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Products used for treatment of water intended for human consumption — Natural zeolite

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Products used for treatment of water intended for human consumption - Natural zeolite

Produits utilisés pour le traitement de l'eau destinée à la consommation humaine - Zéolite naturelle

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Natürlicher Zeolith

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Foreword

This document (EN 16070:2014) 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of an identical text or by endorsement, at the latest by September 2014 and conflicting national standards shall be withdrawn at the latest by September 2014.

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Introduction

In respect of potential adverse effects on the quality of water intended for human consumption, caused by the product covered by this European Standard:

a) this European Standard provides no information as to whether the product may be used without restriction in any of the Member States of the EU or EFTA;

b) it should be noted that, while awaiting the adoption of verifiable European criteria, existing national regulations concerning the use and/or the characteristics of this product remain in force.

NOTE Conformity with this European Standard does not confer or imply acceptance or approval of the product in any of the Member States of the EU or EFTA. The use of the product covered by this European Standard is subject to regulation or control by National Authorities.

1 Scope

This European Standard is applicable to natural zeolites used for treatment of water intended for human consumption. It describes the characteristics of natural zeolites and specifies the requirements and the corresponding test methods for natural zeolites. It gives information on their use in water treatment. The natural zeolites included in this standard are clinoptilolite, chabasite and phillipsite/analcime.

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 12901:1999, *Products used for treatment of water intended for human consumption - Inorganic supporting and filtering materials - Definitions*

EN 12902:2004, *Products used for treatment of water intended for human consumption - Inorganic supporting and filtering materials - Methods of test*

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

EN ISO 14911, *Water quality - Determination of dissolved Li+, Na+, NH4+, K+, Mn2+, Ca2+, Mg2+, Sr2+ and Ba2+ using ion chromatography - Method for water and waste water (ISO 14911)*

3 Terms, definitions and symbols

For the purposes of this document, the terms, definitions and symbols given in EN 12901:1999 apply.

4 Description

4.1 Identification

4.1.1 Chemical name(s)

Crystalline hydrated aluminosilicate.

4.1.2 Synonym or common names

— Zeolite;

— Hydrated sodium, potassium, calcium, magnesium aluminosilicate.

4.1.3 Chemical formula

General formula:

$$M_xD_y[Al_{x+2y}Si_zO_{2x+4y+2z}]\ n\ H_2O$$

where

M = Na, K, Li;

D = Ca, Mg;

x/y/z = 1 to 6;

n = 1 to 6.

4.1.4 CAS Registry number [1)]

1318-02-1

12173-10-3

12271-42-0

12251-32-0

61027-84-7

1318-10-1

4.1.5 EINECS reference [2)]

215-283-8

4.2 Commercial form

Natural zeolites are dry granular products, available in different particle sizes.

4.3 Types of natural zeolites

There are over 200 different zeolites with about 30 natural zeolites. However, not all natural zeolites can be used in the treatment of water intended for human consumption. Given the particular characteristics needed for this type of application, only three species are identified: clinoptilolite, chabazite and phillipsite/analcime. These zeolites are available in large quantities in Europe, with no trace of hazardous or toxic elements.

5 Physical properties

5.1 Appearance

The product consists of dry, free flowing granules. The product has a granular shape and a rough texture.

The product shall be generally homogeneous and shall be visibly free of extraneous matter.

5.2 Particle size distribution

The particle size distribution shall be determined on samples taken at the point of manufacture using the method of test given in EN 12902.

NOTE 1 The particle size can decrease during transportation and handling.

The particle size distribution shall be described by either:

a) effective size: (d_{10}) with a maximum deviation of ± 5 %;

median size: (d_{50}) with a maximum deviation of ± 5 %;

1) Chemical Abstracts Service Registry Number.

2) European Inventory of Existing Commercial Chemical Substances.

uniformity coefficient: (U) shall be less than 1,5;

minimum size: (d_1) with a maximum deviation of ± 5 %;

or

b) by particle size range and by mass fraction of oversize and undersize particles (see A.2.2.1).

The maximum permitted mass fractions of oversize and undersize are 5 %.

NOTE 2 Other values might be necessary for certain applications.

