BS EN 902:2016

BSI Standards Publication

Chemicals used for treatment of water intended for human consumption — Hydrogen peroxide

National foreword

This British Standard is the UK implementation of EN 902:2016. It supersedes BS EN 902:2009 which is withdrawn.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 2016

ISBN 978 0 580 86577 0

ICS 13.060.20; 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 30 June 2016.

Amendments/corrigenda issued since publication

Date	Text affected

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 902

May 2016

ICS 71.100.80

Supersedes EN 902:2009

English Version

Chemicals used for treatment of water intended for human consumption - Hydrogen peroxide

Produits chimiques utilisés pour le traitement de l'eau destinée à la consommation humaine - Peroxyde d'hydrogène

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Wasserstoffperoxid

This European Standard was approved by CEN on 18 March 2016.

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COMITÉ EUROPÉEN DE NORMALISATION
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Ref. No. EN 902:2016 E

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European foreword

This document (EN 902:2016) 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of an identical text or by endorsement, at the latest by November 2016, and conflicting national standards shall be withdrawn at the latest by November 2016.

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 902:2009.

Significant technical differences between this edition and EN 902:2009 are as follows:

a) deletion of the reference to EU Directive 67/548/EEC of June 27,1967 in order to take into account the latest Directive in force (see [1]);

b) use of the changed classification and labelling (see [3]).

According to the CEN/CENELEC Internal Regulations, the national standards organizations 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:

1) 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;

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 1 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.

NOTE 2 This product is a biocide and needs to comply with the relevant legislation in force. In the European Union, at the time of publication, this legislation is REGULATION (EU) No 528/2012 [1].

1 Scope

This European Standard is applicable only to hydrogen peroxide and not to mixtures with other chemicals used for treatment of water intended for human consumption. It describes the characteristics of hydrogen peroxide and specifies the requirements and the corresponding test methods for hydrogen peroxide. It gives information on its use in water treatment. It also determines the rules relating to safe handling and use (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, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

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

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

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

ISO 8288, *Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods*

ISO 9174, *Water quality — Determination of chromium — Atomic absorption spectrometric methods*

3 Description

3.1 Identification

3.1.1 Chemical name

Hydrogen peroxide.

3.1.2 Synonym or common name

None.

3.1.3 Relative molecular mass

34,02.

3.1.4 Empirical formula

H_2O_2.

3.1.5 Chemical formula

H-O-O-H.

3.1.6 CAS Registry Number[1]

7722-84-1.

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[1] Chemical Abstracts Service Registry Number.

3.1.7 EINECS reference[2)]

231-765-0.

3.2 Commercial form

The product is supplied as an aqueous solution.

3.3 Physical properties

3.3.1 Appearance and odour

The product is colourless liquid, slightly pungent odour.

3.3.2 Density

The density of hydrogen peroxide is given in Table 1.

Table 1 — Density

Solution concentration Mass fraction in%	**Density** g/ml at 20 °C
20	1,075
30	1,114
35	1,132
50	1,195
60	1,241
70	1,289

3.3.3 Solubility in water

The product is miscible with water in all proportions.

3.3.4 Vapour pressure

The vapour pressure of hydrogen peroxide depending on concentration is given in Table 2.

Table 2 — Vapour pressure

Solution concentration Mass fraction in %	**Vapour pressure** kPa at 20 °C
20	2,0
30	1,8
35	1,7
50	1,3
60	1,1
70	0,8

3.3.5 Boiling point at 100 kPa[3)]

The boiling point of hydrogen peroxide depending on concentration is given in Table 3.

[2)] European Inventory of Existing Commercial Chemical Substances.

[3)] 100 kPa = 1 bar.

Table 3 — Boiling point

Solution concentration Mass fraction in %	**Boiling point** °C at 100 kPa
20	103
30	106
35	108
50	114
60	119
70	125

3.3.6 Crystallization point

The crystallization point of hydrogen peroxide depending on concentration is given in Table 4.

Table 4 — Crystallization point

Solution concentration Mass fraction in %	**Crystallization point** °C
20	– 14,6
30	– 25,7
35	– 32,5
50	– 51
60	– 55
70	– 37

3.3.7 Specific heat

The specific heat of hydrogen peroxide depending on concentration is given in Table 5.

Table 5 — Specific heat

Solution concentration Mass fraction in %	**Specific heat** kJ/(kg.K)
20	3,82 at 20 °C
50	3,32 at 20 °C
100	2,63 at 25 °C

3.3.8 Viscosity, dynamic

The viscosity of hydrogen peroxide depending on concentration is given in Table 6.

Table 6 — Viscosity

Solution concentration Mass fraction in %	**Viscosity** MPa.s at 20 °C
20	1,04
30	1,07
35	1,10
50	1,17
60	1,20
70	1,24
100	1,25

3.3.9 Critical temperature

The critical temperature of pure hydrogen peroxide is 457 °C.

3.3.10 Critical pressure

The critical pressure of pure hydrogen peroxide is 21,7 mPa.

3.3.11 Physical hardness

Not applicable.

3.4 Chemical properties

Hydrogen peroxide is a weak acid.

According to species in solution, it is an oxidizing agent (E° = 1,776 V) or a reducing agent (E ° = 0,682 V).

