BS EN 12174:2013

BSI Standards Publication

Chemicals used for treatment of water intended for human consumption — Sodium hexafluorosilicate

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National foreword

This British Standard is the UK implementation of EN 12174:2013. It supersedes BS EN 12174:2006 which is withdrawn.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.

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English Version

Chemicals used for treatment of water intended for human consumption - Sodium hexafluorosilicate

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

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Natriumhexafluorsilikat

This European Standard was approved by CEN on 28 March 2013.

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Contents

Foreword

This document (EN 12174:2013) 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of an identical text or by endorsement, at the latest by November 2013, and conflicting national standards shall be withdrawn at the latest by November 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 12174:2006.

Significant differences between this edition and EN 12174:2006 are as follows:

- the replacement of warning and safety precautions notes by labelling according to Regulation (EC) No 1272/2008.

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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 document:

a) this document 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 document 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 document is subject to regulation or control by National Authorities.

1 Scope

This European Standard is applicable to sodium hexafluorosilicate used for treatment of water intended for human consumption. It describes the characteristics of sodium hexafluorosilicate and specifies the requirements and the corresponding test methods for sodium hexafluorosilicate. It gives information on its use in water treatment. It also determines the rules relating to safe handling and use of sodium hexafluorosilicate (see Annex B).

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 ISO 3696:1995, *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 4281, *Sodium hexafluorosilicate for industrial use — Determination of free acidity and total hexafluorosilicate content — Titrimetric method*

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

ISO 5444, *Sodium fluorosilicate for industrial use — Determination of loss in mass at 105 degrees C*

ISO 5993, *Sodium hydroxide for industrial use — Determination of mercury content — Flameless atomic absorption spectrometric method*

ISO 6206, *Chemical products for industrial use — Sampling — Vocabulary*

ISO 6353-1, *Reagents for chemical analysis — Part 1: General test methods*

ISO 8213, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*

3 Description

3.1 Identification

3.1.1 Chemical name

Disodium hexafluorosilicate.

3.1.2 Synonym or common name

Sodium silicofluoride, sodium hexafluorosilicate.

3.1.3 Relative molecular mass

188,055.

3.1.4 Empirical formula

Na_2SiF_6.

3.1.5 Chemical formula

Na_2SiF_6.

3.1.6 CAS-Registry Number[1]

16893-85-9.

3.1.7 EINECS reference[2]

240-934-8.

3.2 Commercial form

The product is a crystalline powder.

3.3 Physical properties

3.3.1 Appearance and odour

The product is a colourless, odourless fine crystalline powder.

3.3.2 Density

The particle density of the crystals is 2,8 g/cm^3 at 20 °C.

The bulk density of the product is approximately 1,5 g/cm^3 at 20 °C.

3.3.3 Solubility

The solubility of the product in water is 7 g/l at 20 °C.

3.3.4 Vapour pressure

Not applicable.

3.3.5 Boiling point at 100 kPa[3]

Not applicable.

3.3.6 Crystallisation point

The product melts above 500 °C.

3.3.7 Specific heat

Not known.

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1) Chemical Abstracts Service Registry Number.

2) European Inventory of Existing Commercial Chemical Substances.

3) 100 kPa = 1 bar.

3.3.8 Viscosity, dynamic

Not applicable.

3.3.9 Critical temperature

Not applicable.

3.3.10 Critical pressure

Not applicable.

3.3.11 Physical hardness

Not applicable.

3.4 Chemical properties

The pH value of a saturated aqueous solution (7 g/l) of sodium hexafluorosilicate is approximately 10.

Sodium hexafluorosilicate reacts with acids to form hydrofluoric acid.

4 Purity criteria

4.1 General

This document specifies the minimum purity requirements for sodium hexafluorosilicate 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, required dosage, contents of other impurities and additives used in the products not stated in this document.

Limits have been given for impurities and chemicals 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.

4.2 Composition of commercial product

The content of sodium hexafluorosilicate shall not be less than a mass fraction of 98 % (Na_2SiF_6).

