BS EN 900:2014

BSI Standards Publication

Chemicals used for treatment of water intended for human consumption — Calcium hypochlorite

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National foreword

This British Standard is the UK implementation of EN 900:2014. It supersedes BS EN 900:2007 which is withdrawn.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.

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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 - Calcium hypochlorite

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

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Calciumhypochlorit

This European Standard was approved by CEN on 22 May 2014.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 900:2014) 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of an identical text or by endorsement, at the latest by January 2015, and conflicting national standards shall be withdrawn at the latest by January 2015.

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 900:2007.

Significant technical differences between this edition and EN 900:2007 are as follows:

a) deletion of the maximum sodium chloride content and of its relevant method of determination;

b) replacement of warning and safety precautions notes by labelling according to REGULATION (EC) No 1272/2008.

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

With respect to 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 1 Conformity with this European Standard does not confer or imply acceptance or approval of the products in any of the Member States of the EU or EFTA. The use of the products covered by this European Standard is subject to regulation or control by National Authorities.

NOTE 2 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 Directive 98/8/EC [1]).

1 Scope

This European Standard is applicable to calcium hypochlorite used for the treatment of water intended for human consumption. It describes the characteristics of calcium hypochlorite and specifies the requirements and the corresponding test methods for calcium hypochlorite. It provides information on its use in water treatment. It also determines the rules relating to safe handling and use of calcium hypochlorite (see Annex B).

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)*

EN ISO 12846:2012, *Water quality - Determination of mercury - Method using atomic absorption spectrometry (AAS) with and without enrichment (ISO 12846:2012)*

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*

ISO 8288, *Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods*

ISO 9174, *Water quality — Determination of chromium — Atomic absorption spectrometric methods*

3 Description

3.1 Identification

3.1.1 Chemical name

Calcium hypochlorite.

3.1.2 Synonym or common name

None.

3.1.3 Relative molecular mass

142,99.

3.1.4 Empirical formula

$Ca(ClO)_2$.

3.1.5 Chemical formula

$Ca(ClO)_2$.

3.1.6 CAS Registry Number [1)]

7778-54-3.

3.1.7 EINECS reference [2)]

231-908-7.

3.2 Commercial form

The product is available as a granular solid or in the form of tablets.

3.3 Physical properties

3.3.1 Appearance

The product is white free-flowing granules or white tablets.

3.3.2 Density

The bulk density is approximately 0,8 g/cm^3 to 1 g/cm^3 for loose granular material and 1,2 g/cm^3 to 1,3 g/cm^3 for tablets, while the density of one tablet is approximately 1,7 g/cm^3 to 1,9 g/cm^3.

3.3.3 Solubility in water

The solubility is 180 g/l at 25 °C.

3.3.4 Vapour pressure

Not applicable.

3.3.5 Boiling point at 100 kPa [3)]

Not applicable.

3.3.6 Melting point

Not applicable as the product decomposes at 177 °C.

3.3.7 Specific heat

Not known.

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1) Chemical Abstracts Service Registry Number.

2) European Inventory of Existing Commercial Chemical Substances.

3) 100 kPa = 1 bar.

3.3.8 Viscosity, dynamic

Not applicable.

3.3.9 Critical temperature

Not applicable.

3.3.10 Critical pressure

Not applicable.

3.3.11 Physical hardness

Not applicable.

3.4 Chemical properties

Solutions of calcium hypochlorite are alkaline. The pH value of a solution of concentration 10 g/l is about 11,5 at 25 °C.

Calcium hypochlorite is a strong oxidant and chlorination agent. It reacts with acids or acidic salts to form chlorine, and can form explosive nitrogen chlorides with ammonia and ammonia compounds. In the presence of inflammable substances, it causes fires and explosions of organic compounds, oxidation reactions occur with the release of heat and of moisture, and it is also highly corrosive to most metals.

4 Purity criteria

4.1 General

This European Standard specifies the minimum purity requirements for calcium hypochlorite 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, then the user, and when necessary the relevant authorities, shall be notified.

Users of the product should check the national regulations 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 this 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 in 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 product shall contain a minimum of a mass fraction of 65,5 % of calcium hypochlorite (equivalent to an available active chlorine content of at least a mass fraction of 65 %).

Dissolution quality, calculated as available chlorine which is obtainable within 1 min after dissolution in water, shall not be less than a mass fraction of 45,5 %.

The water content at the time of delivery should not exceed a mass fraction of 16 % of the product. As the test method is usually not conducted by the user without danger of explosion, the manufacturer should guarantee

to maintain this value. If necessary a test laboratory may be requested to carry out this test. This determination should be carried out by specialists only.

4.3 Impurities and main by-products

The content of water-insoluble matter shall not exceed a mass fraction of 6 % of the product.

NOTE 1 The water insoluble matter consists mainly of carbonates.

NOTE 2 Calcium chlorate can be present as a by-product of the production process.

4.4 Chemical parameters

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

Table 1 — Chemical parameters

Parameter		**Limit in mg/kg of available chlorine**	
		Type 1	**Type 2**
Arsenic (As)	max.	5	10
Cadmium (Cd)	max.	5	10
Chromium (Cr)	max.	15	15
Mercury (Hg)	max.	5	7
Nickel (Ni)	max.	8	10
Lead (Pb)	max.	15	15
Antimony (Sb)	max.	15	15
Selenium (Se)	max.	20	20
		Limit in g/kg of available chlorine	
Bromate [a]	max.	2,1	4,2
NOTE Cyanide, which does not exist in a strong oxidizing medium such as calcium hypochlorite is not a relevant chemical parameter. Pesticides and polycyclic aromatic hydrocarbons are not by-products of the manufacturing process. For parametric values of calcium hypochlorite on trace metal content in drinking water, see [2].			
[a] Bromate is a by-product of the manufacturing process.			

