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ICS 71.100.80

EAST AFRICAN STANDARD

Chemicals used for treatment of water intended for human consumption — Chlorine

Draft for comments only — Not to be cited as East African Standard

EAST AFRICAN COMMUNITY

Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in East Africa. It is envisaged that through harmonized standardization, trade barriers which are encountered when goods and services are exchanged within the Community will be removed.

In order to meet the above objectives, the EAC Partner States have enacted an East African Standardization, Quality Assurance, Metrology and Test Act, 2006 (EAC SQMT Act, 2006) to make provisions for ensuring standardization, quality assurance, metrology and testing of products produced or originating in a third country and traded in the Community in order to facilitate industrial development and trade as well as helping to protect the health and safety of society and the environment in the Community.

East African Standards are formulated in accordance with the procedures established by the East African Standards Committee. The East African Standards Committee is established under the provisions of Article 4 of the EAC SQMT Act, 2006. The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the private sectors and consumer organizations. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the procedures of the Community.

Article 15(1) of the EAC SQMT Act, 2006 provides that "Within six months of the declaration of an East African Standard, the Partner States shall adopt, without deviation from the approved text of the standard, the East African Standard as a national standard and withdraw any existing national standard with similar scope and purpose".

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

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Introduction

In the preparation of this East African Standard, the following source was consulted extensively:

BS EN 937:2009, *Chemicals used for treatment of water intended for human consumption — Chlorine*

Assistance derived from this source and others inadvertently not mentioned is hereby acknowledged.

Draft for comments only — Not to be cited as East African Standard

BRITISH STANDARD

BS EN 937:2009

Chemicals used for treatment of water intended for human consumption — Chlorine

ICS 13.060.20; 71.100.80

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BSi
British Standards

National foreword

This British Standard is the UK implementation of EN 937:2009. It supersedes BS EN 937:1999 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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EUROPEAN STANDARD
 NORME EUROPÉENNE
 EUROPÄISCHE NORM

EN 937

June 2009

ICS 71.100.80

Supersedes EN 937:1999

English Version

**Chemicals used for treatment of water intended for human
 consumption - Chlorine**

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

Produkte zur Aufbereitung von Wasser für den
 menschlichen Gebrauch - Chlor

This European Standard was approved by CEN on 13 May 2009.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

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



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Foreword

This document (EN 937:2009) 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 December 2009, and conflicting national standards shall be withdrawn at the latest by December 2009.

This document supersedes EN 937:1999.

Differences between this edition and EN 937:1999 are mainly editorial to harmonize the text with other standards in this series and more precisely as follows:

- a) deletion of reference to EU Directive 80/778/EEC of July 15, 1980 in order to take into account the latest Directive in force (see [1]);
- b) deletion of Hg as Chemical Parameter in Table 1.

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 chlorine used for treatment of water intended for human consumption. It describes the characteristics of chlorine and specifies the requirements and the corresponding test methods for chlorine. It gives information on its use in water treatment.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

ISO 1552, *Liquid chlorine for industrial use — Method of sampling (for determining only the volumetric chlorine content)*

ISO 2120, *Liquid chlorine for industrial use — Determination of the content of chlorine by volume in the vaporized product*

ISO 2121, *Liquid chlorine for industrial use — Determination of water content — Gravimetric method*

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

3 Description

3.1 Identification

3.1.1 Chemical name

Chlorine.

3.1.2 Synonym or common name

Liquid chlorine.

3.1.3 Relative molecular mass

70,91.

3.1.4 Empirical formula

Cl₂.

3.1.5 Chemical formula

Cl₂.

3.1.6 CAS Registry Number ¹⁾

7782-50-5.

3.1.7 EINECS reference ²⁾

231-959-5.

3.2 Commercial form

Pressurized liquefied gas.

3.3 Physical properties

3.3.1 Appearance

Liquid chlorine is a clear, amber coloured liquid. Chlorine gas is greenish yellow, 2,5 times heavier than air. It has a suffocating and characteristic odour.

