BS EN 13752:2012

Incorporating corrigendum June 2013

BSI Standards Publication

Products used for treatment of water intended for human consumption — Manganese dioxide

National foreword

This British Standard is the UK implementation of EN 13752:2012. It supersedes BS EN 13752:2009, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/59, Chemicals for drinking water treatment.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Published by BSI Standards Limited 2013

ISBN 978 0 580 82766 2

ICS 71.100.80

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2013.

Amendments/corrigenda issued since publication

Date	Text affected
30 June 2013	Implementation of CEN correction notice 19 December 2012: Title updated

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 13752

August 2012

ICS 71.100.80

English Version

Products used for treatment of water intended for human consumption - Manganese dioxide

Produits chimiques utilisés pour le traitement de l'eau destinée à la consommation humaine - Dioxyde de manganèse

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Mangandioxid

This European Standard was approved by CEN on 13 July 2012.

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Ref. No. EN 13752:2012: E

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Foreword

This document (EN 13752:2012) 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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

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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 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 manganese dioxide used for treatment of water intended for human consumption. It describes the characteristics of manganese dioxide and specifies the requirements and the corresponding test methods for manganese dioxide. It gives information on its use in water treatment. Two classes of product are specified: Class 1 with hardness greater than or equal to 6 Mohs, Class 2 with hardness less than 6 Mohs.

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, *Products used for treatment of water intended for human consumption – Inorganic supporting and filtering materials – Methods of test*

EN ISO 385, *Laboratory glassware – Burettes (ISO 385)*

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

ISO 6333, *Water quality – Determination of manganese – Formaldoxime spectrometric method*

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

Manganese dioxide.

4.1.2 Synonym or common names

Manganese(IV) oxide, pyrolusite.

4.1.3 Chemical formula

MnO_2.

NOTE Manganese dioxide used as a catalytic filtering medium is a natural ore, usually pyrolusite. Manganese dioxide ores differ widely in their chemical composition depending on their origin. Most are composed of manganese dioxide together with silica, alumina, iron oxide and numerous other elements present in varying proportions which might affect mechanical strength.

4.1.4 CAS Registry number [1]

Manganese dioxide: 1313-13-9.

4.1.5 EINECS reference [2]

Manganese dioxide: 215-202-6.

4.2 Commercial form

Manganese dioxide is a granular material usually available in three size ranges: 0,355 mm to 0,850 mm, 0,50 mm to 1,00 mm and 0,5 mm to 3,00 mm.

5 Physical properties

5.1 Appearance

The product is a granular material varying in colour from dark brown to dark grey to black, depending upon its origin. It consists of amorphous grains which can be rounded or sub-angular.

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 described by either:

a)

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

— uniformity coefficient: (U) less than 1,5 for Class 1 and Class 2 products with particles smaller than or equal to 1 mm; and less than 2,5 for Class 1 and Class 2 products with particles greater than 1 mm.

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

NOTE The particle size can decrease during transportation and handling.

b) or, particle size range and mass fraction of oversize and undersize particles according to application.

The maximum contents of oversize and undersize shall be a mass fraction of 10 % for application of the product in multimedia filters and a mass fraction of 5 % for use in single media filters. See A.2.3 for examples of available particle sizes that are used.

Other values can be necessary for certain applications.

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1) Chemical Abstracts Service Registry Number.

2) European Inventory of Existing Commercial Chemical Substances.

5.3 Density

5.3.1 Bulk density loose

The bulk density loose shall be in the range of 1 750 kg/m^3 to 1 850 kg/m^3 for Class 1 and Class 2 products with particles smaller than or equal to 1 mm and 1 800 kg/m^3 to 2 200 kg/m^3 for Class 1 and Class 2 products with particles greater than 1 mm.

5.3.2 Bulk density packed

The bulk density packed shall be in the range of 1 950 kg/m^3 to 2 050 kg/m^3 for Class 1 and Class 2 products with particles smaller than or equal to 1 mm and 2 000 kg/m^3 to 2 400 kg/m^3 for Class 1 and Class 2 products with particles greater than 1 mm.