5 Test methods

5.1 Sampling

Observe the general recommendations of ISO 3165 and take account of ISO 6206. Prepare the laboratory sample(s) required by the relevant procedure described in ISO 8213.

5.2 Analysis

5.2.1 Determination of calcium hypochlorite content (main product)

5.2.1.1 Principle

Calcium hypochlorite reacts with potassium iodide to release iodine in the presence of acetic acid. The iodine is titrated with sodium thiosulfate standard volumetric solution in the presence of starch indicator solution.

NOTE 1 It detects all oxidizing agents being active in a weak acidic solution, i.e. hypochlorite/chlorine, iodate, and partially chloramines, Fe(III), etc. Not covered under these conditions are bromate and chlorate.

NOTE 2 The titration may also be carried out potentiometrically, with automatic titration, in which case the addition of soluble starch is unnecessary.

5.2.1.2 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to grade 3 as specified in EN ISO 3696.

5.2.1.2.1 Potassium iodide crystals (KI).

5.2.1.2.2 Glacial acetic acid.

5.2.1.2.3 Hydrochloric acid solution.

Concentrated hydrochloric acid density ρ (HCl) = 1,16 g/ml diluted 1 + 1 by volume with water.

5.2.1.2.4 Sodium thiosulfate standard volumetric solution, $c(Na_2S_2O_3.5H_2O)$ = 0,1 mol/l.

Standard volumetric solutions are commercially available, which might have to be diluted.

Alternatively a standard volumetric solution may be prepared by the following procedure: Dissolve 24,8 g $Na_2S_2O_3.5H_2O$ in a 1 000 ml one-mark volumetric flask in about 0,75 l water. After the temperature has equalized make up to the mark with water and mix thoroughly.

To standardize: Weigh, to the nearest 0,1 mg, 3,600 g (m) of dry potassium iodate. Dissolve in water in a 1 000 ml one-mark volumetric flask, make up to the mark with water and mix (standard reference solution c(1/6 KIO_3) = 0,1 mol/l). Place 200 ml of water in a 500 ml stoppered conical flask, add (2 ± 0,5) g of potassium iodide and stir to dissolve. Then introduce by means of a pipette, 10,0 ml of sodium thiosulfate solution for standardization, add (15 ± 1) ml of hydrochloric acid solution (5.2.1.2.3) and (5 ± 1) ml of starch solution (5.2.1.2.5). Titrate immediately with the potassium iodate standard reference solution until the appearance of a blue coloration persisting for at least 30 s. Record the volume *(V)* of iodate used.

The concentration, c, of the sodium thiosulfate standard volumetric solution ($Na_2S_2O_3.5H_2O$), expressed in moles per litre is given by the following formula:

$$C = \frac{m}{V \times 49,0317} \quad (1)$$

where

m is the mass, in milligrams, of potassium iodate (KIO_3) weighed;

V is the volume, in millilitres, of the sodium thiosulfate standard volumetric solution used.

5.2.1.2.5 Starch solution, mass fraction 1 %.

Make a slurry with (1 ± 0,1) g starch and (5 ± 1) ml water. Add (90 ± 5) ml boiling water to the slurry. Stir to dissolve it and cool the solution. This solution needs refrigeration to avoid the decomposition of the starch which results in a vague end point. Keep the solution cool and use it within one week.

Commercial indicators for iodine titration exist and may be used in place of the described starch solution provided that their efficiency has been previously tested.

5.2.1.3 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

5.2.1.3.1 Laboratory sonic vibrator.

5.2.1.4 Procedure

5.2.1.4.1 Test portion

Weigh, to the nearest 0,1 mg, 3,5 g of the laboratory sample (m_1) into a tarred stoppered weighing bottle.

5.2.1.4.2 Determination

Transfer the test portion to a 500 ml volumetric flask with 300 ml of water, stopper, and place in the sonic vibrator for 10 min, swirling it occasionally until the test portion is in a solution. Make up to the mark with water.

Place a magnetic stirring bar into the volumetric flask and begin mixing. Transfer 25 ml, while the test portion is being stirred and without allowing any insoluble matter to settle out, into the 500 ml conical flask.

Add 100 ml of water and 2 g of potassium iodide (5.2.1.2.1), and mix to dissolve. Add 8 ml of glacial acetic acid (5.2.1.2.2), stir and titrate immediately with the sodium thiosulfate standard volumetric solution (5.2.1.2.4) to a light yellow colour. Add 3 ml of the starch solution (5.2.1.2.5) and continue titration to the disappearance of the blue black colour. Record the volume V_1, of the sodium thiosulfate standard volumetric solution used.

5.2.1.5 Expression of results

The chlorine (Cl_2) content, w_1, expressed as a mass fraction in %, is given by the following formula:

$$w_1 = \frac{V_1 \times c \times 35{,}453 \times 20 \times 100}{m_1} \qquad (2)$$

where

V_1 is the volume, in millilitres, of the sodium thiosulfate solution (5.2.1.2.4) used for the titration;

c is the concentration, in moles per litre, of the sodium thiosulfate standard volumetric solution (5.2.1.2.4);

m_1 is the mass, in milligrams, of the test portion (5.2.1.4.1);

35,453 is the mass in milligrams of chlorine (Cl_2) corresponding to 1,00 ml of sodium thiosulfate solution $c(Na_2S_2O_3.5H_2O)$ = 1,000 mol/l.

The $Ca(ClO)_2$ content, w_2, expressed as a mass fraction of product, is given by the following formula:

$$w_2 = \frac{w_1 \times 3{,}574\,6}{3{,}5453} \quad (3)$$

5.2.1.6 Repeatability limit

The absolute difference between two single test results, obtained under repeatability conditions, shall not be greater than the repeatability value, r, as calculated from the following formula:

$$r = 0{,}01 \times z \quad (4)$$

where

z is the mean of the two results, expressed in mass fraction in percent (%).

