Chemicals used for water treatment intended for human consumption — Polyamines

ICS 13.060.20; 71.100.80

British Standards

National foreword

This British Standard is the UK implementation of EN 1409:2008. It supersedes BS EN 1409:1998 which is withdrawn.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.

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

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 January 2009

ISBN 978 0 580 58027 7

Amendments/corrigenda issued since publication

Date	Comments

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1409

January 2008

ICS 71.100.80

Supersedes EN 1409:1998

English Version

Chemicals used for water treatment intended for human consumption - Polyamines

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

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Polyamine

This European Standard was approved by CEN on 10 November 2007.

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 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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 1409:2007: E

Contents

Page

Foreword

This document (EN 1409:2008) has been prepared by Technical Committee CEN/TC164 “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 July 2008, and conflicting national standards shall be withdrawn at the latest by July 2008.

This document supersedes EN 1409:1998.

Significant technical differences between this edition and EN 1409:1998 are as follows:

(a) reduction in the limit values for 1,3-dichloro-2-propanol and 2,3-dichloro-1-propanol in 4.3;

(b) introduction of a limit for 3-monochloropropane-1,2-diol (3-MCPD) in 4.3;

(c) reduction of the limit value for epichlorohydrin in 4.4;

(d) modification of the test method for epichlorohydrin, dichloropropanol isomers and 3-MCPD;

(e) updating of the reference to the drinking water directive from 80/778/EEC to 98/83/EC.

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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and 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 Member States of the EU or EFTA;

b) it should be noted that, while awaiting the adoption of verifiable European criteria, existing national regulations concerning the use and/or the characteristics of this product remain in force.

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

1 Scope

This European Standard is applicable to polyamines used for treatment of water intended for human consumption. It describes the characteristics and specifies the requirements and the corresponding test methods for polyamines. It gives information on its use in water treatment.

2 Normative references

The following referenced documents are indispensable for the application of this document. 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 3696:1987)*

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

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

3 Description

3.1 Identification

3.1.1 Chemical name

(di)methylamine - epichlorohydrin resin.

3.1.2 Synomyms or common names

- polyamine;
- polyalkylene amine;
- polyquaternary salts.

NOTE The more general terms "cationic polymer", "cationic polyelectrolyte", "cationic flocculant" and "quaternary ammonium polyelectrolyte" are used but can also cover other chemicals referred to in other European Standards.

3.1.3 Relative molecular mass

In the range of 10 000 g/Mol to 1 000 000 g/Mol.

3.1.4 Empirical formula

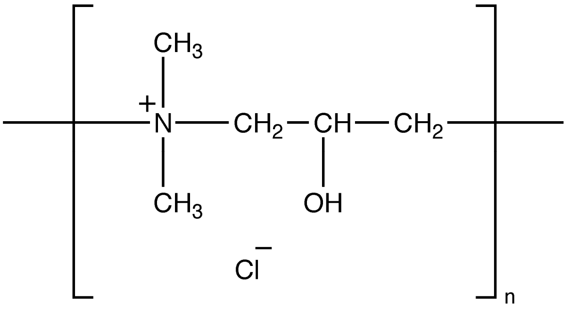
$-(C_aH_bN_cO_dCl_e)_n-$

where

- a, b, c, d and e are variable depending on the reactants used and on their molar ratios.

3.1.5 Chemical formulae

The following formula illustrates typical structures formed when dimethylamine is reacted with epichlorohydrin.



$$\left[-\overset{CH_3}{\underset{CH_3}{\overset{+}{N}}}-CH_2-\underset{OH}{CH}-CH_2- \right]_n \quad Cl^-$$

3.1.6 CAS Registry Numbers [1]

25988-97-0

68583-79-1

42751-79-1

NOTE This list is not exhaustive: polyamines with other CAS numbers are also used for water treatment.

3.1.7 EINECS reference [2]

EINECS does not list polyamines because EINECS reference numbers do not exist for polymers; conformity to EINECS being assessed on the basis of the monomers of which they are composed.