3.3.2 Density

Liquid: 1,409 g/ml at 20 °C.

Gas:

- 3,169 kg/m³ at 101.3 kPa at 0 °C;
- 2,945 kg/m³ at 101.3 kPa at 20 °C.

3.3.3 Solubility (in water)

7,26 g/l at 20 °C and 100 kPa.

3.3.4 Vapour pressure

669 kPa at 20 °C.

3.3.5 Boiling point at 100 kPa ³⁾

- 34 °C.

3.3.6 Liquefaction point

- 101 °C at 100 kPa.

¹⁾ Chemical Abstracts Service Registry Number.

²⁾ European Inventory of Existing Commercial Chemical Substances.

³⁾ 100 kPa = 1 bar.

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3.3.7 Specific heat

Liquid : 920 J/(kg.K) at - 34 °C.

Gas : 475 J/(kg.K) at 0 °C.

3.3.8 Viscosity (dynamic)

Gas : 1 333 x 10⁻⁸ Pa.s at 20 °C.

Liquid : 4,78 x 10⁻⁴ Pa.s at - 34 °C.

3.3.9 Critical temperature

144 °C.

3.3.10 Critical pressure

7 710,83 kPa.

3.3.11 Physical hardness

Not applicable.

3.4 Chemical properties

Chlorine is a very strong oxidizing agent and can react violently with some gases such as hydrogen. Almost all metals form chlorides in the presence of chlorine. Organic compounds including mineral oils and greases react very quickly with chlorine.

The standard redox potential of gaseous chlorine in neutral aqueous solution at 25 °C and 101.3 mbar is:



4 Purity criteria

4.1 General

This European Standard specifies the minimum purity requirements for chlorine 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, contents of other impurities and additives used in the product not stated in the product standard.

Limits have been given for impurities and chemicals 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 leads 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 product shall contain at least a volume fraction of 99,5 % chlorine.

4.3 Impurities and main by-products

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

Table 1 — Impurities

| Impurities | Limit in mg/kg of product |
|--|---------------------------|
| Water (H ₂ O) | 20 |
| Nitrogen trichloride (NCl ₃) | 20 ^a |
| ^a Valid for containers with a net mass of max. 1000 kg liquid chlorine. For tanks with a higher capacity the limit shall be lowered to 10 mg/kg due to safety concerns (see [4] for further details). | |

NOTE 1 Other by-products consist of inert gases (nitrogen, oxygen, carbon dioxide, hydrogen) in variable proportion which are not relevant for the purposes described in this European Standard.

NOTE 2 Carbon tetrachloride, which is used in some chlorine manufacturing plants as an auxiliary solvent in chlorine processing, and other chlorinated hydrocarbons originating from rubberised or plastic piping might be present in traces in chlorine, but are not relevant due to the low dosage of chlorine to water intended for human consumption. Chlorine can also contain traces of bromine, depending on the purity of the salt used in the electrolytic process and the subsequent chlorine processing.

4.4 Chemical parameters

Heavy metals, which might be present in traces in liquid chlorine, are no relevant chemical parameters for gaseous chlorine that is applied in water treatment. Cyanide, pesticides and polycyclic aromatic hydrocarbons are not by-products of the manufacturing process.

NOTE For the purpose of this standard, "chemical parameters" are those defined in the EU Directive 98/83/EC of November 3, 1998 ([1]). For their parametric values in drinking water see [1]."

5 Test methods

5.1 General

The composition of chlorine is usually controlled and monitored by the supplier and not intended to be performed by users. The methods given for sampling and analysis are intended for use in case of dispute, and shall be carried out by very competent personnel only.

NOTE Due to the potential safety risks when performing sampling and analysis, it is strongly recommended to take advice of the chlorine producers or specialised laboratories.

5.2 Sampling

Take a sample of liquid chlorine, taking account of ISO 6206, in accordance with the following techniques:

- ISO 1552 for the determination of chlorine content;
- ISO 2121 for the determination of water content.

