

Products used for treatment of water intended for human consumption — Bone charcoal

The European Standard EN 14456:2004 has the status of a
British Standard

ICS 71.100.80

National foreword

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English version

Products used for treatment of water intended for human consumption - Bone charcoal

Produits utilisés pour le traitement de l'eau destinée à la consommation humaine - Charbon animal

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Tierkohle

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Foreword

This document (EN 14456:2004) 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of an identical text or by endorsement, at the latest by December 2004, and conflicting national standards shall be withdrawn at the latest by December 2004.

Annex A is informative.

This document includes a Bibliography.

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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 standard :

- 1) this 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 ;
- 2) 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 the 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 bone charcoal used for the treatment of water intended for human consumption. It describes the characteristics of bone charcoal and specifies the requirements and the corresponding test methods for bone charcoal. It gives information on its use in water treatment.

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

EN 12902, *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 - Specifications and test methods (ISO 3696:1987).*

ISO 4793, *Laboratory sintered (fritted) filters - Porosity grading, classification and designation.*

3 Terms and definitions

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

4 Description

4.1 Identification

4.1.1 Chemical name(s)

Bone charcoal.

4.1.2 Synonym or common names

Animal charcoal, bone black.

4.1.3 Chemical formula

$C + Ca_{10}(PO_4)_6(OH)_2$.

4.1.4 CAS Registry Number¹⁾

8021-99-6.

1) Chemical Abstracts Service Registry Number.

4.1.5 EINECS Reference²⁾

232-421-2.

4.2 Commercial form

Bone charcoal is available in various size ranges.

4.3 Appearance

The product consists of black granules of irregular shape.

5 Physical properties**5.1 Particle size distribution**

The particle size distribution shall be described by either :

- | | |
|------------------------|----------|
| a) effective size | d_{50} |
| uniformity coefficient | U |
| minimum size | d_1 |

or

- b) particle size range and mass fraction of oversize and undersize particles.

The proportion of oversize and undersize particles shall be within the manufacturer's declared values.

5.2 Density

The bulk density (loose) shall be within $\pm 10\%$ of the value specified by the manufacturer or supplier.

NOTE The bulk density (loose) should be in the range of 600 kg/m^3 to 670 kg/m^3 .

6 Chemical properties**6.1 General**

Bone charcoal is a carbonaceous adsorbent, manufactured by controlled carbonisation in a reducing atmosphere, from selected grades of cattle-bone.

The product consists of a carbon component (about mass fraction 10 %) contained within a hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) structure.

2) European Inventory of Existing Commercial Chemical Substances.

Both the carbon component and the hydroxyapatite structure have an internal porosity, and each separate component accounts for about half the total surface area of the product.

6.2 Purity criteria

6.2.1 General

This European Standard specifies the minimum purity requirements for bone charcoal 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 can be present and, if so, this shall be notified to the user and when necessary to relevant authorities.

NOTE Users of this product should satisfy themselves that 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 products not stated in the product standard, and other relevant factors.

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 a change in the production process or raw materials leads to significant quantities of impurities, by-products or additives being present, this shall be notified to the user.

6.2.2 Main impurities and by-products

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

Table 1 — Main impurities and by-products

Impurity		Mass fraction, %
Acid-insoluble ash	max.	5
Water	max.	8

6.2.3 Water-extractable polycyclic aromatic hydrocarbons (PAH)

When tested in accordance with 8.2.5 the total PAH concentration in each extract shall not exceed 0,02 µg/l.

7 Specific properties

Bone charcoal, because of its hydroxyapatite structure, exhibits a buffering capability which imparts alkalinity to any water treated by the product.

Unlike conventional activated carbons, which are manufactured from cellulosic materials and comprise amorphous elementary carbon, bone charcoal is made from bone which is crystalline in form and the product itself retains much of this crystallinity.

8 Test methods

8.1 Sampling

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

8.2 Analysis

8.2.1 Particle size distribution

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

8.2.2 Bulk density loose

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

8.2.3 Water content

The water content shall be determined in accordance with EN 12902.

8.2.4 Carbon and acid-insoluble ash determination

8.2.4.1 Principle

The carbon content and acid-insoluble ash of a sample of bone charcoal are determined jointly. A known mass of ground bone charcoal is digested in acid and the residue filtered, washed, dried and incinerated. The carbon content is determined by mass difference before and after incineration, while acid-insoluble ash is obtained from the mass of residue after incineration.

8.2.4.2 Reagents

8.2.4.2.1 All reagents shall be of a recognized analytical grade and the water used shall conform to grade 3 in accordance with EN ISO 3696.

8.2.4.2.2 Hydrochloric acid, concentrated ($\rho = 1,18 \text{ g/ml}$).

8.2.4.3 Apparatus

8.2.4.3.1 Filter crucibles, sintered, porosity grade P 100 ($40 \mu\text{m}$ to $100 \mu\text{m}$) in accordance with ISO 4793.