The concentration of sodium hexafluorosilicate shall be within ± 5 % of the manufacturer's declared value.

4.3 Impurities and main by-products

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

Table 1 — Impurities

Impurity		**Limit** in mass fraction in % of commercial product
Water- insoluble matter	max.	0,5
Moisture	max.	0,3

4.4 Chemical parameters

The product shall conform to the requirements specified in Table 2.

Table 2 — Chemical parameters

Parameter		Limit mg/kg of commercial product
Antimony (Sb)	max.	80
Arsenic (As)	max.	400
Cadmium (Cd)	max.	40
Chromium (Cr)	max.	400
Lead (Pb)	max.	400
Mercury (Hg)	max.	10
Nickel (Ni)	max.	400
Selenium (Se)	max.	80
NOTE Other chemical parameters and indicator parameters are not relevant in sodium hexafluorosilicate because the raw materials used in the manufacturing process are free of them. For parametric values of sodium hexafluorosilicate on trace metal content in drinking water, see [1].		

5 Test methods

5.1 Sampling

Observe the general recommendations of ISO 3165 and take account of ISO 6206.

Prepare the laboratory sample(s) required by the relevant procedure described in ISO 8213.

NOTE Sodium hexafluorosilicate presents a toxic hazard through inhalation of dust (see B.1).

5.2 Analyses

5.2.1 Main product

The sodium hexafluorosilicate content shall be determined in accordance with ISO 4281.

This standard includes the determination of free acid.

5.2.2 Impurities

5.2.2.1 Insoluble matters

5.2.2.1.1 Procedure

Weigh and transfer to a beaker 2 g of the sample, which has previously been dried at 105 °C to constant mass.

Dissolve in 500 ml of hot water. Generally, 15 min to 30 min is sufficient time.

Filter through a tared Gooch crucible or a tared fritted-glass filter of medium porosity (porosity P 16 according to ISO 4793). Wash with at least six separate 25 ml portions of boiling water, allowing the crucible to drain between washings. Dry the crucible or filter at 105 °C to constant mass.

5.2.2.1.2 Expression of results

The content of insoluble matters, W_1, expressed as mass fraction in % is given by the following formula:

$$W_1 = \frac{m_1}{m_2} \text{x}\, 100 \quad (1)$$

where

m_1 is the mass, in grams, of the filter residue;

m_2 is the mass, in grams, of the test portion.

5.2.2.2 Moisture content

The content of moisture shall be determined in accordance with ISO 5444.

5.2.3 Chemical parameters

5.2.3.1 General

The content of chemical parameters shall be determined using the procedures specified in Table 3.

Table 3 — Procedures for the determination of chemical parameters

Element	Reference	Method	Wavelength nm	Flame
As	See 5.2.3.3	Hydride AAS	193,7	n.a
Sb	See 5.2.3.3	Hydride AAS	217,6	n.a
Cd	ISO 6353-1, MG 29 See 5.2.3.2	AAS	228,8	air-acetylene
Cr	ISO 6353-1, MG 29 See 5.2.3.2	AAS	357,8	air-acetylene
Pb	ISO 6353-1, MG 29 See 5.2.3.2	AAS	217,0 or 283,3	air-acetylene
Ni	ISO 6353-1, MG 29 See 5.2.3.2	AAS	232,0	Oxidising Air-acetylene
Se	See 5.2.3.3	Hydride AAS	196,0	n.a
Hg	In accordance with ISO 5993	flameless AAS	253,6	n.a
n.a. not applicable. AAS Atomic absorption spectrometry.				

5.2.3.2 Determination of cadmium (Cd), chromium (Cr), lead (Pb) and nickel (Ni)

5.2.3.2.1 Principle

The elements cadmium (Cd), chromium (Cr), lead (Pb) and nickel (Ni) are determined using atomic absorption spectrometry with standard additions method in taking account of ISO 6353-1.