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.

5.2.2 Dissolution quality (available chlorine after 1 min)

5.2.2.1 Principle

A representative sample is stirred in water for 1 min and immediately titrated iodometrically. Dissolved calcium hypochlorite reacts with iodide in the presence of acetic acidic to release iodine, which is titrated with sodium thiosulfate standard volumetric solution.

NOTE The titration may be carried out manually with the addition of starch solution as a visual indicator, or potentiometrically with automatic titration and without an indicator.

5.2.2.2 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to grade 3 as specified in EN ISO 3696.

5.2.2.2.1 Sodium thiosulfate standard volumetric solution, $c(Na_2S_2O_3.5H_2O)$ = 0,1 mol/l (see 5.2.1.2.4).

5.2.2.2.2 Potassium iodide, virtually free of iodate.

5.2.2.2.3 Glacial acetic acid.

5.2.2.2.4 Starch indicator solution, mass fraction 1 % (see 5.2.1.2.5).

5.2.2.3 Apparatus

Ordinary laboratory apparatus and glassware.

5.2.2.4 Procedure

From a representative sample of the product weigh 1,5 g to the nearest 1 mg (m_2). Add the test portion to a beaker containing 1 000 ml of water (at a temperature of 20 °C to 25 °C) and by use of a magnetic stirrer agitate the contents.

After 1 min stop the stirrer and remove a 25 ml aliquot by use of a pipette. Transfer to a conical flask containing 100 ml of water, add 2 g of potassium iodide crystals (5.2.2.2.2), 8 ml of acetic acid (5.2.2.2.3) and titrate the liberated iodine with sodium thiosulfate standard volumetric solution (5.2.2.2.1) to a light yellow colour. Add 3 ml of the starch indicator solution (5.2.2.2.4) and continue titration to the disappearance of the blue-black colour. Record the volume (V_2) of the sodium thiosulfate standard volumetric solution used.

5.2.2.5 Expression of results

The available chlorine which is released within 1 min, (w_3), expressed as a mass fraction in % of product is given by the following formula:

$$w_3 = \frac{V_2 \times c \times 35{,}453 \times 40 \times 100}{m_2} \quad (5)$$

where

c is the concentration in moles per litre of sodium thiosulfate standard volumetric solution (5.2.2.2.1);

V_2 is the volume, in millilitres, of the sodium thiosulfate standard volumetric solution (5.2.2.2.1);

m_2 is the mass, in milligrams, of the test portion;

35,453 is the mass in milligrams of chlorine (Cl_2) corresponding to 1,00 ml of sodium thiosulfate standard volumetric solution $c(Na_2S_2O_3.5H_2O)$ = 1,000 mol/l.

5.2.2.6 Repeatability limit

The absolute difference between two single test results, obtained under repeatability conditions, shall not be greater than the repeatability value, r, as calculated from the following formula:

$$r = 0{,}01\ z \quad (6)$$

where

z is the mean of the two results, expressed in mass fraction in percent (%).

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.

5.2.3 Impurities

5.2.3.1 Insoluble matter

5.2.3.1.1 Principle

A representative sample of calcium hypochlorite is dissolved into water. The insoluble matter is separated by filtration then dried and weighed.

5.2.3.1.2 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

5.2.3.1.2.1 Sintered glass crucible of porosity P40 (pores size between 16 µm to 40 µm).

5.2.3.1.2.2 Oven capable of being controlled at (105 ± 3) °C.

5.2.3.1.3 Procedure

Weigh approximately 10 g of the representative sample (m_3) to the nearest 0,01 g and dissolve in 1 000 ml of water by stirring for 30 min. Then filter the solution under vacuum through a dried and weighed glass filter (5.2.3.1.2.1). After the filtration, wash the residue with 20 ml of water and remove excess water by filtering

under vacuum. Dry the residue at (105 ± 3) °C in the oven (5.2.3.1.2.2) until the mass remains constant and weigh it (m_4) after cooling in a desiccator.

5.2.3.1.4 Expression of results

The insoluble matter expressed as a mass fraction in %, w_4 in the product is given by the following formula:

$$w_4 = \frac{m_4}{m_3} \times 100 \quad (7)$$

where

m_4 is the mass, in grams, of the residue;

m_3 is the mass, in grams, of the test portion.

5.2.3.2 Water content

WARNING If the product is overheated (see 6.5.2), a violent decomposition or explosion can occur. This test shall only be carried out by experienced laboratory staff (usually by the manufacturing company) and not by the end users.

The determination of the water content of calcium hypochlorite may be achieved by drying a sample at temperature between 105 °C and 110 °C until the mass of dried residue remains constant.

5.2.4 Chemical parameters

5.2.4.1 General

Determination of antimony (Sb), arsenic (As), cadmium (Cd), chromium (Cr), lead (Pb), nickel (Ni) and selenium (Se).

5.2.4.2 Principle

The elements arsenic, antimony, cadmium, chromium, lead, nickel and selenium are determined by atomic absorption spectrometry.

5.2.4.3 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to the grade 2 specified in EN ISO 3696.

5.2.4.3.1 Nitric acid, concentrated, density ρ = 1,42 g/ml.

5.2.4.4 Procedure

5.2.4.4.1 Test portion

Weigh, to the nearest 0,001 g, 10 g (m_5) of the laboratory sample into a 100 ml one-mark volumetric flask.

5.2.4.4.2 Test solution

Add 1 ml of nitric acid (5.2.4.3.1) to the test portion; dilute with a few millilitres of water and mix. Make up to volume with water and homogenize.

5.2.4.4.3 Determination

Determine the content of elements in the test solution (5.2.4.4.2) in accordance with the following methods:

— **Cd, Ni and Pb**: In accordance with ISO 8288, Method A;

— **Cr**: In accordance with ISO 9174;

— **As, Se and Sb**: In accordance with the method given in Annex C.