5.3 Analysis

5.3.1 Determination of chlorine content

The chlorine content shall be determined in accordance with ISO 2120.

5.3.2 Determination of water content

The water content shall be determined in accordance with ISO 2121.

5.3.3 Determination of nitrogen trichloride content

The nitrogen trichloride content shall be determined in accordance with Annex C.

6 Labelling - Transportation - Storage

6.1 Means of delivery

Chlorine shall be delivered in transportable pressure equipments (cylinders, pressure drums, tanks, portable tanks, etc.), (see [8]).

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 ⁴⁾

At the time of publication of this European Standard the following labelling requirements shall apply to chlorine:

- symbols and indications of danger:
-

⁴⁾ See [2].

- T : Toxic
- N : Dangerous for the environment
- nature of special risks:
 - R 23 : Toxic by inhalation;
 - R 36/37/38 : Irritating to eyes, respiratory system and skin;
 - R 50 : Very toxic to aquatic organisms
- safety advice:
 - S 7/9: Keep container tightly closed and in a well-ventilated place;
 - S 45: In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible);
 - S 61: Avoid release to the environment - Refer to special instructions / Safety data sheet.

NOTE 1 Annex I of the Directive 67/548/EEC see [2] 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. The labelling information given above covers the 30th ATP (adaptation to technical progress) of the EU Directive [2]."

NOTE 2 Chlorine used for treatment of water intended for human consumption is applied as a biocidal product. Therefore additional labelling requirements according to Directive 98/8/EC (see [3]) shall be applied.

6.3 Transportation regulations and labelling

Chlorine is listed as UN Number ⁵⁾ 1017.

ADR ⁶⁾/RID ⁷⁾: class 2, classification code 2 TC, labels : 2.3 + 8+ environmentally hazardous substance mark, hazard n°.:268.

IMDG ⁸⁾: class 2.3, marine pollutant: yes, EmS: F-C, S-U.

IATA ⁹⁾: Not permitted.

6.4 Marking

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

- the name: "chlorine", the trade name, the grade and type;

⁵⁾ United Nations Number.

⁶⁾ European Agreement concerning the International Carriage of Dangerous Goods by Road.

⁷⁾ Regulations concerning International Carriage of Dangerous Goods by Rail.

⁸⁾ International Maritime transport Dangerous Goods Code.

⁹⁾ International Air Transport Association Dangerous Goods Regulations.

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- the net mass;
- the name and the address of supplier and/or manufacturer;
- the statement "This product conforms to EN 937".

6.5 Storage

6.5.1 Storage conditions

Keep containers with chlorine tightly closed and store in a cool, dry and well ventilated place. Tightly screw on the valve outlet protection seal and the valve protection cap when storing. Prevent cylinders from falling over. Protect from heat and direct sunlight, the temperature of the container not exceeding 50 °C.

6.5.2 Long term stability

Stable.

6.5.3 Storage incompatibilities

See 3.4.

Annex A (informative)

General information on chlorine

A.1 Origin

A.1.1 Raw materials

Alkali chlorides (sodium chloride or potassium chloride) or hydrogen chloride, water.

A.1.2 Manufacturing process

Electrolysis of alkali chloride solutions or hydrochloric acid.

A.2 Use

A.2.1 Function

Disinfectant, removal of ammonia compounds, oxidizing of sulfides, oxidizing of iron(II) to iron(III).

A.2.2 Form in which the product is used

It is used as delivered. Chlorine is released off the pressurised containers either as gas or as liquid that is vaporised externally by adequate vaporising equipment.

A.2.3 Treatment dose

The treatment dose depends on the composition of the raw water. Care should be taken not to exceed a maximum concentration of active chlorine at the water tap, usually a few tenths of 1 mg/l.

A.2.4 Means of application

Gaseous chlorine is fed into the water stream by adequate injection devices (usually vacuum injection devices).

A.2.5 Secondary effects

Excess of dosage can lead to slight pH lowering due to generation of hydrochloric acid.

Oxidation of organic compounds; the formation of halogenated organic substances, especially trihalomethanes, is possible.

