### BS EN 15040:2014



# **BSI Standards Publication**

Chemicals used for treatment of water intended for human consumption — Antiscalants for membranes — Phosphonic acids and salts



BS EN 15040:2014 BRITISH STANDARD

#### National foreword

This British Standard is the UK implementation of EN 15040:2014. It supersedes BS EN 15040:2006 which is withdrawn.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.

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

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

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# EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

**EN 15040** 

March 2014

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Supersedes EN 15040:2006

#### **English Version**

# Chemicals used for treatment of water intended for human consumption - Antiscalants for membranes - Phosphonic acids and salts

Produits chimiques pour le traitement de l'eau destinée à la consommation humaine - Produits antitartre pour membranes - Acides phosphoniques et sels

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Antiscalants für Membranen -Phosphonsäuren und deren Salze

This European Standard was approved by CEN on 5 January 2014.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION COMITÉ EUROPÉEN DE NORMALISATION EUROPÄISCHES KOMITEE FÜR NORMUNG

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#### **Foreword**

This document (EN 15040:2014) 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 September 2014 and conflicting national standards shall be withdrawn at the latest by September 2014.

This document supersedes EN 15040:2006.

Significant technical differences between this edition and EN 15040:2006 are as follows:

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

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.

WARNING – The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

#### Introduction

In respect of potential adverse effects on the quality of water intended for human consumption, caused by the products covered by this European Standard:

- a) this European Standard provides no information as to whether the products 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 these products remain in force.

NOTE Conformity with this European Standard does not confer or imply acceptance or approval of the products in any of the Member States of the EU or EFTA. The use of the products covered by this European Standard is subject to regulation or control by National Authorities.

#### 1 Scope

This European Standard is applicable to phosphonic acids and salts used as antiscalants for membranes in the treatment of water intended for human consumption. It describes the characteristics and specifies the requirements and the corresponding analytical methods for phosphonic acids and salts. It gives information on their use as antiscalants for membranes 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 1233, Water quality — Determination of chromium — Atomic absorption spectrometric methods

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

EN ISO 5961, Water quality - Determination of cadmium by atomic absorption spectrometry (ISO 5961)

EN ISO 11885, Water quality - Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885)

EN ISO 11969, Water quality - Determination of arsenic - Atomic absorption spectrometric method (hydride technique) (ISO 11969)

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

ISO 2997, Phosphoric acid for industrial use — Determination of sulphate content — Method by reduction and titrimetry

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

ISO 3360, Phosphoric acid and sodium phosphates for industrial use (including foodstuffs) — Determination of fluorine content — Alizarin complexone and lanthanum nitrate photometric method

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

ISO 6703-1, Water quality — Determination of cyanide — Part 1: Determination of total cyanide

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

ISO 8288:1986, Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods

ISO 9965, Water quality — Determination of selenium — Atomic absorption spectrometric method (hydride technique)

#### 3 Description

#### 3.1 Identification

#### 3.1.1 Chemical name

- a) Morpholinomethane diphosphonic acid,
- b) Aminotrismethylene phosphonic acid,
- c) Hydroxyethane diphosphonic acid,
- d) Diethylene triamine pentamethylene phosphonic acid,
- e) Ethylene diamine tetramethylene phosphonic acid,
- f) Ethanol aminobismethylene phosphonic acid,
- g) Phosphonobutane tricarboxylic acid,
- h) Hexamethylenediamine tetramethylene phosphonic acid.

These acids can also be used as sodium, potassium, and ammonium salts.

#### 3.1.2 Synonym or common names

- a) MOMP,
- b) ATMP,
- c) HEDP,
- d) DETAPMP,
- e) EDATMP,
- f) EABMP,
- g) PBTC,
- h) HDTMP

#### 3.1.3 Relative molecular mass

- a) 260,15
- b) 299,04
- c) 206,02
- d) 572,95
- e) 436,06
- f) 249,02
- g) 270,82

h) 492,23

#### 3.1.4 Empirical formula

- a)  $C_5H_{13}O_7P_2N$ ,
- b)  $C_3H_{12}O_9P_3N$ ,
- c)  $C_2H_8O_7P_2$ ,
- d)  $C_9H_{28}O_{15}N_3P_5$ ,
- e)  $C_6H_{20}O_{12}N_2P_4$ ,
- f)  $C_4H_{13}O_7P_2N$ ,
- g)  $C_7H_{11}O_9P$ ,
- h)  $C_{10}H_{28}N_2O_{12}P_4$

