BS EN 896:2012

BSI Standards Publication

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

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National foreword

This British Standard is the UK implementation of EN 896:2012. It supersedes BS EN 896:2005 which is withdrawn.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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Compliance with a British Standard cannot confer immunity from legal obligations.

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EN 896

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English Version

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

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

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Natriumhydroxid

This European Standard was approved by CEN on 16 September 2012.

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Foreword

This document (EN 896:2012) 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of an identical text or by endorsement, at the latest by May 2013, and conflicting national standards shall be withdrawn at the latest by May 2013.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 896:2005.

Significant technical differences between this edition and EN 896:2005 are as follows:

a) Modification of 6.2 on labelling, deletion of the reference to EU Directive 80/778/EEC of 15 July 1980 in order to take account of the latest Directive in force.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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

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

EN ISO 12846:2012, *Water quality — Determination of mercury — Method using atomic absorption spectrometry (AAS) with and without enrichment (ISO 12846:2012)*

ISO 979, *Sodium hydroxide for industrial use — Method of assay*

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling*

ISO 3196; *Sodium hydroxide for industrial use — Determination of carbonates content — Titrimetric method*

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

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

Sodium hydroxide.

3.1.2 Synonym or common name

Caustic soda.

3.1.3 Relative molecular mass

40,0.

3.1.4 Empirical formula

NaOH.

3.1.5 Chemical formula

NaOH.

3.1.6 CAS Registry Number[1)]

1310-73-2.

3.1.7 EINECS reference[2)]

215-185-5.

3.2 Commercial forms

The product is available as flakes, pearls, solid, or as an aqueous solution of different concentrations.

3.3 Physical properties

3.3.1 Appearance

Solid: the product is white, deliquescent.

Liquid: the product is a clear solution, slightly turbid colourless solution, slightly viscous.

3.3.2 Density

Solid : the density of this product is 2,1 g/cm^3.

The bulk density of pearls is 1,2 kg/dm^3.

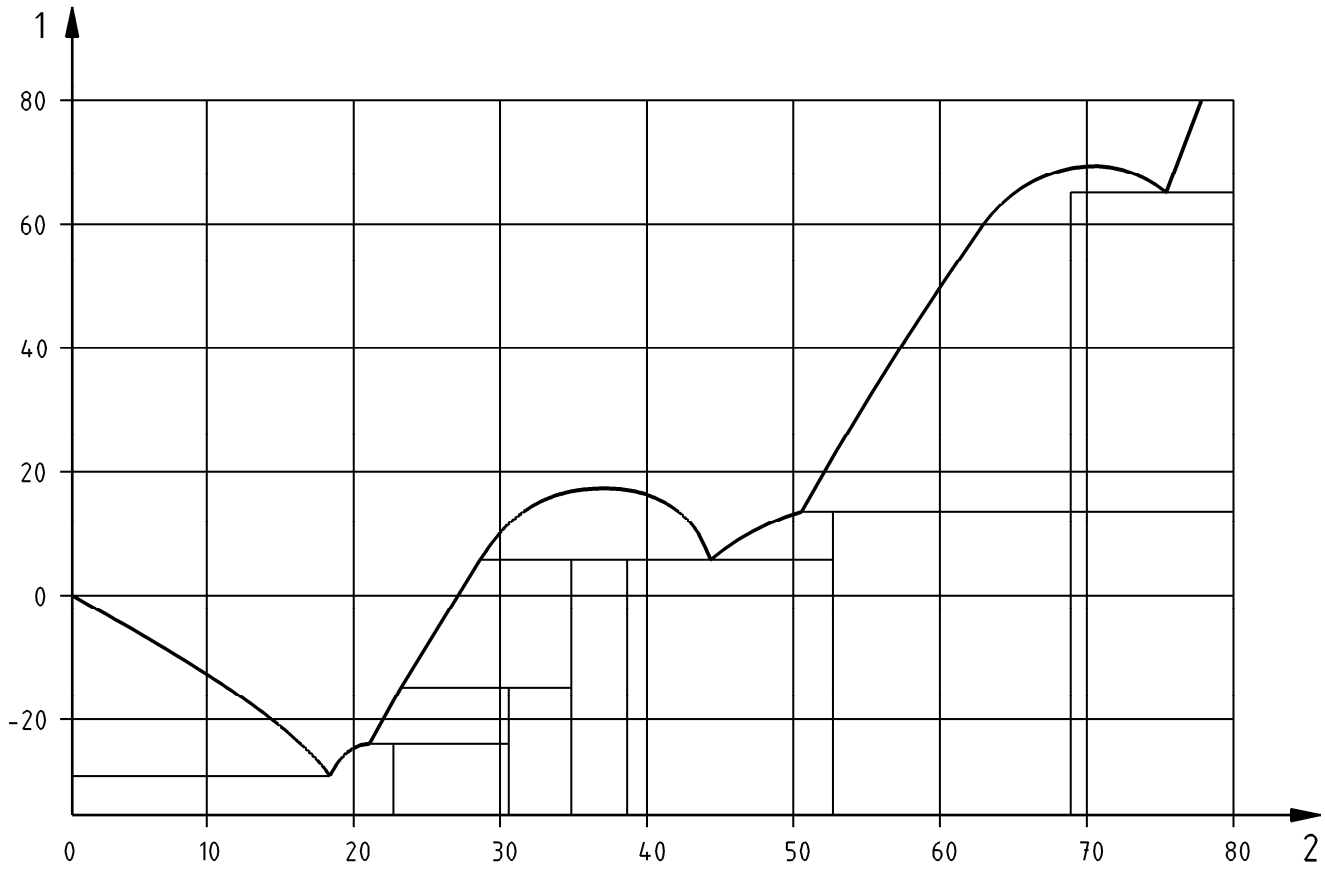
Liquid : the density of solution is 1,52 g/ml for a product concentration of mass fraction of 50 % at 20 °C.

