



SLOVENSKI STANDARD

SIST EN 12485:2001

01-december-2001

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

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

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

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

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Ta slovenski standard je istoveten z: EN 12485:2001

ICS:

13.060.20	Pitna voda	Drinking water
71.100.80	Kemikalije za čiščenje vode	Chemicals for purification of water

SIST EN 12485:2001

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EUROPEAN STANDARD

EN 12485

NORME EUROPÉENNE

EUROPÄISCHE NORM

April 2001

ICS 71.100.80

English version

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

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

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This European Standard was approved by CEN on 21 January 2001.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This European Standard 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of an identical text or by endorsement, at the latest by October 2001, and conflicting national standards shall be withdrawn at the latest by October 2001.

The annexes A and B are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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1 Scope

This European standard describes methods used of the analyses of calcium carbonate, high-calcium lime and half-burnt dolomite used to treat water for human consumption.

NOTE Of the reference methods described, the atomic spectroscopy methods are preferred to the conventional methods. If methods other than those described are used, it should be demonstrated that the results are equivalent to those of the reference methods.

The schematic diagram of the chemical analysis is given in annex A (Figures A.1 to A.4).

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 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*.

EN ISO 3696, *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, *Laboratory sintered (fritted) filters - Porosity grading, classification and designation*.

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results - Part 2 : Basic method for the determination of repeatability and reproducibility of a standard measurement method*.

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 analysis of half-burnt dolomite, calcium carbonate and high calcium lime and the principle of each method are listed in Table 1.

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 12518
Carbon dioxide	6.2	Gravimetry	EN 1017, EN 12518
Residue insoluble in hydrochloric acid	6.3	Acidimetry, Gravimetry	EN 1018
Water-soluble calcium oxide or calcium hydroxide	6.4	Acidimetric titration	EN 12518
Water-insoluble constituents	6.5	Gravimetry	EN 12518
Free calcium oxide	6.6	Extraction, acidimetric titration	EN 1017
Calcium and magnesium	6.7	Complexometric titration	EN 1017, EN 1018
Sulfur	6.8	Barium sulfate gravimetry	EN 1017
Magnesium, Silicium, Aluminium, Iron, Manganese	7.1	AAS (flame)	EN 1017, EN 1018
	or 7.2	or ICP-OES	EN 1017, EN 12518 EN 1017, EN 12518 EN 1017, EN 12518 EN 12518
Sulfate	7.2	ICP-OES	EN 1017
Lead, Cadmium, Chromium, Nickel	8.1	AAS (flameless)	EN 1017, EN 1018, EN 2518
	or 8.2	or ICP-OES	
Arsenic, Antimony, Selenium	8.3	AAS (hydride)	EN 1017, EN 1018, EN 12518
		AAS (hydride)	
Mercury	8.4	AAS (cold-vapour technique)	EN 1017, EN 1018, EN 12518

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 1l.

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 ± 5) °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 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 (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 shall be prepared in accordance with the instructions given in this standard.

This 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 using a mean-value control chart (see 3.9). This is the most reliable method of detecting errors in calibration due, for example, to:

- incorrect preparation of the standards and of the reagents ;
- dilution errors ;
- ageing of standards, reagents and reference material ; and
- increase in concentration as a result of evaporation.

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 per cent by mass, c (% (m/m)), while those for trace elements shall be reported, in milligrams per kilogram, as the mean of two determinations. In general, analytical values shall be reported to three significant figures.

EXAMPLES

$$c(\text{CaO}) = 91,2 \% (m/m) ;$$

$$c(\text{SiO}_2) = 3,70 \% (m/m) ;$$

$$c(\text{MnO}_2) = 0,15 \% (m/m) ;$$

$c(\text{Cr}) = 0,32 \text{ mg/kg}$;

$c(\text{Hg}) = 0,05 \text{ mg/kg}$.

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 products and in EN 1017 for half-burnt dolomites, the results have to be converted to the form in which the requirements are stated in the above standards.

The paragraphs 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}	6.9.2.2
EN 1018	CaO MgO	CaCO ₃ MgCO ₃	6.9.2.1
EN 12518	CO ₂	CaCO ₃	6.9.2.3

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 refer to the standard used and contain the following information:

- a reference to the method used ;
- a complete identification of the sample ;
- expression of the results (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.

3.9 Quality assurance procedures

The methods of chemical analysis described in this standard shall include the quality criteria listed as product requirements in the individual material standards in the form of analytical results. For this purpose, any test method shall determine correct and precise values of the criteria. This will necessitate establishing suitable in-house quality assurance procedures for every test method, particularly for the methods of determining trace elements.

These procedures shall include the following:

- standard working instructions ;

- calibration instructions ;
- keeping of quality control charts.

Standard working instructions shall record in writing how recurring laboratory examinations are to be carried out. Calibration instructions shall include the checking of :

- blanks ;
- calibration standards ;
- the accuracy of recovery ;
- measurement variations ;
- the linearity of analytical functions ; and
- working range, sensitivity of the method, of method standard deviation and of the limit of determination.

Quality control charts shall be used in the trace element analysis methods employing AAS and ICP-OES to check the accuracy. Blank-value, mean-value and accuracy-of-recovery control charts are suitable for this purpose. The blank-value control chart can be used to detect:

- impurities in the reagents ;
- impurities originating from reaction vessels and instruments ; and
- errors in instrument parameters (e.g. base-line drift, short-term stability, long-term stability).