5.3 Density (bulk density loose)

The bulk density loose shall be in the range of:

— 800 kg/m^3 to 1 100 kg/m^3 for a clinoptilolite type zeolite;

— 700 kg/m^3 to 850 kg/m^3 for a chabazite type zeolite;

— 1 000 kg/m^3 to 1 100 kg/m^3 for a phillipsite/analcime type zeolite.

6 Chemical and mineralogical properties

6.1 General

This European Standard specifies the minimum purity requirements for natural zeolite used for the treatment of water intended for human consumption. Limits are given for impurities commonly present in the product. Depending on the raw material and the manufacturing process, other impurities may be present and, if so, this shall be notified to the user and when necessary to relevant authorities.

Users of this product should check the national regulations in order to clarify whether it is of appropriate purity for treatment of water intended for human consumption, taking into account raw water quality, contents of other impurities and additives used in the product not stated in this product standard.

Limits have been given for impurities and chemical parameters where these are likely to be present in significant quantities from the current production process and raw materials. If the production process or raw materials lead to significant quantities of impurities, by-products or additives being present, this shall be notified to the user.

The composition of the commercial product shall conform to Table 1.

Table 1 — Composition of commercial product

Parameters		Limit in mass fraction %
Mass loss at 150 °C	max.	7
Ignition loss at 650 °C	max.	14

After filling, washing and commissioning of a filter system producing drinking water, natural zeolite should not increase the concentrations of chemical parameters (see[1]).

NOTE Water extractable substances, determined in accordance with the method for granular materials given in EN 12902, can be used to estimate the leaching of the chemicals specified in EN 12902.

6.2 Capacity

The cations that maintain the electronic neutrality of the zeolite structure can be exchanged. This is a selective cation exchange according to the zeolite affinity for the replacing cation. The total cation exchange capacity and the selectivity are specific to each type of zeolite. This property makes the zeolites especially useful and efficient for cation elimination or to achieve control of their concentration in water. Ammonium can be used as a quality standard to determine the Cation Exchange Capacity (CEC) of a natural zeolite.

The total CEC shall be not less than 1,2 mg of ammonium per gram zeolite.

7 Test methods

7.1 Sampling

Prepare the laboratory sample(s) required by the relevant procedures described in EN 12902.

7.2 Analysis

7.2.1 Particle size distribution

The particle size distribution shall be determined in accordance with EN 12902.

7.2.2 Bulk density loose

The bulk density loose shall be determined in accordance with EN 12902.

7.2.3 Mass loss at 150 °C

It is assumed that water is the only material present in the natural zeolite that is volatile at this temperature.

The mass loss shall be determined by the method for water content, heating to 150 °C, in accordance with EN 12902.

7.2.4 Precision - repeatability limit

The absolute difference between two single test results, obtained under repeatability conditions, shall not exceed the repeatability limit, *r*, in more than 1 in 20 cases.

$$r = 0{,}07 \times X_1 \quad (1)$$

where

X_1 is the measured value.

7.2.5 Ignition loss at 650 °C

7.2.5.1 Principle

Heat the sample to constant mass at 650 °C. It is assumed that water, including chemically bound water and carbon dioxide produced from carbonates, are the only materials present in natural zeolite that are volatile at this temperature.

At 650 °C, the loss of water is practically complete.

7.2.5.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

7.2.5.2.1 Muffle furnace, capable of being controlled at (650 ± 25) °C.

7.2.5.2.2 High temperature metal or ceramic crucible with lid.

7.2.5.2.3 Desiccator.

7.2.5.2.4 Analytical balance, having an accuracy of ± 0,1 mg.

7.2.5.3 Procedure

Ignite the crucible and the lid (7.2.5.2.2) in the muffle furnace at (650 ± 25) °C for 1 h. Place the crucible and the lid in the desiccator, cool to room temperature and weigh to the nearest 0,1 mg; note the mass m_0.

Place a 5 g to 10 g sample of natural zeolite into the pre-ignited crucible. Close the crucible and weigh immediately to the nearest 0,5 mg (m_1). Remove the lid and place the crucible and lid in the muffle furnace at (650 ± 25) °C and leave for 3 h. Place the crucible in the desiccator, cover it with the lid and allow to cool to room temperature. When cool, weigh to the nearest 0,5 mg (m_2).