A.2.6 Removal of excess product

The most practical method will be the use of sulfur dioxide or an aqueous solution of a sulfite compound. Other methods can utilise activated carbon or hydrogen peroxide.

Annex B (normative)

General rules relating to safety

B.1 Rules for safe handling and use

The supplier shall provide current safety instructions.

NOTE Further safety and technical information can be obtained from Euro Chlor ([5,6,7,8,9]).

B.2 Emergency procedures

B.2.1 General

The following information is only a very brief summary of actions to be taken in case of emergency.

Facilities using chlorine shall have an emergency plan with safety instructions for all kinds of incidents which potentially could give rise to emissions of hazardous chlorine.

B.2.2 First aid

Contact with skin: Wash immediately with plenty of water. Seek medical advice.

On inhalation of chlorine gas: Move affected person into fresh air, keep warm and allow to rest. Call a physician immediately. As soon as practicable treat initially with a cortisone spray metered dose inhaler. If there is difficulty in breathing, give oxygen. In case of respiratory arrest, apply ventilation with respiratory device or perform mouth to nose or mouth to mouth respiration.

B.2.3 Spillage

Put on breathing apparatus. Isolate contaminated area by means of water curtains. Call local fire brigade.

B.2.4 Fire

Chlorine is non-flammable but strongly oxidising and very corrosive and toxic, posing an extra hazard with fires. Containers or pipes filled with liquid chlorine can burst when exposed to a fire or heat, causing emissions of large amounts of very hazardous chlorine gas. If heated higher than ca. 120 °C chlorine filled containers and pipes made of mild steel might self-ignite and burst (chlorine iron fires).

Cool fire-endangered containers and pipes with water. Use appropriate means to extinguish surrounding fire.

Annex C (normative)

Determination of nitrogen trichloride (molecular absorption spectrometry)

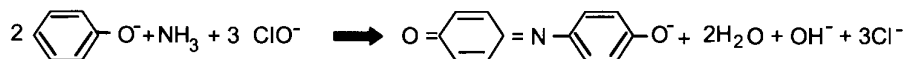
C.1 General

This method applies to products with a nitrogen trichlorine content within the range of 0,2 mg to 200 mg per kilogram of liquid chlorine.

C.2 Principle

Discharge of chlorine from the liquid phase into a sample vessel containing hydrochloric acid and cooled to - 60 °C. Evaporation of the chlorine in the presence of hydrochloric acid $c(\text{HCl}) = 8 \text{ mol/l}$ and scrubbing the chlorine gas with hydrochloric acid $c(\text{HCl}) = 12 \text{ mol/l}$ with which nitrogen trichloride reacts to form ammonium chloride. Removal of the dissolved chlorine, careful neutralization of the hydrochloric acid solution and determination of the ammonium ion by spectrometry of the indophenol complex.

C.3 Reaction



C.4 Reagents

C.4.1 All reagents shall be of a recognized analytical grade and the water used shall conform to grade 3 purity in accordance with EN ISO 3696 and be free of ammonia.

C.4.2 Sodium hydroxide solution, $c(\text{NaOH}) = 6 \text{ mol/l}$.

C.4.3 Hydrochloric acid, $c(\text{HCl}) = 12 \text{ mol/l}$.

NOTE Hydrochloric acid with a guaranteed low ammonia content is not commercially available. In case of problems, hydrochloric acid should be prepared by absorbing hydrogen chloride gas in water.

C.4.4 Hydrochloric acid, $c(\text{HCl}) = 8 \text{ mol/l}$.

NOTE Hydrochloric acid with a guaranteed low ammonia content is not commercially available. In case of problems, hydrochloric acid should be prepared by absorbing hydrogen chloride gas in water.

C.4.5 Trichloroethylene, C_2HCl_3 .

C.4.6 Carbon dioxide, solid.

C.4.7 Nitrogen, under pressure.

C.4.8 Ammonia solution $c(\text{NH}_3) = 50 \text{ } \mu\text{g/ml}$.