Oxidizing agent:

$H_2O_2 + 2H^+ + 2e^- --- > 2H_2O$

Reducing agent:

$H_2O_2 --- > O_2 + 2H^+ + 2e^-$

NOTE 1 It can be activated by ultraviolet light, ozone or metals to generate free radicals.

NOTE 2 Singlet oxygen can be obtained by reaction of hydrogen peroxide with hypochlorite ion.

4 Purity criteria

4.1 General

This European Standard specifies the minimum purity requirements for hydrogen peroxide 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 product not stated in this product standard.

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 hydrogen peroxide is usually available in concentrated solution with concentration within the range of mass fraction of 20 % to 70 %. Diluted products are also available.

The concentration of hydrogen peroxide shall be equal to or greater than the manufacturer specified value.

4.3 Impurities and main by-products

Not applicable.

4.4 Chemical parameters

The product shall conform to the requirements specified in Table 7.

Table 7 — Chemical parameters

Parameter		Limit in mg/kg of product (mass fraction 100 %)	
		Type 1	**Type 2**
Arsenic (As)	max.	0,5	1
Cadmium (Cd)	max.	0,5	1
Chromium (Cr)	max.	0,5	1
Mercury (Hg)	max.	0,5	1
Nickel (Ni)	max.	1	5
Lead (Pb)	max.	0,5	1
Antimony (Sb)	max.	0,5	1
Selenium (Se)	max.	0,5	1

NOTE Cyanide which does not exist in a strong oxidizing medium such as hydrogen peroxide is not a relevant chemical parameter. Pesticides and polycyclic aromatic hydrocarbons are not by-products of the manufacturing process. For parametric values of chemical parameters in drinking water, see [2].

5 Test methods

5.1 Sampling

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

5.2 Analysis

5.2.1 Determination of hydrogen peroxide content (main product)

5.2.1.1 Principle

Titration of a test portion of hydrogen peroxide in acidic medium with a potassium permanganate standard volumetric solution.

5.2.1.2 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.

5.2.1.2.1 Sulfuric acid, solution, free from reducing substance, 440 g/l:

Check for the absence of reducing substances by adding two drops of the potassium permanganate solution (5.2.1.2.2) to 1 l of the sulfuric acid solution. The pink coloration shall persist for at least 30 min.

5.2.1.2.2 Potassium permanganate, standard volumetric solution, $c(KMnO_4) = 0{,}1$ mol/l.

Weigh about 16 g of potassium permanganate ($KMnO_4$) and dissolve in 1 l of water.

Mix the solution and store it in a coloured, ground glass stoppered bottle for at least 10 days before standardizing.

The standardization shall be carried out in accordance with the following procedure:

a) Preparation of the sodium oxalate, standard volumetric solution, $c(Na_2C_2O_4) = 0{,}25$ mol/l:

Dry the sodium oxalate ($Na_2C_2O_4$) at 200 °C for 24 h and allow to cool in a desiccator. Weigh to the nearest 0,000 1 g, 16,75 g of this product and dissolve it in 200 ml of the sulfuric acid solution (5.2.1.2.1). Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

Store the solution in a coloured, ground glass stoppered bottle. Store for at least 3 days before using and discard after 30 d.

b) Determination of the concentration:

Introduce 50 ml (V_1) of the sodium oxalate solution (a) into a 500 ml conical flask and add 45 ml of water and 15 ml of the sulfuric acid solution (5.2.1.2.1).

Heat the mixture to 40 °C by means of a hot water bath. Using a burette, quickly add 38 ml of the potassium permanganate solution (5.2.1.2.2.). Discoloration shall occur after shaking for several seconds; if necessary raise the temperature of the test solution slightly. Continue adding the potassium permanganate solution, drop by drop, until a pink coloration is obtained which persists for 30 s. Note the volume V_2.

c) Calculation:

The concentration (c_2) of the potassium permanganate solution (5.2.1.2.2) expressed in moles per litre is given by the following formula:

$$c_2 = 2/5 \times (c_1 \times V_1)/V_2 \qquad (1)$$

where

c_1 is the concentration, in moles per litre, of the sodium oxalate solution used for the titration in c) ($c_1 = 0{,}25$ mol/l);

V_1 is the volume, in millilitres, of the sodium oxalate solution used for the titration in b) ($V_1 = 50{,}00$ ml);

V_2 is the volume, in millilitres, of the potassium permanganate solution used for the titration in b).

5.2.1.3 Apparatus

Ordinary laboratory apparatus and glassware together with glass containers, about 10 mm in diameter and 20 mm long.

5.2.1.4 Procedure

5.2.1.4.1 Test portion

Depending on the concentration of the test sample, weigh, to the nearest 0,000 1 g, into one of the clean glass containers (5.2.1.3) the mass of the test sample (m_0) indicated below:

- about 1,1 g for H_2O_2 solutions with concentration of mass fraction 35 %;
- about 0,75 g for H_2O_2 solutions with a concentration of mass fraction 50 %;
- about 0,55 g for H_2O_2 solutions with a concentration of mass fraction 70 %.

