CDC 6 (3945) DTZS

ICS 71.100.80

DRAFT TANZANIA STANDARD

Polyamines for treatment of water intended for human consumption — Specification

Draft for public comments only

TANZANIA BUREAU OF STANDARDS

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2nd Edition 2026

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Foreword

This Tanzania Standard was published under the authority of the Board of Director of Tanzania Bureau of Standards.

Tanzania Bureau of Standards (TBS) is the statutory national standards body for Tanzania established by the Standards Act Cap. 130.

Tanzania Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. The Technical Committees work under the supervision of Divisional (sectoral) Committees. The Standards are developed in accordance with the Guide and Procedure for Development of Tanzania Standards and TZS 0, Guide for presentation of Tanzania Standards.

Tanzania Standards are subject to review, to keep pace with science and technological advances. Users of the Tanzania Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

This Tanzania Standard was developed under the supervision of the Chemical Divisional Standards Committee [*CDC*]. The Technical Committee responsible for the standard is CDC 6-Water quality.

This second cancels and replaces the first edition (*TZS 1409:2011*), which has been technically revised.

This Tanzania Standard has been prepared with the assistance drawn from:

> BS EN 1409:2023, Polyamines for treatment of water intended for human consumption – Specification, published by European Union

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Polyamines for treatment of water intended for human consumption — Specification

1 Scope

This draft Tanzania Standard specifies the requirements, sampling and test methods for polyamines used for treatment of water intended for human consumption.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

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

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

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

ISO 8213: 1986, Chemical products for industrial use – Sampling techniques – Solid chemical products in the form of particles varying from powders to coarse lumps

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Description

4.1 Identification

4.1.1 Chemical name(s)

(di)methylamine - epichlorohydrin resin.

4.1.2 Synonym(s) or common name(s)

— Polyamine;

— Polyalkylene amine;

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— Polyquaternary salts.

NOTE The more general terms: “quaternary ammonium polyelectrolyte”, “cationic polymer”, “cationic polyelectrolyte”, “polymer coagulant” and “cationic flocculant” are used, but can also cover other chemicals referred to in other standards.

4.1.3 Relative molecular mass

Typically, in the range of 10000 to 1 million Daltons.

4.1.4 Empirical formulae

Non-ionic polyacrylamide:

$(C_aH_bN_cO_dCl_e)_n$

where

n is variable depending on the product;

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

4.1.5 Chemical formula

The following formula (Figure 1) illustrates typical structures formed when dimethylamine is reacted with epichlorohydrin.

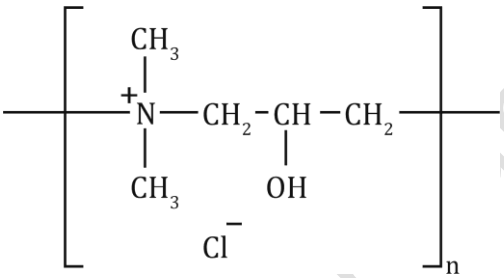


Figure 1 — Dimethylamine reaction with epichlorohydrin

Where

n is variable depending on the product

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4.1.6 CAS Registry Numbers [1])

The following is an exemplary list of CAS Registry Numbers for typical anionic or non-ionic polyacrylamides:

— 25988-97-0

— 68583-79-1

— 42751-79-1

— 31568-35-1

— 52722-38-0

— 42751-81-5

— 68409-70-1

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

1) Chemical Abstracts Service Registry Number.

4.1.7 EINECS reference [2])

The conformity of polymers to EINECS is assessed on the basis of the monomers of which they are composed. Thus, EINECS reference numbers do not exist for polymers.

Polymers are exempt from registration according to EU Regulation 1907/2006/EC (see [3]), REACH.

Monomer substance(s) and any other substance(s) in the form of monomeric units and chemically bound substance(s) may have to be REACH registered according to Article 6 of EU Regulation 1907/2006/EC.

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

4.2 Commercial form

Polyamines as specified in this standard are aqueous solutions, the concentration (active content) of which is approximately 20 percent to 50 percent mass fraction (see 7.2.2.2).

5 Physical properties

5.1 Appearance

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

5.2 Density

The density of the solution depends on the concentration. A typical value is 1.16 g/ml for 50 % mass fraction polyamine at 20 °C.

5.3 Solubility

The product is miscible with water at all concentrations.