Dissolve 315 mg of ammonium chloride, NH_4Cl , in water and dilute to 100 ml in a volumetric flask. Dilute 5,0 ml of this solution to 100 ml in a volumetric flask. Always use a freshly prepared diluted solution.

C.4.9 Phenol solution, $c(\text{C}_6\text{H}_5\text{OH}) = 10 \text{ g/l}$.

Dissolve 5 g of phenol, $\text{C}_6\text{H}_5\text{OH}$, and 50 mg of sodium pentacyanonitrosylferrate(III) dihydrate $\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}] \cdot 2\text{H}_2\text{O}$, in 500 ml of water. Store the solution in an amber glass bottle. The solution is stable for 1 month.

C.4.10 Sodium hypochlorite solution.

Add to 15 ml of sodium hydroxide solution, $c(\text{NaOH}) = 4 \text{ mol/l}$, 1,5 ml of sodium hypochlorite solution, $c(\text{Cl}_2) = 160 \text{ g/l}$ and dilute to 500 ml with water. Store the solution in an amber glass bottle at $4 \text{ }^\circ\text{C}$. The solution is stable for 1 month. It is important to ensure that the available chlorine content of the sodium hypochlorite is at the stated value.

C.4.11 Sodium hydroxide solution, $c(\text{NaOH}) = 10 \text{ mol/l}$.

C.4.12 Sodium hydroxide solution, $c(\text{NaOH}) = 0,1 \text{ mol/l}$.

C.4.13 Sulfuric acid, $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/l}$.

C.4.14 Sodium chloride solution, $c(\text{NaCl}) = 300 \text{ g/l}$.

Dissolve 300 g of NaCl , in water and dilute to 1 l.

C.4.15 Boiling chips.

C.5 Apparatus

C.5.1 Sample cylinder ¹⁰⁾, volume 0,5 l, provided with a valve ¹¹⁾.

NOTE The presence of nitrogen prevents complete filling of the sample cylinder with liquid chlorine. A residual gas volume approximately 15 % (V/V) is left if the cylinder is filled to a pressure of approximately 700 kPa. Large changes in temperature should be avoided during transport.

C.5.2 Device for discharge and evaporation of liquid chlorine (see Figure C.1).

C.5.3 Water jet vacuum pump or vacuum line.

C.5.4 Nitrogen cylinder or line with pressure regulator.

C.5.5 Nitrogen supply with ball joint S 13.

C.5.6 Metal fittings.

¹⁰⁾ Hoke HS 500 (steel 188) is an example of a suitable product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN/CENELEC of this product.

¹¹⁾ Hoke Y 3001 H is an example of a suitable product available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN/CENELEC of this product.

NOTE With fittings of Gyrolok or Swagelok¹²⁾, tubes of various materials can be connected.

C.5.7 Ball joints S 13.

C.5.8 Spectrometer.

C.5.9 Cell, path length 1 cm.

C.5.10 Bath with boiling water.

C.5.11 pH-meter, with combined glass/reference electrode.

C.5.12 Disposable pipette.

C.6 Sampling and samples

Discharge the sample directly from the sample point in the plant into the sample cylinder (C.5.1). Use valves and fittings as shown in Figure C.1.

The sampling procedure shall be carried out so that the supply tube can be purged with liquid chlorine.

It is important to avoid the application of materials affecting the decomposition of NC_3 , particularly materials containing copper. Avoid also contact with (stopcock) grease, organic material and UV radiation.

Analyse the sample immediately after sampling, unless it is established that no decomposition takes place in the sample cylinder. Proceed with the analysis in a fume cupboard.

C.7 Procedure

C.7.1 Calibration solutions

Transfer to six 50 ml volumetric flasks, using a disposable pipette, 0 ml; 0,10 ml; 0,25 ml; 0,50 ml; 0,75 ml and 1,0 ml of standard ammonia solution (C.4.8) respectively (0 μg ; 5,0 μg ; 12,5 μg ; 25 μg ; 37,5 μg and 50 μg of ammonia). Add 25 ml of sodium chloride solution (C.4.14) and proceed according to C.7.4 (Absorbance = 0 to 1).