3.2 Commercial form

Polyamines as specified in this standard are aqueous polymer solutions in the form of viscous liquids, the concentration (active content) of which is approximately 30 percent to 50 percent mass fraction.

3.3 Physical properties

3.3.1 Appearance

The product is a clear, colourless to amber-coloured liquid.

3.3.2 Density

The density of the solution depends on concentration. A typical value is 1,16 g/ml for 50 % (*m/m*) polyamine at 20 °C.

3.3.3 Solubility

The products are miscible with water at all concentrations.

---

[1] Chemical Abstracts Service Registry Number.

[2] European Inventory of Existing Commercial Chemical Substances.

3.3.4 Vapour pressure

A typical value is 2,4 kPa for 50 % mass fraction polyamine at 20 °C.

3.3.5 Boiling point at 100 kPa[3)]

A typical value is 101 °C for 50 % mass fraction polyamine.

3.3.6 Freezing point

A typical value is - 7 °C for 50 % mass fraction polyamine.

3.3.7 Specific heat

A typical value is 3,13 kJ/kg K for 50 % mass fraction polyamine.

3.3.8 Viscosity, dynamic

The viscosity is dependent on molecular mass and active content. A typical value is 400 mPa.s for 50 % mass fraction polyamine at 20 °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

Polyamines are non hazardous materials and not intrinsically reactive. However, in common with many other organic compounds, a strong exothermic reaction will occur if they are brought into contact with strong acid or strong oxidizing agent.

NOTE In dilute solution there can be a reaction with, or destruction by, some of the disinfection and oxidizing agents used in water treatment.

4 Purity criteria

4.1 General

This European Standard specifies the minimum purity requirements for polyamines 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.

NOTE 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 and contents of other impurities and additives used in the product not stated in the 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 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 following requirements shall apply to polyamines:

- there shall be no visible insoluble gel or extraneous matter;
- pH shall be in the range 4 to 7.

4.3 Impurities and main by-products

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

Table 1 — Impurity/by - product

Impurity / by-product	Limits (mg/kg of active product)
3-monochloropropane-1,2-diol	40
1,3-Dichloro-2-propanol	40
2,3-Dichloro-1-propanol	40

Based on the raw materials and manufacturing process (see A.1), there are no significant concentrations of additional reactants or by-products which are relevant to the application of these products in drinking water treatment.

4.4 Chemical parameters

The product shall contain no more than 10 mg of epichlorohydrin (CAS Number 106-89-8) per kilogram of active ingredient.

NOTE Other chemical parameters and indicator parameters as listed in EU Directive 98/83/EC (see [1]) are not relevant to polyamines because the raw materials used in the manufacturing process are free of them and they are not by-products of the manufacturing process.

5 Test methods

5.1 Sampling

5.1.1 General

For sampling the recommendations given in ISO 3165 and ISO 6206 shall be followed.

A representative sample of the liquid product, of sufficient mass, shall be obtained immediately after manufacture or from a newly opened container(s). The sample shall be clearly labelled with product name/code, batch number, type of container(s) sampled and date sampled. Reference samples shall be retained for the storage life of the product as claimed by the manufacturer/supplier.

5.1.2 Sampling from drums and bottles

5.1.2.1 General

5.1.2.1.1 Mix the contents of the 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.

5.1.2.1.2 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 5.1.2.1.3.

5.1.2.1.3 Examine the surface of the liquid. If there are signs of surface contamination, take samples from the surface as described in 5.1.2.2; otherwise, take samples as described in 5.1.2.3.

5.1.2.2 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.

5.1.2.3 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 Sampling from tanks and tankers

From each access point, take samples as follows:

c) from the surface of the liquid, using a ladle as described in 5.1.2.2;

d) from the bottom of the tank or tanker, using a sampling tube as described in 5.1.2.3 or using specially designed bottom-sampling apparatus;

e) 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 General

Unless otherwise specified all reagents shall be of a recognised analytical grade. The water used shall conform to grade 2 specified in EN ISO 3696.

5.2.2 Main product

5.2.2.1 pH measurement

Measure the pH of the undiluted product, at a temperature of 20 °C, using a pH meter. Before making the measurement, calibrate the apparatus using a reference buffer solution of pH about 7,0.