3.3.3 Solubility in water

The product is highly soluble at all temperatures above 20 °C (partial crystallization occurs above concentration of mass fraction of 55 %, (see Figure 1).

1) Chemical Abstracts Service Registry Number.

2) European Inventory of Existing Commercial Chemical Substances.



Key

1 temperature in °C.

2 NaOH concentration in mass fraction in %

Figure 1 — Solubility of sodium hydroxide

3.3.4 Vapour pressure

Solution of concentration of mass fraction of 50 %

- 120 Pa at 20 °C;
- 450 Pa at 40 °C;
- 5 000 Pa at 80 °C.

3.3.5 Boiling point at 100 kPa[3)]

145 °C for a solution of concentration of mass fraction of 50 %.

3.3.6 Crystallization point

+ 12 °C for a solution of concentration of mass fraction of 50 % (see Figure 1).

3.3.7 Specific heat

3 220 J/(kg K) at 20 °C for a solution of concentration of mass fraction of 50 %.

3.3.8 Viscosity (dynamic)

For a solution of concentration of mass fraction of 50 %:

- 100 Pa.s at 20 °C;
- 25 Pa.s at 40 °C;
- 5 Pa.s at 80 °C.

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 solutions of sodium hydroxide are strongly alkaline.

Dilution of sodium hydroxide is very exothermic.

4 Purity criteria

4.1 General

This European Standard specifies the minimum purity requirements for sodium hydroxide 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.

3) 100 kPa = 1 bar.

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 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 leads 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 product shall contain not less than a mass fraction of 96 % of NaOH for the solid form. Typical concentration for solutions of sodium hydroxide is either a mass fraction of 50 % or 30 % , and shall be in any case within the manufacturer's stated tolerance.

4.3 Impurities and main by-products

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

The concentration limits refer to pure NaOH mass fraction of 100 %.