5.4 Vapour pressure

A typical value is 2.4 kPa for 50 % mass fraction polyamine at 20 °C.

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5.5 Boiling point at 100 kPa [3])

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

5.6 Freezing point

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

5.7 Specific heat

A typical value is 3.13 kJ/kg K for 50 % mass fraction polyamine.

5.8 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.

5.9 Critical temperature

Not applicable.

5.10 Critical pressure

Not applicable.

5.11 Physical hardness

Not applicable.

6. Chemical properties

6.1 General

Polyamines are a non-hazardous material and not intrinsically reactive. However, in common with many other organic compounds, a strong exothermic reaction will occur if it is brought into contact with strong acids or oxidizing agents.

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

6.2 Purity criteria

6.2.1 General

This document 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.

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.

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.

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6.2.2 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.

6.3 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.

NOTE Various parameters can be checked as part of assessment of product quality (see 7.2.2).

6.4 Chemical parameters

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

polymer.

NOTE Other chemical parameters and indicator parameters as listed in EU Directive 2020/2184 (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.

7 Sampling

7.1 Sampling

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.

7.1.1 General

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.

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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 7.1.2.1.

Examine the surface of the liquid. If there are signs of surface contamination, take samples from the surface as described in 7.1.2.2; otherwise, take samples as described in 7.1.2.3.

7.1.2.2 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.

7.1.2.3 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.

7.1.2.4 Sampling from tanks and tankers

From each access point, take samples as follows:

— from the surface of the liquid, using a ladle as described in 7.1.2.2;

— from the bottom of the tank or tanker, using a sampling tube as described in 7.1.2.3 or using specially designed bottom-sampling apparatus;

— from one or more positions, depending on the overall depth, between the bottom and the surface using a weighted sampling can.

8. Test Method

Methos of test shall be carried in accordance to annex A

9 Labelling - transportation – storage

9.1 Means of delivery

The product shall be delivered in suitable containers, e.g. bulk containers, 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.

9.2 Transportation regulations and labelling

Polyamines are not classified as hazardous for transport and does not therefore have a UN number, hazard class, packaging group or require UN-certified containers. The user shall be aware of the incompatibilities between transported products.

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10. Marking

Each container and the accompanying documents shall be marked with the following information:

— name “Polyamine”;

— trade name;

— net mass;

— name and address of supplier and/or manufacturer;

— date of manufacturing;

8.4 Storage

8.4.1 Long term stability

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

8.4.2 Storage incompatibilities

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

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Annex A

(Normative)

Test method

A.2 Analyses

A.2.1 General

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

A.2.2 Main product

A.2.2.1 Measurement of pH

The measurement of the pH is carried out on the undiluted product, at a temperature of 20°C, using a pH meter. Before making the measurement, the apparatus shall be calibrated using a reference buffer solution of pH about 7,0.

A.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 formula:

$C_0 = C_1 - C_2$

where

C_0 is the percent by mass of polyamine;

C_1 is the percent by mass of dry solids (see A.2.2.3);

C_2 is the percent by mass of inorganic metal salt (see A.2.3.2);

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 volatilisation at the ash determining step.

A.2.2.3 Determination of dry solids content

A.2.2.3.1 Principle

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

A.2.2.3.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

A.2.2.3.2.1 Balance, with an accuracy of 0.1 mg.

A.2.2.3.2.2 Oven, capable of maintaining (110 ± 1) °C vented to fume cupboard;

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A.2.2.3.2.3 Desiccator containing dried silica gel; A.2.2.3.2.4 Porcelain crucible, 57 mm diameter.

A.2.2.3.3 Procedure

Place porcelain crucible (A.2.2.3.2.4) in oven at 110 °C (A.2.2.3.2.2) for at least 10 min.

Remove crucible from oven, place in desiccator (A.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 1 g 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.

A.2.2.3.4 Expression of results

A.2.2.3.4.1 Method of calculation

The dry solids content, C1, expressed as a percentage by mass of the product, is given by the following formula:

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

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.

A.2.2.3.4.2 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 formula:

r = 0.05 z

where

r is the repeatability value;

z is the mean of the two results, expressed in percent by mass.

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.

A.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.

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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 infrared spectroscopic analysis, depending on the product and manufacturer/supplier.