5.2.2.2 Determination of active content

The proportion of active ingredient, i.e. polyamine, in a sample of product is determined by a procedure in which it is assumed that the product is an aqueous solution containing polyamine and inorganic metal salt. The active content, expressed as a percentage by mass of polyamine, is given by the following equation:

$$C_0 = C_1 - C_2$$

where

— C_0 is the percent by mass of polyamine;

— C_1 is the percent by mass of dry solids (see 5.2.2.3);

— C_2 is the percent by mass of inorganic metal salt (see 5.2.3.1).

The result shall be expressed to two decimal places.

NOTE The procedure will tend to overestimate the active content slightly if there are ammonium salts present in the product, as these are lost by volatilization at the ash determining step.

5.2.2.3 Determination of dry solids content

5.2.2.3.1 Principle

The product is heat dried and the mass difference determined gravimetrically.

5.2.2.3.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

5.2.2.3.2.1 Balance, with an accuracy of 0,1 mg.

5.2.2.3.2.2 Oven, capable of maintaining (110 ± 1) °C vented to fume cupboard.

5.2.2.3.2.3 Desiccator, containing dried silica gel.

5.2.2.3.2.4 Porcelain crucible, 57 mm diameter.

5.2.2.3.3 Procedure

Place a porcelain crucible (5.2.2.3.2.4) in an oven at 110 °C (5.2.2.3.2.2) for at least 10 min.

Remove the crucible from the oven, place in a desiccator (5.2.2.3.2.3) and allow to cool for at least 10 min.

Weigh the crucible to the nearest 0,1 mg.

Shake the sample in its container to ensure that it is homogeneous.

Add 1g to 2 g test portion of the product sample to the crucible and weigh to the nearest 0,1 mg.

Place crucible in oven at 110 °C for 2 h.

After this time, transfer crucible directly from the oven to desiccator and allow to cool for at least 10 min.

Weigh the crucible containing the dry residue to the nearest 0,1 mg.

5.2.2.3.4 Expression of results

5.2.2.3.4.1 Method of calculation

The dry solids content, C_1, expressed as a percentage by mass of the product, is given by the following equation:

$$C_1 = \frac{(m_3 - m_1)}{(m_2 - m_1)} \times 100$$

where

— m_1 is the mass, in grams, of the crucible;

— m_2 is the mass, in grams, of the crucible and wet sample;

— m_3 is the mass, in grams, of the crucible and dried sample.

The result shall be expressed to two decimal places.

5.2.2.3.4.2 Precision

The absolute difference between two single test results, obtained under repeatability conditions (see note), should not be greater than the repeatability value, *r*, as calculated from the following equation:

$$r = 0{,}05\ z$$

where

— z is the mean of the two results, expressed in % (*m/m*).

NOTE Repeatability conditions are where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

5.2.2.4 Test methods for assessment of product quality

If additional requirements are agreed between the customer and the manufacturer/supplier, the latter shall provide the necessary test methods, if requested, so that the customer can carry out his own quality checks. A certificate of analysis shall be provided by the manufacturer/supplier, if requested.

NOTE A number of physical/chemical measurements can be used by manufacturers to ensure the consistent quality of products delivered to customers. For example, solution viscosity is commonly measured, this being done under strictly controlled conditions. The viscosity value obtained provides a reliable indication of relative molecular mass when comparing batches of a particular product, but has no significance in absolute terms, since it is highly dependent on the composition of the product, the solution preparation procedure, the measuring device and test conditions used. Other tests which can be carried out include determination of ionic charge and infra-red spectroscopic analysis, depending on the product and manufacturer/supplier.

5.2.3 Impurities

5.2.3.1 Determination of salt content

WARNING: Toxic vapours might be released during the ignition of the test material. Use a furnace which is vented to a fume cupboard.

5.2.3.1.1 Principle

A known mass of wet sample is dried in accordance with 5.2.2.3. The salt content is then determined gravimetrically following ignition of the dry residue in a furnace at 600 °C.