5.2.1.4.2 Determination

Introduce 60 ml of water and 15 ml of the sulfuric acid solution (5.2.1.2.1) into a 500 ml conical flask. Add the potassium permanganate solution (5.2.1.2.2), drop by drop, shaking continuously, until a slight pink coloration is obtained which persists for 30 s (one drop is usually sufficient).

Add the test portion in the glass container and titrate with the potassium permanganate standard volumetric solution (5.2.1.2.2) until a pink coloration is obtained which persists for 30 s. Record the volume (V_3).

NOTE The determination can be easily automated using automatic titration apparatus which gives an end point in 15 s to 20 s.

5.2.1.5 Expression of results

The concentration of hydrogen peroxide (c_3), expressed in mass fraction in % is given by the following formula:

$$c_3 = 5/2 \times (c_2 \times V_3 \times M)/(m_0 \times 10) \qquad (2)$$

where

V_3 is the volume, in millilitres, of the potassium permanganate standard volumetric standard volumetric solution (5.2.1.2.2) used for the determination;

c_2 is the concentration, in moles per litre, of the potassium permanganate standard volumetric solution (5.2.1.2.2);

m_0 is the mass, in grams, of the test portion (5.2.1.4.1);

M is the molecular mass of hydrogen peroxide (M = 34,01 g/mol).

Express the result to the second significant figure.

5.2.2 Chemical parameters

5.2.2.1 Determination of antimony (Sb), arsenic (As), cadmium (Cd), chromium (Cr), lead (Pb), nickel (Ni) and selenium (Se)

5.2.2.1.1 Principle

The elements arsenic, antimony, cadmium, chromium, lead, nickel and selenium are determined by atomic absorption spectrometry.

5.2.2.1.2 Reagents

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

5.2.2.1.2.1 Nitric acid, concentrated, density ρ = 1,42 g/ml.

5.2.2.1.3 Procedure

5.2.2.1.3.1 Test portion

Weigh, to the nearest 0,001 g, 20 g (m) from the laboratory sample into a glass beaker.

5.2.2.1.3.2 Test solution

Evaporate until a wet residue is obtained, cool, add 1 ml of nitric acid (5.2.2.1.2.1), dilute with a few millilitres of water, transfer quantitatively to a 100 ml one-mark volumetric flask and dilute to volume with mater and mix.

Carry out the evaporation carefully and not to dryness in order to avoid possible losses of arsenic and selenium.

5.2.2.1.3.3 Determination

Determine the content of chemical parameters in the test solution (5.2.2.1.3.2) in accordance with the following methods:

1) Cd, Ni and Pb: in accordance with ISO 8288, Method A;
2) Cr: in accordance with ISO 9174;
3) As, Se and Sb: in accordance with the method given in Annex C.

These methods are providing an interim result (y) expressed in milligrams per litre which needs to be converted to give the final concentration according to Formula (3) in 5.2.2.1.3.4.

5.2.2.1.3.4 Expression of results

From the interim results (y) determined (see 5.2.2.1.3.3), the content, C_1, of each chemical parameter in the laboratory sample expressed in milligrams per kilogram of hydrogen peroxide 100 %, is given by the following formula:

$$C_1 = y \times \frac{V}{m} \times \frac{100}{c_3} \tag{3}$$

where

y is the interim result (5.2.2.1.3.3);

V is the volume, expressed in millilitres, of the test solution (5.2.2.1.3.2) (= 100 ml);

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

c_3 is the concentration of hydrogen peroxide in mass fraction (5.2.1.5).

5.2.2.2 Determination of mercury content (Hg)

5.2.2.2.1 Principle

The element mercury is determined by flameless atomic absorption spectrometry in accordance with EN ISO 12846.

5.2.2.2.2 Reagents

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

5.2.2.2.2.1 Potassium permanganate solution, $c(KMnO_4)$ = 50 g/l.

5.2.2.2.2.2 Sulfuric acid, concentrated, density ρ = 1,84 g/ml.

5.2.2.2.2.3 Hydroxylammonium chloride solution, $c(NH_2OH.HCl)$ = 100 g/l.

5.2.2.2.2.4 Potassium dichromate solution, $c(K_2Cr_2O_7)$ = 4 g/l in volume fraction 50 % of nitric acid solution.

5.2.2.2.3 Procedure

5.2.2.2.3.1 Test portion

Weigh, to the nearest 0,01 g, 10 g (m_1) from the laboratory sample, into a glass beaker.

5.2.2.2.3.2 Test solution

Quantitatively transfer the test portion to a washing flask (e.g. Durand bottle), capacity 250 ml, the gas inlet of which is made of a porous frit. Dilute the contents of the washing flask with water to obtain a total volume of 100 ml. Transfer to a volumetric flask (solution A).

Pipette, accurately 10 ml of the solution A. Transfer to a 250 ml conical flask and add 60 ml of water, 20 ml of a potassium permanganate solution (5.2.2.2.2.1) and five 1 ml portions of sulfuric acid (5.2.2.2.2.2). Heat and keep boiling for 10 min. Allow to cool. Just dissolve the precipitate (MnO_2) with hydroxylammonium chloride (5.2.2.2.2.3), add 5 ml of the potassium dichromate solution (5.2.2.2.2.4) and transfer to a 100 ml (V_T) volumetric flask. Dilute to the mark with water and mix.

5.2.2.2.3.3 Determination

Proceed as described in EN ISO 12846.