The mean-value control chart shall be used to check the correctness:

- of the calibration with standard solutions ; and
- the control sample.

The accuracy-of-recovery control chart determines the accuracy of analytical results by including matrix effects which are revealed by a proportional and systematic error in the calibration function.

The precision of analytical results can be determined using the range control chart.

4 Determination of screen oversize of high-calcium lime

4.1 Air-jet sieving method

4.1.1 General

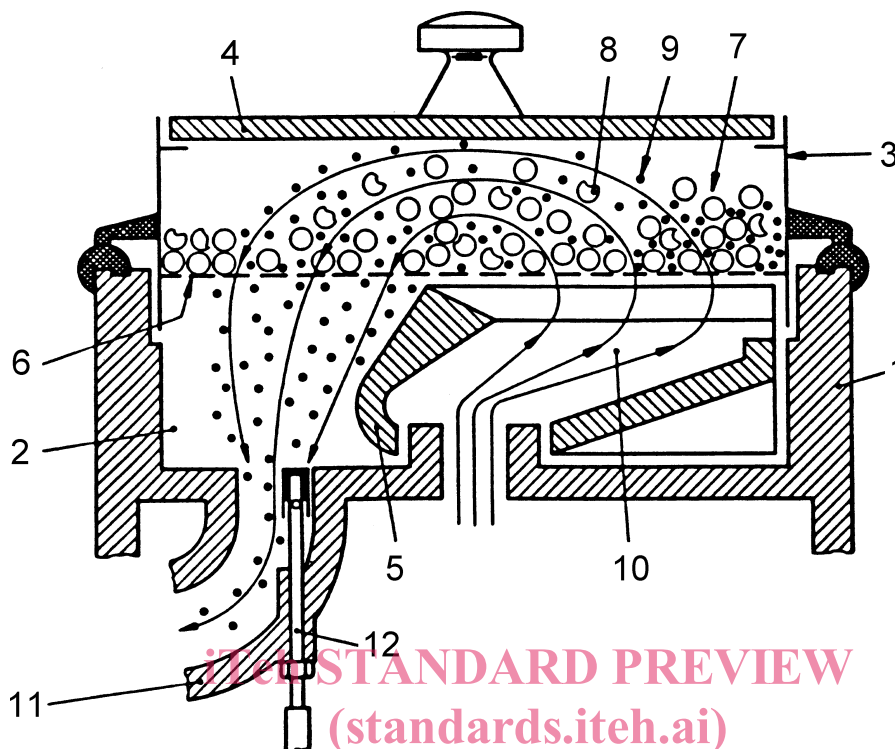
The method is used to determine the retention on sieving of high-calcium quicklime and hydrated lime as specified in EN 12518.

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.



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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

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, for cleaning the mesh of the sieves. A camel hair brush is suitable.

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

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

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 the 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.

4.1.4 Expression of results

The mass retained on each sieve expressed as a per cent by mass, $m(P_{0,60 \text{ or } 0,09})$, (% (m/m)), 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 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 ± 5) °C.

4.2.2.6 Two sintered glass filter crucibles, of porosity 2 (as specified in ISO 4793) (maximum porosity diameter 40 µm to 100 µm) 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 ± 5) °C for 1h. 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 ± 5) °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 a per cent by mass, $m(P_{0,60 \text{ or } 0,09})$, (% (m/m)), 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.

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 limes 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 ; and
- calcium carbonate as specified in EN 1018 in order to determine magnesium when the per cent by mass of MgO is less than 1 % (m/m).

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).

5.1.3 Reagents

5.1.3.1 Lithium tetraborate, $\text{Li}_2\text{B}_4\text{O}_7$.

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 $0,1 \text{ mg}$ of the sample into a platinum-gold crucible and add $1,5 \text{ g}$ of lithium tetraborate (5.1.3.1). Roast at $(1000 \pm 25) \text{ }^\circ\text{C}$ 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 $100 \text{ }^\circ\text{C}$. Then transfer the solution to a 250 ml volumetric flask with water (3.3) and, after cooling to room temperature, add each 10 ml of the appropriate reagent solutions (7.1.4.6 and 7.1.4.7 for the AAS measurement) or 1 ml of scandium solution (7.4.2.2, 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 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, $\rho = 1,16 \text{ g/ml}$.

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, NH_4Cl .

5.2.4 Apparatus

Ordinary laboratory apparatus and the following :

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 μm) 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 μm) 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).

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5.3 Microwave digestion with nitric acid

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5.3.1 General

The purpose of this method of digestion is to release trace elements from the accompanying matrix and determine the content of the various elements. The method is applied to high-calcium limes as specified in EN 12518, to calcium carbonate as specified in EN 1018 and to half-burnt dolomite as specified in EN 1017.

5.3.2 Principle

The sample is weighed into a perfluoroalkoxyethylene (PFA) beaker and nitric acid is added. After the mixture has been placed in a microwave apparatus, digestion is carried out. Any undissolved component is removed by filtering and the supernatant is used to determine the elements concerned.

5.3.3 Reagents

5.3.3.1 Nitric acid, $\rho = 1,40$ g/ml.

5.3.4 Apparatus

Ordinary laboratory apparatus and the following:

5.3.4.1 Microwave digestion apparatus, equipped with PFA digestion vessels and a programmable pressure and temperature control unit. Performance ≥ 600 W.

5.3.4.2 50 ml polypropylene vessel with screw lid.

5.3.4.3 Filtration apparatus.