Table 1 — Impurities

Impurity	**Limit in mass fraction in % of NaOH**
Sodium chloride (NaCl) [a] max.	2,4
Sodium carbonate (Na_2CO_3) [b] max.	0,4
Sodium chlorate ($NaClO_3$) [c] max.	0,7
a) Too high concentrations can cause problems with some ion exchange resins. b) Sodium carbonate is formed in contact with atmospheric carbon dioxide. c) The presence of any oxidizing agent in sodium hydroxide is to be avoided.	

4.4 Chemical parameters

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

Table 2 — Chemical parameters

Parameter		Limit in mg/kg of NaOH	
		Type 1	**Type 2**
Arsenic (As)	max.	2	10
Cadmium (Cd)	max.	1	5
Chromium (Cr)	max.	1	10
Mercury (Hg)	max.	0,1	1
Nickel (Ni)	max.	2	10
Lead (Pb)	max.	5	20
Antimony (Sb)	max.	5	5
Selenium (Se)	max.	5	5
NOTE Cyanides, pesticides and polycyclic aromatic hydrocarbons are not relevant in sodium hydroxide. For parametric values of sodium hydroxide on trace metal content in drinking water, see [1].			

5 Test methods

5.1 Sampling

Prepare the laboratory sample(s) required by the relevant procedure described in ISO 8213, observe the recommendations of ISO 3165 and also take into account ISO 6206. The nature of caustic alkalis requires special care at all points of sampling and preparation for analysis. Sampling techniques shall be such as to limit or prevent atmospheric exposure since sodium hydroxides, either as aqueous solutions or as anhydrous products, rapidly absorb moisture and carbon dioxide (and other acid gases) from the atmosphere. Additional precautions are necessary if trace constituents are to be determined.

NOTE For sampling liquids see [2].

5.2 Analyses

5.2.1 Main product

5.2.1.1 Total alkalinity

The total alkalinity shall be determined by titration with an acid standard volumetric solution in accordance with ISO 979.

5.2.1.2 Caustic alkalinity

The caustic alkalinity equals the total alkalinity as NaOH (see 5.2.1.1) minus the alkalinity as Na_2CO_3 multiplied by 0,755 determined in accordance with ISO 3196.

5.2.2 Impurities

5.2.2.1 Sodium chloride

The sodium chloride content shall be determined by potentiometric titration with silver nitrate solution (see B.1).

5.2.2.2 Sodium carbonate

The sodium carbonate content shall be determined by the titrimetric method, in accordance with ISO 3196.

5.2.2.3 Sodium chlorate

The sodium chlorate content shall be determined by ionic chromatography (see B.2).

5.2.3 Chemical parameters

5.2.3.1 Principle

The elements antimony, arsenic, cadmium, chromium, lead, nickel and selenium are determined by inductively coupled plasma optical emission spectrometry. Mercury is determined by cold vapour atomic absorption spectrometry.

5.2.3.2 Arsenic

The arsenic content shall be determined by inductively coupled plasma optical emission spectrometry (ICP/OES) (see B.3).

5.2.3.3 Cadmium

The cadmium content shall be determined by inductively coupled plasma optical emission spectrometry (ICP/OES) (see B.3).

5.2.3.4 Chromium

The chromium content shall be determined by inductively coupled plasma optical emission spectrometry (ICP/OES) (see B.3).

5.2.3.5 Nickel

The nickel content shall be determined by inductively coupled plasma optical emission spectrometry (ICP/OES) (see B.3).

5.2.3.6 Lead

The lead content shall be determined by inductively coupled plasma optical emission spectrometry (ICP/OES) (see B.3).

5.2.3.7 Antimony

The antimony content shall be determined by inductively coupled plasma optical emission spectrometry (ICP/OES) (see B.3).

5.2.3.8 Selenium

The selenium content shall be determined by inductively coupled plasma optical emission spectrometry (ICP/OES) (see B.3).

5.2.3.9 Mercury

The mercury content shall be determined by cold vapour atomic absorption spectrometry in accordance with EN ISO 12846 (see B.4).

6 Labelling – Transportation – Storage

6.1 Means of delivery

Sodium hydroxide can be delivered in rail or road tankers, in containers, in drums or in plastics bags.