6 Chemical properties

For the composition of the commercial product, see A.2.1.

After filling, washing and commissioning of a filter system producing drinking water, manganese dioxide should not increase the concentrations of chemical parameters above the regulated values (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.

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 products not stated in the product standard.

7 Specific properties

The oxidation capacity of manganese dioxide shall be at least 500 bed volumes when tested according to the method described in 8.2.4.

NOTE For a Class 1 product, a greater oxidation capacity may be specified.

8 Test methods

8.1 Sampling

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

8.2 Analysis

8.2.1 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.

8.2.2 Bulk density loose

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

8.2.3 Bulk density packed

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

8.2.4 Oxidation capacity

8.2.4.1 Principle

A solution of manganese sulfate is passed through a bed of "regenerated" manganese dioxide. Portions of the effluent are analysed for manganese and the cumulative volume, until breakthrough of manganese, is determined.

8.2.4.2 Reagents

8.2.4.2.1 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.

8.2.4.2.2 Manganese sulfate stock solution, *c*(Mn) = 5 mg/l

Dissolve 1,00 g of sodium hydrogen carbonate ($NaHCO_3$) in water and add 0,154 g of manganese sulfate monohydrate ($MnSO_4 \cdot H_2O$). Dilute to 10,0 l with water and adjust the pH to 7,0 ± 0,5 using dilute sulfuric acid or sodium hydroxide solution as required.

8.2.4.2.3 Manganese sulfate test solution, *c*(Mn) = 1 mg/l

Dilute 2 l of the manganese sulfate stock solution (8.2.4.2.2) to 10 l with water and adjust the pH to 7,0 ± 0,5 using dilute sulfuric acid or sodium hydroxide solution as required.

8.2.4.2.4 Sodium hypochlorite solution, approximately 1 200 mg/l Cl_2

Dilute 10 ml of sodium hypochlorite solution, available chlorine concentration approximately 120 g/l, to 1 l with water.

8.2.4.3 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

8.2.4.3.1 Glass burette of 50 ml capacity conforming to the requirements of EN ISO 385.

8.2.4.3.2 Graduated measuring cylinder, 200 ml capacity.

8.2.4.3.3 Borosilicate glass bottle, 10 l capacity.

8.2.4.4 Preparation of test sample

Take approximately 100 g of the manganese dioxide and sieve to obtain that fraction of the material sized between 0,355 mm and 0,850 mm, using the procedure for determination of particle size described in EN 12902. Use this fraction for the determination.

Take approximately 20 g of the manganese dioxide (0,355 mm to 0,850 mm), place in a 250 ml beaker and gently wash with successive portions of water until all adherent fines have been removed. Dry the beaker and contents to constant mass at (100 ± 5) °C.

Place a loose plug of glass wool in the bottom of the burette (8.2.4.3.1) and add the manganese dioxide to a depth of approximately 50 mm. Connect the nozzle of the burette to a cold water tap by means of suitable rubber tubing and backwash the manganese dioxide in the burette vigorously ensuring complete fluidisation of the bed in order to remove any ultra-fine material not previously removed. When any remaining fines have been washed off, turn off the tap and drain down the water in the burette to just above the surface of the manganese dioxide bed. To avoid entrapment of air, ensure the water level does not fall below the top of the manganese dioxide bed.

Tap the burette gently with a glass rod fitted with a rubber policeman to compact the manganese dioxide and measure the depth of the bed of manganese dioxide and the internal diameter of the burette and calculate the volume of the bed of manganese dioxide in litres.

Add sodium hypochlorite solution (8.2.4.2.4) to the burette and allow to drain out drop wise until the manganese dioxide has been in contact with the sodium hypochlorite solution for 10 min. Drain the sodium hypochlorite solution until the liquid level is just above the surface of the manganese dioxide bed. Fill the burette with water, open the tap fully and rinse the manganese dioxide for about 10 min to remove residual sodium hypochlorite solution.

