

BS EN 937:2016



BSI Standards Publication

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

**National foreword**

This British Standard is the UK implementation of EN 937:2016. It supersedes BS EN 937:2009 which is withdrawn.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.

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Published by BSI Standards Limited 2016

ISBN 978 0 580 91012 8

ICS 13.060.20; 71.100.80

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 August 2016.

**Amendments/corrigenda issued since publication**

Date	Text affected
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EUROPEAN STANDARD

**EN 937**

NORME EUROPÉENNE

EUROPÄISCHE NORM

May 2016

ICS 71.100.80

Supersedes EN 937:2009

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 18 March 2016.

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## European foreword

This document (EN 937:2016) 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of an identical text or by endorsement, at the latest by November 2016 and conflicting national standards shall be withdrawn at the latest by November 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 937:2009.

Differences between this edition and EN 937:2009 are editorial in order to harmonize the text with other standards in this series:

- a) deletion of reference to Directive 67/548/EEC of 27th June 1967 in order to take into account the latest Regulation in force (see [2]);
- b) amendment of subclause 6.2 according to [2];
- c) deletion of reference to Directive 98/8/EC of 16 February 1998 in order to take into account the latest Regulation in force (see [3]).

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, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the 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.

This product is a biocide and needs to comply with the relevant legislation in force. In the European Union, at the time of publication, this legislation is Regulation (EU) No 528/2012 [3].

## 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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)*

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

Cl<sub>2</sub>.

#### 3.1.5 Chemical formula

Cl<sub>2</sub>.

#### 3.1.6 CAS Registry Number <sup>1)</sup>

7782-50-5.

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<sup>1)</sup> Chemical Abstracts Service Registry Number.

### 3.1.7 EINECS reference <sup>2)</sup>

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/m<sup>3</sup> at 101.3 kPa at 0 °C;
- 2,945 kg/m<sup>3</sup> 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 <sup>3)</sup>

- 34 °C.

### 3.3.6 Liquefaction point

- 101 °C at 100 kPa.

### 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:  $1\,333 \times 10^{-8}$  Pa.s at 20 °C.

Liquid:  $4,78 \times 10^{-4}$  Pa.s at - 34 °C.

### 3.3.9 Critical temperature

144 °C.

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<sup>2)</sup> European Inventory of Existing Commercial Chemical Substances.

<sup>3)</sup> 100 kPa = 1 bar.



### 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.

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 <sub>2</sub> O)	20
Nitrogen trichloride (NCl <sub>3</sub> )	20 <sup>a</sup>
<sup>a</sup> Valid for containers with a net mass of max. 1 000 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 gases (nitrogen, oxygen, carbon dioxide, hydrogen) in variable proportions 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.

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 equipment (cylinders, pressure drums, tanks, portable tanks, etc.). (See [7]).

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 Labelling in accordance with EU legislation <sup>4)</sup>

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



Figure 1 — GHS04

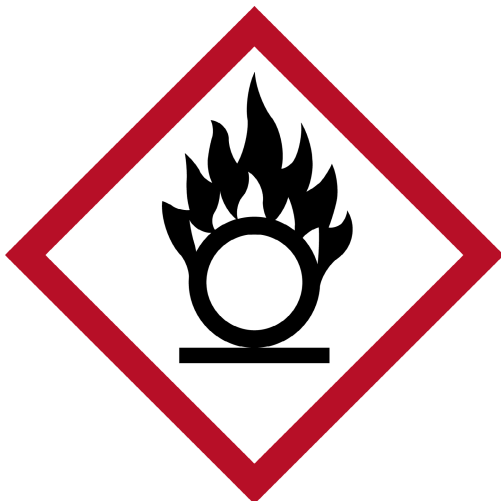


Figure 2 — GHS03

— Signal Word:

**Danger**

— Hazard Statements:

H270 May cause or intensify fire; oxidiser

H331 Toxic if inhaled

H319 Causes serious eye irritation

H335 May cause respiratory irritation

H315 Causes skin irritation

H400 Very toxic to aquatic life

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<sup>4)</sup> See [2].



Figure 3 — GHS06



Figure 4 — GHS09

The Regulation [2] contains a list of substances classified by the EU. Substances not listed in this Regulation should be classified on the basis of their intrinsic properties according to the criteria in the Regulation by the person responsible for the marketing of the substance.

Chlorine used for treatment of water intended for human consumption is applied as a biocidal product. Therefore, additional labelling requirements according to Regulation (EU) No 528/2012 (see [3]) shall be applied.

### 6.3 Transportation regulations and labelling

Chlorine is listed as UN Number <sup>5)</sup> 1017.

ADR <sup>6)</sup>/RID <sup>7)</sup> : class 2, classification code 2 TOC, labels: 2.3 + 5.1 + 8 + environmentally hazardous substance mark, hazard n°.:265.

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

IATA <sup>9)</sup> : Not permitted.

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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.