In order that the purity of the products 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 hydroxide at the date of the publication of this standard.

4) See [3]

Hazard label

Figure 2 GHS 05

– Signal word :

Danger

– Hazard statement:

H 314 : causes severe skin burns and eye damage

Precautionary statements ("P statements") should be provided by the company being responsible for the marketing of the substance. They should be indicated on the packaging label and in the extended safety data sheet (eSDS) of the substance.

The regulation [3], 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 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.

6.3 Transportation regulations and labelling

Sodium hydroxide solid is listed as UN Number[5]: 1823.

Sodium hydroxide solution is listed as UN Number: 1824.

RID[6]/ADR[7] : - class 8, classification code C6, packing group II for solid;

- class 8, classification code C5, packing group II for solution.

IMDG[8] : Class 8, packing group II.

IATA [9] : Class 8, packing group II.

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[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.

[9] International Air Transport Association.

6.4 Marking

The marking shall include the following information:

— name " sodium hydroxide", trade name and type;

— net mass;

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

— statement " this product conforms to EN 896".

6.5 Storage

6.5.1 Material

Avoid contact with aluminium, zinc or galvanised steel material. Mild steel, polyester or polypropylene (polypropene) are suitable materials. To avoid any iron contamination in the product, a suitable lining of the steel tank may be used.

6.5.2 Long term stability

Absorption of carbon dioxide from the ambient air leads to the formation of sodium carbonate.

6.5.3 Storage incompatibilities

Avoid contact with metals such as zinc, aluminium, copper, tin or their alloys, which produce hydrogen. Violent reaction is to be expected when sodium hydroxide comes in contact with concentrated acids, and organic chemicals, particularly chlorinated hydrocarbons.

Annex A
(informative)

General information on sodium hydroxide

A.1 Origin

A.1.1 Raw materials

Sodium chloride solution: For alternative production route: sodium carbonate and calcium hydroxide.

A.1.2 Manufacturing process

Electrolysis of sodium chloride solution (brine) in a mercury cell, a membrane cell or a diaphragm cell. Can also be produced by caustification of sodium carbonate with calcium hydroxide.

A.2 Use

A.2.1 Function

Sodium hydroxide is mainly used as a neutralizing agent, for adjustment of pH value, as a softening agent, for alkalinity adjustment or as a regenerant for ion exchange resins.

A.2.2 Form in which it is used

Sodium hydroxide is mainly used as delivered, or diluted if more convenient.

A.2.3 Treatment dose

The treatment dose is depending of the application or the initial pH and the buffer capacity of the water.

A.2.4 Means of application

The product is usually applied using a metering pump or a dissolving tank.

A.2.5 Secondary effects

Temperature rise at the injection point.

A.2.6 Removal of excess product

Excessive pH values can be readjusted by adding acids (such as sulfuric acid).

Annex B
(normative)

Analytical methods

B.1 Determination of sodium chloride (potentiometric titration)

B.1.1 General

This method applies to products with a chloride content within the range of mass fraction of 0,5 % to 2 %.

B.1.2 Principle

Chloride is determined by potentiometric titration with silver nitrate using a silver electrode.

NOTE An automatic titrator or a manual system can be used to determine the end point.

B.1.3 Reagents

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

B.1.3.1 Nitric acid (HNO_3), density $\rho \approx 1{,}40$ g/ml

B.1.3.2 Silver nitrate standard volumetric solution $\rho(AgNO_3) = 0{,}1$ mol/l

B.1.3.3 Phenolphthalein indicator solution 10 g/l in ethanol volume fraction 95 %

B.1.4 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

B.1.4.1 **Automatic titrator or pH meter** used in mV mode (reading of scale in millivolts)

B.1.4.2 **Silver combined electrode** with double junction

B.1.4.3 **Burette**, automatic or manual type

B.1.5 Procedure

B.1.5.1 Test portion

Weigh, to the nearest 0,01 g, a mass of laboratory sample corresponding to 10 g of NaOH.

