BRITISH STANDARD

BS EN 1410:2008

Chemicals used for treatment of water intended for human consumption — Cationic polyacrylamides

ICS 13.060.20; 71.100.80

BSI British Standards

National foreword

This British Standard is the UK implementation of EN 1410:2008. It supersedes BS EN 1410:1998 which is withdrawn.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.

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Chemicals used for treatment of water intended for human consumption - Cationic polyacrylamides

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

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Kationische Polyacrylamide

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

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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 1410:2008) has been prepared by Technical Committee CEN/TC 164 “Water supply”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2008, and conflicting national standards shall be withdrawn at the latest by July 2008.

This document supersedes EN 1410:1998.

Significant technical differences between this edition and EN 1410:1998 are as follows:

(a) reduction in the limit value for acrylamide from 250 mg/kg to 200 mg/kg in 4.4;

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

(c) provision of more information on treatment dose in Annex A.

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 the Member States of the EU or EFTA;

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

NOTE Conformity with this European 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 cationic polyacrylamides used for treatment water intended for human consumption. It describes the characteristics of cationic polyacrylamides and specifies the requirements and the corresponding test methods for cationic polyacrylamides. It gives information on their 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*

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

3 Description

3.1 Identification

3.1.1 Chemical names

Copolymer of acrylamide and amine ester or amide.

3.1.2 Synonym or common name

Cationic polyacrylamide.

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

3.1.3 Relative molecular mass

Typically in the range of 1 to 20 million.

3.1.4 Empirical formula

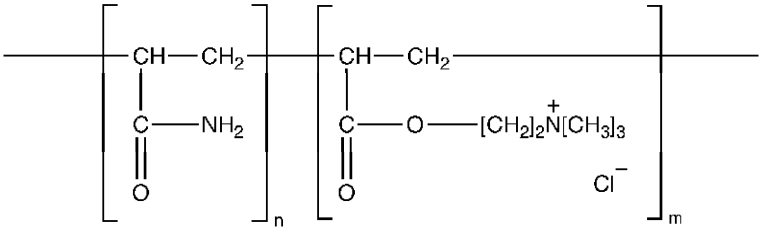
$$-(C_3H_5NO)_x-(C_aH_bN_cO_dA)_y-$$

where

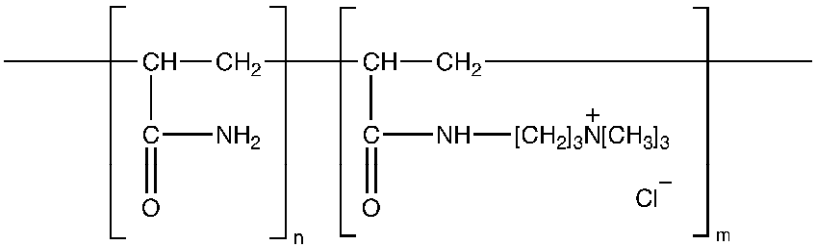
A	is a negative ion;
a, b, c and *d*	are variable depending on the cationic monomer;
x and *y*	are variable depending on the product.

3.1.5 Chemical formulae

Copolymer of acrylamide and amine ester



Copolymer of acrylamide and amine amide



where

n and *m*	are variables depending on the product.

3.1.6 CAS Registry Numbers [1)]

The following is a list of CAS Registry Numbers for typical cationic polyacrylamide polymers.

— 69418-26-4

— 26006-22-4

— 35429-19-7

— 25568-39-2

— 60162-07-4

[1)] Chemical Abstracts Service Registry Number.

— 51410-72-1

— 52285-95-7

— 68227-15-6

— 55216-72-3

— 26796-75-8

— 45021-77-0

3.1.7 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.

3.1.8 Commercial form

Cationic polyacrylamide as specified in this standard is available as a solid containing a small amount of residual moisture.

3.2 Physical properties

3.2.1 Appearance

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

3.2.2 Density

The bulk density of the product is typically in the range 0,5 g/cm^3 to 0,8 g/cm^3.

3.2.3 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.

3.2.4 Vapour pressure

Not applicable.

3.2.5 Boiling point at 100 kPa [3)]

Not applicable.

3.2.6 Melting point

The product decomposes at approximately 200 °C.

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2) European Inventory of Existing Commercial Chemical Substances.

3) 100 kPa = 1 bar.

3.2.7 Specific heat

Not applicable.

3.2.8 Viscosity, dynamic

Not applicable.

3.2.9 Critical temperature

Not applicable.

3.2.10 Critical pressure

Not applicable.

3.2.11 Physical hardness

Not applicable.

3.3 Chemical properties

Cationic polyacrylamide is 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 in the dry state with a strong acid or 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 cationic 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.