7.2.5.4 Expression of results

The ignition loss, X_2, expressed as a percentage mass fraction of dried product, is given by the formula:

$$X_2 = \frac{100 \times (m_1 - m_2)}{(m_1 - m_0)} \quad (2)$$

where

m_0 is the mass, in grams, of the empty crucible and lid;

m_1 is the mass, in grams, of the crucible and lid plus the sample;

m_2 is the mass, in grams, of the crucible and lid plus the ignited sample.

7.2.5.5 Precision - repeatability limit

The absolute difference between two single test results, obtained under repeatability conditions, shall not exceed the repeatability limit, *r,* in more than 1 in 20 cases.

$$r = 0{,}07 \times X_2 \quad (3)$$

7.2.6 Capacity

7.2.6.1 Principle

A sample of the zeolite is added to an ammonium chloride solution and the decrease of ammonium in the solution is determined after two hours. It is assumed that the ammonium capacity is a measure of the overall cation exchange capacity of the zeolite.

7.2.6.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

7.2.6.2.1 Volumetric flask of capacity 1 000 ml.

7.2.6.2.2 Erlenmeyer flask of capacity 500 ml.

7.2.6.2.3 Analytical balance, having an accuracy of ± 1 mg.

7.2.6.2.4 Laboratory shaker.

7.2.6.3 Reagents

7.2.6.3.1 General

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

7.2.6.3.2 Sodium chloride solution 2 mol/l

Dissolve 117 g sodium chloride in distilled water, transfer to a 1 l volumetric flask (7.2.6.2.1) and dilute to the mark with distilled water.

7.2.6.3.3 Ammonium chloride solution 1 mol/l

Dissolve 53,5 g ammonium chloride in distilled water, transfer to a 1 l volumetric flask (7.2.6.2.1) and dilute to the mark with distilled water.

7.2.6.4 Procedure

Add a 10 g (exactly weighed, m_0) dry sample of natural zeolite into an Erlenmeyer flask containing 300 ml of the sodium chloride solution. Immediately after adding the zeolite, begin shaking the Erlenmeyer flask and continue shaking for 24 h. Separate the zeolite from the saline solution by filtration. Rinse with distilled water. Place the zeolite in an Erlenmeyer flask containing 300 ml of distilled water. Shake for 30 min, filter and repeat the operation twice more.

Place 3 ml of the ammonium chloride solution in the Erlenmeyer flask. Dilute with water to 300 ml. Determine the ammonium concentration (c_0) according to EN ISO 14911. Immediately after adding the zeolite begin shaking the Erlenmeyer flask and continue shaking for 2 h. Immediately after shaking, filter the sample and measure the ammonium concentration in the filtrate (c_t).

7.2.6.5 Expression of results

The cation exchange capacity *CEC*, expressed as mg ammonium removed by 1 g of natural zeolite is given by the formula:

$$CEC = \frac{0{,}3 \times (c_0 - c_t)}{m_0} \quad (4)$$

where

c_0 is the ammonium concentration before adding the sample of natural zeolite, expressed in milligrams per litre (mg/l);

c_t is the ammonium concentration in the filtrate after shaking, expressed in milligrams per litre (mg/l);

m_0 is the mass of natural zeolite added, expressed in grams (g).

8 Labelling, transportation and storage

8.1 Means of delivery

Natural zeolite shall be delivered in bags (of plastic or paper materials), or semi-bulk containers or silo lorries.

In order that the purity of the product is not affected, the means of delivery shall not have been used previously for any different product or it shall have been specially cleaned and prepared before use.

8.2 Labelling according to the EU legislation [3)]

Natural zeolite is not listed within Annex VI of Regulation (EC) No 1272/2008 at the date of publication of this European Standard.

NOTE The legislation [2] contains a list of substances classified by the EU. Substances not listed in this regulation should be classified on the basis of their intrinsic properties according to the criteria in the regulation by the person responsible for the marketing of the substance.

8.3 Transportation regulations and labelling

Natural zeolite is not classified as a dangerous product for road, rail, sea, or air transportation at the date of publication of this standard. Natural zeolite is not listed under a UN number [4)].