B.1.5.2 Determination

Transfer the test portion to a 250 ml conical flask. Add 100 ml of water, 2 or 3 drops of phenolphthalein (B.1.3.3) and dissolve using a magnetic stirrer.
Neutralize the solution with the nitric acid (B.1.3.1) and add 2 or 3 drops in excess.
After cooling, insert the silver combined electrode (B 1.4.2) in the solution and titrate with the silver nitrate standard volumetric solution (B.1.3.2), either manually using the pH meter (B 1.4.1) in the mV mode or with an automatic titrator (B 1.4.1), following the manufacturer's instructions for titration and end point determination.

Perform a blank titration on the same volume of nitric acid used for the acidification of the test portion.

NOTE For manual titration, it is recommended to record the mV readings obtained after each addition of the silver nitrate standard volumetric solution, to plot a graph showing the volumes of standard volumetric solution used versus the mV readings and to determine the volume corresponding to the end-point at the point of the inflection.

B.1.6 Expression of results

The chloride content $\rho_{(Cl)}$, expressed in mass fraction in % is given by the following formula:

$$\rho(CI) = \frac{(V_1 - V_2) \times \rho \times 0{,}035453 \times 100}{m} \qquad \text{(B.1)}$$

where

V_1 is the volume in millilitres of silver nitrate standard volumetric solution used for the sample;

V_2 is the volume in millilitres of silver nitrate standard volumetric solution used for the blank;

ρ is the concentration, in moles per litre, of the silver nitrate standard volumetric solution used;

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

0,035453 is the mass in grams of chloride corresponding to 1,00 ml of silver nitrate solution $\rho(AgNO_3)$=1,000 mol/l.

B.2 Determination of sodium chlorate (ion chromatography)

B.2.1 General

This method applies to products with sodium chlorate ($NaClO_3$) contents within the range of 2 g/kg to 20 g/kg.

B.2.2 Principle

Direct determination of the chlorate ion in a diluted sample solution by ionic chromatography apparatus equipped with a chemical suppression device and a conductimetric detector.

B.2.3 Reagents

All reagents shall be of recognized analytical grade and the water used shall have a conductivity lower than 56 µS/cm.

B.2.3.1 Eluant solution, solution containing Na_2CO_3 and $NaHCO_3$.

B.2.3.2 Regenerant solution, $\rho_{(H_2SO_4)}$ = 0,025 mol/l.

B.2.3.3 Sodium chlorate standard stock solution, $\rho_{(NaClO_3)}$ = 1 000 µg/ml.

Dissolve (0,2000 ± 0,0001) g of $NaClO_3$, with water. Dilute with water to 200 ml in a volumetric flask and mix.

B.2.4 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

B.2.4.1 Ionic chromatograph equipped with:

a) chemical suppressor;

b) conductivity detector;

c) anion separation column and precolumn: Consists of polystyrene/divinylbenzene substrate agglomerated with aminated anion exchange latex;

d) data logger/plotter.

B.2.4.2 Cartridge of cation exchange resin (H^+ form) type "ON-GUARD H^+ [10)] or equivalent (cation exchange capacity ≈1,8 mmol H^+).

B.2.5 Procedure

B.2.5.1 Test portion

Weigh, to the nearest 0,001 g, 0,5 g of the laboratory sample.

B.2.5.2 Test solutions

Transfer the test portion to a 200 ml volumetric flask, dilute to the mark with the eluant solution (B 2.3.1) and mix. This is the first test solution.

Prepare a second test solution by dilution of 25 ml of the first test solution with the eluant solution (B.2.3.1) in a 100 ml volumetric flask.

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10) ON-GUARD H^+ is the trade name of a product supplied by DIONEX. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN/CENELEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

B.2.5.3 Calibration solutions

Prepare calibration solutions by diluting the chlorate stock solution (B.2.3.3) with the eluant solution (B.2.3.1) according to the Table B.1.

