

# **SLOVENSKI STANDARD**

## **SIST EN 12485:2011**

**01-november-2011**

**Nadomešča:**  
**SIST EN 12485:2001**

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**Kemikalije, ki se uporabljajo za pripravo pitne vode - Kalcijev karbonat, visoko kalcijevo apno in polžgan dolomit, magnezijev oksid in kalcij-magnezijev karbonat - Preskusne metode**

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

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Calciumcarbonat, Weißkalk und halbgebrannter Dolomit - Analytische Verfahren

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

**Ta slovenski standard je istoveten z: EN 12485:2010**

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EUROPEAN STANDARD  
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**EN 12485**

April 2010

ICS 71.100.80

Supersedes EN 12485:2001

English Version

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

Produits chimiques utilisés pour le traitement de l'eau  
destinée à la consommation humaine - Carbonate de  
calcium, chaux et dolomie semi calcinée - Méthodes  
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Produkte zur Aufbereitung von Wasser für den  
menschlichen Gebrauch - Calciumcarbonat, Weißkalk,  
halbgebrannter Dolomit, Magnesiumoxid und  
Calciummagnesiumcarbonat - Analytische Verfahren

This European Standard was approved by CEN on 18 March 2010.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

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# Contents

Page

Foreword.....	3
1 Scope .....	4
2 Normative references .....	4
3 General requirements.....	4
3.1 Number of determinations .....	4
3.2 Methods for analysis .....	4
3.3 Sample preparation .....	6
3.4 Reagents.....	6
3.5 Glassware .....	7
3.6 Expression of results .....	7
3.7 Repeatability and reproducibility limits .....	8
3.8 Test report .....	8
4 Determination of screen oversize of high-calcium lime .....	8
4.1 Air-jet sieving method .....	8
4.2 Wet sieving method .....	10
5 Preparation of test solutions.....	12
5.1 Fusion with lithium tetraborate .....	12
5.2 Extraction with hydrochloric acid .....	13
5.3 Microwave digestion with nitric acid .....	14
5.4 Pressurized digestion with nitric acid .....	14
6 Conventional methods of determining major and minor constituents .....	15
6.1 Determination of free water .....	15
6.2 Loss on ignition at 450 °C .....	16
6.3 Determination of carbon dioxide.....	17
6.4 Determination of residue insoluble in hydrochloric acid .....	18
6.5 Determination of content of water soluble calcium oxide or calcium hydroxide (reference method).....	19
6.6 Determination of sugar soluble calcium oxide or calcium hydroxide (alternative method).....	21
6.7 Water-insoluble matter.....	23
6.8 Determination of free CaO .....	24
6.9 Determination of calcium oxide and magnesium oxide .....	25
6.10 Determination of sulfate.....	28
6.11 Determination of solubility index by conductivity .....	29
6.12 Calculation of the composition of a commercial product .....	32
7 Determination of constituents by modern techniques .....	34
7.1 Determination of minor constituents by AAS flame technique .....	34
7.2 Determination of major and minor constituents by ICP-OES .....	38
8 Determination of trace elements .....	43
8.1 Determination of lead, cadmium, chromium and nickel by AAS graphite tube technique .....	43
8.2 Determination of lead, cadmium, chromium and nickel by ICP-OES.....	49
8.3 Determination of arsenic, antimony and selenium by AAS hydride technique .....	53
8.4 Determination of mercury by cold-vapour technique .....	56
Annex A (informative) Analytical scheme.....	60
Annex B (informative) Table B.1: Precision data for the test methods .....	66
Bibliography.....	67

## Foreword

This document (EN 12485:2010) has been prepared by Technical Committee CEN/TC 164 "Water supply", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2010, and conflicting national standards shall be withdrawn at the latest by October 2010.

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.

This document supersedes EN 12485:2001.

Annexes A and B are informative.

Significant technical differences between this edition and EN 12485:2001 are as follows:

- a) addition of a method for determination of sugar-soluble calcium oxide or calcium hydroxide (see 6.6);
- b) addition of a method for determination of solubility index (see 6.11).

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

## EN 12485:2010 (E)

## 1 Scope

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

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

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

NOTE Schematic diagrams of the analyses are given in Annex A (Figures A.1 to A.6).

## 2 Normative references

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

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

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

EN 1018, *Chemicals used for treatment of water intended for human consumption — Calcium carbonate*

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

prEN 16003, *Chemicals used for treatment of water intended for human consumption — Calcium magnesium carbonate*

prEN 16004, *Chemicals used for treatment of water intended for human consumption — Magnesium oxide*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

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

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

## 3 General requirements

### 3.1 Number of determinations

Two analyses shall be carried out to determine the various constituents (see Clause 5 to Clause 8, see also 3.6).