## 6.4 Marking

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

- the name: "chlorine", the trade name, the grade and type;
- 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 sulphides, 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 pressurized containers either as gas or as liquid that is vaporized externally by adequate vaporizing 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 sulphur dioxide or an aqueous solution of a sulphite compound. Other methods can utilize activated carbon or hydrogen peroxide.

## **Annex B** (informative)

### **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 (see [5, 6, 7, 8]).

#### **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 oxidizing 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 (informative)

### Determination of nitrogen trichloride (molecular absorption spectrometry)

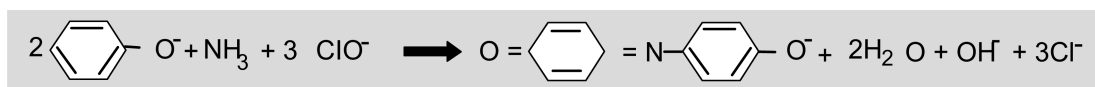
#### C.1 General

This method applies to products with a nitrogen trichloride 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,  $\text{C}_2\text{HCl}_3$ .

**C.4.6** Carbon dioxide, solid.

**C.4.7** Nitrogen, under pressure.

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



Dissolve 315 mg of ammonium chloride,  $\text{NH}_4\text{Cl}$ , 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 °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 <sup>10)</sup>, volume 0,5 l, provided with a valve <sup>11)</sup>.

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.

NOTE With fittings of Gyrolok or Swagelok<sup>12)</sup>, tubes of various materials can be connected.

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 European Standard and does not constitute an endorsement by CEN 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 European Standard and does not constitute an endorsement by CEN of this product.

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

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  $\text{NCl}_3$ , 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 ( $0 \mu\text{g}$ ;  $5,0 \mu\text{g}$ ;  $12,5 \mu\text{g}$ ;  $25 \mu\text{g}$ ;  $37,5 \mu\text{g}$  and  $50 \mu\text{g}$  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 ( $m_1$ ).

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  $-60 \text{ }^\circ\text{C}$ .

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 °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 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.

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.

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  $\mu\text{g}$  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.

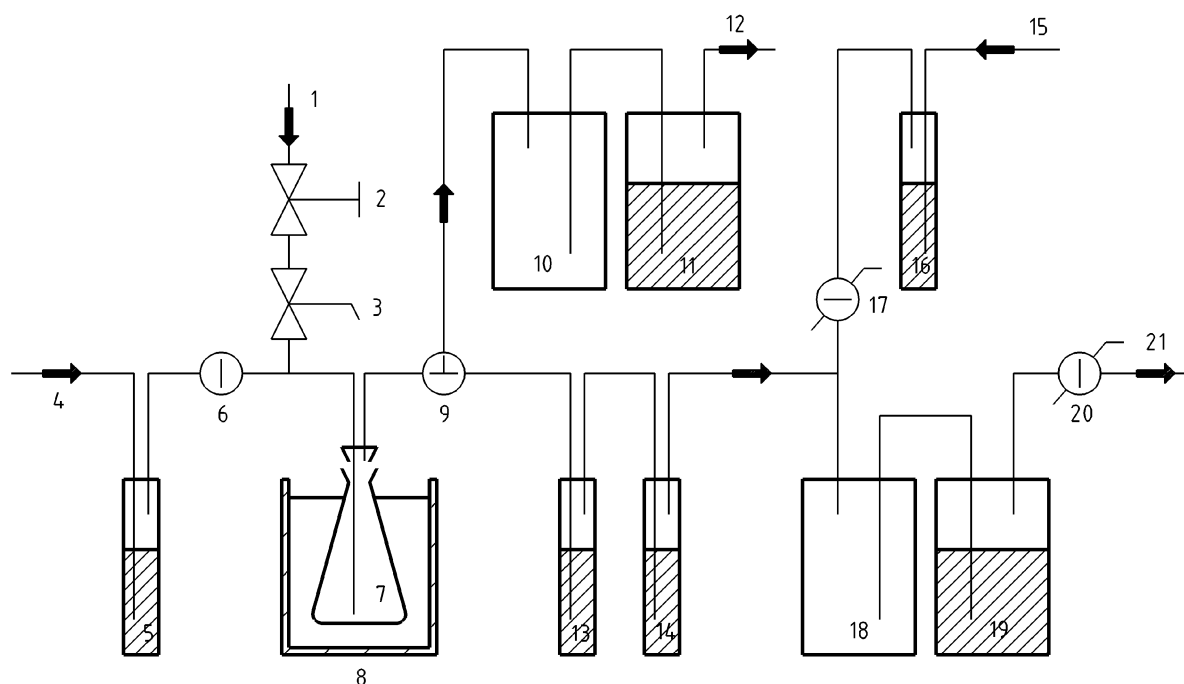
If the colour of the test solution complex is different from that of the calibration solutions, interfering bodies other than iron are present. A volume of 50 ml of the test and reagent blank solutions should be distilled separately using a boiled-out ammonia distillation apparatus containing 5 ml of 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 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  $\text{NCl}_3$  determinations at the lowest levels, check the daily blank value and calculate the corresponding limit of quantisation.



### 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  $\text{NCl}_3/\text{NH}_3$ .

**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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