5.2.3.2.2 Reagents

All reagents shall be of a recognised analytical grade and the water used shall conform to the grade 3 specified in EN ISO 3696:1995.

5.2.3.2.2.1 Standard solution (100 µg/l Cd, Cr, Pb or Ni)

The standard solution shall be freshly prepared on the day of use by individual dilution of a stock solution. This stock solution with a Cd, Cr, Pb or Ni content of at least 1 mg/l shall be made by dilution of standard solutions of Cd, Cr, Pb and Ni, which are available from all major suppliers of laboratory chemicals. This stock solution shall be kept in containers of tetrafluoroethylene-hexafluoropropylene copolymer (FEP), polytetrafluoroethylene (PTFE) or polyethylene (PE).

The stock solution should not be kept for longer than four weeks.

5.2.3.2.3 Apparatus

Ordinary laboratory apparatus and the following:

5.2.3.2.3.1 Atomic absorption spectrometer with the measurement parameters specified in Table 3.

5.2.3.2.4 Procedure

5.2.3.2.4.1 Test portion

Weigh 1 g to the nearest 0,01 mg of the laboratory sample into a 100 ml one-mark volumetric flask and make up to the mark at 20 °C with water.

5.2.3.2.4.2 Determination

The reference solutions shall be made by spiking the sample with the standard solutions, which contain stepwise increasing contents of the elements to be determined.

NOTE The amount of internal standard to be added can be estimated from a preliminary investigation, determining roughly the element content of the test sample from simple calibration.

The steps in which internal standards are added shall be at least as high as the estimated content of the test sample. With the spectrometer (5.2.3.2.3.1), carry out the measurement with the parameters specified in Table 3 in accordance with the manufacturer's instructions.

Repeat the procedure with all reagents and the same volume of standard solution to be added using water in place of the sample as a blank determination.

5.2.3.2.5 Expression of results

Prepare a calibration curve using the measured absorbencies of the spiked measurement solutions.

Read the concentration of each element in the test solution by extrapolation of the correlation line to absorbance A = 0 (see Figure 1). Similarly determine the element concentration of the blank solution (see Figure 2) and subtract from the result obtained for the test solution.

Alternatively, the evaluation may be carried out by linear regression. Additional dilution steps shall be compensated in the calculation.

This interim result (y) expressed in micrograms per litre which is converted to give the final concentration according to 5.2.3.2.6.

5.2.3.2.6 Calculation

From the interim result (y) determined (see (5.2.3.2.5) the content, W_2. of each element in the laboratory sample, expressed in milligrams per kilogram of commercial product is given by the following formula.

