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

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

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

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

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This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 164.

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prEN 12485:2023 (E)**European foreword**

This document (prEN 12485:2023) has been prepared by Technical Committee CEN/TC 164 “Water supply”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 12485:2010.

Annexes A and B are informative. For further information on methodology see [2] to [6].

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

- a) Table 1 – XRF methods are added;
- b) changed for requirements of water quality;
- c) loss on ignition 550 °C changed to 500 °C;
- d) unglazed porcelain or platinum crucible changed in glazed porcelain or platinum crucible.

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1 Scope

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

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

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.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

EN 1017:2014+A1:2017, *Chemicals used for treatment of water intended for human consumption — Half-burnt dolomite*

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

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

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

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

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

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

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

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

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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4 General requirements

4.1 Number of determinations

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

4.2 Methods for analysis

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

The requirement values for free MgO and free Mg(OH)₂ in half-burnt dolomite shall be expressed as free MgO in accordance with EN 1017. The same requirement is related to free CaO. The requirement value for MgO in magnesium oxide shall be expressed as MgO in dry substance in accordance with EN 16004. Therefore, the analysis of half-burnt dolomite and magnesium oxide shall be performed on a sample which is bound-water free. Before starting the chemical analysis, the loss on ignition at 550 °C shall be determined as described in 7.2. The analysis itself shall be performed on the material obtained after the determination of the loss on ignition.

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

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

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

Table 1 — Methods for analysis

Determination	Method	Principle	Standard
Screen oversize	5.1	Air-jet sieving	EN 12518, EN 16409
	5.2	Wet sieving	EN 12518
Free water	7.1	Gravimetry	EN 1018, EN 12518, EN 16003, EN 16409
Loss on ignition at 500 °C	7.2	Gravimetry	EN 1017, EN 12518, EN 16004, EN 16409
Carbon dioxide	7.3	Gravimetry	EN 1017, EN 12518, EN 16004, EN 16409
Residue insoluble in hydrochloric acid	7.4	Acidimetry, Gravimetry	EN 1018, EN 16003
Water-soluble calcium oxide or calcium hydroxide	7.5	Acidimetric titration	EN 12518

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

4.3 Sample preparation

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

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

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When sampling milk of lime, the material from which the sample is to be taken shall be thoroughly mixed with an electrically driven stirrer of adequate power. The milk of lime shall be dried before being analysed chemically as described in 6.1.

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

4.4 Reagents

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

EN ISO 3696 defines three types of water quality (essential requirements given below):

- Grade 3 suitable for most laboratory wet chemistry work (electrical conductivity < 0,5 mS/m (at 25 °C));
- Grade 2 very low in contaminants and suitable for sensible analytical work (electrical conductivity < 0,1 mS/m (at 25 °C));
- Grade 1 essentially free from contaminants and suitable for most stringent analytical requirements (electrical conductivity < 0,01 mS/m (at 25 °C));
- In all cases the concentration of the element(s) to be determined shall be low enough not to influence the results of the analysis. This can be verified by analysing the blank solution.

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

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

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

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

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

4.5 Glassware

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

4.6 Expression of results

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

EXAMPLE:

- (CaO) = 91,2 %;
- (SiO₂) = 3,70 %;
- (MnO₂) = 0,15 %;
- (Cr) = 0,32 mg/kg;
- (Hg) = 0,05 mg/kg.

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

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

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

Table 2 — Methods for calculation of the results

Requirements from standard	Parameter	Required value for	Calculation described in
EN 1017	CaO _{free} , CaO CO ₂ MgO	CaCO ₃ MgCO ₃ MgO _{free}	7.12.2.3
EN 1018	CaO MgO	CaCO ₃ MgCO ₃	7.12.2.1
EN 12518	CO ₂	CaCO ₃	7.12.2.4
EN 16004	MgO, loss on ignition at (1 050 ± 25) °C and (500 ± 25) °C	(CO ₂ and H ₂ O)	7.12.2.5
EN 16003	MgO	CaMg(CO ₃) ₂ MgCO ₃	7.12.2.2
EN 16409	CaO, MgO	CaO + MgO	7.12.2.6

4.7 Repeatability and reproducibility limits

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

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4.8 Test report

The report shall contain the following information:

- a reference to the method used;
- a complete identification of the sample;
- results obtained and the method of expression used (see 4.6)
- sample pretreatment, e.g. method of digestion (see Clause 6);
- any deviation from this standard and an indication of any circumstances which can have affected the results.