5.2.2.2.4 Expression of result

The interim result for mercury content (y) expressed in milligrams per litre is given by the following general formula:

$$y = y_A \times \frac{V_T}{10} \tag{4}$$

where

y_A is the result obtained, for the concentration of mercury in solution A, expressed in milligrams per litre;

V_T is the volume in millilitres of the test solution.

The content of mercury, C_2, in milligrams per kilogram of hydrogen peroxide 100 % is given by the following formula:

$$C_2 = y \times \frac{10}{m_1} \times \frac{100}{c_3} \qquad (5)$$

where

m_1 is the mass, expressed in grams, of the test portion;

c_3 is the concentration of hydrogen peroxide in mass fraction (5.2.1.5).

6 Labelling - Transportation - Storage

6.1 Means of delivery

Hydrogen peroxide shall be delivered in:

a) polyethylene drums up to 225 l for hydrogen peroxide concentration not greater than a mass fraction of 50 %;

b) intermediate bulk containers if hydrogen peroxide concentration is not greater than a mass fraction of 50 %;

c) aluminium or stainless steel tanks of up to 25 t capacity for all concentrations.

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 (see Table 8) shall apply to hydrogen peroxide at the date of publication of this standard.

[4)] See [2].

Table 8 — Labelling requirements

Solutions of hydrogen peroxide concentration	Hazard statements	Hazard pictograms	Signal word
$C \geq 70$ %	H271: May cause fire or explosion; strong oxidizer H314: Cause severe skin burns and eye damage H335: May cause respiratory irritation	 **Figure 1 — GHS03**  **Figure 2 — GHS05**  **Figure 3 — GHS07**	Danger

Solutions of hydrogen peroxide concentration	Hazard statements	Hazard pictograms	Signal word
50 % ≤ C < 70 %	H272: May intensify fire; oxidizer H314: Cause severe skin burns and eye damage H335: May cause respiratory irritation	 **Figure 4 — GHS03**  **Figure 5 — GHS05**  **Figure 6 — GHS07**	Danger

Solutions of hydrogen peroxide concentration	Hazard statements	Hazard pictograms	Signal word
35 % ≤ C < 50 %	H315: Causes skin irritation H318: Causes serious eye damage H335: May cause respiratory irritation	 Figure 7 — GHS05  Figure 8 — GHS07	Danger
8 % ≤ C < 35 %	H318: Causes serious eye damage	 Figure 9 — GHS05	Danger
5 % ≤ C < 8 %	H319: Causes serious eye irritation	 Figure 10 — GHS07	Warning
C ≤ 5 %	-	-	-
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 legislation [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

The following transportation regulations and labelling (see Table 9) shall apply to hydrogen peroxide at the date of publication of this standard.

Table 9 — Transportation regulations and labelling

	H_2O_2 concentration in % (m/m)		
	> 8 to 20	**> 20 to 60**	**> 60**
UN-Number [a]	2984	2014	2015
RID [b] /ADR [c]	Class 5.1 Classification code O1 Packing group III	Class 5.1 Classification code OC1 Packing group II	Class 5.1 Classification code OC1 Packing group I
IMDG [d]	Class 5.1	Class 5.1	Class 5.1
IATA [e]	Class 5.1	> 20–40: Class 5.1 > 40: prohibited	Prohibited

[a] United Nations Number.

[b] Regulations concerning International carriage of Dangerous goods by rail.

[c] European Agreement concerning the international carriage of Dangerous goods by Road.

[d] International Maritime transport of Dangerous Goods.

[e] International Air Transport Association.

6.4 Marking

The marking shall include the following:

- name “hydrogen peroxide”, trade name and type;
- net mass;
- name and address of the supplier and/or manufacturer;
- statement “this product conforms to EN 902”.

6.5 Storage[5)]

6.5.1 Containers

The product shall be stored in containers made of suitable material, vented to avoid any pressure build-up, in a cool place away from any heat sources and protected from sunlight.

6.5.2 Long term stability

The product is stable for long periods without any significant loss of active oxygen (less than 2 % relative per year).

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5) See [4].

6.5.3 Storage incompatibilities

The product shall not be allowed to come into contact with combustible organic materials (e.g. paper, cloth, saw dust, solvents, lubricants), salts of e.g. copper, chromium, iron, vanadium, tungsten, manganese, molybdenum and platinum, metals like tin, brass and mild steel, and reducing compounds such as sulphites and hydrosulphites, acids or alkalis.

Annex A
(informative)

General information on hydrogen peroxide

A.1 Origin

A.1.1 Raw materials

Hydrogen peroxide is manufactured from hydrogen and atmospheric oxygen.

A.1.2 Manufacturing process

It is produced from hydrogen and oxygen (air) by the use of anthraquinonoïd compounds as an autooxidation catalyst.

A.2 Use

A.2.1 Function

Its functions in water treatment are as an oxidant to remove oxidizable impurities and as a disinfectant.

NOTE Hydrogen peroxide can either be used alone for these purposes or in accordance with existing national regulations in conjunction with other systems (e.g. ozone, ultraviolet light, or any activating systems) as hydroperoxide radical generators.

A.2.2 Form in which it is used

It is used in aqueous solution.