8.2.4.5 Procedure

Pass water through the burette and adjust the setting of the tap to give a flow rate of 16 ml/min.

NOTE This corresponds to a velocity of 2,77 mm/s.

Drain the water to just above the surface of the manganese dioxide bed and, leaving the tap at the same setting, pass the manganese sulfate test solution (8.2.4.2.3) through the burette at the same flow rate. Collect the effluent in 200 ml aliquots in the measuring cylinder (8.2.4.3.2) and analyze for manganese content in accordance with ISO 6333. When the concentration of manganese in the effluent reaches 0,05 mg/l, stop the flow of manganese sulfate solution. Reject the aliquot having a manganese concentration greater than 0,05 mg/l and note the total volume of manganese sulfate solution passed. Carry out the procedure in duplicate, using a fresh portion of the “regenerated” and dried manganese dioxide for each test.

8.2.4.6 Expression of results

The oxidation capacity of the manganese dioxide, C, expressed as bed volumes of test solution treated, is given by the following formula:

$$C = \frac{V}{V_1} \tag{1}$$

where

V is the mean volume, in litres, of water with a manganese content less than 0,05 mg/l in the two runs;

V_1 is the volume, in litres, of the bed of manganese dioxide.

9 Labelling, transportation and storage

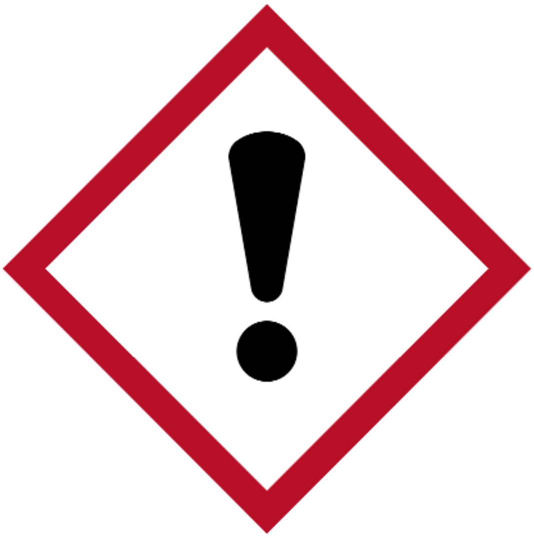
9.1 Means of delivery

Class 1 manganese dioxide shall be delivered in big bags or in bulk. Class 2 manganese dioxide shall be delivered in bags of 25 kg to 50 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 Labelling according to the EU legislation

The following labelling requirements shall apply to manganese dioxide at the date of publication of this standard.

Hazard pictogram  **Figure 1 — GHS07**	— Signal word: **Warning** — Hazard statements : H302: Harmful if swallowed H332: Harmful if inhaled

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 placing the product on the market. Classification and labelling is carried out in compliance with [2].

9.3 Transportation regulations and labelling

At the date of publication of this European Standard, manganese dioxide is not listed under a UN number [3]; it is not a dangerous cargo.

9.4 Marking

The marking shall include the following:

— the name "manganese dioxide", trade name and class;

— the net mass;

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

— the statement "This product conforms to EN 13752".

[3] United Nations number.

9.5 Storage

9.5.1 Long term chemical stability

Manganese dioxide can be stored for an unlimited period of time.

9.5.2 Storage incompatibility

Manganese dioxide shall not be allowed to come into contact with hydrochloric acid; contact with this chemical produces chlorine gas. Avoid storing with any chloride salt jointly with an acid, sulfuric acid, or hydrogen sulfates.

Annex A
(informative)

General information on manganese dioxide

A.1 Origin

A.1.1 Raw material

Manganese dioxide exists in nature in several varieties, not all of which are suitable for use as catalytic filtration materials. The catalytic activity of manganese dioxide varies greatly and is generally unrelated to its manganese content.

A.1.2 Manufacturing process

Manganese dioxide is produced by mining, crushing and screening rock deposits.

A.2 Typical properties

A.2.1 Chemical composition

The chemical composition depends on the origin. Typical values are given in Table A.1.