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 cationic polyacrylamide shall be free of any visible extraneous matter.

NOTE Various parameters can be checked as part of assessment of product quality (see 5.2.2).

4.3 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.

4.4 Chemical parameters

The product shall contain no more than 200 mg of acrylamide monomer per kilogram of product.

NOTE Other chemical parameters and indicator parameters as listed in EU Directive 98/83/EC (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.

5 Test methods

5.1 Sampling

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

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.

5.2 Analyses

5.2.1 General

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

5.2.2 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 infra-red spectroscopic analysis, depending on the product and manufacturer/supplier.

5.2.3 Impurity: residual acrylamide monomer content

5.2.3.1 Principle

Acrylamide monomer is extracted from the cationic polyacrylamide sample into a mixture of water and acetone 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.

5.2.3.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

5.2.3.2.1 For extraction

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

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

5.2.3.2.1.3 Laboratory shaker or tumbler.

5.2.3.2.1.4 Measuring cylinders.

5.2.3.2.1.5 Syringes, 2 ml capacity.

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

5.2.3.2.1.7 Laboratory grinder.

5.2.3.2.1.8 Woven-wire sieve, aperture size 1 000 µm.

5.2.3.2.2 For analysis

5.2.3.2.2.1 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).

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

5.2.3.2.2.3 Microlitre syringe.

5.2.3.3 Reagents

5.2.3.3.1 Helium gas, high purity, at a line pressure of 0,006 9 kPa.

5.2.3.3.2 Acrylamide monomer (electrophoresis grade).

5.2.3.3.3 Acetone.

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

5.2.3.3.5 Sodium dihydrogen phosphate dihydrate.

[4)] FISONS PLRP S 100A is the trade name of a product supplied by FISONS. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown, to lead to the same results.

5.2.3.3.6 Eluent

Weigh, to the nearest 0,001 g, 6,240 g of sodium dihydrogen phosphate dihydrate (5.2.3.3.5) and dissolve with about 500 ml of water. Transfer quantitatively to a 2 000 ml volumetric flask and make up to the mark with water and mix.

Adjust the pH to 3,0 with phosphoric acid (5.2.3.3.4). 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 (5.2.3.3.1) through the solution.

5.2.3.3.7 Acetone/water solvent

Measure 800 ml of acetone (5.2.3.3.3) into a 1 l measuring cylinder (5.2.3.2.1.4). Then measure 200 ml of water into a 250 ml measuring cylinder and pour into the first measuring cylinder containing the acetone. Thoroughly mix, without violent agitation. Degas by use of an ultrasonic bath or by passing helium through the mixture and store in an amber glass reagent bottle.

5.2.3.3.8 Acrylamide monomer,standard stock solution (1 000 mg/l)

Weigh, to the nearest 0,001 g, 0,5 g of acrylamide monomer (5.2.3.3.2) and dissolve with about 200 ml of acetone/water solvent (5.2.3.3.7).

Transfer to a 500 ml volumetric flask and make up to the mark with solvent (5.2.3.3.7). Store in a tightly-stoppered glass reagent bottle in a refrigerator. The solution is stable for at least four weeks.

5.2.3.3.9 Acrylamide monomer, standard solution (100 mg/l)

Introduce with a pipette (5.2.3.2.2.2) 25 ml of the 1 000 mg/l stock solution into a 250 ml volumetric flask and make up to the mark with acetone/water solvent (5.2.3.3.7). This solution shall be prepared weekly and stored in a refrigerator.

5.2.3.4 Procedure

WARNING: Acrylamide monomer is toxic and shall be handled with care; avoid inhalation, skin contact and ingestion.

5.2.3.4.1 Preparation of test sample

Samples containing more than 10 % (*m/m*) of particles with a particle size greater than 1 000 μm shall be reduced, using a laboratory grinder (5.2.3.2.1.7), until all the material passes a 1 000 μm aperture test sieve (5.2.3.2.1.8). The resultant material is the test sample A.

5.2.3.4.2 Extraction of test portion

Weigh, to the nearest 0,001 g, 4 g of the test sample (A) into a 125 ml glass bottle (5.2.3.2.1.1) and pipette 40 ml of acetone/water solvent.

Screw on the vial cap tightly, and agitate on a mechanical shaker or tumbler (5.2.3.2.1.3) for a minimum of 16 h.

If necessary, the liquid extract (test solution B) can be drawn off, by means of a syringe (5.2.3.2.1.5) or pipette, and stored in a vial in a refrigerator for a maximum of one week before analysis is carried out.