8.2.4.3.2 Drying oven capable of being maintained at $(105 \pm 5) \text{ }^\circ\text{C}$.

8.2.4.3.3 Furnace capable of being maintained at $800 \text{ }^\circ\text{C}$ to $850 \text{ }^\circ\text{C}$.

8.2.4.3.4 Desiccator.

8.2.4.3.5 Hotplate.

8.2.4.4 Procedure

Grind a sample of at least 50 g until all of the product will pass through a sieve of nominal aperture size $250 \mu\text{m}$. Weigh, to an accuracy of 1 mg, about 5 g of ground sample and transfer quantitatively to a 400 ml beaker. Add 75 ml of water, followed by 25 ml of hydrochloric acid (8.2.4.2.2). Mix the reactants by swirling.

Cover the beaker with a watch glass, place on the hotplate (8.2.4.3.5) and bring to the boil. Maintain the state of boiling for 20 min, occasionally washing down the walls of the beaker with a fine jet of water to ensure that all of the sample is subjected to acid digestion.

Filter the contents of the beaker, without cooling, under vacuum through a previously conditioned crucible (8.2.4.3.1). Rinse the beaker with hot water at least twice, adding the rinsings to the crucible. Wash the residue on the filter with hot water.

Remove the crucible and dry in the oven (8.2.4.3.2) at $(105 \pm 5) \text{ }^\circ\text{C}$ for 3 h. After cooling in a desiccator (8.2.4.3.4), reweigh. Place the filter crucible in a furnace (8.2.4.3.3) maintained at $800 \text{ }^\circ\text{C}$ to $850 \text{ }^\circ\text{C}$ for a period of 2 h, then cool in the desiccator and reweigh.

8.2.4.5 Expression of results

The content of carbon, X_1 , expressed as percentage mass fraction of product is given by the following equation :

$$X_1 = \frac{(m_1 - m_2) \times 100}{m} \quad \dots(1)$$

where

m is the mass of the test portion, in grams ;

m_1 is the mass of the crucible plus test portion after drying at 105 °C, in grams ;

m_2 is the mass of the crucible plus test portion after incinerating at 800 °C to 850 °C, in grams.

The content of acid-insoluble ash, X_2 , expressed as percentage mass fraction of product is given by the following equation :

$$X_2 = \frac{(m_2 - m_3) \times 100}{m} \quad \dots(2)$$

where

m is the mass of the test portion, in grams ;

m_3 is the mass of the dried crucible, in grams ;

m_2 is the mass of the crucible plus test portion after incinerating at 800 °C to 850 °C, in grams.

8.2.5 Water-extractable polycyclic aromatic hydrocarbons

8.2.5.1 Principle

A sample of bone charcoal is placed in a glass column, rinsed and contacted with water for 24 h. The water is drained off and analysed for polycyclic aromatic hydrocarbons (PAH). This is repeated with fresh water for a further 24 h contact period.

8.2.5.2 Reagents

Water conforming to grade 2 in accordance with EN ISO 3696.

8.2.5.3 Apparatus

Ordinary laboratory apparatus and the following.

Two glass columns for leaching tests.

8.2.5.4 Procedure

Carry out the determination in duplicate using two columns run in parallel. The amount of product (bed volume) to be used (and hence the size of column required) shall be adequate to produce sufficient leachate to enable the specified analytical tests to be conducted. The determination shall be carried out following any pre-treatment specified by the manufacturer for the normal use of the product. The temperature of the water shall be maintained at (20 ± 1) °C throughout the determination.

Place 1 bed volume of water in the empty column and allow to stand for (24 ± 1) h. Drain the water into a glass bottle and retain for analysis; this constitutes the 'blank'.

Place a sample of bone charcoal in the column and add one bed volume of water. Drain the water to rinse the bed. Repeat until the sample has been rinsed with a total of 20 bed volumes of water. Leave the last volume of water in contact with the sample for (24 ± 1) h. Drain the water into a glass bottle and retain for analysis; this constitutes the

'first 24 h leachate'. Add a further bed volume of water, leave in contact with the sample for (24 ± 1) h, drain and collect ; this constitutes the 'second 24 h leachate'.

Analyse all samples and blanks for total PAH (NOTE 1) using a method of analysis that as a minimum is capable of measuring concentrations in the region of the limit value with:

- trueness $\leq 25\%$ of limit value (NOTE 2)
- accuracy $\leq 25\%$ of limit value (NOTE 3)
- limit of detection $\leq 25\%$ of limit value (NOTE 4)

NOTE 1 Polycyclic Aromatic Hydrocarbons : the sum of the detected concentrations of fluoranthene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, benzo(ghi)perylene, indeno(1,2,3-cd)pyrene

NOTE 2 The difference between the mean value of a large number of measurements and the true value.

