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Anionic and non-ionic polyacrylamides for treatment of water intended for human consumption — Specification

Draft for public comments only

TANZANIA BUREAU OF STANDARDS

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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 1410:2011*), which has been technically revised.

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

> BS EN 1407:2023, Chemicals used for treatment of water intended for human consumption – Anionic and non-ionic polyacrylamides, published by European Union

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Anionic and non-ionic polyacrylamides for treatment of water intended for human consumption — Specification

1 Scope

This draft Tanzania Standard specifies the requirements, sampling and test methods for anionic and non-ionic polyacrylamides 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, 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)

Copolymer of acrylamide and acrylic acid salt, or homopolymer of acrylamide.

4.1.2 Synonym(s) or common name(s)

Anionic polyacrylamide or non-ionic polyacrylamide.

NOTE The more general terms: “anionic (or non-ionic) polymer”, “anionic polyelectrolyte” and “anionic (or non-ionic) flocculant” are used but can also cover other chemicals referred to in other standards.

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4.1.3 Relative molecular mass

Typically, in the range of 1 million to 20 million Daltons.

4.1.4 Empirical formulae

Non-ionic polyacrylamide:

— - (C_3H_5NO) x -

Anionic polyacrylamide:

— - (C_3H_5NO) x - $(C_3H_3O_2A)$ y -

where

A is a positive ion;

x and y are variables depending on the product;

4.1.5 Chemical formulae

The following formula illustrates typical structures of:

— non-ionic polyacrylamide (Figure 1):

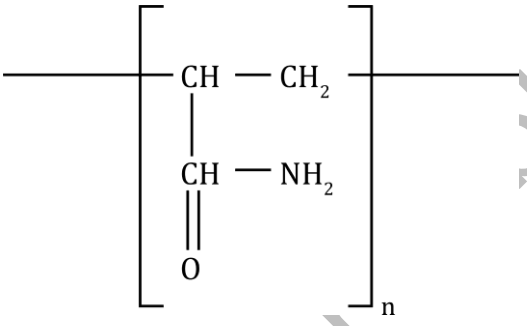


Figure 1 — Non-ionic polyacrylamide

— anionic polyacrylamide (Figure 2):

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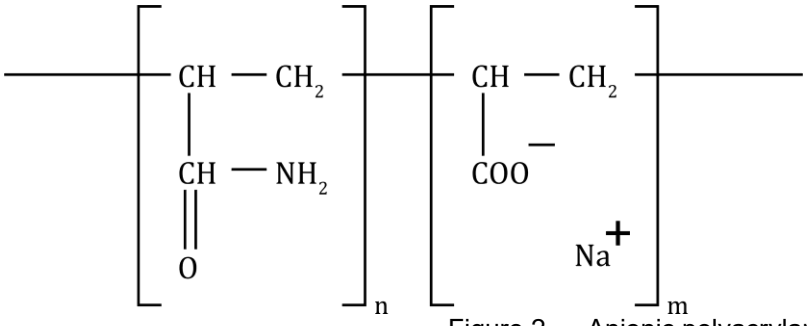


Figure 2 — Anionic polyacrylamide

Where

A is a positive ion;

x and y are variables depending on the product;

4.1.6 *CAS Registry Numbers*

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

— 25987-30-8

— 55554-55-7

— 25085-02-3

— 26100-47-0

— 9003-05-8

— 9003-04-7

— 9003-03-6

Note CAS Registry Number is Chemical Abstracts Services Registry Number.

4.1.7 *EINECS reference*

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.

Note EINECS is European Inventory of Existing Commercial Chemical Substances.

4.2 Commercial form

Anionic or non-ionic polyacrylamides as specified in this standard are available as solids containing a small amount of residual moisture.

5. Physical properties

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5.1 Appearance

The product is a white or off-white solid in the form of granule, flake or powder.

5.2 Bulk density

The bulk density of the product is typically in the range 0.5 g/cm^3 to 0.9 g/cm^3.

5.3 Solubility

The product is soluble in cold water. Its solubility is limited only by viscosity, with a gel being formed at concentrations of approximately 20 g/l and above.

5.4 Vapour pressure

Not applicable.

5.5 Boiling point at 100 kPa^3)

Not applicable.

5.6 Melting point

The product will decompose typically at temperature above 200 °C.

5.7 Specific heat

Not applicable.

5.8 Viscosity dynamic

Not applicable.

5.9 Critical temperature

Not applicable.

5.10 Critical pressure

Not applicable.

5.11 Physical hardness

Not applicable.

6. Chemical properties

6.1 General

Anionic and non-ionic polyacrylamides 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 in the dry state with a 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.