These methods will give an interim result (y) expressed in milligrams per litre which needs to be converted to give the final concentration according to the formula in 5.2.4.4.4.

5.2.4.4.4 Expression of results

From the interim result (y) determined (see 5.2.4.4.3), the content, w_5, of each element in the laboratory sample, expressed in milligrams per kilogram of available chlorine is given by the following formula:

$$w_5 = y \times \frac{V}{m_5} \times \frac{100}{w_1} \tag{8}$$

where

y is the interim result (5.2.4.4.3);

V is the volume, expressed in millilitres, of the test solutions (5.2.4.4.2) (= 100 ml);

m_5 is the mass, expressed in grams, of the test portion (5.2.4.4.1);

w_1 is the available chlorine content in mass fraction of product (5.2.1.5).

5.2.4.5 Determination of the mercury content (Hg)

5.2.4.5.1 Principle

The element mercury is determined by flameless atomic absorption spectrometry in accordance with EN ISO 12846:2012, Clause 7.

5.2.4.5.2 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to the grade 3 specified in EN ISO 3696.

5.2.4.5.2.1 Potassium permanganate solution, $c(KMnO_4)$ = 50 g/l.

5.2.4.5.2.2 Sulfuric acid, concentrated, density ρ = 1,84 g/ml.

5.2.4.5.2.3 Hydroxylammonium chloride solution, $c(NH_2OH.HCl)$ = 100 g/l.

5.2.4.5.2.4 Potassium dichromate solution, $c(K_2Cr_2O_7)$ = 4 g/l in 50 % (V/V) nitric acid solution.

5.2.4.5.3 Procedure

5.2.4.5.3.1 Test portion

Weigh, to the nearest 0,01 g, 10 g (m_6) of the laboratory sample, into a glass beaker.

5.2.4.5.3.2 Test solution

Quantitatively transfer the test portion to a washing flask (e.g. Durand bottle), capacity 250 ml, the gas inlet of which is made of a porous frit. Dilute the contents of the washing flask with water to obtain a total volume of 100 ml. Transfer to a volumetric flask (solution A).

Accurately pipette 10 ml of the solution A. Transfer to a 250 ml conical flask and add 60 ml of water, 20 ml of a potassium permanganate solution (5.2.4.5.2.1) and five 1 ml portions of sulfuric acid (5.2.4.5.2.2). Heat and keep boiling for 10 min. Allow to cool. Dissolve the precipitate (MnO_2) with hydroxylammonium chloride (5.2.4.5.2.3), add 5 ml of the potassium dichromate solution (5.2.4.5.2.4) and transfer to a 100 ml volumetric flask. Make up to the mark with water and mix.

5.2.4.5.3.3 Determination

Proceed as described in EN ISO 12846:2012, Clause 7.

5.2.4.5.4 Expression of result

The interim result for mercury content (y) expressed in milligrams is given by the following formula:

$$y = y_A \times \frac{V_T}{10} \quad (9)$$

where

y_A is the result obtained, for the concentration of mercury in solution A, expressed in milligrams per litre;

V_T is the volume in millilitres of the test solution.

The content of mercury, C_1, in milligrams per kilogram of available chlorine is given by the following formula:

$$C_1 = y \times \frac{10}{m_6} \times \frac{100}{w_1} \quad (10)$$

where

m_6 is the mass, expressed in grams, of the test portion;

w_1 is the available chlorine content in mass fraction (5.2.1.5).

6 Labelling - Transportation - Storage

6.1 Means of delivery

Calcium hypochlorite shall be delivered in plastic-coated steel drums, plastic pails or polyethylene bottles.

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

6.2 Labelling according to the EU legislation [4)]

The following labelling requirements shall apply to calcium hypochlorite at the date of the publication of this European Standard.

— Signal word:

Danger

— Hazard statements:

H272 May intensify fire; oxidiser

H 314 Causes severe skin burns and eye damage

H 302 Harmful if swallowed

H400 Very toxic to aquatic life

The regulation [3] 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.

6.3 Transportation regulations and labelling

Calcium hypochlorite is listed as:

— UN Number [5)] 1748 for granular, calcium hypochlorite mixture, dry;

— UN Number 1748 for tablets, calcium hypochlorite mixture, dry;

NOTE The above products have more than a mass fraction of 39 % available chlorine.

— UN Number [5)] 2880 for granular, calcium hypochlorite, hydrated or calcium hypochlorite, hydrated mixture;

— UN Number 2880 for tablets, calcium hypochlorite, hydrated or calcium hypochlorite, hydrated mixture;

NOTE The above products contain a minimum of mass fraction 5,5 % of water but no more than 16 % of water.

— UN Number 3485 [5)] for granular, calcium hypochlorite mixture, dry;

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4) See [3].

5) United Nations Number.

— UN Number 3485 for tablets, calcium hypochlorite mixture, dry;

NOTE The above products have more than a mass fraction of 39 % available chlorine.

— UN Number 3487 [5)] for granular, calcium hypochlorite, hydrated or calcium hypochlorite, hydrated mixture;

— UN Number 3487 for tablets, calcium hypochlorite, hydrated or calcium hypochlorite, hydrated mixture;

NOTE The above products contain a minimum of mass fraction 5,5 % of water but no more than 16 % of water.

— RID [6)]: class 5.1, classification code O2, packing group II (III for tablets);

— ADR [7)]: class 5.1, classification code O2, packing group II (III for tablets);

— IMDG [8)]: class 5.1;

— IATA [9)]: class 5.1.

6.4 Marking

The marking shall include the following:

— name “calcium hypochlorite”, trade name, grade and type;

— net mass;

— name and the address of supplier and/or manufacturer;

— statement “this product conforms to EN 900”.