NOTE 3 The standard deviation (within a batch and between batches) of the spread of results about the mean.

NOTE 4 The lowest concentration of an analyte in a sample that can be determined quantitatively. Limit of detection is calculated as 4,65 times the relative within-batch standard deviation of a blank sample.

8.2.5.5 Expression of results

The content of water-extractable PAH, X_3 , expressed in micrograms per litre of water, is given by the equation :

$$X_3 = c_1 - c_0 \quad \dots(3)$$

where

c_0 is the concentration, in micrograms per litre, of PAH in the blank ;

c_1 is the concentration, in micrograms per litre, of PAH in the extract ;

9 Labelling, transportation and storage

9.1 Means of delivery

Bone charcoal is delivered in paper or polypropylene sacks containing 25 kg or 50 kg or in semi-bulk bags containing 500 kg to 1 000 kg.

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.

9.2 Risk and safety labelling according to EU Directives³

Bone charcoal is not listed as a dangerous substance at the date of publication of this standard.

NOTE Annex I of the Directive 67/548/EEC on Classification, packaging and labelling of dangerous substances and its amendments and adaptations in the European Union contains a list of substances classified by the EU. Substances not in this Annex I should be classified on the basis of their intrinsic properties according to the criteria in the Directive by the person responsible for the marketing of the substance.

³ See [1].

9.3 Transportation regulations and labelling

Bone charcoal is not classified as dangerous for transportation by road, rail, sea or air. Bone charcoal is not listed under a UN⁴⁾ number.

9.4 Marking

The marking shall include the following :

- name “Bone charcoal”, trade name and commercial grade ;
- net mass ;
- name and the address of the supplier and/or manufacturer ;
- statement “this product conforms to EN 14456”.

9.5 Storage

9.5.1 Long term chemical stability

Bone charcoal does not deteriorate on storage and is non-hazardous.

9.5.2 Storage incompatibilities

The product shall be kept away from strong oxidants.

4) United Nations Number.

Annex A (informative)

General information on bone charcoal

A.1 Origin

A.1.1 Raw materials

Bone charcoal is produced from selected grades of sun-dried crushed cattle bone.

A.1.2 Manufacturing process

The product is manufactured by the dry distillation of sun dried crushed cattle bone in a reducing atmosphere using indirect heat.

During this carbonisation process at temperatures reaching 1 000 °C, a porous structure is developed in both the carbon component and the hydroxyapatite structure.

No other chemicals or additives are used during manufacture.

A.2 Typical properties

A.2.1 Chemical composition

A typical analysis of bone charcoal is as shown in Table A.1.

Table A.1 — Typical analysis

Parameter	Mass fraction, %
Carbon	8,5 to 11
Acid-insoluble ash	5 max.
Moisture	8 max.
Hydroxyapatite	70 to 76

A.2.2 Physical properties

Typical values are as shown in Table A.2.

Table A.2 — Physical properties

Total surface area	100 m ² /g
Carbon surface area	50 m ² /g
Pore size distribution	7,5 nm to 60 000 nm
Pore volume	0,225 cm ³ /g

A.3 Hydraulic characteristics

A.3.1 Headloss during filtration

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

A.3.2 Expansion during up-flow washing

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

A.4 Use

A.4.1 Function

Bone charcoal is used to remove colour, taste, odour and wide range of metals, fluoride and certain other organic and inorganic contaminants.

A.4.2 Means of application

Bone charcoal can be used in any fixed-bed mode, e.g. slow sand type filtration, rapid gravity filtration or in pressure filters. The product should, after filling, be either washed or back-washed with a minimum of 20 bed volumes of clean treated water before being put into service.

A.5 Rules for safe handling and use

It is recommended to handle the product so as to avoid dust formation. When handling dry product the use of overalls, gloves, goggles and a dust mask is recommended.

A.6 Emergency procedures

A.6.1 First aid

In case of skin contact, it is recommended to wash with soap and water.

In case of eye contact, it is recommended to flush with plenty of water and seek medical advice.

In case of inhalation, it is recommended to move to fresh air. If large amounts are inhaled it is recommended to seek medical advice.

In case of ingestion, if large amounts are ingested it is recommended to seek medical advice.

A.6.2 Spillage

It is recommended to sweep up and to discard in a refuse container.

A.6.3 Fire

Any extinguishing media can be used.

Self-contained breathing apparatus should be worn because carbon dioxide and carbon monoxide can be produced during combustion.

Bibliography

[1] 67/548/EEC - Council Directive of 27th June 1967 on the approximation of the laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances and its amendments and adaptations.

[2] 98/83/EC, Council Directive of 3 November 1998 on the quality of water intended for human consumption.

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