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6.2 Purity criteria

6.2.1 General

This document specifies the minimum purity requirements for anionic and non-ionic polyacrylamides 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.

6.2.2 Impurities and main by-products

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 anionic or non-ionic polyacrylamide shall be free of any visible extraneous matter.

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 200 mg of acrylamide monomer 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 polyacrylamides 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

Sampling shall be in accordance with ISO 8213 and the recommendations given in ISO 3165 and ISO 6206 shall be followed.

Note: A representative sample of the solid product, of sufficient mass, shall be obtained immediately after manufacture or from a newly opened package(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.

8 Test methods

Methods of test shall be carried out as prescribed in the annexes

9. Packaging – Marking and storage

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9.1 Packaging

The product shall be packed in suitable containers to protect the polymer from moisture, contamination, heat and physical damage, e.g. bulk containers, sacks, drums, cans, or bottles. Drums and sacks shall have a moisture barrier, e.g. an internal polyethylene liner.

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 Marking

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

— Name of the product, "Anionic polyacrylamide" or "Non-ionic polyacrylamide";

— trade name;

— net mass;

— name and address of supplier and/or manufacturer;

— Instruction for use and

— The word “Store in cool and dry place”

9.2.1 Transportation and labelling

Anionic and non-ionic polyacrylamides are not classified as hazardous for transport and do not therefore have UN-certified, hazard class, packaging group or require UN-certified containers

9.3 Storage

9.3.1 Long term stability

Due to its slight hygroscopic nature, product is best stored in a cool dry place, with low humidity and away from high temperatures. From the moment of receipt by the customer, the product has to be stable at least for 12 months. Follow supplier's advice.

9.3.2 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 methods

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

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

A.2.3 Impurity: Residual acrylamide monomer content

A.2.3.1 Principle

Acrylamide monomer is extracted from the anionic or non-ionic polyacrylamide sample into a mixture of water and methanol which softens the polymer but does not dissolve it. The extract is analysed by high- performance liquid chromatography (HPLC) using ultraviolet detection. Identification is made by comparison with an external standard and concentration determined by peak area measurements and ratio.

A.2.3.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

A.2.3.2.1 For extraction

A.2.3.2.1.1 Glass bottles (approximately 125 ml capacity) with polytetrafluoroethylene (PTFE) lined screw caps.

A.2.3.2.1.2 Balance, with an accuracy of 0,1 mg.

A.2.3.2.1.3 Laboratory shaker or tumbler. 7.2.3.2.1.4Measuring cylinders.

A.2.3.2.1.5 Syringes, 2 ml capacity.

A.2.3.2.1.6 Disposable syringe filters, 25 mm diameter, fitted with polyvinylidene fluoride (PVDF) membrane, pore size 0,45 µm.

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A.2.3.2.1.7 Laboratory grinder.

A.2.3.2.1.8 Woven-wire sieve, aperture size 1 000 µm.

A.2.3.2.2 For analysis

A.2.3.2.2.1 High-performance liquid chromatograph fitted with a constant flow solvent delivery system, a remote sample injection valve, a 5 µl loop, a column oven, a variable wavelength ultraviolet spectrometric detector, a chart recorder or electronic integrator. Use a column of 150 mm and 4,6 mm internal diameter with packing (FISONS PLRP-S 100A 4)), 5 µm column or equivalent).

A.2.3.2.2.2 Balance, with an accuracy of 0,1 mg.

A.2.3.2.2.3 Volumetric flasks.

A.2.3.2.2.4 Pipettes, with an accuracy of 0,03 ml, with suitable filling device.

A.2.3.2.2.5 Microlitre syringe.

A.2.3.3 Reagents

A.2.3.3.1 Helium gas, high purity, at a line pressure of 0,0069 kPa.

A.2.3.3.2 Acrylamide monomer (electrophoresis grade).

A.2.3.3.3 Methanol.

A.2.3.3.4 Phosphoric acid, 88 % (m/m), density ρ = 1,75 g/ml.

A.2.3.3.5 Sodium dihydrogen phosphate dihydrate.

A.2.3.3.6 Eluent.

Weigh, to the nearest 0,001 g, 6,240 g of sodium dihydrogen phosphate dihydrate (A.2.3.3.5) and dissolve with about 500 ml of water. Transfer quantitatively to a 2 000 ml volumetric flask (A.2.3.2.2.3) and make up to the mark with water and mix.

Adjust the pH to 3,0 with phosphoric acid (A.2.3.3.4). Pour a little of the eluent into a small beaker to check the pH then discard. This will avoid contamination of the bulk.

Degas by use of an ultrasonic bath or by-passing helium (A.2.3.3.1) through the solution.

A.2.3.3.7 Methanol/water solvent.