C.7.2 Discharge and evaporation (see Figure C.1)

Transfer 10 ml of hydrochloric acid (C.4.4.) to the sample vessel (7 in Figure C.1). Fill the absorption vessels (11) and (19) with 1 l of sodium hydroxide solution (C.4.2) and determine the mass of vessel (19) plus the contents (m_1).

NOTE 1 In 1 l of sodium hydroxide solution, $c(\text{NaOH}) = 6 \text{ mol/l}$, a quantity of 200 g of chlorine can be absorbed theoretically. It is recommended to absorb not more than 150 g.

Transfer trichloroethylene (C.4.5) into the Dewar vessel in which the sample vessel is immersed to the conical joint. Cool the trichloroethylene with carbon dioxide (C.4.6) to a temperature of - 60 °C.

¹²⁾ Gyrolok or Swagelok is an example of a suitable material available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by GEN/GENELEC of this product.

NOTE 2 The temperature should not be decreased to a value less than $-60\text{ }^{\circ}\text{C}$, in order to prevent solidification of hydrochloric acid, $c(\text{HCl}) \approx 8\text{ mol/l}$.

Place the sample vessel into the Dewar vessel. Assemble the device as shown in Figure C.1.

Open the valves (2) and (3) and discharge about 100 ml (about 150 g) of liquid chlorine into the cooled sample vessel. Adjust the sample supply in such a way that only a small amount of gaseous chlorine is passed via stopcock (9) and guard vessel (10) and absorbed in the sodium hydroxide solution (C.4.2) in vessel (11). Close the valves (2) and (3), disconnect the adsorption system (10 + 11) and the sample cylinder (1) and remove the sample cylinder.

Connect the sample vessel to two Durand (gas washing) bottles (13) and (14), each containing 25 ml of hydrochloric acid (C.4.3). Connect the chlorine absorption system (18 + 19) to the gas washing bottle (14). Connect the gas washing bottle (16), containing 25 ml of water, via a T-piece and a regulating stopcock (17), to the guard vessel (18). Connect the chlorine absorption vessel (19) to a water jet pump (C.5.3). Switch on the water jet pump and adjust the flow with the help of the stopcocks (17) and (20) in such a way that air is drawn through the absorption system (18 + 19) and the water containing gas washing bottle (16).

Remove the Dewar vessel (8) and replace it by a beaker. Allow the liquid chlorine to evaporate with a flow of 50 l/h to 100 l/h. Add, if necessary, water at about $30\text{ }^{\circ}\text{C}$ to the beaker under the sample vessel (7) in order to maintain this evaporation rate. Take care, with the help of stopcocks (17) and (20), that at all times some air is drawn through gas washing bottle (16). Disconnect, after complete evaporation of the chlorine, the water jet pump (C.5.3) and the gas washing bottle (16) and close stopcock (17). By opening stopcock (6), connect a nitrogen supply (C.5.5) via gas washing bottle (5), containing 25 ml of sulfuric acid (C.4.13), to the sample vessel (7).

Pass nitrogen through the sample vessel (7), the gas washing bottles (13) and (14) and the absorption system (18 + 19) until no more chlorine can be detected beyond gas washing bottle (14).

NOTE 3 Chlorine can be detected by a wet potassium iodide-starch paper.

Disconnect all joints. Determine again the mass of absorption vessel (19) and contents (m_2). Transfer the contents of the sample vessel (7) and of the gas washing bottles (13) and (14) quantitatively to a 250 ml beaker. Take care that the concentration of hydrochloric acid after this transfer is higher than 6 mol/l.

NOTE 4 During the neutralization step the pH should not exceed the value of 8 in order to prevent losses of ammonia. If the liquid chlorine sample is contaminated with iron(III), iron(III) hydroxide will be precipitated and should be filtered off and washed before diluting the solution to volume.