6.5 Storage

6.5.1 General

The product shall be stored in airtight containers in a cool, dry and well-ventilated room.

6.5.2 Long term stability

Heat stability: The stability of solid calcium hypochlorite depends on the water content.

At temperatures above 177 °C, decomposition is rapid with the evolution of oxygen and heat and thus increasing the risks of pressure build-up to blow off the lid or rupture the container.

Decomposition also occurs at temperatures maintained above about 50 °C for longer periods.

NOTE Decomposition products are calcium chloride ($CaCl_2$), oxygen (O_2) and chlorine (Cl_2).

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6) Regulations concerning International carriage of Dangerous goods by rail.

7) European Agreement concerning the international carriage of Dangerous goods by Road.

8) International Maritime transport of Dangerous Goods.

9) International Air Transport Association.

Chemical stability:

Contamination can initiate a vigorous chemical reaction resulting in fire and/or explosion.

6.5.3 Storage incompatibilities

Keep away from acids, acidic salts, ammonia and ammonium compounds, inflammable substances, organic compounds and moisture.

Annex A
(informative)

General information on calcium hypochlorite

A.1 Origin

A.1.1 Raw materials

Calcium hypochlorite is manufactured from chlorine, calcium hydroxide and sodium hydroxide.

A.1.2 Manufacturing process

It is produced by drying a filter cake of neutral calcium hypochlorite dihydrate that is usually prepared from calcium hydroxide (hydrated lime) ($Ca(OH)_2$), sodium hydroxide (caustic soda) (NaOH), and chlorine (Cl_2).

A.2 Use

A.2.1 Function

Its functions in water treatment are the removal of ammonium compounds, the oxidation of sulfides, the oxidation of iron (II) to iron (III) and as a disinfectant.

A.2.2 Form in which it is used

It is used in aqueous solutions, usually as a mass fraction of 1 % to 4 %.

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 available chlorine in the water supply, usually a few tenths of 1 mg/l.

A.2.4 Means of application

It is applied using a metering pump, from a dissolving tank.

A.2.5 Secondary effects

The secondary effects include the following:

— slight increase in pH;

— slight increase in the chloride content;

— odour and colour removal;

— oxidation of organic compounds;

— formation of halogenated organic substances, especially trihalomethanes, is possible;

— local precipitation of carbonate at injection point.

A.2.6 Removal of excess product

Excess active chlorine can be removed by utilising a reducing agent such as sulfur dioxide gas or an aqueous solution of a sulfite compound.

Passing through activated carbon is also effective.

Annex B
(normative)

General rules relating to safety

B.1 Rules for safe handling and use

The supplier shall provide current safety instructions.

B.2 Emergency procedures

B.2.1 First aid

In case of contact with the skin, rinse with copious amounts of water, remove contaminated clothing.

In case of contact with the eyes or mucous membranes, rinse immediately with copious of amounts of water for at least 10 min and consult a doctor (danger of irreversible damage to the eye).

In case of inhalation get to the fresh air and consult a doctor.

If swallowed wash out the mouth with water and give water to drink. Do not induce vomiting (danger of perforation). Do not apply neutralization agents. Consult a doctor immediately.

B.2.2 Spillage

Wear respiratory and eye protection equipment and chemical resistant protecting gloves. Do not drain into waste water piping. Collect spilt solid product in separate plastic containers, do not accumulate.

Contain spilt solution and absorb with inert absorbing material (kieselguhr, universal absorbent, etc. - do not use sawdust!). Solutions may be reduced with either sodium sulfite, sodium hydrogen sulfite, hydrogen peroxide or sodium thiosulfate.

Disposal shall be carried out in accordance with the local regulations. Clean contaminated tools by rinsing with plenty of water.

B.2.3 Fire

The material is not combustible, but due to the formation of oxygen as a decomposition by-product it will support combustion. Use air-independent respiratory equipment for fire fighting. Use water to extinguish fire and to cool containers exposed to fire. Do not use dry chemical extinguishers containing ammonium compounds, e.g. monoammonium phosphate.

Annex C
(normative)

Determination of arsenic, antimony and selenium (atomic absorption spectrometry hydride technique)

SAFETY PRECAUTIONS: Arsenic, antimony and selenium and their hydrides are toxic. Handle with care.

C.1 General principle

Arsenious acid, antimonic acid and selenious acid, the As(III), Sb(III) and Se(IV) oxidation states of arsenic, antimony and selenium, respectively, are instantaneously converted by sodium borohydride reagent in acid solution to their volatile hydrides. The hydrides are purged continuously by argon or nitrogen into an appropriate atomizer of an atomic absorption spectrometer and converted to the gas-phase atoms. The sodium borohydride reducing agent, by rapid generation of the elemental hydrides in an appropriate reaction cell, minimizes dilution of the hydrides by the carrier gas and provides rapid, sensitive determinations of arsenic, antimony and selenium.

The sample is digested to solubilize particulate As, Sb and Se. The digested solutions are treated separately for determination of As, Sb and Se to convert them to As(III), Sb(III) and Se(IV) oxidation states respectively.

C.2 Interferences

Interferences are minimized because the As, Sb and Se hydrides are removed from the solution containing most potential interfering substances. Slight response variations occur when acid matrices are varied. Control these variations by treating standards and samples in the same manner. Low concentrations of noble metals (approximately 100 µg/l of Ag, Au, Pt, Pd, etc.) concentrations of Cu, Ni and Pb at or greater than 1 mg/l, and concentrations between 0,1 mg/l and 1 mg/l of hydride-forming elements (Bi, Sn and Te) can suppress the response of As, Sb and Se hydrides due to the formation of mixed metal – As-Sb or -Se compounds. The presence of As, Sb and Se in each other's matrices can cause similar suppression. Reduced nitrogen oxides resulting from HNO_3 digestion and nitrite also can suppress instrumental response for all elements. Large concentrations of iodide interfere with the Se determination by reducing Se to its elemental form. Do not use any glassware for determining Se that has been used with iodide reduction of As(V).