5.2.3.1.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

5.2.3.1.2.1 Balance, with an accuracy of 0,1 mg.

5.2.3.1.2.2 Muffle furnace, capable of maintaining (600 ± 20) °C vented to fume cupboard.

5.2.3.1.2.3 Desiccator containing dried silica gel.

5.2.3.1.2.4 Oven, capable of maintaining (110 ± 1) °C.

5.2.3.1.3 Procedure

Place the crucible, containing the dry residue from 5.2.2.3 in the furnace at 600 °C (5.2.3.1.2.2) for at least 30 min. Remove the crucible and place in an oven at 110 °C (5.2.3.1.2.4) for at least 30 min, then place in a desiccator (5.2.3.1.2.3) until cool and reweigh to the nearest 0,1 mg.

5.2.3.1.4 Expression of results

5.2.3.1.4.1 Method of calculation

The salt content, C_2, expressed as a percentage by mass of the product, is given by the following equation:

$$C_2 = \frac{(m_4 - m_1)}{(m_2 - m_1)} \times 100$$

where

— m_1 is the mass, in grams, of the crucible;

— m_2 is the mass, in grams, of the crucible and wet sample;

— m_4 is the mass, in grams, of the crucible and ashed sample.

5.2.3.1.4.2 Precision

The absolute difference between two single test results, obtained under repeatability conditions (see note), should not be greater than the repeatability value, r, as calculated from the following equation:

— $r = 0{,}13\ z$

where

— z is the mean of the two results, expressed in % (*m/m*).

NOTE Repeatability conditions are where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

5.2.3.2 Determination of epichlorohydrin, 1,3-dichloro-2-propanol, 2,3-dichloro-1-propanol and 3-monochloropropane-1, 2-diol contents.

WARNING: Epicholorhydrin is flammable with a poisonous vapour. It is toxic by inhalation, skin absorption and ingestion and is irritating to the eyes, skin and respiratory system. Eye contact can cause conjunctivitis. It can cause cancer. Avoid exposure and keep closed containers in a well-ventilated place.

1,3-dichloro-2-propanol and 2,3-dichloro-1-propanol are irritant and toxic by inhalation, skin absorption and ingestion. Avoid exposure and keep closed containers in a well-ventilated place.

3-monochloropropane-1,2-diol is flammable with a poisonous vapour. It is toxic by skin absorption and ingestion and is irritating to the eyes, skin and respiratory system. It can cause cancer. Avoid exposure and keep closed containers in a well-ventilated place.

Tert-Butyl methyl ether is highly flammable. Keep container tightly closed in a cool, well-ventilated place. Take precautions against static electricity discharges. Keep away from sources of ignition. Do not breathe vapour.

5.2.3.2.1 Principle

Epichlorohydrin, 1,3-dichloro-2-propanol, 2,3-dichloro-1-propanol and 3-monochloropropane-1, 2-diol are extracted from the polymer with tert-Butyl methyl ether then separated and quantified using gas chromatography (GC).

It is not required to include epichlorohydrin to be analysed in the final product due to the fact that 1,3-dichloro-2 propanol, 2,3-dichloro-1-propanol and 3 monochloropropane-1,2 diol are all by-products of ECH.

5.2.3.2.2 Reagents

5.2.3.2.2.1 *Epichlorohydrin* (ECH) CAS No. (106-89-8).

5.2.3.2.2.2 *1,3-Dichloro-2-propanol* (1,3 DCP) CAS NO. (96-23-1).

5.2.3.2.2.3 *2,3-Dichloro-1-propanol* (2,3 DCP) CAS No. (616-23-9).

5.2.3.2.2.4 *Tert-Butyl methyl ether* (TBME) CAS No. (1634-04-4).

5.2.3.2.2.5 *3-monochloropropane-1,2-diol* (MCPD) CAS No. (96-24-2)

NOTE In respect of 5.2.3.2.2.1, 5.2.3.2.2.2, 5.2.3.2.2.3 and 5.2.3.2.2.5 a certificate of analysis giving the assay of the reagent can be obtained from the supplier.