#### 3.1.5 Chemical formula

- a)  $C_5H_{13}O_7P_2N$ ,
- b)  $C_3H_{12}O_9P_3N$ ,
- c)  $C_2H_8O_7P_2$ ,
- $d) \quad C_9 H_{28} O_{15} N_3 P_5, \\$
- $e) \quad C_6 H_{20} O_{12} N_2 P_4, \\$
- f)  $C_4H_{13}O_7P_2N$ ,
- g)  $C_7H_{11}O_9P$ ,
- h)  $C_{10}H_{28}N_2O_{12}P_{4.}$

#### 3.1.6 CAS Registry Number 1)

- a) 32545-75-8,
- b) 6419-19-8,
- c) 2809-21-4,
- d) 15827-60-8,
- e) 1429-50-1,
- f) 5995-42-6,
- g) 37971-36-1,

<sup>1)</sup> Chemical Abstracts Service Registry Number.

#### BS EN 15040:2014 EN 15040:2014 (E)

h) 23605-74-5.

#### 3.1.7 EINECS reference 2)

- a) 251-094-7,
- b) 229-146-5,
- c) 220-552-8,
- d) 239-931-4,
- e) 215-851-5,
- f) 227-833-4,
- g) 253-733-5,
- h) 245-781-0.

#### 3.2 Commercial forms

The phosphonic acids and salts are available in a number of different forms (see 3.3.1).

Different commercial forms, solids or dissolved in water are possible. All concentrations mentioned refer to the active matter (active acid or active salt) and shall be calculated accordingly.

#### 3.3 Physical properties

#### 3.3.1 Appearance

Solid: The product is a yellowish or white powder or granulate.

Liquid: The product is a colourless, light yellow, pale yellow, amber or brown solution.

#### 3.3.2 Density

Solid: The bulk density of the product varies from 500 g/dm<sup>3</sup> to 1200 g/dm<sup>3</sup>.

Liquid: The density of solution is typically 1,1 g/ml to 1,46 g/ml for a product concentration from mass fraction 22 % to 60 % of active matter at 20 °C.

#### 3.3.3 Solubility in water

Solid: the solubility is approximately 300 g/l solution (Na $_4$ HEDP) at 25 °C, and approximately 420 g/l solution (Na $_7$ DETAPMP) at 25 °C.

Liquid: it is miscible in all proportions with pure water.

#### 3.3.4 Vapour pressure

Not applicable.

<sup>2)</sup> European Inventory of Existing Commercial Chemical Substances.

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#### 3.3.5 Boiling point at 100 kPa 3)

Not applicable.

#### 3.3.6 Melting point

Not applicable.

#### 3.3.7 Specific heat

Not known.

#### 3.3.8 Viscosity (dynamic)

For the solid product it is not applicable.

For the liquid the viscosity is equal from 4 mPa.s to 170 mPa.s for a product concentration of 50 g/l.

#### 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 phosphonic acids and solutions of phosphonic acid salts have acidic to alkaline reactions. The pH value of an aqueous solution of a mass fraction of 1 % is approximately between 2 to 12.

#### 4 Purity criteria

#### 4.1 General

This European Standard specifies the minimum purity requirements for phosphonic acids and salts used as antiscalants for membranes for the treatment of water intended for human consumption. Limits are given for impurities commonly present in the products. 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 these products 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 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.

 $<sup>^{(3)}</sup>$  100 kPa = 1 bar.

#### 4.2 Composition of commercial product

The products shall conform to the following requirements on a dry mass basis:

phosphorus content expressed as 35,07-95,27 % PO<sub>4</sub> dry mass basis.

#### 4.3 Impurities and main by-products

The phosphonic acids and salts shall conform to the requirements specified in Table 1.

Table 1 — Limits of impurities

Impurity		Limit	
		mg/kg of dry product	
Sulfate (SO <sub>4</sub> <sup>2-</sup> )	max.	500	
Fluoride (F <sup>-</sup> )	max.	10	

#### 4.4 Chemical parameters

Content of various metals depends on the origin of the raw materials, most of these elements are present only as traces.