Table A.1 — Chemical composition of ordinary manganese dioxide

Parameter	Limit as mass fraction of the product %			
	Class 1		Class 2	
	Minimum	Maximum	Minimum	Maximum
MnO_2	80	90	52	79
Fe_2O_3	1	10	5	15
SiO_2	2	10	2	10
CaO	0	3	0	5
MgO	0	3	0	5
Al_2O_3	1	5	-	-

A.2.2 Mechanical strength

The mechanical strength of manganese dioxide depends mostly on purity: the higher the purity, the higher the mechanical strength. Hardness is measured on the Mohs scale. The mechanical strength of Class 1 manganese dioxide is medium and the mechanical strength of Class 2 manganese dioxide is low. With Class 1 product (hardness greater than or equal to 6 Mohs) the filtering medium is resistant to attrition; it will not be consumed during washing of the filter and annual loss in mass will be around 3 % and will not exceed 5 %. With Class 2 product (hardness lower than 6 Mohs), the filtering medium is more sensitive to attrition and should be considered as a consumable product.

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 and attempting to do so can result in the generation of excessive fines as a result of attrition with sand particles if a mixed bed of sand and manganese dioxide is washed too vigorously. When washing beds containing manganese dioxide, the bed should be expanded to a just-fluidised condition and no more.

Hardness can be determined by fixing the particles on a plate, then performing an abrasion test to classify the hardness using Mohs scale. This allows classification of the different materials according to their mechanical strength and especially regarding consumption of material during operation. The existing methods for determination of abrasion do not lead to exact results regarding the behaviour of filter media during operation. They can be used only for comparison of different filter media.

A.2.3 Alternative description of particle size distribution

Examples of particle size distribution described by different particle size ranges and a permissible mass percentage of oversize and undersize product, are given in Table A.2.

Table A.2 — Examples of particle size range

Particle size range mm	**Permissible mass fraction** [a] %			
	Class 1		**Class2**	
	Undersize	**Oversize**	**Undersize**	**Oversize**
0,355 to 0,850	-	-	5	5
0,50 to 1,00	-	-	5	5
0,5 to 3,0	5	5	-	-

[a] Additionally, no more than a mass fraction of 10 % should pass the test sieve of next wider aperture to the stated bottom size; i.e. for 0,355 mm to 0,850 mm product, no more than a mass fraction of 10 % should pass a 0,425 mm sieve and for 0,50 mm to 1,00 mm product, no more than a mass fraction of 10 % should pass a 0,600 mm test sieve.

Other particle size ranges may be specified.

A.2.4 Absolute density

The density of manganese dioxide depends on its origin. Most manganese dioxide ores have an absolute density of 3,9 g/cm^3 to 4,3 g/cm^3 but some varieties have an absolute density as low as 3,5 g/cm^3.

A.3 Use

A.3.1 Function

Manganese dioxide is used as a catalytic filtering material for the removal of iron and manganese from water.

A.3.2 Method of use

Manganese dioxide is used as an addition to silica sand, usually in proportions of between 10 % to 50 % volume fraction.

When Class 1 product with large particles (average of 1 mm to 2 mm) is used in addition to silica sand, the manganese dioxide forms a distinct layer, which remains separated from the sand during operation. Backwashing (air alone or air plus water) restores this separation, because of the differences in particle size and density of the two materials.

With Class 2 product it is important that the manganese dioxide and sand are intimately mixed after backwashing and for this reason the size grading of the manganese dioxide is chosen so that, having regard to the different densities of sand and manganese dioxide, the two will mix thoroughly. Normally, with manganese dioxide having absolute density in the range 3,9 g/cm^3 to 4,0 g/cm^3 the size range used is 0,355 mm to 0,850 mm. This grade is used with 0,5 mm to 1,0 mm or 0,6 mm to 1,18 mm sand of absolute density 2,65 g/cm^3. If a larger grade of sand (e.g. 1,00 mm to 2,0 mm) or a manganese dioxide of lower density is used, the sizing of manganese dioxide is usually increased to 0,5 mm to 1,0 mm.