5.2.3.2.2.6 *ECH standard stock solution*. Weigh (0,100 ± 0,05) g W of ECH into a small glass sample bottle, and make up to (100,0 ± 0,5) g T with TBME. Cap the bottle and shake it vigorously to mix the contents.

The concentration of ECH in milligrams per kilogram is calculated as follows:

$$C(ECH) = W \times \frac{P \times 10}{T}$$

where

P is the assay of ECH, expressed as per cent by mass [%(m/m)].

5.2.3.2.2.7 Calibration solutions of ECH $A_{1,}$ weigh (0,500 ± 0,05)g M_1 of C(ECH) 5.2.3.2.2.6 into a small glass bottle and make up to (100 ± 0,2) g with TBME.

5.2.3.2.2.8 Calibration solutions of ECH $A_{2,}$ weigh (1,000 ± 0,05) g M_2 of C(ECH) 5.2.3.2.2.6 into a small glass bottle and make up to (100 g ± 0,2) g with TBME.

5.2.3.2.2.9 Calibration solutions of ECH $A_{3,}$ weigh (2,500 ± 0,05) g M_3 of C(ECH) 5.2.3.2.2.6 into a small glass bottle and make up to (100 g ± 0,2)g with TBME.

5.2.3.2.2.10 Concentration of ECH in the calibration solutions is calculated as follows:

For 5.2.3.2.2.7, $A_1 = \frac{C(ECH) \times M_1}{100 \pm 0,2}$

Substitute M_2 and M_3 in the equation for concentration of standards A_2 and A_3.

5.2.3.2.2.11 *Stock solution of MCPD*.

Weigh (0,25 ± 0,05) g (G_1) of MCPD 5.2.3.2.2.5 into a small bottle and make up to (25 ± 0,2) g (S_1) of TBME. Cap the bottle and shake vigorously to mix the contents. Concentration C(MCPD) is calculated as below:

$$C(MCPD) = \frac{G_1}{S_1} \times 100 \times P = CM$$

where P = assay of MCPD (% m/m).

5.2.3.2.2.12 *1,3 DCP and 2,3 DCP standard stock solution*.

Weigh (0,500 ± 0,05) g (G_2) of 1.3 DCP 5.2.3.2.2.2 and (0,500 ± 0,05) g (G_3) of 2,3 DCP 5.2.3.2.2.3 into a glass bottle and make up to (25 ± 0,2) g (S_2) with TBME, cap and shake vigorously to mix.

$$C(1{,}3DCP) = \frac{G_2}{S_2} \times 100 \times P = C_1$$

$$C(2{,}3DCP) = \frac{G_3}{S_2} \times 100 \times P = C_2$$

P represents assay of 1,3 and 2,3 DCP % m/m.

5.2.3.2.2.13 *Mixed standard stock of 1,3 DCP; 2,3 DCP and MCPD*.

Weigh (10,000 0 ± 0,05) g (D) of 1,3; 2,3 DCP stock (5.2.3.2.2.12) into a glass bottle and (2,000 0 ± 0,05) g (MP) of MCPD stock (5.2.3.2.2.11). Add (28,00 ± 0,05) g (S_3) of TBME.

Thoroughly mix the solution and calculate the concentration of the impurity in the mixed stock standard as below:

$$C(1{,}3DCP) = \frac{D}{S_3} \times C_1 = C_{m1}$$

$$C(2{,}3DCP) = \frac{D}{S_3} \times C_2 = C_{m2}$$

$$C(MCPD) = \frac{MP}{S_3} \times CM = C_{m3}$$

5.2.3.2.2.14 *Calibration solution A*.

Take (0,400 00 ± 0,05) g (G_4) of 5.2.3.2.2.13 add (39,6 ± 0,05) g (S_4) of TBME.

5.2.3.2.2.15 *Calibration solution B*.

Take (1,0 ± 0,05) g (G_5) of 5.2.3.2.2.13 add (39,0 ± 0,05) g (S_5) of TBME.

5.2.3.2.2.16 *Calibration solution C*.

Take (4,0 ± 0,05) g (G_6) of 5.2.3.2.2.13 add (36,0 ± 0,05) g (S_6) of TBME.