Proceed according to C.7.3.

C.7.3 Test solution

Place the beaker in an ice/water bath and using a magnetic stirrer, agitate the solution slowly. Suspend a pH electrode in the cooled solution and add sodium hydroxide solution (C.4.11) slowly and continuously from a burette with the delivery below the liquid level in the beaker; the addition shall take 4 min to 5 min. Thereafter add the alkali in very small increments up to pH 2,5, and complete the neutralization using a dilute sodium hydroxide solution (C.4.12). In order to avoid losses of ammonia during neutralization it is essential to avoid overshooting in this titration.

Transfer the neutralized solution completely to a 100 ml volumetric flask, allow the solution to reach room temperature, dilute to volume and mix.

NOTE During the neutralization step the pH should not exceed the value of 8 in order to prevent losses of ammonia. If the liquid chlorine sample is contaminated with iron(III), iron(III) hydroxide will be precipitated and should be filtered off and washed before diluting the solution to volume.

Proceed according to C.7.4.

C.7.4 Determination

Transfer a volume V of test solution (25 ml maximum), containing not more than 50 μg of ammonia, into a 50 ml volumetric flask and dilute to 25 ml if necessary. Add 5 ml of phenol solution (C.4.9) and 5 ml of sodium hypochlorite solution (C.4.10) and mix after each addition. Dilute to volume with water, mix and, with the stoppers set loosely in position, place the flask in a boiling water bath so that the test solution is covered by the boiling water. After 10 min in the water bath, allow to cool to room temperature.

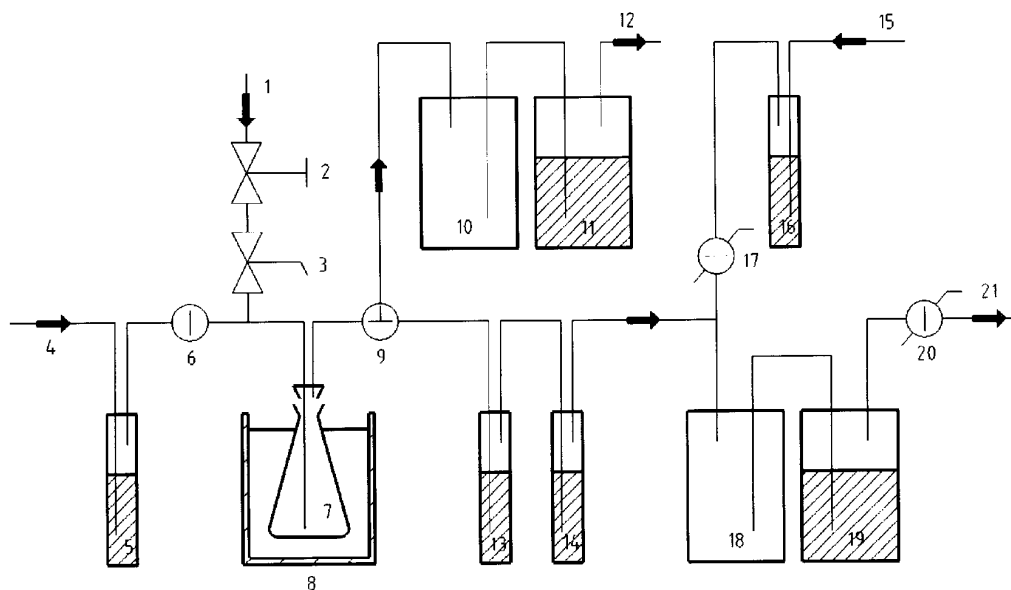
NOTE 1 If the colour of the test solution complex is different from that of the calibration solutions, interfering bodies other than iron are present. A volume of 50 ml of the test and reagent blank solutions should be distilled separately using a boiled-out ammonia distillation apparatus containing 5 ml of sodium hydroxide solution [$c(\text{NaOH}) = 10 \text{ mol/l}$]. A volume of 49 ml of distillate should be collected in a 50 ml volumetric flask containing 1 ml of hydrochloric acid. The contents of the flasks should be mixed and the development and measurement of the colour should be repeated according to C.7.4.