### 3.2 Methods for analysis

The methods to be used for the analysis of half-burnt dolomite, calcium carbonate, high calcium lime, magnesium oxide and calcium magnesium carbonate and the principle of each method are listed in Table 1.

The requirement values for free MgO and free  $\text{Mg}(\text{OH})_2$  in half-burnt dolomite shall be expressed as free MgO in accordance with EN 1017. The same requirement is related to free CaO. The requirement value for MgO in magnesium oxide shall be expressed as MgO in dry substance in accordance with prEN 16004. Therefore,

the analysis of half-burnt dolomite and magnesium oxide shall be performed on a sample which is bound-water free. Before starting the chemical analysis, the loss on ignition at 450 °C shall be determined as described in 6.2. The analysis itself shall be performed on the material obtained after the determination of the loss on ignition.

The requirement values for calcium carbonate, hydrated lime and calcium magnesium carbonate shall be expressed in dry substance in accordance with EN 1018, EN 12518 and prEN 16003. Therefore, the analysis of these products shall be performed on a sample which has been dried in accordance with the procedure described in 6.1.

For the determination of the water-soluble content of high-calcium lime, the method in 6.5 is considered as the reference method. The sugar method described in 6.6 can be used as an alternative method if it is demonstrated that the results obtained with this method are equivalent to those of the reference method.

**Table 1 — Methods for analysis**

Determination	Method	Principle	Standard
Screen oversize	4.1	Air-jet sieving	EN 12518
	4.2	Wet sieving	EN 12518
Free water	6.1	Gravimetry	EN 1018, EN 12518, prEN 16003
Loss on ignition at 450 °C	6.2	Gravimetry	EN 1017, EN 12518, prEN 16004
Carbon dioxide	6.3	Gravimetry	EN 1017, EN 12518, prEN 16004
Residue insoluble in hydrochloric acid	6.4	Acidimetry, Gravimetry	EN 1018, prEN 16004
Water-soluble calcium oxide or calcium hydroxide	6.5	Acidimetric titration	EN 12518
Sugar-soluble calcium oxide or calcium hydroxide	6.6	Acidimetric titration	EN 12518
Water-insoluble constituents	6.7	Gravimetry	EN 12518
Free calcium oxide	6.8	Extraction, acidimetric titration	EN 1017, prEN 16004
Calcium oxide and magnesium oxide	6.9	Complexometric titration	EN 1017, EN 1018, prEN 16003, prEN 16004
Sulfate	6.10	Gravimetry	EN 1017
Solubility index	6.11	Conductivity	EN 12518
Magnesium	7.1 or 7.2	AAS (flame) or ICP-OES	EN 1017, EN 1018, prEN 16003, prEN 16004
Silicium, Aluminium, Iron,	7.1 or 7.2	AAS (flame) or ICP-OES	EN 1017, EN 12518, prEN 16003, prEN 16004

Table 1 (continued)

Determination	Method	Principle	Standard
Manganese	7.2	ICP-OES	EN 12518
Sulfur	7.2	ICP-OES	EN 1017
Lead, Cadmium, Chromium, Nickel	8.1 or 8.2	AAS (flameless) or ICP-OES	EN 1017, EN 1018, EN 12518, prEN 16003, prEN 16004
Arsenic, Antimony, Selenium	8.3	AAS (hydride)	EN 1017, EN 1018, EN 12518, prEN 16003, prEN 16004
Mercury	8.4	AAS (cold-vapour technique)	EN 1017, EN 1018, EN 12518, prEN 16003, prEN 16004

### 3.3 Sample preparation

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

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

When sampling milk of lime, the material from which the sample is to be taken shall be thoroughly mixed with an electrically driven stirrer of adequate power. The milk of lime shall be dried at  $(105 \pm 5) ^\circ\text{C}$  (see 6.1) before being analysed chemically.

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

### 3.4 Reagents

All reagents shall be of a recognized analytical grade appropriate for the method being used. The water used shall conform to grade 3 in accordance with EN ISO 3696:1995 unless otherwise specified in the method. The concentration of the analytes in the water and reagents shall be negligible compared with the lowest concentration to be determined.