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

Table 2 — Chemical parameters

Parameter	Limit		
		mg/kg of dry product	
Antimony (Sb)	max.	3	
Arsenic (As)	max.	3	
Cadmium (Cd)	max.	3	
Chromium (Cr)	max.	10	
Cyanide (CN⁻)	max.	5	
Lead (Pb)	max.	10	
Mercury (Hg)	max.	1	
Nickel (Ni)	max.	10	
Selenium (Se)	max.	3	
NOTE Pesticides and	d polycyclic a	aromatic hydrocarbons are not	

NOTE Pesticides and polycyclic aromatic hydrocarbons are no relevant in these products [1]

#### 5 Test methods

#### 5.1 Sampling

#### 5.1.1 General

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

#### 5.1.2 Solid

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

#### 5.1.3 Liquid

#### 5.1.3.1 Sampling from drums and bottles

#### 5.1.3.1.1 General

- **5.1.3.1.1.1** Mix the contents of each container to be sampled by shaking the container, by rolling it or by rocking it from side to side, taking care not to damage the container or spill any of the liquid.
- **5.1.3.1.1.2** If the design of the container is such (for example, a narrow-necked bottle) that it is impracticable to use a sampling implement, take a sample by pouring after the contents have been thoroughly mixed. Otherwise, proceed as described in 5.1.3.1.1.3.
- **5.1.3.1.1.3** Examine the surface of the liquid. If there are signs of surface contamination, take samples from the surface as described in 5.1.3.1.2; otherwise, take samples as described in 5.1.3.1.3.

#### 5.1.3.1.2 Surface sampling

Take a sample using a suitable ladle. Lower the ladle into the liquid until the rim is just below the surface, so that the surface layer runs into it. Withdraw the ladle just before it fills completely and allow any liquid adhering to the ladle to drain off. If necessary, repeat this operation so that, when the other selected containers have been sampled in a similar manner, the total volume of sample required for subsequent analysis is obtained.

#### 5.1.3.1.3 Bottom sampling

Take a sample using an open sampling tube, or a bottom-valve sampling tube, suited to the size of container and the viscosity of the liquid.

When using an open sampling tube, close it at the top and then lower the bottom end to the bottom of the container. Open the tube and move it rapidly so that the bottom of the tube traverses the bottom of the container before the tube is filled. Close the tube, withdraw it from the container and allow any liquid adhering to the outside of the tube to drain off.

When using a bottom-valve sampling tube, close the valve before lowering the tube into the container and then proceed in a similar manner to that when using an open sampling tube.

#### 5.1.3.2 Sampling from tanks and tankers

From each access point, take samples as follows:

- a) from the surface of the liquid, using a ladle as described in 5.1.3.1.2;
- b) from the bottom of the tank or tanker, using a sampling tube as described in 5.1.3.1.3 or using a specially designed bottom-sampling apparatus;
- c) from one or more positions, depending on the overall depth, between the bottom and the surface using a weighted sampling can.

#### 5.2 Analyses

#### 5.2.1 Phosphate content (main product)

#### 5.2.1.1 **General**

The determination of the phosphate content expressed in (PO<sub>4</sub>) shall be determined in accordance with the Zimmermann method.

Phosphonic acids and salts are determined by means of organic phosphate analysis. Phosphonic acids and salts have a strong C-P bond which is highly hydrolysis-stable. This bond shall be split by oxidation before the quantitative analysis of phosphate can be carried out.

#### 5.2.1.2 Principle

Conversion of organic phosphate to orthophosphate by oxidation and reaction of orthophosphate with salts of molybdate in acid solution and in the presence of methylaminophenol (a reducing agent) to a blue coloured complex. By the addition of citric acid the formation of silica molybdate is prevented (Zimmermann method).

#### 5.2.1.3 Reagents

#### 5.2.1.3.1 General

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

- **5.2.1.3.2 Hydrochloric acid**, concentrated, density  $\rho = 1.18$  g/ml.
- **5.2.1.3.3** Sulfuric acid, solution,  $c(H_2SO_4) = 6 \text{ mol/l.}$

Add 500 ml of water to a 1 l beaker. Cautiously add, with continuous stirring and cooling, 250 ml of sulfuric acid,  $\rho$  =1,84 g/ml. Mix well and allow the solution to cool to room temperature.

#### 5.2.1.3.4 Ammonium peroxodisulfate

#### **5.2.1.3.5 Orthophosphate**, standard stock solution

Dissolve 250,8 mg of Na<sub>2</sub>HPO<sub>4</sub> with water in a 1 000 ml volumetric flask, dilute to the mark and mix.

#### 5.2.1.3.6 Orthophosphate, standard solution

Dilute the standard stock solution (5.2.1.3.5) with water to 1+10.

#### 5.2.1.3.7 Reducing agent

Add 20 g of methylaminophenol, 100 g of sodium pyrosulfite and 20 g of citric acid into a 1 000 ml volumetric flask. Dissolve with water, dilute to the mark and mix. Store the solution in a cool and dark place.