5.2.3.2.2.17 Concentrations of DCP and MCPD in calibration solutions

Concentration of the calibration solutions is calculated as follows:

$$C\ (1{,}3\ \text{DCP})\ \text{in A} = C_{m1} \times \frac{G_4}{S_4}$$

$$C\ (2{,}3\ \text{DCP})\ \text{in A} = C_{m2} \times \frac{G_4}{S_4}$$

$$C\ (\text{MCPD})\ \text{in A} = C_{m3} \times \frac{G_4}{S_4}$$

Substitute masses of the mixed calibration solution taken for Standards B and C and calculate as above.

5.2.3.2.3 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

5.2.3.2.3.1 *Gas chromatograph*, with flame ionisation detector (FID).

5.2.3.2.3.2 *Integrator* / data capture by integration.

5.2.3.2.3.3 *Balance*, with an accuracy of 0,1 mg.

5.2.3.2.3.4 *Column*, HP Ultra 2 (5% diphenyl 95% dimethyl-siloxane) 25 m × 0,2 mm film thickness, 0,33 µm, or equivalent.

5.2.3.2.3.5 *Syringe*, GC, 10 µl.

5.2.3.2.3.6 *Glass bottle*, circa 120 ml capacity, with polytetrafluorethylene (PTFE) lined screw caps.

5.2.3.2.3.7 *Laboratory shaker*.

5.2.3.2.3.8 *Pipettes*. Disposable Pasteur pipettes.

5.2.3.2.3.9 *Glass vials*, 2ml, with septum, crimped or screw cap.

5.2.3.2.4 Procedure

5.2.3.2.4.1 *Test portion and sample extraction*.

A sample extraction solution is prepared for subsequent GC analysis as follows:

Weigh a test portion of (25,0 ± 0,1)g (M_p) of polymer sample (5.2.2.2) into a clean glass bottle (5.2.3.2.3.6) and add (20,0 ± 0,1) g (M_t) of TMBE (5.2.3.2.2.4). Cap the bottle and, by means of laboratory shaker (5.2.3.2.3.7), agitate the contents vigorously for 5 min.

Allow to stand for a few minutes until the two layers have completely separated.

Draw off some of the TMBE layer with a pipette (5.2.3.2.8) and transfer to a 2ml glass vial (5.2.3.2.3.9) for storage in a refrigerator until required for analysis.

5.2.3.2.4.2 *Analytical procedure*.

Instrument settings: extraction solutions, calibration and blank solutions are analysed by GC apparatus (5.2.3.2.3.1). To ensure good separation of ECH the following settings and conditions are used:

Oven/column temperature programme:

Initial temperature = 40 °C for 3.2 min.

Rate at 10 °C/min to 60 °C for 3 min.

Rate at 45 °C/min to 250 °C for 2 min.

Injector temperature = 250 °C.

Detector temperature = 300 °C.

Injection volume = 2 µl.

Carrier gas = hydrogen.

Column head pressure = 100 kPa.

Total flow = 90ml/min.

5.2.3.2.4.3 *Retention times*.

To determine retention times, prepare separate solutions in TBME of ECH, 1,3 DCP; 2,3 DCP and MCPD. Inject with a syringe (5.2.3.2.3.5) 2 µl of one solution into the gas chromatograph (5.2.3.2.3.1) and record the retention time of the resulting peak. Repeat this procedure with all the other solutions.

5.5.3.2.4.4 *Blank determination.*

Inject 2µl of TMBE solvent into the gas chromatograph to obtain a blank reading. (This ensures that there is no interfering peak present.)

5.2.3.2.4.5 *Calibration.*

For calibrations solutions A (5.2.3.2.2.14), B (5.2.3.2.2.15) and C (5.2.3.2.2.16) in turn, inject 2µl of each solution into the gas chromatograph and measure the areas of the resulting peaks.

If confirmation of ECH is required, a separate calibration graph should be obtained from standard solutions prepared as specified in 5.2.3.2.2.7, 5.2.3.2.2.8 and 5.2.3.2.2.9.