Table B.1 - Calibration solutions

Calibration solution No	Sodium chlorate concentration mg/l
0	0
1	4
2	8
3	12
4	16

B.2.5.4 Determination

B.2.5.4.1 Test solutions pretreatment

Proceed as follows to eliminate the OH^- ions from the test solution(s).

Flush 25 ml of water through a cartridge containing cation exchange resin on H^+ form (B 2.4.2).

Slowly push 10 ml of the test solution through the cartridge. Reject the first millilitre and use the rest for the determination.

B.2.5.4.2 Preparation of the apparatus

Set all instrument parameters of ionic chromatograph (B.2.4.1) in accordance with the operating manual of the instrument's manufacturer with the following conditions:

- eluant (B 2.3.1) flow rate: 2 ml/min;
- regenerant (B 2.3.2) flow rate: 2,5 ml/min;
- full scale of conductivity: 30 µS/cm;
- residual conductivity: < 18 µS/cm;
- number of replicates: 3.

B.2.5.4.3 Measurements

Measure each solution at least three times in order to obtain a relative standard deviation lower than 1 %.

Calibrate the instrument with the five calibration solutions.

Measure the test solution (first or second) having a chlorate concentration located more or less in the middle of the range of the calibration curve.

B.2.6 Expression of results

The chlorate content $\rho(NaClO_3)$ of the sample expressed in grams per litre, is given by the formula:

$$\rho_1(NaClO_3) = \frac{200 \times \rho_1}{1\,000\ m} \quad \text{(B.2)}$$

in case of undiluted test solution (first),

or

$$\rho_2(NaClO_3) = \frac{800 \times \rho_1}{1\,000\ m} \quad \text{(B.3)}$$

in case of diluted test solution (second),

where

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

ρ_1 is the concentration of sodium chlorate in the measured test solution expressed in milligrams per litre.

B.3 Determination of antimony, arsenic, cadmium, chromium, lead, nickel and selenium (inductively coupled plasma optical emission spectrometry (ICP/OES))

B.3.1 General

The concentration range covered for each element is given in Table B.2.

Table B.2 - Concentration range

Element	Concentration range, mg/kg of product
Cr, Cd	0,2 to 40
Ni	0,5 to 40
As, Sb, Se	1 to 40
Pb	2 to 40

B.3.2 Principle

Dissolution of the sample with nitric acid and direct nebulization of the acid solution into inductively coupled argon plasma formed by a high frequency. Measurement of the radiations at specific wavelengths using background correction and internal standardization.

B.3.3 Reagents

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

Store all prepared solutions in polyethylene or polytetrafluoruethylene (PTFE) flasks to prevent contamination.

B.3.3.1 **Nitric acid solution**, $\rho \approx 1{,}40$ g/ml, mass fraction 65 %.

B.3.3.2 **Hydrochloric acid solution,** $\rho \approx 1{,}19$ g/ml, mass fraction 37 %.

B.3.3.3 **Sodium chloride solution,** $\rho(NaCl) = 250$ g/l.
Dissolve 250 g of NaCl (very high purity grade) with water and transfer to a 1 000 ml volumetric flask. Add 10 ml of nitric acid (B.3.3.1), make up to the mark with water and mix.

B.3.3.4 **Scandium (internal standard) solution,** $\rho(Sc) = 50$ mg/l.
Transfer 50 ml of a scandium stock solution [$\rho(Sc) = 1\,000$ mg/l] and 10 ml nitric acid (B.3.3.1) to a 1 000 ml volumetric flask. Make up to the mark with water and mix.

B.3.3.5 **As, Cd, Cr, Ni, Pb, Sb or Se elements stock solution**, ρ(element) = 1 000 mg/l commercial solution.

B.3.3.6 **Multi-element solution**, ρ(As, Cd, Cr, Ni, Pb, Sb, Se) = 100 mg/l
Transfer 10 ml of each stock solution (B.3.3.5) and 10 ml of hydrochloric acid (B.3.3.2) to a 100 ml volumetric flask, make up to the mark with water and mix.