The concentrated liquids used for the reagents in this standard have the following densities ( $\rho$ ) (in grams per millilitre at  $20 ^\circ\text{C}$ ):

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

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



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

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

This European Standard makes no stipulations relating to the shelf life of stock, standard and reference solutions. In the case of stock solutions having an element concentration of 1 g/l, the manufacturer generally specifies a shelf life of one year. It is advisable to check the calibration solutions regularly.

### 3.5 Glassware

Glass containers and pipettes shall be cleaned with hot dilute nitric acid immediately before use and then rinse with water. If determining trace elements rinse with grade 2 water.

### 3.6 Expression of results

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

#### EXAMPLE

(CaO) = 91,2 %;

(SiO<sub>2</sub>) = 3,70 %;

(MnO<sub>2</sub>) = 0,15 %;

(Cr) = 0,32 mg/kg;

(Hg) = 0,05 mg/kg.

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https://standards.iteh.ai/catalog/standards/sist/e89d845b-0722-4a95-b561-a6a038cbd8fa/sist-en-12485-2011](https://standards.iteh.ai/catalog/standards/sist/e89d845b-0722-4a95-b561-a6a038cbd8fa/sist-en-12485-2011)

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

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

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

Table 2 — Methods for calculation of the results

Requirements from standard	Parameter	Required value for	Calculation described in
EN 1017	CaO <sub>free</sub> , CaO CO <sub>2</sub> MgO	CaCO <sub>3</sub> MgCO <sub>3</sub> MgO <sub>free</sub>	6.12.2.3
EN 1018	CaO MgO	CaCO <sub>3</sub> MgCO <sub>3</sub>	6.12.2.1
EN 12518	CO <sub>2</sub>	CaCO <sub>3</sub>	6.12.2.4
prEN 16004	MgO, loss on ignition at (1 000 ± 50) °C and (450 ± 25) °C	(CO <sub>2</sub> and H <sub>2</sub> O)	6.12.2.5
prEN 16003	MgO	CaMg(CO <sub>3</sub> ) <sub>2</sub>	6.12.2.2

### 3.7 Repeatability and reproducibility limits

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

### 3.8 Test report

The report shall contain the following information:

- a reference to the method used; [SIST EN 12485:2011](https://standards.iteh.ai/catalog/standards/sist/e89d845b-0722-4a95-b561-a6a038cbd8fa/sist-en-12485-2011)
- a complete identification of the sample; <https://standards.iteh.ai/catalog/standards/sist/e89d845b-0722-4a95-b561-a6a038cbd8fa/sist-en-12485-2011>
- results obtained and the method of expression used (see 3.6)
- sample pretreatment, e.g. method of digestion (see Clause 5);
- any deviation from this standard and an indication of any circumstances which can have affected the results.

## 4 Determination of screen oversize of high-calcium lime

### 4.1 Air-jet sieving method

#### 4.1.1 General

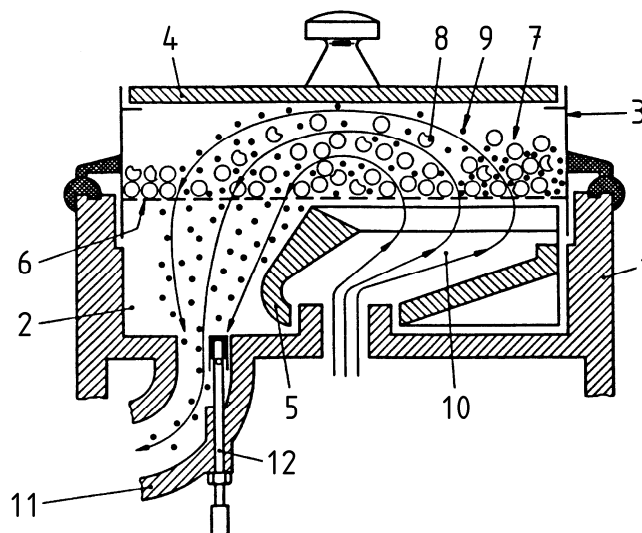
The method is used to determine the retention on sieving of high-calcium quicklime and hydrated lime as specified in EN 12518.

The particle size distribution of high-calcium quicklime and hydrated lime can be determined using airjet sieving apparatus.

For quicklime, the method is suitable for particles which substantially pass a 2,0 mm test sieve. For hydrated lime, the method can be used to determine the particle size distribution of the agglomerates of very fine particles produced by the manufacturing process. This method uses test sieves with aperture sizes of 0,60 mm and 0,09 mm.

