

DRAFT UGANDA STANDARD

Second Edition
2021-mm-dd

Chemicals used for treatment of water intended for human use — Polyamines — Specification



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Foreword

Uganda National Bureau of Standards (UNBS) is a parastatal under the Ministry of Trade, Industry and Cooperatives established under Cap 327, of the Laws of Uganda, as amended. UNBS is mandated to coordinate the elaboration of standards and is

- (a) a member of International Organisation for Standardisation (ISO) and
- (b) a contact point for the WHO/FAO Codex Alimentarius Commission on Food Standards, and
- (c) the National Enquiry Point on TBT Agreement of the World Trade Organisation (WTO).

The work of preparing Uganda Standards is carried out through Technical Committees. A Technical Committee is established to deliberate on standards in a given field or area and consists of key stakeholders including government, academia, consumer groups, private sector and other interested parties.

Draft Uganda Standards adopted by the Technical Committee are widely circulated to stakeholders and the general public for comments. The committee reviews the comments before recommending the draft standards for approval and declaration as Uganda Standards by the National Standards Council.

The committee responsible for this document is Technical Committee UNBS/TC 5, Chemicals and environment.

This second edition cancels and replaces the first edition (US 926:2012), which has been technically revised.

Introduction

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.

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Chemicals used for treatment of water intended for human use — Polyamines — Specification

1 Scope

This draft Uganda Standard specifies the requirements and methods of sampling and test for polyamines used for water treatment intended for human use.

2 Normative references

The following referenced documents 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 3165, Sampling of chemical products for industrial use — Safety in sampling

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

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

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

ISO 1392, Determination of crystallizing point — General method

ISO 3146:2000, Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods

ASTM D2879 - 86, Standard Test Method for Vapor Pressure-Temperature Relationship and Initial Decomposition Temperature of Liquids by Isoteniscope

ASTM E537 - 20, Standard Test Method for Thermal Stability of Chemicals by Differential Scanning Calorimetry

ASTM E1269 – 11, Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry

US 1847:2017, Standard Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

3 Terms and definitions

There are no normative references in this document.

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

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

4 Requirements

4.1 General requirements

Polyamines shall be Clear, colourless to amber-coloured liquid. No visible insoluble gel or extraneous matter, and soluble with water at all concentrations in all proportions.

4.2 Specific requirements

Polyamines shall conform to the specific requirements given in table 1 when tested in accordance to the methods described therein.

Table 1— Specific requirements for polyamines

Parameter	Requirement	Test method
Specific gravity, at 20°C.	1.16 - 1.24	US 1847:2017
Vapour pressure, kPa at 20°C	2.4	ASTM D 2879-86
Boiling point at 100 kPa	101°C	ASTM E 537-20
Freezing point	-7°C	ISO 1392
Specific heat capacity, kJ/kg	3.13	ASTM E1269 - 11
pH (undiluted)	4 – 7	Annex A
Bulk Brookfield Viscosity (mPa.s)	70 - 8000	Annex D
Active content, % (w/w)	30 - 50	Annex B
Epichlorohydrin, mg/kg of active ingredient , max	20	Annex C
1,3-Dichloro-2-propanol, mg/kg of active ingredient, max	1000	
2,3-Dichloro-1-propanol, mg/kg of active ingredient, max	500	

NOTE 1 Density of the solution depends on the concentration.

NOTE 2 Viscosity is dependent on molecular mass and active content

5 Sampling

5.1 General

Sampling shall be carried out in accordance with ISO 3165 and ISO 6206. 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.

5.2 Sampling from drums and bottles

5.2.1 General

5.2.1.1 Mix the contents of the container to be sampled by shaking the container or by rolling it or rocking it from side to side, taking care not to damage the container or spill any of the liquid.

5.2.1.2 If the design of the container is such (for example 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 5.2.1.3.

5.2.1.3 Examine the surface of the liquid. If there no signs of surface contamination, take samples from the surface as described 5.2.2; otherwise take samples as described in 5.2.3.

5.2.2 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.

5.2.3 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 to the top and the 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.