Measure 800 ml of methanol (A.2.3.3.3) into a 1 l measuring cylinder (A.2.3.2.1.4). Then measure 200 ml of water into a 250 ml measuring cylinder (A.2.3.2.1.4) and pour into the first measuring cylinder containing the methanol. Thoroughly mix, without violent agitation (a reduction in volume will occur; this is normal).

Degas by use of an ultrasonic bath or by-passing helium through the mixture and store in an amber glass reagent bottle.

A.2.3.3.8 Acrylamide monomer, standard stock solution (1 000 mg/l).

Weigh, to the nearest 0.001 g, 0,5 g of acrylamide monomer (A.2.3.3.2) and dissolve with about 200 ml of methanol/water solvent (A.2.3.3.7).

Transfer to a 500 ml volumetric flask (A.2.3.2.2.3) and make up to the mark with methanol/water solvent (A.2.3.3.7). Store in a tightly stoppered glass reagent bottle in a refrigerator. The solution is stable for at least four weeks.

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A.2.3.3.9 Acrylamide monomer, standard solution (20 mg/l).

Introduce with a pipette (A.2.3.2.2.4) 5 ml of the 1 000 mg/l stock solution into a 250 ml volumetric flask (A.2.3.2.2.3) and make up to the mark with methanol/water solvent (A.2.3.3.7). This solution shall be prepared weekly and stored in a refrigerator.

A.2.3.4 Procedure

WARNING — Acrylamide monomer is toxic if swallowed, harmful if inhaled or in contact with skin. It causes skin and serious eye irritations. It may cause an allergic skin reaction. It is cancerogenic, mutagenic and toxic for reproduction and it causes damage to peripheral nervous system through prolonged or repeated exposure. It shall be handled with care; avoid inhalation, skin contact and ingestion.

A.2.3.4.1 Preparation of test sample

Samples containing more than 10 % (m/m) of particles with a particle size greater than 1000 µm shall be reduced, using a laboratory grinder (A.2.3.2.1.7), until all the material passes a 1 000 µm aperture test sieve (A.2.3.2.1.8). The resultant material is the test sample A.

A.2.3.4.2 Extraction of test portion

Weigh, to the nearest 0.001 g, 4 g of the test sample (A) into a 125 ml glass bottle (A.2.3.2.1.1) and pipette 40 ml of methanol/water solvent.

Screw on the vial cap tightly and agitate on a mechanical shaker or tumbler (A.2.3.2.1.3) for a minimum of 16 h.

If necessary, the liquid extract (test solution B) can be drawn off, by means of a syringe (A.2.3.2.1.5) or pipette, and stored in a vial in a refrigerator for a maximum of one week before analysis is carried out.

A.2.3.4.3 Determination

A.2.3.4.3.1 Chromatographic conditions

Analyse the extraction solution, blank solution and calibration solution by HPLC using the apparatus (7.2.3.2.2.1) with the following conditions:

— mobile phase/eluent: 0,02 mol/l sodium dihydrogen phosphate at pH 3 (A.2.3.3);

— flow rate: 1.5 ml/min;

— column temperature: 40 °C;

— wavelength: at maximum absorbance in the region of 220 nm.

A.2.3.4.3.2 Analysis of extraction solution

Using a second syringe (A.2.3.2.2.5) draw off an aliquote of the liquid extract (test solution B) as follows. Fit syringe filter (A.2.3.2.1.6) and needle, expel all air and insert into the injection port of the 5 µl sample loop. Flush the loop with approximately half the syringe contents before injecting onto the column.

Measure the area of the peak which has a retention time corresponding to that of the acrylamide standard (the procedure assumes that the detector response is a linear function of concentration, but linearity shall first be established by the operator by means of a calibration graph).

A.2.3.4.3.3 Blank determination

Repeat the operations in A.2.3.4.3.2 omitting the filtered liquid extract and substituting with the methanol/water solvent. Inject two blank solutions with each set of samples.

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A.2.3.4.3.4 Calibration

Repeat the operations in A.2.3.4.3.2 omitting the filtered liquid extract and substituting with the 20 mg/l standard solution. Run a calibration after every four or five samples to check the performance of the column.

If the peak areas of consecutive solutions vary by more than 10 % (relative) then remedial action shall be taken, following usual HPLC procedures.

A.2.3.5 Expression of results

A.2.3.5.1 Method of calculation

The residual acrylamide monomer content (CA) expressed in milligrams per kilogram of the laboratory sample is calculated as follows:

$$C_A = \frac{(A_T - A_B) \times c \times V}{(A_S - A_B) \times m}$$

where

A_T is the peak area of the test solution B;

A_B is the peak area of the blank solution;

A_S is the peak area of the standard solution (A.2.3.3.9);

c is the concentration, in milligrams per litre, of the standard solution;

m is the mass, in grams, of the test sample A (A.2.3.4.1);

V is the volume, in millilitres, of the solvent; here V = 40 ml.