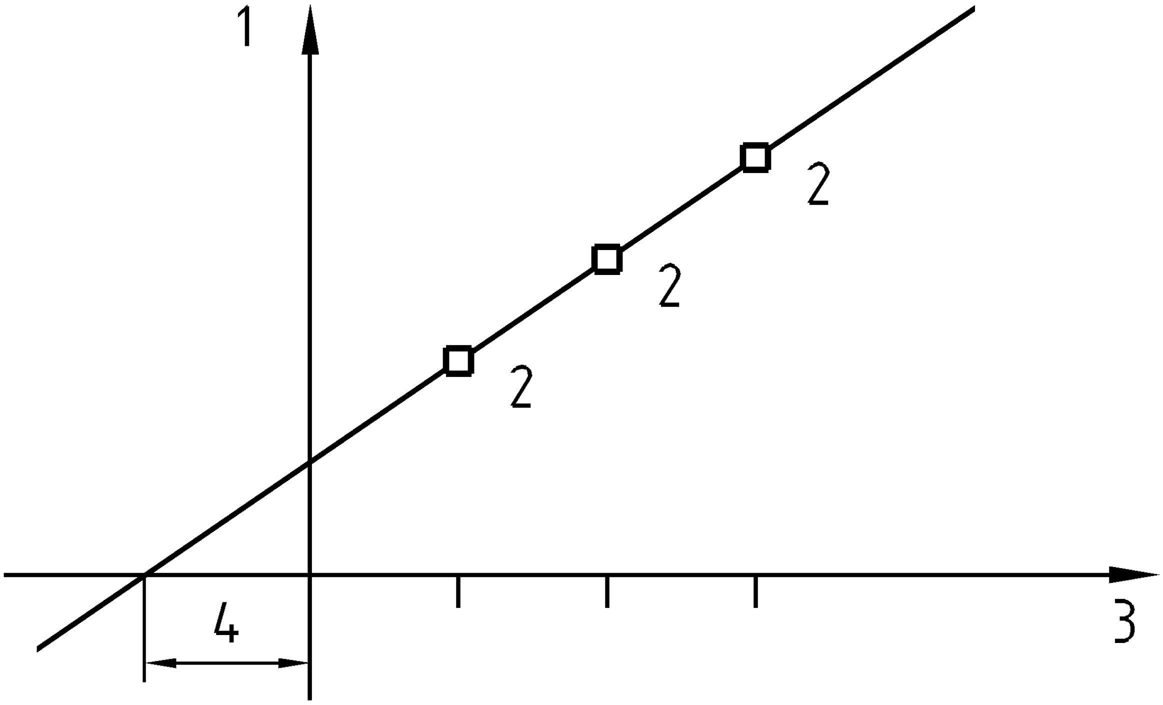
$$W_2 = \frac{y \times V_2 \ \times 1000}{m_2} \tag{2}$$

where

y is the interim result (5.2.3.2.5);

V_2 is the volume, expressed in millilitres, of the test solution;

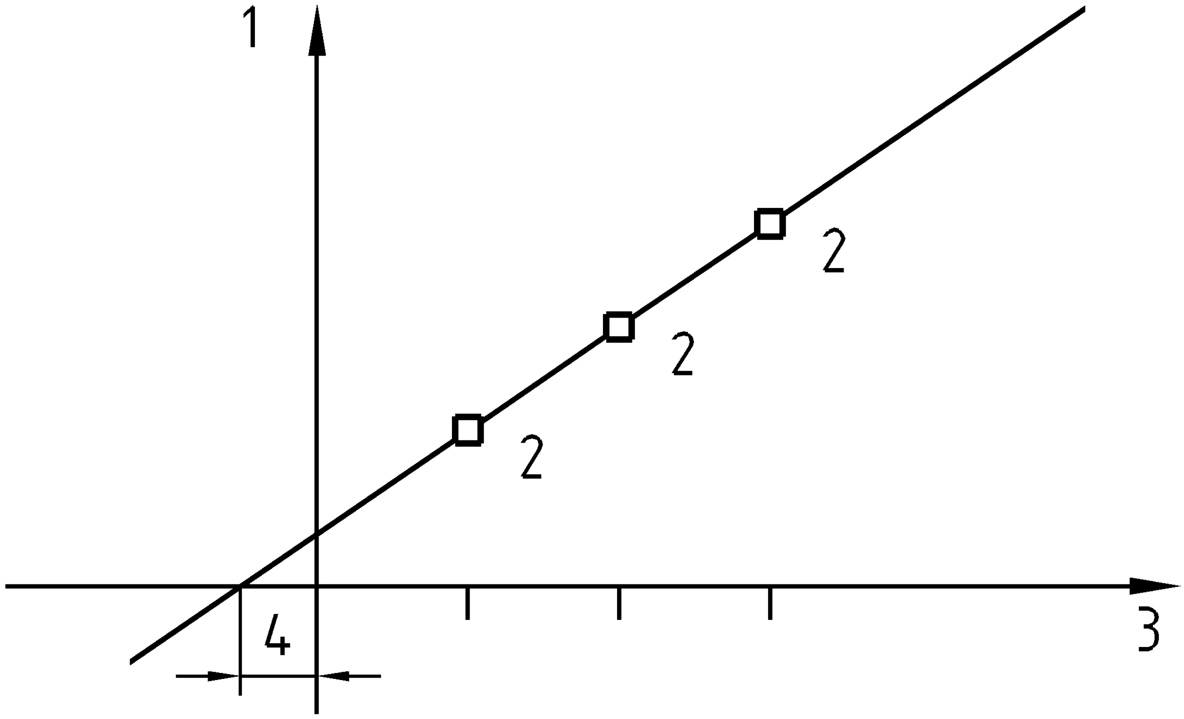
m_2 is the mass, expressed in grams, of the test portion.