#### 4.1.2 Apparatus

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



#### Key

- 1 housing
- 2 dish
- 3 sieve drum
- 4 lid
- 5 slit-nozzle
- 6 sieve

- 7 test sample
- 8 oversize material
- 9 undersize material
- 10 air jet
- 11 air discharge
- 12 pressure gauge socket, with dust hood

**Figure 1 — Air-jet sieving apparatus**

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

**NOTE** The effective operation of some makes of air-jet apparatus can require non-standard sieve frames and additional gaskets. This is permissible, provided the sieving medium and general method of construction comply with the requirements of this standard.

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

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

**4.1.2.5** Soft brush.

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

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

**4.1.2.8** Drying oven (optional), thermostatically controlled to maintain a temperature of  $(105 \pm 5) ^\circ\text{C}$ .

## EN 12485:2010 (E)

## 4.1.3 Procedure

Weigh to the nearest 1 mg ( $10 \pm 0,5$ ) g of quicklime ( $m$ ) or ( $25 \pm 0,5$ ) g of hydrated lime ( $m$ ). Fit the test sieve with the aperture size 0,09 mm into the apparatus and transfer all of the test portion onto the sieve mesh. Take care not to lose any of the test portion.

Fit the lid and switch on the apparatus. Check that the vacuum created is above the minimum value stated in the manufacturer's instructions, and that the slit nozzle is rotating properly.

If material adheres to the lid of the apparatus, gently tap the centre of the lid with the mallet.

If the material agglomerates under the action of the air-jet, interrupt the sieving process, and break up the agglomerates with the soft brush.

After ( $5 \pm 0,2$ ) min, switch off the apparatus and carefully remove the sieve. Transfer the material retained on the sieve into a tray or other suitable container. Carefully clean the mesh of the sieve over the tray using a soft brush.

Determine the mass of the residue, including the material brushed from the sieve mesh, and record the mass to the nearest 1 mg.

Refit the sieve into the apparatus and transfer all of the residue back to the sieve mesh. Repeat the weighing and sieving stages until the sieving end-point has been achieved, and record the end-point mass to the nearest 1 mg. The sieving end-point is defined as being when not more than 0,2 % of the mass of the original test portion passes through the sieve in 1 min.

Fit the 0,60 mm test sieve into the apparatus, and repeat the weighing and sieving stages until the mass of the residue confirms that the sieving end-point has been reached. Record the end-point mass to the nearest 1 mg.

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#### 4.1.4 Expression of results

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

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

where

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

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

## 4.2 Wet sieving method

### 4.2.1 General

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

### 4.2.2 Apparatus

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

**4.2.2.2 500 ml wide-mouthed bottle, or other suitable vessel** which can be sealed with a stopper.

**4.2.2.3 200 mm diameter 0,60 mm and 0,09 mm aperture size test sieves.**

**4.2.2.4 5 mm bore rubber or plastics tubing.**

**4.2.2.5 Ventilated drying oven** (optional), **thermostatically controlled** to maintain a temperature of  $(105 \pm 5) ^\circ\text{C}$ .

**4.2.2.6 Two sintered glass filter crucibles, of porosity 2** (as specified in ISO 4793:1980) (maximum porosity diameter  $40 \mu\text{m}$  to  $100 \mu\text{m}$ ) with filter flask and adapter funnel for crucibles.

**4.2.2.7 Desiccator.**

**4.2.2.8 Wash bottle.**

**4.2.2.9 Filtration apparatus.**

### 4.2.3 Procedure

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

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

Wash the residue on the 0,09 mm mesh test sieve for  $(5 \pm 0,2)$  min. Transfer the residue to a tared filter crucible, dry at  $(105 \pm 5) ^\circ\text{C}$  until constant mass is reached, cool and reweigh as described above.

### 4.2.4 Expression of results

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

$$m(P_{0,60 \text{ or } 0,09}) = \frac{m_1 \times 100}{m} \quad (2)$$

where

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

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

## EN 12485:2010 (E)

## 5 Preparation of test solutions

### 5.1 Fusion with lithium tetraborate

#### 5.1.1 General

Fusion with lithium tetraborate is used to dissolve:

- high-calcium lime as specified in EN 12518 in order to determine silicon, aluminium, iron and manganese;
- half-burnt dolomite as specified in EN 1017 in order to determine magnesium, silicon, aluminium and iron;
- calcium carbonate as specified in EN 1018 in order to determine calcium and magnesium;
- magnesium oxide as specified in prEN 16004 in order to determine magnesium, silicon, aluminium and iron;
- calcium magnesium carbonate as specified in prEN 16003 in order to determine calcium and magnesium.

