liquid blends (results of supplier tests; requirements in brackets)

- **Composition:**
 - Calcium chloride:Weight %
 - Magnesium chloride:Weight %
 - Sodium chloride:Weight %
 - Potassium chloride:Weight %
 - Total chloride based salts:Weight % (min. 16)
- **Sulphate:**Weight % (for blends with CaCl₂ max. 0,5)
 -Weight % (for blends with MgCl₂ max. 1,0, measured in a 10 weight % solution)
- **Water insoluble matter:** Weight % (max. 0,2)
- **pH:**(5 - 11)
- **Density (20 °C):**.....kg/dm³
- **Heavy metals, etc.:**

Al (Aluminium)*	mg/kg (≤ 50)
As (Arsenic)	mg/kg (≤ 2,5)
Cd (Cadmium)	mg/kg (≤ 2)
Co (Cobalt)	mg/kg (≤ 2)
Cr (Chromium)	mg/kg (≤ 5)
Cu (Copper)	mg/kg (≤ 5)

Hg (Mercury)	mg/kg ($\leq 0,5$)
Ni (Nickel)	mg/kg (≤ 5)
Pb (Lead)	mg/kg (≤ 5)
Zn (Zinc)	mg/kg (≤ 20)
Hydrocarbons*	mg/kg (≤ 100)
TOC*	mg/kg

*data necessary if the product is not of natural origin

Other information (additives, etc.):

.....

.....

.....

.....

Date, signature and stamp of supplier

Annex B (normative)

Sampling

B.1 Solid form

B.1.1 Package shipments

Select not less than three containers at random from the shipment. For bags, take 1,0 kg samples by means of a sampling tube or other method that will ensure a sample that is representative of the material in the bag. Penetrate with the sampling tube or other method at least 300 mm into the bag.

B.1.2 Bulk shipments

For solid products in bulk lots the sampling shall be in accordance with the methods described in EN 932-1.

Select samples from at least three locations in the shipment. Scrape aside the top layer to a depth of approximately 300 mm. Use a sampling tube or other method according to EN 932-1, to obtain a sample extending from the cleared surface to a maximum depth. Each sample shall contain at least 1,0 kg of material.

Use caution during the sampling operation to avoid exposing the sample unduly to atmospheric moisture. Immediately and thoroughly mix the individual samples to form a representative composite sample of material and store in a sealed glass or suitable plastic container.

B.2 Liquid form

Obtain a sample of at least 500 ml from the bulk shipping container or storage tank or during discharge. Recirculate the solution in the tank until it is homogenous, and then take one or more samples by means of an appropriate sampling device.

Use caution during the sampling operation to avoid exposing the sample unduly to atmospheric moisture. If more than one sample is taken, immediately and thoroughly mix the individual samples to form a representative composite sample of material and store in a sealed glass or suitable plastic container.

B.3 Labelling and distribution of samples

The container with the samples shall have labels as follows:

- trade name of the product;
- address of the point of sampling;
- date of the sampling;
- number of the sampling report.

The sample shall be sent to the test laboratory. If more than one sample is taken, the other samples are for tests by the supplier and used as retain samples.

B.4 Sampling report

The sampling report should include the following information:

- purchasing organization;
- trade name of product;
- active ingredient;
- name and address of supplier;
- date of delivery (delivery note);
- quantity of the delivered product;
- date of sampling;
- point of sampling;
- responsible organization for sampling;
- persons present during sampling;
- short description of sampling procedure;
- number of samples;
- weight of sample;
- addressee of the samples;
- requested tests;
- signature of persons present during sampling.

Annex C (normative)

Analytical methods — Determination of calcium chloride by complexometry

C.1 Scope

This test method covers the determination of calcium chloride in the range from 0 to 100 weight % in liquid and solid products.

C.2 Principle

Calcium in an alkaline solution is titrated with standard ethylenediaminetetraacetate solution, using calcein as an indicator. The colour change is from green to orange.

C.3 Reagents

All reagents shall be of recognized analytical grade and the water used shall conform to grade 2 in accordance with EN ISO 3696.

C.3.1 EDTA solution, $c = 0,1$ mol/l.