5.3 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 5.2.2;
- b) from the bottom of the tank or tanker, using a sampling tube as described in 5.2.3 or using specially designed bottom-sampling apparatus; and
- c) from one or more positions, depending on the overall depth, between the bottom and the surface using a weighted sampling can.

6 Packaging and transportation

The product shall be delivered in suitable containers, for example in bulk containers, drums, cans or bottles.

In order that the purity of the product is not affected, the means of delivery for bulk containers shall not have been used previously for any different product or it shall have been specially cleaned and prepared before use.

7 Labelling

7.1 Risk and safety labelling

Depending on the 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 the general labelling to be applied to packages see 7.2.

7.2 General labelling

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

- a) name of product "POLYAMINE";
- b) trade name and grade;
- c) net mass;
- d) name and address of supplier and/or manufacturer;
- e) country of origin;
- f) storage instructions such as "KEEP AWAY FROM STRONG ACIDS, STRONG OXIDIZING AGENTS AND DIRECT SUNLIGHT";
- g) date of manufacturing or packaging;
- h) shelf life; and
- i) appropriate R- and S-phrases.

Annex A (normative)

Determination of pH

A.1 General

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

A.2 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 pH meter using at least a buffer solution of pH 4.0 and 7.0

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Annex B (normative)

Determination of active content

B.1 General

The proportion of active ingredient, that is, 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 formula:

$$C_0 = C_1 - C_2$$

where

C_0 is the per cent by mass [% (m/m)] of polyamine;

C_1 is the per cent by mass [% (m/m)] of dry solids; and

C_2 is the per cent by mass [% (m/m)] of inorganic metal salt.

The results shall be expressed to two decimal places.

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

B.2 Determination of dry solids content

B.2.1 Principle

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

B.2.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

B.2.2.1 Balance, with an accuracy of 0.1 mg.

B.2.2.2 Oven, capable of maintaining $(110 \pm 1)^\circ\text{C}$ vented to fume cupboard.

B.2.2.3 Desiccator, containing dried silica gel.

B.2.2.4 Porcelain crucible, 57 mm diameter

B.2.3 Procedure

B.2.3.1 Place a porcelain crucible in an oven at 110°C for at least 10 min.

B.2.3.2 Remove the crucible from the oven, place in a desiccator and allow to cool for at least 10 min.

- B.2.3.3** Weigh the crucible to the nearest 0.1 mg.
- B.2.3.4** Shake the sample in its container to ensure that it is homogeneous.
- B.2.3.5** Add 1g to 2g test portion of the product sample to the crucible and weigh to the nearest 0.1 mg.
- B.2.3.6** Place crucible in oven at 110°C for 2 hrs.
- B.2.3.7** After this time, transfer crucible directly from the oven to desiccator and allow to cool for at least 10 min
- B.2.3.8** Weigh the crucible containing the dry residue to the nearest 0.1 mg.

B.2.4 Expression of results

B.2.4.1 Method of calculation

The dry solids content, C_1 , expressed as a percentage by mass of the product, is given by the 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 results shall be expressed to two decimal places.

B.2.5 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 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.

B.3 Determination of salt content.

B.3.1 Principle

A known mass of wet sample is dried in accordance with B.2.3 the salt content is then determined gravimetrically following ignition of the dry residue in a furnace at 600°C.

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

B.3.2 Apparatus.

Ordinary laboratory apparatus and glassware together with the following.

B.3.2.1 Balance, with an accuracy of 0.1 mg.

B.3.2.2 Muffle furnace, capable of maintaining (600 ± 20)°C vented to fume cupboard.

B.3.2.3 Desiccator, containing dried silica gel.

B.3.2.4 Oven, capable of maintaining (110 ± 1)°C.

B.3.3 Procedure

B.3.3.1 Place the crucible, containing the dry residue in the muffle furnace at 600°C for at least 30 min.

B.3.3.2 Remove the crucible and place in the oven at 110°C for at least 30 min, then place in a desiccator until cool and reweigh to the nearest 0.1 mg.

B.3.4 Expression of results

B.3.4.1 Method of calculation

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

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

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 ash sample.

The results shall be expressed to two decimal places.