Key

1 absorbance A
2 spiking
3 concentration of added standard in micrograms per litre
4 concentration in the test solution in micrograms per litre

Figure 1 — Calculation of the element concentration in the test solution



Key

1 absorbance A

2 spiking

3 concentration of added standard in micrograms per litre

4 concentration in the blank solution in micrograms per litre

Figure 2 — Calculation of the element concentration in the blank solution

5.2.3.3 Determination of arsenic (As), antimony (Sb) and selenium (Se)

5.2.3.3.1 Principle

The elements arsenic, antimony, and selenium are determined by hydride-atomic absorption spectrometry. The elements are reduced by reducing agents (sodium borohydride ($NaBH_4$)) to form the hydrides. These volatile compounds flow through the heated measuring cuvette of an atomic absorption spectrometer where the content of the individual element is determined.

5.2.3.3.2 Reagents

5.2.3.3.2.1 Hydrochloric acid, high purity analytical grade, 30 % (m/m), density ρ = 1,15 g/ml.

5.2.3.3.2.2 Preliminary reduction agent

Dissolve 10 g sodium iodide and 100 g *L*-ascorbic acid, in 1 000 ml of water.

5.2.3.3.2.3 Reduction solution

Dissolve in water $NaBH_4$ and NaOH in concentrations specified in the manufacturer's handbook for the spectrometer.

5.2.3.3.2.4 Standard solution (100 µg/l As, Sb or Se).

The standard solution shall be freshly prepared on the day of use by individual dilution of a stock solution. This stock solution with an As, Sb or Se content of at least 1 mg/l shall be made by dilution of standard solutions of Se, As and Sb which are available from all major suppliers of laboratory chemicals. This stock solution shall be kept in containers of tetrafluoroethylene - hexafluoropropylene copolymer (FEP), polytetrafluorethylene (PTFE) or polyethylene (PE).

The stock solution should be not kept for longer than four weeks.

5.2.3.3.3 Apparatus

5.2.3.3.3.1 Four one-mark volumetric flasks, 1 000 ml.

5.2.3.3.3.2 Nine one-mark volumetric flasks, 10 ml.

5.2.3.3.3.3 Pipettes 5 ml, 10 ml and 100 ml.

5.2.3.3.3.4 Micropipettes, volume adjustable to maximum 500 µl.

5.2.3.3.3.5 Atomic absorption spectrometer with the measurement parameters specified in Table 3.

The width of the slit, the measuring time, flushing with argon before and after the measurement and the reaction time shall be adjusted in accordance with the manufacturer's instructions. The background compensation shall be activated for the measurement of As and Sb, but not for the measurement of Se.

5.2.3.3.4 Procedure

For As (procedure for Sb and Se in parentheses if different from As procedure): weigh a test portion of 0,5 g to the nearest 0,1mg and transfer it into a 1 000 ml one-mark volumetric flask (5.2.3.3.3.1) and make up to the mark at 20 °C with water. Pipette 10 ml (Sb, Se: 100 ml) of this solution into a 1 000 ml one-mark volumetric flask and add 5 ml of HCl (5.2.3.3.2.1) and 5 ml of preliminary reduction agent (5.2.3.3.2.2). Do not add preliminary reduction agent to the flasks for Sb- and Se-determination. Allow 3 h for reaction to occur and fill to the mark with water. Pipette 5 ml of this solution into three 10 ml one-mark volumetric flasks labelled (5.2.3.3.3.2) A, B, C. Add 0,8 ml of HCl (5.2.3.3.2.1) to each flask. For the purpose of internal calibration, add those quantities of standard solutions (5.2.3.3.2.4) as given in Table 4 to the flasks B and C.