Weigh 37,22 g of the dihydrated disodium salt of ethylenediaminetetraacetic (EDTA), place it in a beaker and dissolve in 600 ml to 800 ml of water. Transfer the solution quantitatively into a graduated flask. Make up the volume and mix. Check this solution with the standard solution (1 ml = 0,008 32 g CaCl₂) by transferring a 50 ml aliquot of the latter to a 500 ml Erlenmeyer flask and dilute to 200 ml with water. Proceed as directed in C.5.2. Calculate the CaCl₂ equivalent of the EDTA solution as follows:

$$\text{Calcium chloride equivalent, g/ml} = 0,416/A$$

where

A = millilitres of EDTA solution required for the titration of the CaCl₂ solution.

C.3.2 Calcium standard solution (1 ml = 0,008 32 g CaCl₂).

Weigh 7,500 g of dry calcium carbonate. Place it in a beaker with 150 ml of water. Progressively stir in 180 ml of hydrochloric acid (C.3.4).

Bring to the boil in order to drive off the carbon dioxide, cool, transfer quantitatively into a graduated 1 l flask, make up the volume with water and mix.

C.3.3 Calcein indicator.

Carefully mix in a mortar 1 g of calcein with 100 g sodium chloride. Use 10 mg of this mixture. The indicator changes from green to orange. Titration shall be carried out until an orange is obtained which is free from green tinges.

C.3.4 Hydrochloric acid solution, approximately $c = 1$ mol/l.

C.3.5 Hydrochloric acid solution (1 + 3).

Mix 1 volume of concentrated hydrochloric acid (specific weight 1,19 g/ml) and 3 volumes of water.

C.3.6 Hydroxylamine hydrochloride solution (10 weight %).

Dissolve 10 g of hydroxylamine hydrochloride in 90 ml of water.

C.3.7 Potassium Cyanide.**C.3.8 Sodium hydroxide solution.**

Add slowly 80 g sodium hydroxide (NaOH) in 300 ml of water stirring constantly. Cool, transfer to a 1 l volumetric flask, dilute to volume with water, and mix.

C.3.9 Sugar.**C.4 Apparatus**

Usual laboratory equipment.

C.5 Procedure**C.5.1 Solid samples**

Weigh 100,0 g of sample and wash into a 1 000 ml volumetric flask with water. Add 10 ml of hydrochloric acid (C.3.5) and swirl to dissolve the sample. Cool to room temperature, make to volume with water, and mix. Pipet a 20 ml aliquot into a 500 ml volumetric flask, dilute to volume, and mix. Proceed as in C.5.3.

C.5.2 Liquid samples

Weigh 100,0 g of sample and wash into a 1 000 ml volumetric flask with water. Add 10 ml of hydrochloric acid (C.3.5) and mix. Cool to room temperature, make up to volume with water, and mix. Pipet an aliquot containing about 2 g of CaCl₂ into a 500 ml volumetric flask, dilute to volume, and mix. Appropriate aliquot volumes are indicated in the table below. Interpolate if necessary.

Table C.1 — Appropriate aliquot volumes

Expected CaCl ₂ concentration (weight %)	Aliquot size (ml)
10	200
20	100
30	75
40	50

C.5.3 Titration

Pipet a 100 ml aliquot of the solution prepared in C.5.1 or C.5.2 into a 500 ml Erlenmeyer flask and dilute to about 200 ml with water. Add in order 10 ml of hydroxylamine hydrochloride (C.3.6) and 3 g of sugar. Swirl to dissolve. Add 40 ml of NaOH solution (C.3.8) and swirl to mix. Add 0,1 g of KCN, and swirl

to dissolve and mix. Add about 0,2 g of calcein indicator (C.3.3). Titrate with 0,1 mol/l EDTA solution until the indicator changes from green to orange.

C.6 Calculation

Calculate the calcium chloride concentration as follows:

$$W_{\text{CaCl}_2} = \left[\left(\frac{A \cdot B}{C} \right) \right] \cdot 100 \quad (1)$$

where

- W_{CaCl_2} concentration of calcium chloride in weight %
- A millilitres of EDTA solution required for titration of the sample, ml
- B calcium chloride equivalent of the EDTA solution, g/ml
- C mass of sample in the aliquot used, g

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