B.3.4 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 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.

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Annex C (normative)

Determination of impurities (epichlorohydrin, 1,3-dichloro-2-propanol and 2,3-dichloro-1-propanol) content

C.1 Principle

Epichlorohydrin, 1,3-dichloro-2-propanol and 2,3-dichloro-1-propanol are extracted from the polymer with tertbutylmethylether then separated and quantified using gas chromatography (GC).

WARNING: Epichlorohydrin 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 by inhalation, skin absorption and ingestion. Avoid exposure and keep closed containers in a well-ventilated place.

Tert-butylmethylether 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.

C.2 Reagents

C.2.1 Epichlorohydrin (ECH)

C.2.2 1,3-dichloro-2-propanol, (1,3-DCP)

C.2.3 2,3-dichloro-1-propanol, (2,3-DCP)

C.2.4 Tert-butylmethylether, (TBME)

NOTE In respect of C.2.2.2, C.2.2.3 and C.2.2.4 a certificate of analysis giving the assay of the reagent can be obtained from the supplier.

C.2.5 ECH standard stock solution, weigh (0.1000 ± 0.0002) g of ECH into a small glass sample bottle, and make up to (100.0 ± 0.5) g 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:

$$C(\text{ECH}) = 10 \times P$$

where

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

C.2.6 Calibration solution A, weigh $(0.1\ 000 \pm 0.0002)$ g of 1,3-DCP, $(0.1\ 000 \pm 0.0002)$ g of 2,3-DCP and (5.0000 ± 0.0002) g of ECH standard stock solution into a small glass bottle, and make up to (50.0 ± 0.2) g with TBME. Cap the bottle and shake it vigorously to mix the contents.

C.2.7 Calibration solution B, weigh (0.2000 ± 0.0002) g of 1,3-DCP, (0.2000 ± 0.0002) g of 2,3-DCP and (0.5000 ± 0.0002) g of ECH standard stock solution into a small glass bottle, and make up to (50.0 ± 0.2) g with TBME. Cap the bottle and shake it vigorously to mix the contents

C.2.8 Concentration in calibration solutions, of ECH (C_3), 1,3-DCP and 2,3-DCP (C_4) in milligrams per kilogram, in calibration solutions A and B are calculated as follows:

$$C_3 = (m \times C_5)/50$$

where

m is the mass, in grams (g), of ECH standard stock solution taken;

C_5 is the concentration, in milligrams per kilogram (mg/kg), of ECH in the Standard stock solution.

$$C_4 = (m \times P)/0.005$$

where:

m is the mass, in grams (g), of ECH standard stock solution taken;

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

C.3 Apparatus

C.3.1 Ordinary laboratory apparatus and glassware

C.3.2 Gas chromatograph, with flame ionization detector (FID)

C.3.3 Integrator

C.3.4 Balance, with an accuracy of 0.1 mg

C.3.5 Column, ultra 2 % to 5 % phenylmethyl silicone, 25 mm x 0.2 mm; film thickness, 0.33 μm , or equivalent

C.3.6 Syringe, GC, 10 μL

C.3.7 Glass bottle, circa 120 mL capacity, with polytetrafluorethylene (PTFE) lined screw caps

C.3.8 Pipettes, glass, with suitable filling device

C.3.9 Glass vials, 2 mL, with septum crimped or screw cap

C.4 Procedure

C.4.1 Test portion and sample extraction

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

Weigh test portion of (25.0 ± 0.1) g polymer sample into a clean glass bottle and add (20.0 ± 0.1) g of TBME. Cap the bottle and, by means of a laboratory shaker, agitate the contents vigorously for 5 min.

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

Draw off some of the TBME layer with a pipette and transfer to a 2 mL glass vial for storage in a refrigerator until required for analysis

C.4.2 Analytical procedure

Instruments settings: sample extraction solutions, calibration and blank solutions are analysed by GC apparatus using the following setting and conditions:

Oven/column temperature: programme: initial 40 $^{\circ}\text{C}$, hold for 1 min, ramp at 20 $^{\circ}\text{C}/\text{min}$ to 140 $^{\circ}\text{C}$, hold for 4 min;

Injector temperature: 250 $^{\circ}\text{C}$;

Detector temperature: 300 $^{\circ}\text{C}$;

Injection volume: 2 μL ;

Carrier gas: hydrogen;

Split vent flow: 50 mL/min;

Column head pressure: 35 kPa.