With the spectrometer (5.2.3.3.3.5), carry out the measurement with addition of the reduction agent (5.2.3.3.2.3) and the parameters of measurement in accordance with the manufacturer's instructions for the spectrometer.

Repeat the procedure with all reagents and the same volume of standard solution to be added using water in place of the sample as a blank determination.

Table 4 — Standard solution

	Volume of standard solution to be added		
	As	Sb	Se
Flask B	200 µl	100 µl	200 µl
Flask C	500 µl	200 µl	500 µl

5.2.3.3.5 Expression of results

See 5.2.3.2.5.

5.2.3.3.6 Calculation

See 5.2.3.2.6.

6 Labelling - Transportation - Storage

6.1 Means of delivery

Sodium hexafluorosilicate shall be delivered in suitable containers (not glass), e.g. in drums.

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.

6.2 Labelling according to the EU legislation [4]

The following labelling requirements shall apply to sodium hexafluorosilicate at the date of publication of this document.

Pictograms	
 **Figure 3 — GHS 06**	— Signal word : Danger — Hazard statements : H 301 Toxic if swallowed. H 311 Toxic in contact with skin. H 331 Toxic if inhaled.

NOTE The regulation [2], and its amendments for the purposes of its adaptation to technical and scientific progress, contains a list of substances classified by the EU. Substances not listed in this regulation can 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.

6.3 Transportation regulations and labelling

Sodium hexafluorosilicate is listed as UN Number[5]: 2674.

- RID [6] /ADR [7]: Class 6.1, classification code T5; packing group III.
- IMDG [8]: Class 6.1, packing group III.

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4) See [2].

5) United Nations Number.

6) Regulations concerning International carriage of Dangerous Goods by rail.

7) European Agreement concerning the International carriage of Dangerous Goods by road.

8) International Maritime Transport of Dangerous Goods.

- IATA [9]: Class 6.1, packing group III.

6.4 Marking

The marking shall include the following:

- name 'sodium hexafluorosilicate', trade name;
- net mass;
- name and the address of supplier and/or manufacturer;
- statement "this product conforms to EN 12174".

6.5 Storage

6.5.1 Long term stability

The product is stable when stored in tightly closed containers in a cool and dry place.

6.5.2 Storage incompatibilities

The product shall be kept away from acids, such as hydrochloric acid and sulfuric acid.

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[9] International Air Transport Association.

Annex A
(informative)

General information on sodium hexafluorosilicate

A.1 Origin

A.1.1 Raw materials

The sodium hexafluorosilicate is manufactured from hexafluorosilicic acid and sodium hydroxide or a sodium salt.

A.1.2 Manufacturing process

Hexafluorosilicic acid is neutralised with sodium hydroxide or a sodium salt.

A.2 Use

A.2.1 Function

Sodium hexafluorosilicate is used for the fluoridation of drinking water to increase the resistance of consumers to dental decay.

A.2.2 Form in which it is used

Sodium hexafluorosilicate is used as an aqueous solution (usually 2 g/l).

A.2.3 Treatment dose

The usual treatment dose is that which gives a final concentration of 1 mg/l as F^- (corresponding to 1,65 mg/l as Na_2SiF_6) in the drinking water. It is important to avoid overdosing. In the EU Directive 98/83/EC the parameter value is 1,5 mg/l of fluoride.

A.2.4 Means of application

It is usually applied using a metering pump.

A.2.5 Secondary effects

None.

A.2.6 Removal of excess product

It is practically impossible to remove excess product.

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 immediately with plenty of water.

In case of contact with eyes treat by irrigation with water, with the eyelids held open.

In case of ingestion, seek medical advice immediately.

B.2.2 Spillage

Collect and remove any spillage avoiding the formation of dust.

B.2.3 Fire

The product is not 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 (REACH)

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