5.2.3.2.4.6 *Sample analysis.*

Inject 2µl of sample extraction solution into the gas chromatograph and with the aid of an electronic integrator (5.2.3.2.3.2), measure the areas of the peaks for each of the 3 main impurities, (1,3 DCP; 2,3 DCP; MCPD).

5.2.3.2.5 Expression of results

5.2.3.2.5.1 *Method of calculation*.

The contents of ECH; 1,3 DCP; 2,3 DCP and MCPD, expressed in milligrams per kilogram of active product are given by the following equation:

$$C_6 = \frac{A_1}{A_S} \times C_7 \times \frac{M_T}{M_P} \times \frac{100}{C_0}$$

where

A_1 is the peak area of the impurity in the extraction solution;

A_s is the peak area of the impurity standard;

C_7 is the concentration, in milligrams per kilogram (mg/kg) of the impurity in the calibration solutions A, B or C;

M_T is the mass in grams (g), of TBME (5.2.3.2.4.1);

M_P is the mass in grams (g), of test portion (5.2.3.2.4.1)

C_o is the percent by mass [%(m/m)] of polyamine in the product (5.2.2.2).

OR Values for the impurity can be read off a calibration graph, the dilution factor and active strength of the product taken into account as below:

$$C_6 = \text{Impurity reading} \times \frac{M_T}{M_P} \times \frac{100}{C_0}$$

5.2.3.2.5.2 *Precision*

The absolute difference between two single test results, obtained under repeatability conditions (see note), shall not be greater than the repeatability value, r, as calculated from the following equation:

Epichlorohydrin: r = 0,21 z
1,3- and 2,3-DCP: r = 0,13 z

MCPD: r = 0,26 z

where

z is the mean of the two results, expressed in milligrams per kilogram (mg/kg) of active product.

NOTE Repeatability conditions are conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

6 Labelling - transportation - storage

6.1 Means of delivery

The product shall be delivery in suitable containers, e.g. in bulk containers, drums, cans or bottles.

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 Risk and safety labelling in accordance with the EU Directives

Depending on toxicity test results of the specific product or on the concentration of active ingredient, appropriate R-and S-phrases shall be assigned to polyamines to warn of possible eye irritant hazard. Appropriate risk phrasing shall also be entered on the label or material safety data sheet to the effect that the product is slippery. For minimum labelling to be applied to packages, see 6.4.

NOTE 1 Attention is drawn to EC Directive 67/548/EEC of 27th June 1967 (see [2]).

NOTE 2 Annex I of the Directive 67/548/EEC on Classification, packaging and labelling of dangerous substances and its amendments and adaptations in the European Union contains a list of substances classified by the EU. Substances not in this Annex I should be classified on the basis of their intrinsic properties according to the criteria in the Directive by the person responsible for the marketing of the substance.

6.3 Transportation regulations and labelling

Polyamines are not classified as hazardous for transport and do not therefore have a UN number[4)], hazard class, packaging group or require UN-certified containers.

6.4 Marking

Each container shall be marked with at least the following information:

- name "polyamine", trade name and grade;
- net mass;
- name and address of supplier and/or manufacturer;
- statement "This product conforms to EN 1409".

6.5 Storage

6.5.1 Long term stability

The product is usually stable for at least 12 months. Follow supplier's advice.

6.5.2 Storage incompatibilities

Store away from strong acids (e.g. sulfuric acid) and strong oxidizing agents (e.g. sodium hypochlorite).

[4)] United Nations Numbers.

Annex A
(informative)

General information on polyamines

A.1 Origin

A.1.1 Raw materials

Polyamines are manufactured from dimethylamine or methylamine and epichlorohydrin with, optionally, a multi-functional amine such as ethylene diamine.

A.1.2 Manufacturing process

Polyamines as specified in this standard are produced by the spontaneous reaction of amines with epichlorohydrin in aqueous solution to form an adduct which then polymerizes on heating. The optional use of multi-functional amine serves to introduce branching into the polymer and hence increases molecular mass.

A.2 Use

A.2.1 Function

Polyamines are used in drinking water treatment to effect coagulation and flocculation of colloidal and fine suspended particles and thereby assist their removal. Polyamines are thus usually utilized in the mainstream processes of clarification and filtration, where they can be used as sole flocculants or to supplement the coagulating action of metal salts.