A.2.3 Impurities

A.2.3.1 Determination of epichlorohydrin, 1,3-dichloro-2-propanol, 2,3-dichloro-1-propanol and 3-monochloropropane-1, 2-diol contents

A.2.3.1.1 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 Epichlorohydrin.

A.2.3.1.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

A.2.3.1.2.1 Gas chromatograph, with flame ionization detector (FID);

A.2.3.1.2.2 Integrator / data capture by integration;

A.2.3.1.2.3 Balance, with an accuracy of 0.1 mg;

A.2.3.1.2.4 Column, HP Ultra 2 (5 % diphenyl 95 % dimethyl-siloxane) 25 m × 0.2 mm film thickness, 0.33 µm, or equivalent;

A.2.3.1.2.5 Syringe, GC, 10 µl;

A.2.3.1.2.6 Glass bottle, circa 120 ml capacity, with polytetrafluorethylene (PTFE) lined screw caps;

A.2.3.1.2.7 Laboratory shaker;

A.2.3.1.2.8 Pipettes, disposable Pasteur pipettes;

A.2.3.1.2.9 Glass vials, 2ml, with septum, crimped or screw cap.

A.2.3.1.3 Reagents

A.2.3.1.3.1 Epichlorohydrin (ECH), CAS No. 106-89-8, analytical grade;

A.2.3.1.3.2 1,3-Dichloro-2-propanol (1,3-DCP), CAS No. 96-23-1, analytical grade;

A.2.3.1.3.3 2,3-Dichloro-1-propanol (2,3-DCP), CAS No. 616-23-9, analytical grade;

A.2.3.1.3.4 Tert-Butyl methyl ether (TBME), CAS No. 1634-04-4, analytical grade;

A.2.3.1.3.5 3-Monochloropropane-1,2-diol (MCPD), CAS No. 96-24-2, analytical grade;

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NOTE In respect of Epichlorohydrin, 1,3-Dichloro-2-propanol, 2,3-Dichloro-1-propanol and 3-Monochloropropane-1,2-diol, a certificate of analysis giving the assay of the reagent can be obtained from the supplier.

A.2.3.1.3.6 ECH standard stock solution

Weigh (0.100 ± 0.05) g, W, of ECH (A.2.3.1.3.1) into a small glass sample bottle, and make up to (100.0 ± 0.5) g, T, with TBME (A.2.3.1.3.4). Cap the bottle and shake it vigorously to mix the contents.

The concentration of ECH in milligrams per kilogram, C(ECH), is calculated as follows:

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

where

W is the mass, in grams, of ECH;

P is the assay of ECH, expressed as percent by mass;

T is the mass, in grams, of ECH standard stock solution.

A.2.3.1.3.7 Calibration solutions of ECH, A_1

Weigh (0.500 ± 0.05) g, M_1, of ECH standard stock solution (A.2.3.1.3.6) into a small glass sample bottle, and make up to (100,0 ± 0.2) g, T_2, with TBME (A.2.3.1.3.4). Cap the bottle and shake vigorously to mix the contents.

A.2.3.1.3.8 Calibration solution of ECH, A_2

Weigh (1,000 ± 0.05) g, M_2, of ECH standard stock solution (A.2.3.1.3.6) into a small glass sample bottle, and make up to (100.0 ± 0.2) g, T_2, with TBME (7.2.3.1.3.4). Cap the bottle and shake vigorously to mix the contents.

A.2.3.1.3.9 Calibration solution of ECH, A_3

Weigh (2.500 ± 0.05) g, M_3, of ECH standard stock solution (A.2.3.1.3.6) into a small glass sample bottle, and make up to (100 ± 0,2) g, T_3, with TBME (A.2.3.1.3.4). Cap the bottle and shake vigorously to mix the contents.

The concentration of ECH in the calibration solutions is calculated as follows:

$$C_n - \frac{C(ECH) \times M_n}{T_n}$$

where

n is the number of the corresponding calibration solution;

M_n is the mass, in grams, of C(ECH) added in the Calibration solution, A_n;

T_n is the mass, in grams, of Calibration solution, A_n.

A.2.3.1.3.10 Stock solution of MCPD

Weigh (0.25 ± 0.05) g, G_1, of MCPD (7.2.3.1.3.5) into a small glass sample bottle, and make up to (25.0 ± 0.2) g, S_1, with TBME (A.2.3.1.3.4). Cap the bottle and shake vigorously to mix the contents.