C.3 Reagents

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

C.3.1 Sodium tetrahydroborate (sodium borohydride).

Dissolve 8 g $NaBH_4$ in 200 ml of NaOH, c(NaOH) = 0,1 mol/l. Prepare fresh daily.

C.3.2 Sodium iodide, pre-reductant solution.

Dissolve 50 g Nal in 500 ml water. Prepare fresh daily.

C.3.3 Sulfuric acid, solution $c(H_2SO_4)$ = 9 mol/l.

C.3.4 Sulfuric acid, solution $c(H_2SO_4)$ = 1,25 mol/l.

Cautiously add 35 ml sulfuric acid, density (ρ) = 1,84 g/ml to about 400 ml water, allow to cool, and adjust volume to 500 ml.

C.3.5 Nitric acid, density (ρ) = 1,42 g/ml.

C.3.6 Perchloric acid, density (ρ) = 1,66 g/ml.

C.3.7 Hydrochloric acid, density (ρ) = 1,16 g/ml.

C.3.8 Argon (or nitrogen), commercial grade.

C.3.9 Hydrogen, commercial grade.

C.3.10 Arsenic (III) solutions:

— stock As(III) solution: Dissolve 1,320 g arsenic trioxide, As_2O_3, in water containing 4 g of NaOH. Transfer quantitatively to 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml = 1,00 mg As(III);

— intermediate As(III) solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml stock As (III) solution to the mark with water containing 5 ml hydrochloric acid (C.3.7) and mix ; 1,00 ml = 10,0 µg As(III);

— standard As(III) solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml intermediate As(III) solution to the mark with water containing the same concentration of acid used for sample preservation (2 ml to 5 ml nitric acid (C.3.5)) and mix; 1,00 ml = 0,100 µg As(III). Prepare diluted solutions daily.

C.3.11 Arsenic(V) solutions:

— stock As(V) solution: Dissolve 1,534 g arsenic pentoxide, As_2O_5, in water containing 4 g NaOH. Transfer quantitatively to 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml = 1,00 mg As(V);

— intermediate As(V) solution: Prepare as for As(III) above ; 1,00 ml = 10,0 µg As(V);

— standard As(V) solution: Prepare as for As(III) above; 1,00 ml = 0,100 µg As(V).

C.3.12 Selenium(IV) solutions:

— stock Se(IV) solution: Dissolve 2,190 g sodium selenite, Na_2SeO_3 in water containing 10 ml hydrochloric acid (C.3.7) and transfer quantitatively to 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml = 1,00 mg Se(IV);

— intermediate Se(IV) solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml stock Se (IV) solution to the mark with water containing 10 ml hydrochloric acid (C.3.7) and mix; 1,00 ml = 10,0 µg Se(IV);

— standard Se(IV) solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml intermediate Se(IV) solution to the mark with water containing the same concentration of acid used for sample preservation (2 ml to 5 ml nitric acid (C.3.5)) and mix. Prepare solution daily when checking the equivalent of instrument response for Se(IV) and Se(VI); 1,00 ml = 0,100 µg Se(IV).

C.3.13 Selenium(VI) solutions:

— stock Se(VI) solution: Dissolve 2,393 g sodium selenate Na_2SeO_4 in water containing 10 ml nitric acid (C.3.5). Transfer quantitatively to 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml = 1,00 mg Se(VI);

— intermediate Se(VI) solution: Prepare as for Se(IV) above ; 1,00 ml = 10,0 µg Se(VI);

— standard Se(VI) solution: Prepare as for Se(IV) above ; 1,00 ml = 0,100 µg Se(VI).

C.3.14 Antimony solutions:

— stock Sb solution: Dry 2 g of potassium antimonyl tartrate hemihydrate (antimony potassium tartrate) ($C_4H_4O_7SbK.0,5\ H_2O$) at 100 °C for 1h. Dissolve 1,669 g in water transfer quantitatively to 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml = 1,00 mg Sb;

— intermediate Sb solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml stock Sb solution to the mark with water containing 10 ml hydrochloric acid (C.3.7) and mix; 1,00 ml = 10,0 µg Sb;

— standard Sb solution: Dilute into 1 000 ml one-mark volumetric flask 10 ml intermediate Sb solution to the mark with water containing the same concentration of acid used for sample preservation (2 ml to 5 ml nitric acid (C.3.5)) and mix; 1,00 ml = 0,100 µg Sb. Prepare diluted solutions daily.

C.4 Apparatus

C.4.1 General

Ordinary laboratory apparatus and glassware, together with the following.

C.4.2 Atomic absorption spectrometer

Atomic absorption spectrometer, equipped with gas flow meters for argon (or nitrogen) and hydrogen, As, Sb and Se electrodeless discharge lamps with, background correction at measurement wavelengths and appropriate strip-chart recorder.

NOTE Certain atomic absorption atomizers and hydride reaction cells are available commercially for use with the sodium borohydride reagent.

C.4.3 Atomizer

Use one of the following:

— Boling-type burner [10)] head for argon (or nitrogen)-air entrained-hydrogen flame;

— cylindrical quartz cell, 10 cm to 20 cm long, electrically heated by external Ni-Cr wire to 800 °C to 900 °C;

— cylindrical quartz cell with internal fuel rich hydrogen-oxygen (air) flame.

The transparency of quartz cells deteriorates over several months of use. The transparency can be restored by treatment with 40 % hydrofluoric acid (HF).

SAFETY PRECAUTIONS: Be careful in handling HF which is toxic and corrosive and avoid prolonged contact of quartz with HF.