8.4 Marking

The marking shall include the following:

— the name “Natural zeolite”, trade name and grade;

— the batch number;

— the net mass;

— the name and the address of the supplier and/or manufacturer;

— the statement “This product conforms to EN 16070”.

8.5 Storage

8.5.1 Long term stability

If properly stored the natural zeolite is stable indefinitely.

8.5.2 Storage incompatibility

The natural zeolite shall be kept away from solvents and odorous substances. It shall be stored in a dry and well-ventilated place.

3) See [2].

4) United Nations number.

Annex A
(informative)

General information on natural zeolites

A.1 Origin

A.1.1 Raw material

Sedimentary natural zeolites were formed when volcanic ash was deposited in alkaline/saline lakes/seas millions of years ago. Over time, the pressure, the temperature and the interaction of the volcanic ash with the salts in the lake water altered the ash creating the mineral zeolite.

A.1.2 Manufacturing process

After mining, the natural zeolite is ground and screened to obtain a product with the desired particle size. The product is dried to a free-flowing consistency, bagged for shipment or stored in silo before bagging.

A.2 Typical properties

A.2.1 Chemical and mineralogical composition

A.2.1.1 Chemical composition

The typical chemical composition of the natural zeolites is given in Tables A.1, A.2 and A.3:

Table A.1 — Chemical composition of the natural zeolite chabazite

Parameter	Mass fraction %
Al_2O_3	15,0 to 18,0
CaO	1,0 to 6,0
$Fe_T(Fe_2O_3)$	3,0 to 4,0
K_2O	1,0 to 7,0
MgO	1,0 to 2,5
Na_2O	0,5 to 9,0
SiO_2	51,0 to 63,0
TiO_2	1,0 to 3,0
Ignition loss	6,0 to 14,0

Table A.2 — Chemical composition of the natural zeolite clinoptilolite

Parameter	Mass fraction %
Al_2O_3	11,0 to 13,0
CaO	1,1 to 4,5
$Fe_T(Fe_2O_3)$	0,3 to 2,0
K_2O	2,2 to 4,0
MgO	0,2 to 2,0
Na_2O	0,5 to 4,0
SiO_2	64,0 to 70,0
TiO_2	0,1 to 3,3
Ignition loss	6,0 to 14,0

Table A.3 — Chemical composition of the natural zeolite phillipsite/analcime

Parameter	Mass fraction %
Al_2O_3	11,0 to 14,0
CaO	12,0 to 17,0
$Fe_T(Fe_2O_3)$	8,0 to 11,0
K_2O	2,0 to 4,0
MgO	3,0 to 6,0
Na_2O	0,3 to 3,0
SiO_2	20,0 to 38,0
TiO_2	1,0 to 3,0
Ignition loss	7,0 to 13,0

The content of chemical parameters should not exceed the limits given in Table A.4.

Table A.4 — Chemical parameters

Element	Content in mg/kg
Arsenic (As)	5
Cadmium (Cd)	2
Chromium (Cr)	30
Mercury (Hg)	0,1
Nickel (Ni)	30
Lead (Pb)	30
Antimony (Sb)	5
Selenium (Se)	10

A.2.1.2 Mineralogical composition

The typical mineralogical composition of the natural zeolites is given in Tables A.5, A.6 and A.7:

Table A.5 — Mineralogical composition of the natural zeolite chabazite

Parameter	Content
Main mineral constituent	Chabazite type zeolite
Main mineral content	Chabazite 70 % ± 5 % (Quantitative XRD analysis)
Other minerals	Phillipsite, feldspar, augite, mica, quartz, clays, glass
Undesirable minerals	Erionite

Table A.6 — Mineralogical composition of the natural zeolite clinoptilolite

Parameter	Content
Main mineral constituent	Clinoptilolite type zeolite
Main mineral content	Clinoptilolite 85 % ± 5 % (Quantitative XRD analysis)
Other minerals	Cristobalite, feldspar, celadonite, calcite, quartz, clays, glass
Undesirable minerals	Erionite

Table A.7 — Mineralogical composition of the natural zeolite phillipsite/analcime

Parameter	Content
Main mineral constituent	Phillipsite and analcime type zeolite
Main mineral content	Phillipsite + Analcime > 50 % (Quantitative XRD analysis)
Other minerals	Calcite, iron oxide, glass, augite
Undesirable minerals	Erionite

The natural zeolite should be free of erionite, a toxic fibrous mineral.