Concentration of MCPD, C (MCPD), in the Stock solution of MCPD is calculated as follows:

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

where

P is the assay of MCPD, expressed as percent by mass.

A.2.3.1.3.11 1,3-DCP and 2,3-DCP standard stock solution

Weigh (0.500 ± 0,05) g, G_2, of 1,3-DCP (A.2.3.1.3.2) and (0.500 ± 0,05) g, G_3, of 2,3-DCP (A.2.3.1.3.3) into

a small glass bottle and make up to (25 ± 0,2) g, S_2, with TBME (A.2.3.1.3.4). Cap the bottle and shake vigorously to mix the contents.

The concentrations of 1,3-DCP, C (1,3-DCP), and 2,3-DCP, C (2,3-DCP), in the 1,3-DCP and 2,3-DCP standard stock solution are calculated as follows:

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

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

where

P is the assay of 1,3-DCP and 2,3-DCP, expressed as percent by mass.

A.2.3.1.3.12 Mixed standard stock of 1,3-DCP, 2,3-DCP and MCPD

Weigh (10 ± 0.05) g, D, of 1,3-DCP and 2,3-DCP standard stock solution (A.2.3.1.3.11) into a small glass bottle and (2000 ± 0.05) g, *MP*, of Stock solution of MCPD (A.2.3.1.3.10) and add (28 ± 0.05) g, S_3, with TBME (A.2.3.1.3). Cap the bottle and shake vigorously to mix the contents.

The concentrations of 1,3-DCP, C (1,3-DCP), of 2,3-DCP, C (2,3-DCP), and MCPD, *C* (MCPD), in the Mixed standard stock of 1,3-DCP, 2,3-DCP and MCPD are calculated as follows:

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

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

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

A.2.3.1.3.13 Calibration solution A

Take (0.40 ± 0.05) g, G_4, of Mixed standard stock of 1,3-DCP, 2,3-DCP and MCPD (A.2.3.1.3.12) and add (39.60 ± 0.05) g, S_4, of TBME (A.2.3.1.3.4).

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A.2.3.1.3.14 Calibration solution B

Take (1 ± 0.05) g, G_5, of Mixed standard stock of 1,3-DCP, 2,3-DCP and MCPD (A.2.3.1.3.12) and add (39± 0.05) g, S_5, of TBME (A.2.3.1.3.4).

A.2.3.1.3.15 Calibration solution C

Take (4 ± 0.05) g, G_6, of Mixed standard stock of 1,3-DCP, 2,3-DCP and MCPD (A.2.3.1.3.12) and add (36 ± 0.05) g, S_6, of TBME (A.2.3.1.3.4).

The concentrations of 1,3-DCP, C (1,3-DCP), of 2,3-DCP, C (2,3-DCP), and MCPD, C (MCPD), in the calibration solutions are calculated as follows:

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

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

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

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

A.2.3.1.4 Procedure

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.

A.2.3.1.4.1 Test portion and sample extraction

Weigh a test portion of (25 ± 0.1) g, M_p, of polymer sample (A.2.2.2) into a clean glass bottle (A.2.3.1.2.6) and add (20 ± 0.1) g (M_t) of TMBE (A.2.3.1.3.4). Cap the bottle and, by means of laboratory shaker (A.2.3.1.2.7), 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 (A.2.3.1.2.8) and transfer to a 2 ml glass vial (A.2.3.1.2.9) for storage in a refrigerator until required for analysis.

A.2.3.1.4.2 Determination

a) Chromatographic conditions

Samples, calibration and blank solutions are analysed by gas chromatography using the apparatus (A.2.3.1.2.1). To ensure good separation of ECH the following settings and conditions are used:

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b) Instrument settings

1) 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 = 90 ml/min.

c) Retention times

To determine retention times, prepare separate solutions in TBME (A.2.3.1.3.4) of ECH (A.2.3.1.3.1), 1,3- DCP (7.2.3.1.3.2), 2,3-DCP (A.2.3.1.3.3) and MCPD (A.2.3.1.3.5). Inject with a syringe (A.2.3.1.2.5) 2 µl of one solution into the gas chromatograph (A.2.3.1.2.1) and record the retention time of the resulting peak. Repeat this procedure with all the other solutions.

d) Blank determination

Inject 2µl of TMBE solvent into the gas chromatograph to obtain a blank reading.