A.2.3 Treatment dose

The treatment dose is usually about 20 mg/l but can be up to 35 mg/l of H_2O_2, (expressed as mass fraction of 100 % hydrogen peroxide) depending on the quality of the raw water.

A.2.4 Means of application

It is applied using a metering pump.

A.2.5 Secondary effects

The secondary effects include the following:

- increase in dissolved oxygen;
- decrease in colour or turbidity of water;
- corrosion inhibition by passivating effect.

A.2.6 Removal of excess product

The most practical method is the use of:

- addition of reducing agent;

- activated carbon;
- ultraviolet light.

A.3 Routine analyses

A.3.1 Determination of chemical parameters

A.3.1.1 Preparation of the test solution

A.3.1.1.1 Reagents

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

A.3.1.1.1.1 Sodium hydroxide solution, 100 g/l.

A.3.1.1.1.2 Methyl orange indicator solution, 0,2 g/l.

A.3.1.1.1.3 Hydrochloric acid ρ = 1,18 g/ml.

A.3.1.1.1.4 Potassium iodide.

A.3.1.1.2 Procedure

A.3.1.1.2.1 Test portion

Weigh, to the nearest 0,01 g, about 25 g (*m*) of the laboratory sample into a 100 ml conical flask.

A.3.1.1.2.2 Test solution

Add 2 ml of sodium hydroxide solution (A.3.1.1.1.1), cover the flask with a watch glass and decompose the sample by gentle warming. When decomposition has ceased, boil the solution for 15 min taking care to maintain a volume of about 25 ml by the addition of water. Cool and add five drops of methyl orange indicator solution (A.3.1.1.1.2). Add by means of a dropping pipette, sufficient hydrochloric acid (A.3.1.1.1.3) to neutralize the solution. Add 2 g of potassium iodide (A.3.1.1.1.4) and swirl to dissolve.

Prepare a blank test solution as described above omitting the sample.

A.3.1.2 Determination

Determine the content of chemical parameter in the test solution (A.3.1.1.2.2) in accordance with the following methods:

- As: in accordance with ISO 17378-2 [5];
- Se: in accordance with ISO/TS 17379-2 (AAS) [6].

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

Seek medical advice if signs or symptoms develop.

In case of contact with skin remove any contaminated clothing. Wash skin with plenty of water, use emergency shower if large area of body contaminated. Wash any contaminated clothing well.

In case of contact with eyes rinse out with plenty of water for at least 15 min and seek medical advice quickly.

In case of ingestion do not induce vomiting. Rinse out mouth quickly and then give plenty of milk or water if the victim is conscious.

In case of inhalation: remove from contaminated area to fresh air. Seek medical advice.

B.2.2 Spillage

Dilute with plenty of water.

B.2.3 Fire

Pressure bursts can occur due to gas evolution. Oxygen released on decomposition assist fire. Use a fine water spray to extinguish. Self-contained breathing apparatus can be necessary. Keep product containers cool with plenty of water, working at safe distance.

Annex C
(normative)

Determination of arsenic, antimony and selenium (atomic absorption spectrometry hydride technique)

C.1 Safety precautions

SAFETY PRECAUTIONS — Arsenic, antimony and selenium and their hydrides are toxic. Handle with care.

C.2 General principle

Arsenious acid, antimonic acid and selenious acid, the As(III), Sb(III) and Se(IV) oxidation states of arsenic, antimony and selenium, respectively, are instantaneously converted by sodium borohydride reagent in acid solution to their volatile hydrides. The hydrides are purged continuously by argon or nitrogen into an appropriate atomizer of an atomic absorption spectrometer and converted to the gas-phase atoms. The sodium borohydride reducing agent, by rapid generation of the elemental hydrides in an appropriate reaction cell, minimizes dilution of the hydrides by the carrier gas and provides rapid, sensitive determinations of arsenic, antimony and selenium.

The sample is digested to solubilize particulate As, Sb and Se. The digested solutions are treated separately for determination of As, Sb and Se to convert them to As(III), Sb(III) and Se(IV) oxidation states respectively.

C.3 Interferences

Interferences are minimized because the As, Sb and Se hydrides are removed from the solution containing most potential interfering substances. Slight response variations occur when acid matrices are varied. Control these variations by treating standards and samples in the same manner. Low concentrations of noble metals (approximately 100 µg/l of Ag, Au, Pt, Pd, etc.) concentrations of Cu, Ni and Pb at or greater than 1 mg/l, and concentrations between 0,1 mg/l and 1 mg/l of hydride-forming elements (Bi, Sn and Te) can suppress the response of As, Sb and Se hydrides due to the formation of mixed metal – As-Sb or -Se compounds. The presence of As, Sb and Se in each other's matrices can cause similar suppression. Reduced nitrogen oxides resulting from HNO3 digestion and nitrite also can suppress instrumental response for all elements. Large concentrations of iodide interfere with the Se determination by reducing Se to its elemental form. Do not use any glassware for determining Se that has been used with iodide reduction of As(V).

C.4 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.

C.4.1 **Sodium tetrahydroborate** (sodium borohydride).

Dissolve 8 g $NaBH_4$ in 200 ml of NaOH, $c(NaOH) = 0,1$ mol/l. Prepare fresh daily.