B.3.3.7 **Argon,** the pressure shall not be less than 700 kPa and the argon used may be compressed or liquefied gas.

B.3.4 Apparatus

Ordinary laboratory apparatus and glassware with together the following:

All vessels (glassware, polyethylene (polyethene), polypropylene (polypropene) and polytetrafluorethylene (polytetrafluorethene) (PTFE) flasks) should be washed with hydrochloric acid $\rho(HCl) \approx 6$ mol/l and water successively.

B.3.4.1 Inductively coupled plasma optical emission spectrometer (ICP/OES) fitted with a nebulizer for high salt concentration. This instrument can be simultaneous and/or sequential. The spectrometer parameters and operating conditions are given in Table B.3:

Table B.3 - Parameters and operating conditions of the spectrometer

Parameter	Unit	Specification
Type		monochromator or /and polychromator
Argon humidifier (water)		yes
Argon (B 3.3.7) flows :		
Plasma	l/min	14
Auxiliary	l/min	1,5
Nebulizer (180 kPa)	l/min	0,7
Sample flow	ml/min	1,5
RF power	W	± 1 000
Integration time	s	10

B.3.5 Procedure

B.3.5.1 Test portion

Weigh, to the nearest 0,1 g, 14 g of the laboratory sample.

B.3.5.2 Test solution

Transfer the test portion (B.3.5.1) and 50 ml of water to a 250 ml polyethylene flask. After dissolution, neutralize the solution with hydrochloric acid (B.3.3.2) and add 1 ml nitric acid (B.3.3.1).

After cooling, transfer to a 100 ml volumetric flask, add 5 ml of scandium solution (B.3.3.4), make up to the mark with water and mix.

B.3.5.3 Calibration and verification solutions

Transfer 80 ml of sodium chloride solution (B.3.3.3), 5 ml of scandium solution (B.3.3.4) and the volumes of multi-element solution (B.3.3.6) given in Table B.4 to a series of four 100 ml volumetric flasks. Make up to the mark with water and mix.

Table B.4 - Calibration solutions for the different elements

Calibration solution No	Multi-element solution, ml	Corresponding concentration of each element (As, Cd, Cr, Ni, Pb, Sb, Se) mg/l
1[1)]	0	0
2[2)]	5,0	5,0
3	10,0	10,0
4[3)]	10,0	10,0

1) Blank calibration solution.

2) Linearity standard matching solution.

3) Control solution prepared with different pipettes, flasks and if possible with different stock solutions.

B.3.5.4 Determination

B.3.5.4.1 Preparation of the apparatus

Set all instrument parameters of the optical emission spectrometer (B.3.4.1) in accordance with the operating manual of the instrument's manufacturer.

Prepare the analytical procedure including the lines shown in Table B.5, with background correction, concentrations of calibration solutions 1 and 3 described in (B.3.5.3) and internal standardization (B.3.3.4).

Table B.5 - Wavelength per element

Element	Wavelength nm	
	line	background
As	193,759	193,79
Cd	228,802	228,83
	214,438	-
Cr	267,716	267,75
Ni	231,604	231,63
Pb	220,353	220,38
Sb	217,581	217,61
Se	196,026	196,05
Sc (internal standard)	424,683 or 361,384	- -

B.3.5.4.2 Spectrometric measurements

Repeat the measurements for at least five integration periods.

Rinse with water after each solution.

Calibrate the instrument with the calibration solutions 1 and 3 (B.3.5.3).

Control and check the linearity of the calibration curve by measurement of the following calibration solutions considered as unknown solutions:

- solution 3;
- solution 1;
- solution 1;
- solution 2;
- solution 4;
- solution 3.

Continue the measurements in the following order:

- solution 3;
- solution 1;
- solution 1;
- test solution (B.3.5.2);
- solution 3;

- solution 1 (B.3.5.3.);

- solution 1 (B.3.5.3.).