#### 5.2.1.3.8 Acid molybdate, solution

Add 50 g of ammonium molybdate into a 1 000 ml volumetric flask. Dissolve with 500 ml of water and cautiously add 50 ml of sulfuric acid,  $\rho$  = 1,84 g/ml. Make up to the mark with water and mix well and allow the solution to cool to room temperature.

#### 5.2.1.4 Apparatus

Ordinary laboratory apparatus and:

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#### **5.2.1.4.1** Clean phosphate free glassware should be only used for this determination.

**5.2.1.4.2 Spectrometer, prism-, grating- or filter-type,** capable of accepting optical cells of 4 cm to 5 cm optical path length. The spectrometer shall be suitable for ensuring maximum transmission at approximately 720 nm.

#### 5.2.1.5 Procedure

#### 5.2.1.5.1 Test portion

Weigh, to the nearest 0,001 g, 2,5 g (m) from the laboratory sample into a 100 ml one-mark volumetric flask.

#### **5.2.1.5.2** Test solution

Add 20 ml of water and 2 ml of the hydrochloric acid (5.2.1.3.2) in the volumetric flask containing the test portion (5.2.1.5.1), dissolve and make up to the mark with water and mix.

#### 5.2.1.5.3 Preparation of the test solution

Transfer with a pipette 50 ml of the test solution (5.2.1.5.2) into a 100 ml volumetric flask and add 2 ml of the sulfuric acid (5.2.1.3.3). Then add 0,5 g of ammonium peroxodisulfate (powder) (5.2.1.3.4) and boil gently for 90 min.

#### 5.2.1.5.4 Blank test

Carry out a blank test in parallel with the determination, by the same procedure, using the same quantities of all the reagents as in the determination, but using the appropriate volume of water without the hydrolysis step instead of the test portion.

#### 5.2.1.5.5 Calibration

#### 5.2.1.5.5.1 Preparation of calibration solutions

Transfer, by means of a volumetric pipette, appropriate volumes, for example 1,0 ml; 5 ml; 10 ml; 15 ml; 20 ml; 25 ml; 30 ml; 35 ml; 40 ml; 45 ml and 50 ml, of the orthophosphate standard solution (5.2.1.3.6) to 100 ml volumetric flasks, dilute with water to about 70 ml. These solutions represent phosphate concentrations  $\rho_P = 0.2$  mg/l to 10 mg/l of  $P_2O_5$ .

#### 5.2.1.5.5.2 Colour development

Add to each volumetric flask 5 ml of reducing agent (5.2.1.3.7) and 5 ml of the solution acid molybdate (5.2.1.3.8). Make up to the mark with water and mix well.

#### 5.2.1.5.5.3 Spectrometric measurements

After 20 min, measure the absorbance of each solution using the spectrometer (5.2.1.4.2) at 720 nm. Use water in the reference cell.

#### 5.2.1.5.5.4 Plotting the calibration curve

Plot a curve of absorbance (as the ordinates-axis) against the  $P_2O_5$  content (as the abscissae-axis) in milligrams of  $P_2O_5$  per litre of the calibration solutions. The relationship between absorbance and concentration is linear. Determine the slope of the calibration curve.

#### 5.2.1.5.6 Determination

#### 5.2.1.5.6.1 Colour development

#### EN 15040:2014 (E)

Take the test solution (5.2.1.5.3) prepared into the 100 ml volumetric flask, then add 5 ml of reducing agent (5.2.1.3.7) and 5 ml of acid molybdate solution (5.2.1.3.8), dilute with water to the mark and mix.

#### 5.2.1.5.6.2 Spectrometric measurements

Carry out the measurement as in 5.2.1.5.5.3

#### 5.2.1.5.7 Expression of results

The content of phosphate (PO<sub>4</sub>),  $c_1$ , expressed as mass fraction of the product is given by the equation:

$$c_1 = \frac{(A - A_0)}{f \times m \times 1.493} \times 100 \tag{1}$$

where

A is the absorbance of the test solution;

 $A_0$  is the absorbance of the blank test;

f is the slope of the calibration curve (5.2.1.5.5.4), in I/mg;

m is the mass, in g, of the test portion (5.2.1.5.1).

#### 5.2.2 Impurities

#### 5.2.2.1 Sulfate

The sulfate  $(SO_4^{2-})$  content shall be determined in accordance with ISO 2997.