C.4.2 **Sodium iodide,** prereductant solution.

Dissolve 50 g Nal in 500 ml water. Prepare fresh daily.

C.4.3 Sulfuric acid, solution $c(H_2SO_4) = 9$ mol/l.

C.4.4 Sulfuric acid, solution $c(H_2SO_4) = 1{,}25$ mol/l.

Cautiously add 35 ml sulfuric acid, density (ρ) = 1,84 g/ml to about 400 ml water, let cool, and adjust volume to 500 ml.

C.4.5 Nitric acid, density (ρ) = 1,42 g/ml.

C.4.6 Perchloric acid, density (ρ) = 1,66 g/ml.

C.4.7 Hydrochloric acid, density (ρ) = 1,16 g/ml.

C.4.8 Argon (or nitrogen), commercial grade.

C.4.9 Hydrogen, commercial grade.

C.4.10 Arsenic(III) solutions:

- stock As(III) solution: Dissolve 1,320 g arsenic trioxide, As_2O_3, in water containing 4 g NaOH. Transfer quantitatively to 1000 ml one-mark volumetric flask and dilute to the mark with water and mix; 1,00 ml = 1,00 mg As(III);
- intermediate As(III) solution: Dilute into 1000 ml one-mark volumetric flask 10 ml stock As(III) solution to the mark with water containing 5 ml hydrochloric acid (C.4.7) and mix; 1,00 ml = 10,0 µg As(III);
- standard As(III) solution: Dilute into 1000 ml one-mark volumetric flask 10 ml intermediate As(III) solution to the mark with water containing the same concentration of acid used for sample preservation (2 ml to 5 ml nitric acid (C.4.5)) and mix; 1,00 ml = 0,100 µg As(III). Prepare diluted solutions daily.

C.4.11 Arsenic(V) solutions:

- stock As(V) solution: Dissolve 1,534 g arsenic pentoxide, As_2O_5, in water containing 4 g NaOH. Transfer quantitatively to 1 000 ml one-mark volumetric flask and dilute to the mark with water and mix; 1,00 ml = 1,00 mg As(V);
- intermediate As(V) solution: Pepare as for As(III) above; 1,00 ml = 10,0 µg As(V);
- standard As(V) solution: Prepare as for As(III) above; 1,00 ml = 0,100 µg As(V).

C.4.12 Selenium(IV) solutions:

- stock Se(IV) solution: Dissolve 2,190 g sodium selenite, Na_2SeO_3 in water containing 10 ml hydrochloric acid (C.4.7) and transfer quantitatively to 1 000 ml one-mark volumetric flask and dilute to the mark with water and mix; 1,00 ml = 1,00 mg Se(IV);
- intermediate Se(IV) solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml stock Se(IV) solution to the mark with water containing 10 ml hydrochloric acid (C.4.7) and mix; 1,00 ml = 10,0 µg Se(IV);
- standard Se(IV) solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml intermediate Se(IV) solution to the mark with water containing the same concentration of acid used for sample

preservation (2 ml to 5 ml nitric acid (C.4.5)) and mix. Prepare solution daily when checking the equivalent of instrument response for Se(IV) and Se(VI); 1,00 ml = 0,100 µg Se(IV).

C.4.13 Selenium(VI) solutions:

- stock Se(VI) solution: Dissolve 2,393 g sodium selenate Na_2SeO_4 in water containing 10 ml nitric acid (C.4.5). Transfer quantitatively to 1 000 ml one-mark volumetric flask and dilute to the mark with water and mix; 1,00 ml = 1,00 mg Se(VI);
- intermediate Se(VI) solution: Prepare as for Se(IV) above; 1,00 ml = 10,0 µg Se(VI);
- standard Se(VI) solution: Prepare as for Se(IV) above; 1,00 ml = 0,100 µg Se(VI).

C.4.14 Antimony solutions:

- stock Sb solution: Dry 2 g of potassium antimonyl tartrate hemihydrate (antimony potassium tartrate) ($C_4H_4SbK.0,5H_2O$) at 100 °C for 1h. Dissolve 1,669 g in water transfer quantitatively to 1 000 ml one-mark volumetric flask and dilute to the mark with water and mix; 1,00 ml = 1,00 mg Sb;
- intermediate Sb solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml stock Sb solution to the mark with water containing 10 ml hydrochloric acid (C.4.7) and mix; 1,00 ml = 10,0 µg Sb;
- standard Sb solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml intermediate Sb solution to the mark with water containing the same concentration of acid used for sample preservation (2 ml to 5 ml nitric acid (C.4.5)) and mix; 1,00 ml = 0,100 µg Sb. Prepare diluted solutions daily.

C.5 Apparatus

Ordinary laboratory apparatus (such as spectrometers computer with updated software) and glassware, together with the following:

C.5.1 Atomic absorption spectrometer.

Equipped with gas flow meters for argon (or nitrogen) and hydrogen, As, Sb and Se electrodeless discharge lamps with background correction at measurement wavelengths and appropriate strip-chart recorder.

NOTE Certain atomic absorption atomizers and hydride reaction cells are available commercially for use with the sodium borohydride reagent.

C.5.2 Atomizer.