NOTE This ensures that there is no interfering peak present.

e) Calibration

For calibrations solutions A (A.2.3.1.3.13), B (A.2.3.1.3.14) and C (A.2.3.1.3.15) in turn, inject 2 µl 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 A.2.3.1.3.

f) Sample analysis

Inject 2 µl of sample extraction solution into the gas chromatograph and with the aid of an electronic integrator (A.2.3.1.2.2), measure the areas of the peaks for each of the 3 main impurities (1,3-DCP, 2,3- DCP and MCPD).

A.2.3.1.5 Expression of results

A.2.3.1.5.1 Method of calculation

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

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

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 (7.2.3.1.3.4);

M_P is the mass in grams (g), of test portion (7.2.3.1.3);

C_0 is the percent by mass [%(m/m)] of polyamine in the product (7.2.2.2).

Alternatively, 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:

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

Results shall be expressed to the nearest whole number.

A.2.3.1.5.2 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 formulae:

Epichlorohydrin:

r = 0.21 z

1,3-DCP 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 of 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.

A.2.3.2 Determination of inorganic metal salt content

A.2.3.2.1 Principle

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A known mass of wet sample is dried in accordance with A.2.2.3. The salt content is then determined gravimetrically following ignition of the dry residue in a furnace at 600 °C.

WARNING — Toxic vapours (such as hydrogen chloride gas) may be released during the ignition of the test material.

A.2.3.2.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

A.2.3.2.2.1 Balance, with an accuracy of 0.1 mg;

A.2.3.2.2.2 Muffle furnace, capable of maintaining (600 ± 20) °C vented to fumed cupboard;

A.2.3.2.2.3 Desiccator containing dried silica gel;

A.2.3.2.2.4 Oven, capable of maintaining (110 ± 1) °C vented to fume cupboard.

A.2.3.2.3 Procedure

Place the crucible, containing the dry residue from A.2.2.3 in the furnace at 600 °C (A.2.3.2.2.2) for at least 30 min. Remove the crucible and place in an oven at 110 °C (A.2.3.2.4) for at least 30 min, then place in a desiccator (A.2.3.2.3) until cool and reweigh to the nearest 0.1 mg.

A.2.3.2.4 Expression of results

A.2.3.2.4.1 Method of calculation

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

$$C_2 = \frac{(m_3 - m_1)}{(m_2 - m_1)} \times 100$$

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 ashed sample.

A.2.3.2.4.2 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 formula:

$r = 0.13\ z$

where

z is the mean of the two results, expressed in milligrams per kilogram of 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.

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Annex B
(informative)

General information on polyamines

B.1 Origin

B.1.1 Raw materials

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

B.1.2 Manufacturing process

Polyamines are produced from 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.

B.2 Use

B.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.

B.2. Form in which it is used

Usually, the product is introduced into the process stream as a dilute (1 % mass fraction 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.

B.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. Typically, a level of active product of between 1 mg/l and 2 mg/l is used.

B.2.4 Means of application

Product is usually applied using a metering pump.

B.2.5 Secondary effects

Very slight increase in chloride content.

B.2.6 Removal of excess product

Not applicable.

B.3 Rules for safe handling and use

Before handle the product, the user should refer to the Safety Data Sheet if available. It is the responsibility of the employer to assess the risks according to national and European regulations and to take appropriate measures.

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Good chemical handling practice should be followed at all times.

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

Appropriate special risks should be entered on the safety data sheet to the effect that the product is slippery:

in case of spillage, it should be prevented large quantities from entering drains or watercourses and it should be contained with an inert material such as sand or earth and removed for disposal. The addition of water will render the floor very slippery and dangerous;

eye and hand protections are not normally warranted unless exposure is prolonged. Mild eye and skin irritation can result from extended 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.

B.4 Emergency procedures

B.4.1 First aid

In case of emergency, the user should refer to the Safety Data Sheet if available.

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.

B.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.

B.4.3 Fire

Low fire and explosion risk. As an aqueous solution product will not burn or support combustion easily. The following extinguishing media can be used in the event of a fire evaporating the water content: carbon dioxide, water spray, dry powder, foam. In addition, supplier's recommendations should be consulted.

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