C.4.4 Reaction cell for producing As, Sb or Se hydrides

An example of reaction cell is given in Figure C.1.

[10)] Boling is the name of the inventor of this type of burner for rapid combustion of the hydrides.

NOTE A commercially available system is acceptable if it utilizes liquid sodium borohydride reagents; accepts samples digested in accordance with C.5.3, accepts between 4 mol/l to 6 mol/l (HCl); and is efficiently and precisely stirred by the purging gas and/or a magnetic stirrer.

Irrespective of the hydride reaction cell-atomizer system selected, it shall meet the following quality-control considerations:

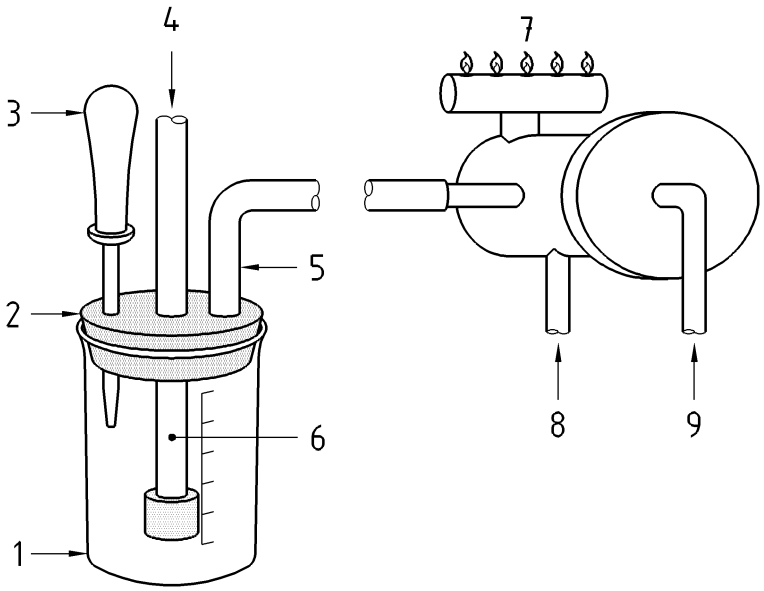
a) it shall provide a precise and reproducible calibration curve between 0 µg/l and 20 µg/l As, Sb or Se and a detection limit between 0,1 µg/l and 0,5 µg/l As, Sb or Se;

b) when carried through the entire procedure, oxidation state couples (As(III) - As(V) or Se(IV) - Se(VI)) shall cause equal instrument response; and

c) sample digestion shall yield 90 % or greater recovery of added As(III), As(V), Se(VI), Se(IV) or Sb.

C.4.5 Dropper and syringe, capable of delivering 0,5 ml to 3,0 ml sodium borohydride reagent.

Exact and reproducible addition is required so that production of hydrogen gas does not vary significantly between determinations.



Key

1	beaker 250ml	6	gas dispersion tube
2	rubber stopper	7	burner
3	dropper	8	hydrogen
4	auxiliary nitrogen	9	nitrogen
5	outlet tube		

Figure C.1 — Reaction cell for producing As, Sb or Se hydrides

C.5 Procedure

C.5.1 Preparation of the apparatus

Connect the inlet of the reaction cell with the auxiliary purging gas controlled by a flow meter. If a drying cell between the reaction cell and the atomizer is necessary, use only anhydrous $CaCl_2$ and not $CaSO_4$, because it can retain SeH_2. Before using the hydride generation/analysis system, optimize operating parameters. Aspirate aqueous solutions of As, Sb and Se directly into the flame to facilitate atomizer alignment. Align quartz atomizers for maximum absorbance. Establish a purging gas flow, concentration and rate of addition of sodium borohydride reagent, solution volume, and stirring rate for optimum instrument response for the chemical species to be analysed. If a quartz atomizer is used, optimize cell temperature. If sodium borohydride reagent is added too quickly, rapid evolution of hydrogen will unbalance the system. If the volume of solution being purged is too large, the absorption signal will be decreased. Recommended wavelengths are 193,7 nm, 196,0 nm and 217,6 nm for As, Se and Sb, respectively.

C.5.2 Preparation of calibration solutions

Transfer 0,00 ml; 1,00 ml; 2,00 ml; 5,00 ml; 10,00 ml; 15,00 ml and 20,00 ml of standard solutions of As(III), Se(IV) or Sb to 100 ml volumetric flasks and make up to volume with water containing the same acid concentration used for sample preservation (commonly 2 ml to 5 ml nitric acid (C.3.5)).This yields calibrations solutions of 0 µg/l, 1 µg/l, 2 µg/l, 5 µg/l, 10 µg/l, 15 µg/l and 20 µg/l As, Se or Sb. Prepare fresh daily.

C.5.3 Preparation of test solutions and standard solutions

Add 50 ml of the sample or As(III), Se(VI) or Sb standard solution to 250 ml beaker. Alternatively, prepare standard solutions by adding aliquots of solutions containing 5 µg As, Se or Sb directly to the beaker and dilute to 50 ml in this beaker, thus achieving a concentration of 100 µg/l of the respective solutions. Add 7 ml sulfuric acid $c(H_2SO_4)$ = 9 mol/l (C.3.3) and 5 ml nitric acid (C.3.5). Add a small boiling chip or glass beads if necessary. Evaporate to SO_3 fumes. Maintain oxidizing conditions at all times by adding small amounts of nitric acid, to prevent solution from darkening.

Maintain an excess of nitric acid until all organic matter is destroyed. Complete digestion is usually indicated by a light-coloured solution. Cool slightly, add 25 ml of water and 1 ml of perchloric acid (C.3.6) and again evaporate to SO_3 fumes to expel oxides of nitrogen.