Use one of the following:

- boling-type burner[6] head for argon (or nitrogen)-air entrained-hydrogen flame;
- cylindrical quartz cell, 10 cm to 20 cm long, electrically heated by external Ni-Cr wire from 800 °C to 900 °C;
- cylindrical quartz cell with internal fuel rich hydrogen-oxygen (air) flame.

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[6] Boling is the name of the inventor of this type of burner for rapid combustion of the hydrides.

The transparency of quartz cells deteriorates over several months of use. The transparency can be restored by treatment with 40 % hydrofluoric acid (HF).

SAFETY PRECAUTIONS — Be careful in handling HF which is toxic and corrosive and avoid prolonged contact of quartz with HF.

C.5.3 Reaction cell for producing As, Sb or Se hydrides.

An example of reaction cell is given in Figure C.1.

NOTE A commercially available system is acceptable if it utilizes liquid sodium borohydride reagents; accepts samples digested in accordance with C.6.3; accepts between 4 mol/l to 6 mol/l (HCl) and is efficiently and precisely stirred by the purging gas and/or a magnetic stirrer.

Irrespective of the hydride reaction cell-atomizer system selected, it shall meet the following quality-control considerations:

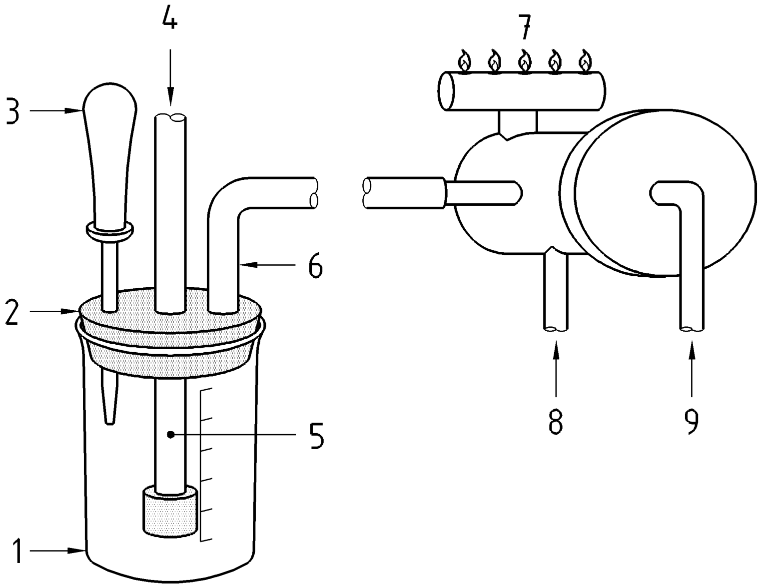
a) it shall provide a precise and reproducible calibration curve between 0 µg/l and 20 µg/l As, Sb or Se and a detection limit between 0,1 µg/l and 0,5 µg/l As, Sb or Se;

b) when carried through the entire procedure, oxidation state couples (As(III) - As(V) or Se(IV) - Se(VI)) shall cause equal instrument response; and

c) sample digestion shall yield 90 % or greater recovery of added As(III), As(V), Se(VI), Se(IV) or Sb.

C.5.4 Dropper and syringe:

Capable of delivering 0,5 ml to 3,0 ml sodium borohydride reagent. Exact and reproducible addition is required so that production of hydrogen gas does not vary significantly between determinations.



Key

1	beaker 250 ml
2	rubber stopper
3	dropper
4	auxiliary nitrogen
5	gas dispersion tube
6	outlet tube
7	burner
8	hydrogen
9	nitrogen

Figure C.1 — Reaction cell for producing As, Sb or Se hydrides

C.6 Procedure

C.6.1 Preparation of the apparatus

Connect inlet of reaction cell with auxiliary purging gas controlled by flow meter. If a drying cell between the reaction cell and atomizer is necessary, use only anhydrous $CaCl_2$ but not $CaSO_4$ because it can retain SeH_2. Before using the hydride generation/analysis system, optimize operating parameters. Aspirate aqueous solutions of As, Sb and Se directly into the flame to facilitate atomizer alignment. Align quartz atomizers for maximum absorbance. Establish purging gas flow, concentration and rate of addition of sodium borohydride reagent, solution volume, and stirring rate for optimum instrument response for the chemical species to be analyzed. If a quartz atomizer is used, optimize cell temperature. If sodium borohydride reagent is added too quickly, rapid evolution of hydrogen will unbalance the system. If the volume of solution being purged is too large, the absorption signal will be decreased. Recommended wavelengths are 193,7 nm 196,0 nm and 217,6 nm for As, Se and Sb, respectively.

C.6.2 Preparation of calibration solutions

Transfer 0,00 ml; 1,00 ml; 2,00 ml; 5,00 ml; 10,00 ml; 15,00 ml and 20,00 ml standard solutions of As(III), Se(IV) or Sb to 100 ml volumetric flasks and make up to volume with water containing the same acid concentration used for sample preservation (commonly 2 ml to 5 ml nitric acid (C.4.5)).This yields calibrations solutions of 0 µg/l, 1 µg/l, 2 µg/l, 5 µg/l, 10 µg/l, 15 µg/l and 20 µg/l As, Se or Sb. Prepare fresh daily.