A.3.3 Oxidation capacity

Manganese dioxide will remove iron from water provided there is sufficient oxygen present (60 % oxygen saturation); this process makes no demand on the manganese dioxide and will continue indefinitely provided the precipitated iron hydroxide is removed from the surface of the manganese dioxide.

Manganese removal is achieved ideally in the presence of chlorine or other strong oxidising agent and, given a sufficient concentration of oxidising agent, will continue indefinitely. In the absence of a strong oxidising agent, the manganese dioxide will itself act as an oxidising agent and oxidise manganese in the water until the surface of the manganese dioxide has been reduced to an inactive lower oxidation state. This fact is utilised in determining the oxidation capacity of manganese dioxide (Clause 8).

When the surface of manganese dioxide has been reduced, it can be restored by attrition during filter backwashing.

Hydrogen peroxide should not be used as the oxidising agent in contact with manganese dioxide as this can result in an excessive manganese concentration in the treated water.

A.3.4 Specific amount

The specific amount of manganese dioxide used depends upon the application and can vary typically between 10 % and 50 % (volume fraction) of the bed of filtering material (see A.4.2).

A.3.5 Means of application

Manganese dioxide is used in open or closed single or multi-layer filters specifically for Class 1 manganese dioxide. Class 2 manganese dioxide may be used in addition to silica sand with which it should mix intimately and not form a discrete layer.

Manganese dioxide is not suitable for the treatment of water having a pH lower than approximately 6,2 unless the pH is adjusted.

A.3.6 Secondary effects

For Class 2, when treating waters which contain more than approximately 0,2 mg/l manganese, the sand in which the manganese dioxide is distributed can become coated with a deposit of manganese dioxide. This occurs as a result of there being more manganese dioxide deposited from the water being treated during the filtration stage than is lost by attrition during the backwash stage. As a consequence, the density of the sand will increase with consequent increase in energy required for backwashing the bed. The sand/manganese dioxide mixture will also increase in particle size and media might have to be removed from the bed in order to avoid exceeding the maximum level. In waters with very low concentrations of manganese, the reverse process occurs and more manganese dioxide is lost through attrition during backwashing than is deposited during manganese removal. Consequently, the manganese dioxide can require topping-up periodically in filters treating such waters.

A.4 Hydraulic characteristics

A.4.1 Interstitial volume

The interstitial volume is approximately a volume fraction of 0,4. If used for calculations the interstitial volume should be measured.

A.4.2 Head loss in filtration

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

Filtration capacity is comparable to sand filtration capacity (depend on velocity of the filtration), therefore existing sand filters can be converted to dual layer filters (repartition between sand and manganese dioxide is calculated according velocity and range size).

A.4.3 Expansion in up-flow washing

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

The backwash regime used for filters containing manganese dioxide should not cause undue attrition of the manganese dioxide; generally the lowest possible backwash velocity consistent with bed fluidisation should be used.

When Class 1 manganese dioxide with larger particles is used, limited expansion of the manganese dioxide occurs during up-flow washing with usual flow rates (≤ 60 m/h). Attrition of manganese dioxide can be encouraged in order to restore oxidation capacity, by using air plus water sequence during backwash.

Annex B
(normative)

General rules relating to safety

B.1 Rules for safe handling and use

The supplier shall provide current safety instructions.

B.2 Emergency procedures

B.2.1 First aid

In case of contact with skin, wash with soap and water.

In case of contact with eyes, flush with plenty of water.

In case of inhalation, move to fresh air.

In case of ingestion, do not induce vomiting. If conscious and alert, rinse mouth with water. If large quantities are swallowed, seek medical advice immediately.

B.2.2 Spillage

Sweep up and discard in refuse container. Avoid mixing with combustible materials.

NOTE Local regulations might apply to the disposal of this product.

B.2.3 Fire

No special requirements apply.

Bibliography

[1] 98/83/EC, Council Directive of 3 November 1998 on the quality of water intended for human consumption, last amended by Regulation (EC) 596/2009.

[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 (REACH).

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