5 Determination of screen oversize of high-calcium lime

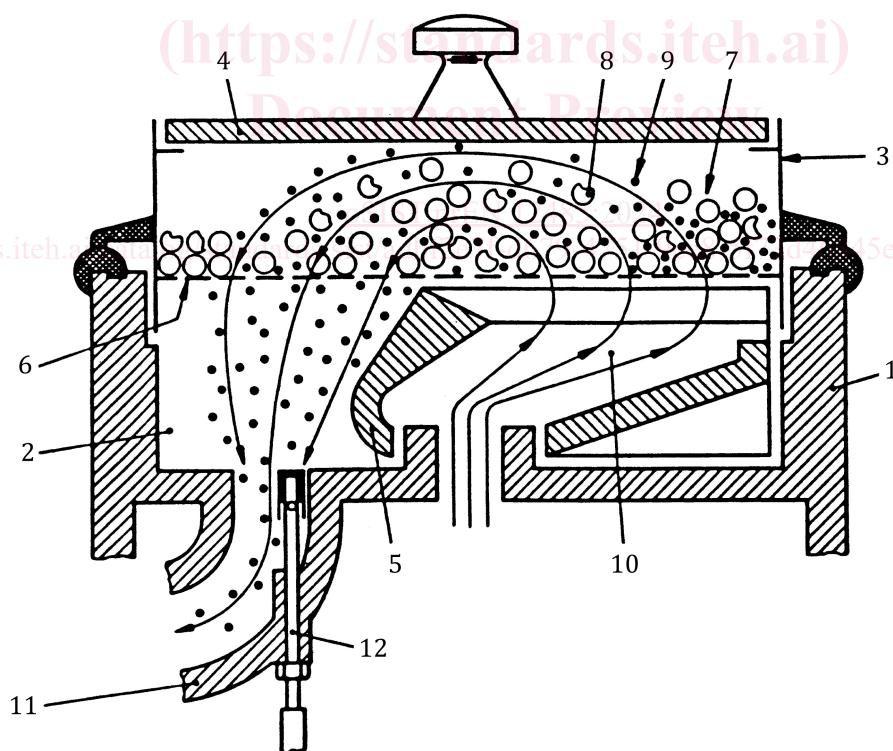
5.1 Air-jet sieving method

5.1.1 General

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

5.1.2 Apparatus

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



Key

1	housing	7	test sample
2	dish	8	oversize material
3	sieve drum	9	undersize material

4	lid	10	air jet
5	slit-nozzle	11	air discharge
6	sieve	12	pressure gauge socket, with dust hood

Figure 1 — Air-jet sieving apparatus

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

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

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

5.1.2.4 Balance, accurate to 0,1 mg.

5.1.2.5 Soft brush.

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

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

5.1.3 Procedure

Weigh an appropriate amount (between 10 g and 50 g pending on the type of material) of the dry sample to the nearest 1 mg (m). Fit the test sieve with the aperture size 0,09 mm into the apparatus and transfer all of the test portion onto the sieve mesh. Take care not to lose any of the test portion.

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

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

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

After $(5 \pm 0,2)$ 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.

5.1.4 Expression of results

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

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

where

m is the mass of the sample, in grams;

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

5.2 Wet sieving method**5.2.1 General**

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

5.2.2 Apparatus

5.2.2.1 Balance, accurate to 0,1 mg.

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

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

5.2.2.4 5 mm bore rubber or plastics tubing.

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

5.2.2.6 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.

5.2.2.7 Desiccator.

5.2.2.8 Wash bottle.

5.2.2.9 Filtration apparatus.

5.2.3 Procedure

Superimpose the 0,60 mm mesh test sieve on the 0,09 mm mesh test sieve and wet the sieves with water. Place a volume of milk of lime containing $(50 \pm 0,5)$ 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.