C.6.3 Preparation of test solutions and standard solutions

Add 50 ml sample or As(III), Se(VI) or Sb standard solution to 250 ml beaker. Alternatively, prepare standard solutions by adding aliquots of solutions containing 5 µg As, Se or Sb directly to the beaker and dilute to 50 ml in this beaker, thus achieving a concentration of 100 µg/l of the respective solutions. Add 7 ml sulfuric acid $c(H_2SO_4) = 9$ mol/l (C.4.3) and 5 ml nitric acid (C.4.5). Add a small boiling chip or glass beads if necessary. Evaporate to SO_3 fumes. Maintain oxidizing conditions at all times by adding small amounts of nitric acid, to prevent solution from darkening.

Maintain an excess of nitric acid until all organic matter is destroyed. Complete digestion usually is indicated by a light-coloured solution. Cool slightly, add 25 ml water and 1 ml of perchloric acid (C.4.6) and again evaporate to SO_3 fumes to expel oxides of nitrogen.

Monitor effectiveness of digestion procedure used by adding 5 ml of a standard arsenic solution, 5 ml of a standard selenium solution or 5 ml of a standard antimony solution to 50 ml sample and measuring recovery. Average recoveries shall be greater than 90 %. Alternatively, use 100 ml micro-Kjeldahl flasks for the digestion of total recoverable arsenic, selenium or antimony, thereby improving digestion effectiveness. After final evaporation of SO_3 fumes, dilute to 50 ml for arsenic measurements or 30 ml for selenium and antimony measurements.

C.6.4 Determination of arsenic with sodium borohydride

To 50 ml digested standard solution or test solution in a 250 ml beaker (see Figure C.1) add 5 ml hydrochloric acid (C.4.7) and mix. Add 5 ml sodium iodide prereductant solution (C.4.2), mix and wait at least 30 min.

NOTE The sodium iodide has not been found necessary for certain hydride reaction cell designs if a 20 % to 30 % loss in instrument sensitivity is not important and variables of solution acid conditions, temperatures, and volumes for production of As(V) and arsine can be controlled strictly. This can require an automated delivery system.

Attach one beaker at a first time to the rubber stopper containing the gas dispersion tube for the purging gas, the sodium borohydride reagent inlet, and the outlet to the atomizer. Turn on strip-chart recorder and wait until the base line is established by the purging gas and all air is expelled from the reaction cell. Add 0,5 ml sodium borohydride reagent (C.4.1). After the instrument absorbance has reached a maximum and returned to the base line, remove beaker, rinse dispersion tube with water, and proceed to the next test solution or standard solution. Periodically compare standard As(III) and As(V) curves for response consistency. Check for presence of chemical interferences that suppress instrument response for arsine by treating a digested sample with 10 µg/l As(III) or As(V) as appropriate. Average recoveries shall be not less than 90 %.

C.6.5 Determination of selenium with sodium borohydride

To 30 ml digested standard solution or test solution, or to 30 ml undigested standard, or sample in a 250 ml beaker, add 15 ml hydrochloric acid (C.4.7) and mix. Heat for a predetermined period at temperature between 90 °C to 100 °C. Alternatively autoclave at 121 °C in capped containers for 60 min, or heat for a predetermined time in open test tubes using a 90 °C to 100 °C hot water bath or an aluminium block digester. Check effectiveness of the selected heating by demonstrating equal instrument responses for calibration curves prepared either from standard Se(IV) or from Se(VI) solutions. Effective heat exposure for converting Se(VI) to Se(IV), with no loss of Se(IV), ranges between 5 min to 60 min when open beakers or test tubes are used. Do not digest standard Se(IV) and Se(Vl) solutions used for this check of equivalency. After prereduction of Se(VI) and Se(IV) attach beakers, one at a time, to the purge apparatus. For each, turn on the strip-chart recorder and wait until the base line is established. Add 0,50 ml sodium borohydride reagent (C.4.1). After the instrument absorbance has reached a maximum and returned to the base line, remove beaker, rinse dispersion tube with water and proceed to the next test solution or standard solution. Check for presence of chemical interferences that suppress selenium hydride instrument response by treating a digested sample with 10 µg/l Se(IV). Average recoveries shall be not less than 90 %.

C.6.6 Determination of antimony with sodium borohydride

To 30 ml digested standard solution or test solution, or to 30 ml undigested standard solution, or test solution in a 250 ml beaker, add 15 ml hydrochloric acid (C.4.7) and mix. Heat for a predetermined period (between 5 min and 60 min) at a temperature between 90 °C to 100 °C. After prereduction of Sb attach beakers, one at a time, to the purge apparatus. For each, turn on the strip-chart recorder and wait until the base line is established. Add 0,50 ml sodium borohydride reagent (C.4.1). After the instrument absorbance has reached a maximum and returned to the base line, remove beaker, rinse dispersion tube with water and proceed to the next test solution or standard solution. Check for presence of chemical interferences that suppress antimony hydride instrument response by treating a digested sample with 10 µg/l Sb. Average recoveries shall be not less than 90 %.

C.7 Calculation

Determine the calibration curve by plotting peak heights of standard solutions versus concentration. Measure peak heights of samples and read concentrations from the calibration curve. If sample was diluted (or concentrated) before sample digestion, apply an appropriate factor.

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