C.4.3 Retention times

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

C.4.4 Sample analysis

Inject 2 µl of sample extraction solution into the gas chromatograph and with the aid of an electronic integrator, measure the areas of the peak of each of three impurities.

C.4.5 Calibration

For calibration solutions A and B in turn, inject 2 µl of solution into the gas chromatograph and measure the areas of the resulting peaks.

C.4.6 Blank determination

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

C.5 Expression of results

C.5.1 Method of calculation

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

$$C_6 = \frac{A_i}{A_s} \times C_7 \times \frac{m_t}{m_p} \times \frac{100}{C_0}$$

where

A_i 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 solution (C_3 and C_4);

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

m_p is the mass, in gram (g), of test portion;

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

Thus:

$$C_6 = \frac{A_i}{A_s} \times C_7 \times \frac{80}{C_0}$$

NOTE The standard solution referred to in the above equation is that which has an impurity peak area closest to that of the extraction solution.

C.5.2 Precision

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

Epichlorohydrin: $r = 0.21z$

1,3- and 2,3-DCP: $r = 0.13z$

where

z is the mean of the two results, expressed in per cent by mass [% (m/m)].

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

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Annex D (normative)

Determination of Bulk Brookfield Viscosity.

D.1 Apparatus

D.1.1 Viscometer, Brookfield, Model LVT or equivalent

D.1.2 Beaker, 180 ml tall form Griffin or other container of equal diameter and depth.

D.2 Procedure

D.2.1 Set up the viscometer without the guard attached. Level the instrument.

D.2.2 Attach the spindle specified by the polyamine supplier. (Note: Left-handed threads.). If no specific spindle is specified by the manufacturer, start with spindle No.1 and proceed through step 9, and then repeat steps 2 through 9, if necessary, with increasing spindle numbers, until a spindle is found that gives a reading in step 9 that is in the middle of the viscometer's scale range.

D.2.3 Pour enough sample into the container to cover the spindle up to the groove.

D.2.4 Adjust the temperature of the sample to $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

D.2.5 Lower the viscometer with the spindle attached in the sample until the surface of the sample is at the groove on the spindle shaft. The spindle should not contact the bottom of the container.

D.2.6 Set the viscometer to the chosen rpm.

D.2.7 Turn on the viscometer motor.

D.2.8 After the needle reaches a steady reading and after at least 10 revolutions, depress the clutch lever on the back of the viscometer to 'freeze' the needle on the scale. With the clutch depressed, stop the viscometer motor when the needle is visible in the viscometer window.

D.2.9 Read and record the position of the needle on the scale.

D.2.10 Repeat steps D.2.7 to D.2.9 to obtain three readings.

D.3 Calculations

D.3.1 Average the three readings

D.3.2 Obtain the Brookfield viscosity by multiply the average reading calculated in step 1 by the factor for that spindle and speed supplied by the viscometer manufacturer.

Bibliography

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- [2] US 926: 2012, *Chemicals used for treatment of water intended for human consumption — Polyamides — Specification*

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Certification marking

Products that conform to Uganda standards may be marked with Uganda National Bureau of Standards (UNBS) Certification Mark shown in the figure below.

The use of the UNBS Certification Mark is governed by the Standards Act, and the Regulations made thereunder. This mark can be used only by those licensed under the certification mark scheme operated by the Uganda National Bureau of Standards and in conjunction with the relevant Uganda Standard. The presence of this mark on a product or in relation to a product is an assurance that the goods comply with the requirements of that standard under a system of supervision, control and testing in accordance with the certification mark scheme of the Uganda National Bureau of Standards. UNBS marked products are continually checked by UNBS for conformity to that standard.

Further particulars of the terms and conditions of licensing may be obtained from the Director, Uganda National Bureau of Standards.



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