#### 5.2.2.2 Fluoride

The fluoride (F<sup>-</sup>) content shall be determined in accordance with ISO 3360.

#### 5.2.3 Chemical parameters

#### 5.2.3.1 **General**

When preparing the products for analysis, it is important to ensure that the chemical parameters are effectively dissolved. The concentration of the solution should be sufficient to permit adequate sensitivity in analysis of the chemical parameters and appropriate steps should be taken to compensate for any matrix interference caused by the concentration of the products.

# 5.2.3.2 Determination of antimony (Sb), arsenic (As), cadmium (Cd), chromium (Cr), cyanide (CN<sup>-</sup>), lead (Pb), mercury (Hg), nickel (Ni) and selenium (Se)

#### 5.2.3.2.1 Principle

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

#### 5.2.3.2.2 Reagents

#### 5.2.3.2.2.1 General

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

**5.2.3.2.2.2 Hydrochloric acid**, concentrated density  $\rho = 1.18$  g/ml.

#### 5.2.3.2.3 Procedure

#### 5.2.3.2.3.1 Test portion

Weigh, to the nearest 0,001 g, 2,5 g (m) from the laboratory sample into a 100 ml one - mark volumetric flask.

#### 5.2.3.2.3.2 Test solution

Add 20 ml of water and 2 ml of the hydrochloric acid (5.2.3.2.2.2), dissolve and make up to the mark with water and mix.

#### 5.2.3.2.3.3 Determination

Determine the content of chemical parameters in the test solution (5.2.3.2.3.2) in accordance with the following methods:

Ni and Pb: in accordance with ISO 8288:1986, method A;

Cd: in accordance with EN ISO 5961;

CN<sup>-</sup>: in accordance with ISO 6703-1;

Cr in accordance with EN 1233;

As: in accordance with EN ISO 11969:

Se: in accordance with ISO 9965;

Sb: in accordance with EN ISO 11885;

Hg: in accordance with EN ISO 12846.

These methods provide an interim result (y) expressed in mg/l which needs to be converted to give the final concentration according to the equation in 5.2.3.2.3.4.

#### 5.2.3.2.3.4 Expression of results

From the interim result (y) determined (see 5.2.3.2.3.3), the content,  $c_3$ , of each element in the laboratory sample, expressed in mg/kg of dry antiscalant products is given by the following equation.

$$c_3 = y \frac{V}{m} \tag{2}$$

y is the interim result (5.2.3.2.3.3);

V is the volume, expressed in ml, of the test solution (5.2.3.2.3.2) ( = 100 ml);

*m* is the mass, expressed in g, of the test portion.

#### 6 Labelling - Transportation - Storage

#### 6.1 Means of delivery

Phosphonic acids and salts can be delivered in bulk, containers, plastic drums, paper bags or big 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 Risk and safety labelling according to the EU legislation 4)

The following labelling requirements shall apply to phosphonic acids and salts at the date of the publication of this standard:



Signal word Warning Classification - Hazard statements: H302: Harmful if swallowed

#### Figure 1 GHS07

The legislation [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 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

Depending on the composition, the commercial products can be subject to transportation regulations. Neutralised products are not listed under a UN number. Acids are listed as UN number<sup>5)</sup> 3265 (corrosive liquid acidic, organic):

- RID<sup>6</sup>): class 8, classification code C3; packing group III;
- ADR<sup>7</sup>): class 8, classification code C3; packing group III;
- IMDG<sup>8)</sup>: class 8;
- IATA<sup>9)</sup>: class 8.

#### 6.4 Marking

The marking shall include the following information:

- name "phosphonic acid" or "phosphonic acid salt", trade name and grade;
- net mass;
- name and the address of the supplier and/or manufacturer;
- statement "this product conforms to EN 15040".

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 Roads.
- 8) International Maritime transport of Dangerous Goods.
- 9) International Air Transport Association.

<sup>4)</sup> See [2] and [3].

### 6.5 Storage

#### 6.5.1 Material

Use plastics or stainless steel depending on product. Avoid contact with other metals.

#### 6.5.2 Long term stability

Phosphonic acids and salts are stable for at least six months.

#### 6.5.3 Storage incompatibilities

For phosphonic acids and salts stored in cool and dry location, the container shall be closed when not in use.

# Annex A (informative)

### General information on phosphonic acids and salts

#### A.1 Origin

#### A.1.1 Raw materials

The raw materials for the production of phosphonic acids are:

formalin solution, organic amine (aqueous solution), acetic acid, phosphorus trichloride, phosphorous acid and acetic acid anhydride.