A.2.2 Physical properties

A.2.2.1 Particle size distribution

Typical particle size distributions are:

0,5 mm to 1 mm;

0,8 mm to 2,0 mm;

0,8 mm to 2,5 mm;

0,7 mm to 2,5 mm;

1 mm to 2,5 mm.

NOTE Other particle size distributions might be necessary for certain applications.

A.2.2.2 Mechanical strength

Abrasion products consist of dust and small particles of material. They are formed during transportation, filling, and washing. Abrasion products are not completely removed by washing.

Friability can be determined according to EN 12902:2004, Annex A.

The percentage losses should not exceed the following values:

a) 750 Impacts

The percentage loss compared with the minimum size d_1 before crushing: 20 %.

The percentage loss compared with the effective size d_{10} before crushing: 25 %.

b) 1 500 Impacts

The percentage loss compared with the minimum size d_1 before crushing: 30 %.

The percentage loss compared with the effective size d_{10} before crushing: 40 %.

A.2.2.3 pH

The pH of a suspension of prewashed natural zeolite in demineralized water (mass fraction 10 %) should be in the range of pH 7,1 to pH 7,7.

A.2.3 Density

A.2.3.1 Absolute density

The absolute density is in the range of 2 200 kg/ m^3 to 2 500 kg/ m^3.

A.2.3.2 Particle density dry

The particle density of the dry product is in the range of:

— 800 kg/m^3 to 1100 kg/m^3 for a clinoptilolite type zeolite;

— 700 kg/m^3 to 850 kg/m^3 for a chabazite type zeolite;

— 1 500 kg/m^3 to 1 800 kg/m^3 for a phillipsite/analcime type zeolite.

A.2.3.3 Particle density wet

The particle density of the wet product is in the range of 1 500 kg/ m^3 to 2 200 kg/ m^3.

A.2.3.4 Bed density

The bed density is 1 200 kg/ m^3 to 1 800 kg/ m^3 (up-washed and drained).

A.3 Use

A.3.1 Function

Natural zeolites can be used in three ways in the treatment of water for human consumption:

— as cation exchanger for the removal of dissolved pollutants such as ammonium, radioactive compounds and heavy metals;

— as filter media for mechanical filtration of water;

— as adsorbent to remove compounds such as ammonia, hydrogen sulfide and some organohalogens and radioactive compounds.

A.3.2 Specific amount

The amount of natural zeolite used depends on the application. Service life can differ depending on inlet water quality. Iron and/or manganese fouling will reduce its lifetime.

A.3.3 Means of application

Natural zeolites are normally used in vertical or horizontal filters.

Clinoptilolite type natural zeolite and phillipsite/analcime type natural zeolite can be used both in pressure and gravity filter systems. Chabazite type zeolite can be used only in gravity filter systems.

A.3.4 Secondary effects

Filtration of iron and manganese.

A.4 Hydraulic characteristics

A.4.1 Interstitial volume

The interstitial volume of the bed after up-washing is approximately 0,2 volume fraction to 0,5 volume fraction. If used for calculations, the interstitial volume should be measured.

A.4.2 Head loss in filtration

Head loss depends on size, shape, size and roughness of particles, filtration rate, filter bed depth, and water temperature.

A.4.3 Expansion during up-flow washing

The expansion during washing depends on flow rate, effective size, density, shape and roughness of particles, and water temperature.

A.5 Rules for safe handling and use

In handling dry natural zeolite, it is recommended to avoid formation of dust and to wear a dust mask.

A.6 Emergency procedures

A.6.1 First aid

In case of contact with skin there is no danger, it is recommended to wash with water.

In case of contact with eyes, it is recommended to flush with plenty of water.

In case of inhalation, it is recommended to move to fresh air.

A.6.2 Spillage

It is recommended to collect any spillage and replace into the original container.

A.6.3 Fire

The product is not flammable or combustible. There are no restrictions on extinguishing media in fire situations.

Bibliography

[1] 98/83/EC, Council Directive of 3 November 1998 on the quality of water intended for human consumption

[2] Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006

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