A.2.2 Form in which it is used

Usually, the product is introduced into the process stream as a dilute (1 % to 10 % mass fraction of product as supplied) solution in order to effect rapid and even dispersion. This can be done either by batch mixing with water in a stirred tank or by metering the product directly and diluting in line prior to addition.

A.2.3 Treatment dose

The treatment dose will vary depending on the quality of the raw water to be treated and can be subject to local regulations. The dose should be selected so as not to exceed the parametric values for chemical parameters as listed in EC Directive 98/83/EC (see [1]) or local regulations. Typically a level of active product of between 1 mg/l and 2 mg/l is used.

A.2.4 Means of application

The product is usually applied using a metering pump.

A.2.5 Secondary effects

Very slight increase in chloride content.

A.2.6 Removal of excess product

Not applicable.

A.3 Rules for safe handling and use

Good chemical handling practice should be followed at all times.

Polyamines do not present a significant health hazard when correctly handled:

- in case of spillage, it should be prevented large quantities from entering drains or watercourses. It should be contained with an inert material, shovelled up and removed for disposal. Residues and small spillages should be hosed away completely with plenty of water;
- eye and hand protection is recommended. Mild eye and skin irritation can result from prolonged contact;
- protective clothing is not required on safety grounds, but overalls are recommended as cleaning can be problematic;
- respiratory protection is not required under normal conditions of use, providing ventilation is adequate.

A.4 Emergency procedures

A.4.1 First aid

If polyamine is in contact with the skin the contaminated area should be washed with copious amounts of soap and water.

If polyamine is in contact with the eyes, they should be rinsed with water for at least 15 min. If irritation persists, medical advice should be sought.

If polyamine is ingested, the mouth should be washed out with water but the affected person should not be allowed to swallow the wash water. Then water should be given to drink. An emetic should not be given. The affected person should be allowed to rest and medical advice should be sought immediately.

In addition to the above, any further advice on the supplier's safety data sheet should be followed.

A.4.2 Spillage

If a large spillage occurs, it should be contained with an inert material, such as sand or earth, to prevent it reaching the drains, and it should then be removed for disposal. Residues or small spillages can be flushed away with water. Spillages should not be disposed in watercourses.

A.4.3 Fire

Low fire and explosion risk. The product will not burn or support combustion easily. The following extinguishing media can be used: carbon dioxide, water spray, dry powder, foam. In addition, supplier's recommendations should be consulted.

Bibliography

[1] 98/83/EC, Council Directive of 3 November 1998 on the quality of water intended for human consumption.

[2] 67/548/EEC, Council Directive of 27th June 1967 on the approximation of the laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances.

BSI - British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: +44 (0)20 8996 9000. Fax: +44 (0)20 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: +44 (0)20 8996 9001. Fax: +44 (0)20 8996 7001 Email: orders@bsigroup.com You may also buy directly using a debit/credit card from the BSI Shop on the Website http://www.bsigroup.com/shop

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact Information Centre. Tel: +44 (0)20 8996 7111 Fax: +44 (0)20 8996 7048 Email: info@bsigroup.com

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: +44 (0)20 8996 7002 Fax: +44 (0)20 8996 7001 Email: membership@bsigroup.com

Information regarding online access to British Standards via British Standards Online can be found at http://www.bsigroup.com/BSOL

Further information about BSI is available on the BSI website at http://www.bsigroup.com.

Copyright

This does not preclude the free use, in the course of implementing the standard, of necessary details such as symbols, and size, type or grade designations. If these details are to be used for any other purpose than implementation then the prior written permission of BSI must be obtained.

Details and advice can be obtained from the Copyright and Licensing Manager. Tel: +44 (0)20 8996 7070 Email: copyright@bsigroup.com

BSI Group
Headquarters 389
Chiswick High Road,
London, W4 4AL, UK
Tel +44 (0)20 8996 9001
Fax +44 (0)20 8996 7001
www.bsigroup.com/
standards