Measure the absorbance (A) versus water in a 1 cm cell at a wavelength of 625 nm.

NOTE 2 The sensitivity of the determination depends slightly on the concentration of sodium chloride.

C.7.5 Blank test determination

Run a blank test determination starting from 10 ml hydrochloric acid $c(\text{HCl}) = 8 \text{ mol/l}$ (C.4.4) + 10 ml hydrochloric acid $c(\text{HCl}) = 12 \text{ mol/l}$ (C.4.3) and measure the absorbance (A_0). For NCl_3 determinations at the lowest levels, check the daily blank value and calculate the corresponding limit of quantitation.



Key

- | | | | |
|----|--|----|---|
| 1 | sample cylinder | 12 | exhaust |
| 2 | main valve | 13 | Durand gas washing bottle, volume 100 ml, amber glass, |
| 3 | needle valve | 14 | see 13. |
| 4 | nitrogen supply | 15 | air inlet |
| 5 | gas washing bottle, volume 100 ml, white glass | 16 | gas washing bottle, volume 100 ml, white glass, filled with water |
| 6 | two-way stopcock, glass | 17 | two-way stopcock, regulating |
| 7 | sample vessel, conical flask, volume 250 ml with stopper, amber glass provided with inlet tube near to the bottom and an outlet tube | 18 | guard vessel, 1,5 l, with gas inlet and outlet tube |
| 8 | Dewar vessel or beaker for cooling or heating of the sample vessel, respectively | 19 | absorption vessel filled with sodium hydroxide solution (C.4.11) |
| 9 | three-way stopcock, glass | 20 | two-way stopcock, regulating |
| 10 | guard vessel, 1,5 l, with gas inlet and outlet tube | 21 | exhaust |
| 11 | absorption vessel containing sodium hydroxide solution (C.4.11), | | |

Figure C.1 — Device for discharge and evaporation

C.8 Calculation and expression of the results

C.8.1 Calibration graph (see Table C.1)

Subtract the absorbance A_0 of the solution containing no added ammonia from the absorbance A of each of the other solutions and plot a graph of corrected absorbance against micrograms of ammonia added.

C.8.2 Test solution

Subtract the absorbance A_0 of reagent blank solution from the absorbance A of the test solution and refer the corrected value to the calibration graph.

The content of nitrogen trichloride $c(\text{NCl}_3)$ expressed in milligrams per kilogram in the liquid chlorine, is given by the formula :

$$c(\text{NCl}_3) = 7,07 \times \frac{m}{(m_2 - m_1) \times V} \quad (\text{C.1})$$

where :

m is the content of ammonia in the test solution aliquot, in micrograms, as read from the calibration graph ;

V is the volume, in millilitres, of the aliquot of the test solution ;

m_1 is the mass, in grams, of the chlorine absorption vessel (19) before sampling ;

m_2 is the mass, in grams, of the chlorine absorption vessel (19) after sampling (see Table C.2).

7,07 is the ratio of molecular mass of NCl_3/NH_3 .

Table C.1 — Calibration graph

| Characteristic | Guide value | Dimension |
|---------------------------------------|-------------|--|
| Intercept (a) | 0,000 | absorbance units |
| Slope (b) | 0,02 | absorbance units per microgram of ammonia in 50 ml of final solution |
| Residual standard deviation (s_e) | 0,004 | absorbance units |
| Coefficient of variation (v_x) | 1 | % |

Table C.2 — Temperature and density of liquid chlorine

| Temperature, °C | Density, g/ml |
|-----------------|---------------|
| - 80 | 1,66 |
| - 60 | 1,62 |
| - 40 | 1,57 |
| - 20 | 1,52 |
| 0 | 1,47 |
| + 20 | 1,41 |
| + 40 | 1,33 |

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- [5] Euro Chlor GEST 94/206: Safe use of chlorine at swimming pools
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