5.2.3.4.3 Determination

5.2.3.4.3.1 Chromatographic conditions

Analyse the extraction solution, blank solution and calibration solution by HPLC using the apparatus (5.2.3.2.2.1) with the following conditions:

- mobile phase/eluent: 0,02 mol/l sodium dihydrogen phosphate at pH 3,0 (5.2.3.3.6);
- flow rate: 1,5 ml/min;
- column temperature: 40 °C;
- wavelength: at maximum absorbance in the region of 220 nm.

5.2.3.4.3.2 Analysis of extraction solution

Using a second syringe (5.2.3.2.1.5) draw off a portion of the liquid extract (test solution B). Fit syringe filter (5.2.3.2.1.6) 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 into 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).

5.2.3.4.3.3 Blank determination

Repeat the operations in 5.2.3.4.3.2 omitting the filtered liquid extract and substituting with the acetone/water solvent. Inject two blanks with each set of samples.

5.2.3.4.3.4 Calibration standard

Repeat the operations in 5.2.3.4.3.2 omitting the filtered liquid extract and substituting with the 100 mg/l standard solution (5.2.3.3.9). Run a calibration with standard solution after every four or five samples to check the performance of the column.

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

5.2.3.5 Expression of results

5.2.3.5.1 Method of calculation

The residual acrylamide monomer content (C_A) 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 (5.2.3.3.9);

c is the concentration in milligramms per litre of the standard solution;

m is the mass in grams of the test sample (A) (5.2.3.4.1);

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

Express the result to the nearest whole number.

5.2.3.5.2 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 equation:

$$r = 0{,}20\ z$$

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.

6 Labelling - transportation - storage

6.1 Means of delivery

The product shall be delivered in suitable containers, 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.

6.2 Risk and safety labelling in accordance with the EU Directives

Cationic polyacrylamides are not classified as a dangerous substance according to EU Directive 67/548/EEC (see [2]).

NOTE 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

Cationic polyacrylamides are not classified as hazardous for transport and do not therefore have a UN number [5)], hazard class, packaging group or require UN-certified containers.

6.4 Marking

The marking shall include the following:

— name "cationic polyacrylamide", trade name and grade;

— net mass;

— name and address of supplier and/or manufacturer;

— statement "This product conforms to EN 1410".

6.5 Storage

6.5.1 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. The product is usually stable for at least 12 months storage at ambient temperatures. 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).

[5)] United Nations Number.

Annex A
(informative)

General information on cationic polyacrylamides

A.1 Origin

A.1.1 Raw materials

Cationic polyacrylamides are manufactured from acrylamide monomer and a cationic monomer, of which the following are the commonest examples:

a) (2-acrylamidoethyl) N-methyl, N-diethylammonium methyl sulfate;

b) (2-acrylamidoethyl) N-methyl, N-diethylammonium chloride;

c) (2-acrylamidoethyl) trimethylammonium chloride;

d) (2-acrylamidoethyl) trimethylammonium methyl sulfate;

e) (2-methacrylamidoethyl) trimethylammonium chloride;

f) (2-methacrylamidoethyl) trimethylammonium methyl sulfate;

g) (3-methacrylamidopropyl) trimethylammonium chloride;

h) (3-acrylamidopropyl) trimethylammonium methyl sulfate.

A.1.2 Manufacturing process

Cationic polyacrylamides are produced by the copolymerization of acrylamide monomer and a cationic acrylic monomer. They vary in regard to their ratio of non-ionic (acrylamide) monomer units to cationic monomer groups and the degree of polymerization (relative molecular mass).

They are produced by free-radical-initiated aqueous bulk copolymerization. The gel so formed is then dried and ground to the required particle size.

A.2 Use

A.2.1 Function

Cationic 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. Cationic 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 cationic 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.

A.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) aqueous solution in order to effect rapid and even dispersion. Initially a more concentrated aqueous solution (0,1 % to 0,5 % mass fraction) 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.

A.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 98/83/EC (see [1]) or local regulations. In order to conform to the parametric value for acrylamide monomer the dose should not exceed 0,5 mg/l. Typically a level between 0,1 mg/l and 0,2 mg/l is used in the mainstream process.

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 cationic polyacrylamide in order to minimize dosage requirement of the latter.

A.2.4 Means of application

Cationic polyacrylamides are usually applied using a metering pump. Sufficient mixing action at the point of addition should be provided to ensure adequate dispersion of the product in the water being treated.

A.2.5 Secondary effects

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

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. 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 protection is 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.

A.4 Emergency procedures

A.4.1 First aid

If cationic polyacrylamide is in contact with the skin, the contamined area should be washed with copious amounts of soap and water.

If cationic 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 cationic 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.

A.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 watercourses.

A.4.3 Fire

Low fire and explosion risk. The 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.

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.

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