#### 5.1.2 Principle

After fusing the sparingly soluble oxide constituents of the lime sample with lithium tetraborate, the fusion cake is dissolved in hydrochloric acid and the solution made up to 250 ml in a volumetric flask. Magnesium, silicon, aluminium, iron and manganese are determined in aliquot parts of this solution by flame-AAS (7.1) or ICP-OES (7.2).

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#### 5.1.3 Reagents

- 5.1.3.1 Lithium tetraborate,  $\text{Li}_2\text{B}_4\text{O}_7$**  SIST EN 12485:2011  
<https://standards.iteh.ai/catalog/standards/sist/e89d845b-0722-4a95-b561-a6a038cbd8fa/sist-en-12485-2011>
- 5.1.3.2 Hydrochloric acid,  $\rho = 1,16 \text{ g/ml}$ .**
- 5.1.3.3 Hydrochloric acid, diluted, (1 + 5).**

#### 5.1.4 Apparatus

Ordinary laboratory apparatus and the following:

- 5.1.4.1 Platinum-gold crucible.**
- 5.1.4.2 Hot plate.**

#### 5.1.5 Procedure

Weigh  $(0,25 \pm 0,02) \text{ g}$  to the nearest 0,1 mg of the sample into a platinum-gold crucible and add 1,5 g of lithium tetraborate (5.1.3.1). Roast at  $(1\,000 \pm 25) ^\circ\text{C}$  until the melt is clear. Remove from the heat, cover the crucible with a watch glass and stand the crucible in water for a few seconds, remove and allow it to stand until it has cooled completely.

Transfer the cold melt to a beaker using approximately 50 ml of hydrochloric acid (5.1.3.3) and dissolve while stirring and heating at  $100 ^\circ\text{C}$ . Then transfer the solution to a 250 ml volumetric flask with water (3.4) and, after cooling to room temperature, add each 10 ml of the appropriate reagent solutions (7.1.2.6 and 7.1.2.7 for the AAS measurement) or 1 ml of scandium solution (7.2.2.2, internal standard solution for the ICP measurement) and make up to the mark with water (digestion solution (I) for AAS and (II) for ICP).

## 5.2 Extraction with hydrochloric acid

### 5.2.1 General

Extraction with hydrochloric acid is used to dissolve calcium carbonate as specified in EN 1018 or calcium magnesium carbonate as specified in prEN 16003 in order to determine calcium and magnesium.

### 5.2.2 Principle

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

### 5.2.3 Reagents

**5.2.3.1 Hydrochloric acid**,  $\rho = 1,16$  g/ml.

**5.2.3.2 Hydrogen peroxide solution**,  $c(\text{H}_2\text{O}_2) = 30\%$  (m/m).

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

**5.2.3.4 Ammonium hydroxide solution**,  $c(\text{NH}_3) = 25\%$  (m/m).

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

**5.2.3.6 Ammonium chloride** ( $\text{NH}_4\text{Cl}$ ).

### 5.2.4 Apparatus

Ordinary laboratory apparatus and the following:  
<https://standards.iteh.ai/catalog/standards/sist/e89d845b-0722-4a95-b561-a0a058c0d01a/sist-en-12485-2011>

**5.2.4.1 Hot plate.**

**5.2.4.2 Magnetic stirrer and magnetic rod.**

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

### 5.2.5 Procedure

Weigh  $(1 \pm 0,1)$  g of the sample to the nearest 1 mg, transfer it to a 250 ml beaker, moisten with 10 ml of water, then gradually add 30 ml of hydrochloric acid (5.2.3.1). Make the solution up to about 100 ml with water then boil it for 10 min. After boiling, filter the solution immediately through a fluted filter paper (particle retention size 2,5  $\mu\text{m}$ ) into a 400 ml beaker and wash the residue well with water.

Add about 4 g of ammonium chloride (5.2.3.6) and a few drops of hydrogen peroxide (5.2.3.3) to the solution, then dilute with about 150 ml of water and heat to boiling. During boiling, add ammonium hydroxide solution (5.2.3.4) to adjust the pH value to 6 to 7 and precipitate aluminium hydroxides and iron hydroxides and the silicic acid.

Continue boiling for 3 min and, after the precipitate has settled, filter the solution immediately through a fluted filter paper (particle retention size 2,5  $\mu\text{m}$ ) into a 500 ml volumetric flask. Wash the filter residue three times with ammonium hydroxide solution (5.2.3.5) and three times with water. After the solution has cooled to room temperature, make the solution up to the mark with water and shake the contents of the flask thoroughly (digestion solution III).