Express the result to the nearest whole number.

A.2.3.5.2 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 formula:

$$r = 0.20\ z$$

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 anionic and non-ionic polyacrylamides

B.1 Origin

B.1.1 Raw materials

Anionic polyacrylamide is manufactured from acrylamide monomer and an anionic monomer, of which the commonest examples are sodium acrylate and ammonium acrylate.

Non-ionic polyacrylamides are manufactured from acrylamide monomer.

B.1.2 Manufacturing process

Anionic or non-ionic polyacrylamide is produced by free radical initiated aqueous bulk copolymerization. The gel so formed is then dried and ground to the required particle size.

Anionic polyacrylamides are produced by the copolymerization of acrylamide monomer and an anionic acrylic monomer. They vary with regard to their ratio of non-ionic (acrylamide) monomer units to anionic monomer units and the degree of polymerization (relative molecular mass).

Non-ionic polyacrylamides are produced by the polymerization of acrylamide monomer alone. They usually have slight anionic charge due to a tendency to hydrolyse during manufacture, and they vary in the level of this weak anionic charge and the degree of polymerization.

Due to the similar chemical characteristics of anionic and non-ionic polyacrylamides both substances are included in this document.

B.2 Use

B.2.1 Function

Anionic and non-ionic polyacrylamides are used in drinking water treatment to facilitate the removal of colloidal and fine suspended particles. They are effective when used in conjunction with metal salts in the removal of turbidity and colour. Anionic and non-ionic polyacrylamides are thus utilized in the mainstream processes of clarification and filtration, where they usually supplement the coagulating/flocculating action of metal salts, or in the side stream processes of (a) settlement and thickening of wash waters/clarifier sludges and (b) dewatering of sludges, where they are usually applied as sole flocculants.

The choice of product, in terms of anionic charge density and molecular mass, depends on such factors as the properties of the raw water, its pre-treatment and the treatment process used.

Product selection is based on a precept of achieving the required performance at minimum applied dosage.

B.2.2 Form in which it is used

Usually, the product is introduced into the treatment system as a dilute (0.01 % to 0.05 % mass fraction of product as supplied) aqueous solution in order to effect rapid and even dispersion. Initially a more concentrated aqueous solution (0.1 % to 0.5 % mass fraction of product as supplied) is prepared, this requiring special batch or automatic make up equipment. The solution is then diluted batch wise or in−line to the required concentration. The product supplier will advise on solution preparation procedures and equipment.

Care should be taken of the maturing time of the solution to be indicated by the producer.

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Care should be taken that polyacrylamide solutions are stable only for a limited period and should be used immediately after preparation.

B.2.3 Treatment dose

The treatment dose will vary depending on the quality of raw water to be treated, treatment process and treatment objectives. The dose should be selected so as not to exceed the parametric values for chemical parameters as listed in EU Directive 2020/2184 (see [1]) or local regulations. Typical treatment doses are 0,2 mg/l to 0,5 mg/l, in individual cases even more.

When estimating the total concentration of free acrylamide monomer in the water leaving the treatment plant, the quantity of polymer present in any recirculated process streams should be taken into account as well as the dose added to the mainstream process.

It is normal practice to allow a delay time between addition of metal salt coagulant and anionic or non- ionic polyacrylamide in order to minimize dosage requirement of the latter.

B.2.4 Means of application

Product is usually applied using a metering pump. Sufficient mixing action at the point of addition should be provided to ensure rapid and even dispersion of the anionic and non-ionic polyacrylamides solution in the water being treated.

B.2.5 Secondary effects

Excessive polymer addition could lead to undesirable effects on plant, e.g. clogging of filters. The optimum dose should be determined for each application.

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.

Good chemical handling practice should be followed at all times. Creation of dust should be avoided and the working environment should be kept clean and dry.

Polyacrylamides 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 when wet:

- in case of spillage, it should be swept up dry. The addition of water will render the floor very slippery and dangerous;
- eye and hand protections are not normally warranted unless exposure is prolonged and in dusty conditions. 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 providing ventilation is adequate and dust is controlled.

B.4 Emergency procedures

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B.4.1 First aid

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

If anionic or non-ionic polyacrylamide is in contact with the skin, the contaminated area should be washed with copious amounts of soap and water.

If anionic or non-ionic polyacrylamide 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 anionic or non-ionic polyacrylamide 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 spillage is dry, it should be shovelled, vacuumed or swept up.

If a large spillage becomes wet, 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 water courses.

B.4.3 Fire

Low fire and explosion risk. Products 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.

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