B.3.6 Expression of results

B.3.6.1 Evaluation

If necessary, correct for drift the results obtained with the test solution and control solutions 2 and 4:

- **for baseline drift** by interpolating in time between both second measurements (the first might be cross-contaminated) of the blank calibration solution (solution 1);

- **for sensitivity drift** by interpolating in time between the measurements of the solution 3.

Samples of unknown composition should be tested for the presence of matrix effects, caused by present components other than sodium hydroxide, by the analyte addition technique.

B.3.6.2 Calculation

The element content of the sample, ρ(element) in milligrams per kilogram is given by the formula:

$$\rho(element) = \frac{100 \times \rho_2}{m} \qquad \text{(B.4)}$$

where

m is the mass, in grams, of the test portion (B.3.5.1);

ρ_2 is the corrected concentration of element, in milligrams per litre, in the test solution (B.3.5.2).

B.4 Determination of mercury (cold vapour atomic absorption spectrometry)

B.4.1 General

This method is suitable for the determination of total mercury in sodium hydroxide. The method is applicable to samples whose mercury content is greater than 0,05 mg/kg as Hg.

B.4.2 Principle

After mineralization of the sample with sulfuric acid and potassium permanganate, the total mercury is determined by cold vapour atomic absorption spectrometry as described in EN ISO 12846.

B.4.3 Reagents

See 4.3 of EN ISO 12846:2012.

B.4.4 Apparatus

See 4.4 of EN ISO 12846:2012.

B.4.5 Procedure

B.4.5.1 Test portion

Weigh, to the nearest 0,01 g, 2 g of the laboratory sample.

B.4.5.2 Test solution

Transfer the test portion and 50 ml of water to a 250 ml conical flask. After dissolution, neutralize the solution with hydrochloric acid solution ρ(HCl) = 6 mol/l. Add 1 ml of potassium permanganate solution (50 g/l) and, with care five 1 ml portions of sulfuric acid (ρ = 1,84 g/ml).

Heat and keep boiling for 1 min.

After cooling, dissolve the precipitate of manganese dioxide, dropwise, with the hydroxylamine hydrochloride solution (100 g/l), add 5 ml of potassium dichromate solution (4 g/l), dilute to 100 ml with water in a volumetric flask and mix.

B.4.5.3 Blank test solution

Prepare a blank test solution according to the instructions detailed in B.4.5.2 but omitting the test portion.

B.4.5.4 Calibration solutions

Just before use, prepare a series of six calibration solutions containing 0 mg/l, 1 mg/l, 2,5 mg/l, 5 mg/l, 7,5 mg/l and 10 mg/l of Hg.

To 100 ml of those solutions add 1 ml of potassium permanganate solution and with care five 1 ml portions of sulfuric acid and continue as described in B.4.5.2 for the test solution.

B.4.5.5 Determination

Proceed with the calibration solutions, test solution and blank test solution as described in 4th paragraph of 4.7.1 in EN ISO 12846:2012 beginning at "Transfer in a flask..."

B.4.6 Expression of results

See 4.8 and 4.9 of EN ISO 12846:2012.

Annex C
(normative)

General rules relating to safety

C.1 Rules for safe handling and use

The supplier shall provide current safety instructions.

C.2 Emergency procedures

C.2.1 General

See also 6.2.

C.2.2 First aid

In case of contact with skin rinse with plenty of water.

In case of ingestion, and the victim is conscious, rinse the mouth with water, let the victim drink fresh water or slightly acidified with acetic acid (vinegar). Consult a doctor in all cases immediately.

C.2.3 Spillage

Collect as much as possible in suitable containers. Neutralize with acids, rinse small spillages with plenty of water.

C.2.4 Fire

Sodium hydroxide is not combustible.

Bibliography

[1] 98/83/EC, *Council Directive of 3 November 1998 on the Quality of Water intended for Human Consumption*

[2] ISO 3195, *Sodium hydroxide for industrial use — Sampling — Test Sample — Preparation of the main solution for carrying out certain determinations*

[3] 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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