The raw materials for the production of phosphonic acid salts are:

sodium hydroxide solution, potassium hydroxide solution or ammonia solution.

#### A.1.2 Manufacturing process

Phosphonic acids: Several different reaction steps are involved.

- 1) Phosphorous acid is added to organic amine and heated between 50 °C and 80 °C. Then formalin solution is added and boiled at 110 °C for a period of time.
- Acetic acid is mixed with phosphorus trichloride and boiled at 120 °C. Then water is added (hydrolysis).
   Hydrochloric acid will be removed by distillation.
- Organic amine is heated to 80 °C and a solution of phosphorous acid dissolved in acetic acid anhydride is added. The mixture is boiled for 2 h. Afterwards water is added to cause hydrolysis and the acetic acid is removed by distillation.

Phosphonic acid salts:

salt solutions are produced by neutralisation or mixing of the acid solutions with sodium hydroxide solution, potassium hydroxide solution or ammonia solution.

Powder products:

spray dried salts of the phosphonic acids.

#### A.2 Use

#### A.2.1 Function

Phosphonic acids and salts are used as antiscalants especially for reverse osmosis (RO) and nanofiltration membranes to prevent CaCO<sub>3</sub>, CaSO<sub>4</sub>, BaSO<sub>4</sub>, SrSO<sub>4</sub>, CaF<sub>2</sub> scale deposition and fouling by iron, aluminium, manganese and silicates. They will not pass the membranes and are rejected to the wastewater with the concentrate.

Disposal of waste water should be done in accordance with all applicable local, national, and federal regulations.

In its concentrated form phosphonic acids are corrosive to metals. Corrosion resistant dosing equipment should therefore be used.

#### A.2.2 Form in which it is used

Phosphonic acids and salts are mainly used as a solution and within the range of mass fraction of 22 % to 60 % active matter (free acid).

#### A.2.3 Treatment dose

The prime application for phosphonic acids and salts are for the inhibition of calcium carbonate fouling, frequently in combination with other antiscalants (e.g. polycarboxylic acids). A typical dosage range is from 1 mg/l to 20 mg/l in the make-up water.

#### A.2.4 Means of application

The product can be pumped directly from the containers as supplied or the product can be diluted with permeate water prior to dose into the (RO) feed water stream

Dosage point: feed water prior to the high pressure pumps

Dosage frequency: continuous using a suitably sized dosing pump

Product dosage: neat as supplied or diluted with permeate water

#### A.2.5 Secondary effects

The product has no secondary effects.

#### A.2.6 Removal of excess product

Not applicable.

#### A.2.7 Ecological review

Refer to Safety Data Sheet (SDS) data of the specified product and in particular the standard heading 12 "Ecological information" as defined in ISO 11014-1:2009, Clause 5<sup>10</sup>).

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<sup>&</sup>lt;sup>10)</sup> See [3]

# **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 eyes or skin, rinse immediately with plenty of water.

#### **B.2.2 Spillage**

Remove mechanically as much as possible of the solid product, then rinse the area with plenty of water.

#### B.2.3 Fire

The products are not combustible.

# Annex C (informative)

### Environmental, health and safety precautions within chemical laboratory

When preparing the analytical methods for application of this document, consideration was given to the minimisation of environmental impacts caused by the use of the methods of analysis.

It is the users' responsibility to use safe and proper techniques in handling materials in the methods of analysis specified in this document.

The following list is not exhaustive but users of the analytical methods referred in this document may use it as a guide to the use of safe and proper techniques. They should:

- investigate if European Directives, transposed European legislation and national laws, regulations and administrative provisions apply;
- consult manufacturers/ suppliers for specific details such as material safety data sheets and other recommendations;
- use safety equipment and wear protective clothing, usually goggles and coats, appropriate for the test product and the test chemicals, in all laboratory areas, to ensure the safety of the operator;
- be careful about flammable materials and substances that are toxic and/ or human carcinogens and generally take care during transportation, decanting, diluting and dealing with spillages;
- use a fume cupboard during preparation of organic solvent solutions;
- store, handle and dispose of chemicals in a safe and environmentally satisfactory manner: including chemicals for laboratory test, test specimens, unused solvents and reagents.

### **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)
- [3] 67/548/EEC, Council Directive of 27th June 1967 on the approximation of the regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances and its amendments and adaptations
- [4] ISO 11014-1:2009, Safety data sheet for chemical products Part 1: Content and order of sections



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