Monitor the effectiveness of the digestion procedure used by adding 5 ml of a standard arsenic solution, 5 ml of a standard selenium solution or 5 ml of a standard antimony solution to 50 ml of the sample and measuring recovery. Average recoveries shall be greater than 90 %. Alternatively, use 100 ml micro-Kjeldahl flasks for the digestion of total recoverable arsenic, selenium or antimony, thereby improving digestion effectiveness. After the final evaporation of the SO_3 fumes, dilute to 50 ml for arsenic measurements or 30 ml for selenium and antimony measurements.

C.5.4 Determination of arsenic with sodium borohydride

To 50 ml of the digested standard solution or test solution in a 250 ml beaker (see Figure C.1) add 5 ml hydrochloric acid (C.3.7) and mix. Add 5 ml sodium iodide pre-reductant solution (C.3.2), mix and wait at least 30 min.

NOTE The sodium iodide has not been found necessary for certain hydride reaction cell designs if a 20 % to 30 % loss in instrument sensitivity is not important and variables of solution acid conditions, temperatures, and volumes for production of As(V) and arsine can be strictly controlled. This might require an automated delivery system.

Attach one beaker the first time to the rubber stopper containing the gas dispersion tube for the purging gas, the sodium borohydride reagent inlet, and the outlet to the atomizer. Turn on strip-chart recorder and wait until the base line is established by the purging gas and all air is expelled from the reaction cell. Add 0,5 ml sodium borohydride reagent (C.3.1). After the instrument absorbance has reached a maximum and returned to the base line, remove the beaker, rinse the dispersion tube with water, and proceed to the next test solution or

standard solution. Periodically compare standard As(III) and As(V) curves for response consistency. Check for the presence of chemical interferences that suppress instrument response for arsine by treating a digested sample with 10 µg/l As(III) or As(V) as appropriate. Average recoveries shall be not less than 90 %.

C.5.5 Determination of selenium with sodium borohydride

To 30 ml of the digested standard solution or test solution, or to 30 ml of the undigested standard, or the sample in a 250 ml beaker, add 15 ml hydrochloric acid (C.3.7) and mix. Heat for a pre-determined period at a temperature between 90 °C to 100 °C. Alternatively autoclave at 121 °C in capped containers for 60 min, or heat for a predetermined time in open test tubes using a 90 °C to 100 °C hot water bath or an aluminium block digester. Check the effectiveness of the selected heating by demonstrating equal instrument responses for calibration curves prepared either from standard Se(IV) or from Se(VI) solutions. Effective heat exposure for converting Se(VI) to Se(IV), with no loss of Se(IV), ranges between 5 min to 60 min when open beakers or test tubes are used. Do not digest standard Se(IV) and Se(VI) solutions used for this check of equivalency. After pre-reduction of Se(VI) and Se(IV) attach beakers, one at a time, to the purge apparatus. For each, turn on the strip-chart recorder and wait until the base line is established. Add 0,50 ml sodium borohydride reagent (C.3.1). After the instrument absorbance has reached a maximum and returned to the base line, remove the beaker, rinse the dispersion tube with water and proceed to the next test solution or standard solution. Check for the presence of chemical interferences that suppress the selenium hydride instrument response by treating a digested sample with 10 µg/l Se(IV). Average recoveries shall be not less than 90 %.

C.5.6 Determination of antimony with sodium borohydride

To 30 ml of the digested standard solution or the test solution, or to 30 ml of the undigested standard solution, or to the test solution in a 250 ml beaker, add 15 ml hydrochloric acid (C.3.7) and mix. Heat for a predetermined period (between 5 min and 60 min) between 90 °C to 100 °C. After the pre-reduction of Sb, attach beakers, one at a time, to the purge apparatus. For each, turn on the strip-chart recorder and wait until the base line is established. Add 0,50 ml sodium borohydride reagent (C.3.1). After the instrument absorbance has reached a maximum and has returned to the base line, remove beaker, rinse the dispersion tube with water and proceed to the next test solution or standard solution. Check for the presence of chemical interferences that suppress the antimony hydride instrument response by treating a digested sample with 10 µg/l Sb. Average recoveries shall be not less than 90 %.

C.6 Calculation

Determine the calibration curve by plotting the peak heights of standard solutions versus concentration. Measure the peak heights of samples and read concentrations from the calibration curve. If sample was diluted (or concentrated) before sample digestion, apply an appropriate factor.

C.7 Repeatability limit

The absolute difference between two single test results, obtained under repeatability conditions, shall not be greater than the repeatability value, r, as calculated from the following formula:

$$r = 0{,}001\ z \qquad \text{(C.1)}$$

where

z is the mean of the two results, expressed in mass fraction in percent (%).

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.

Annex D
(informative)

Environmental, health and safety precautions within chemical laboratory

When preparing the analytical methods for the application of this document, consideration was given to minimize any environmental impacts caused by the methods of analysis.

It is the users' responsibility to use safe and proper techniques when handling materials during the methods of analysis specified in this document.

The following list is not exhaustive, however users of the analytical methods referred in this document may use it as a guide for the use of safe and proper techniques. They should:

— investigate if European Directives, transposed European legislation and national laws, regulations and administrative provisions apply;

— consult manufacturers / suppliers for specific details such as material safety data sheets and other recommendations;

— use safety equipment and wear protective clothing, usually goggles and coats, appropriate for the test product and the test chemicals, in all laboratory areas, to ensure the safety of the operator;

— be careful about flammable materials and substances that are toxic and/ or human carcinogens and generally take care during transportation, decanting, diluting and dealing with spillages;

— use a fume cupboard during preparation of organic solvent solutions;

— store, handle and dispose of chemicals in a safe and environmentally satisfactory manner: including chemicals for laboratory test, test specimens, unused solvents and reagents that have to be disposed of.

Bibliography

[1] Directive 98/8/EC of the European Parliament and of the Council on the placing on the market of biocidal products

[2] 98/83/EC: Council Directive of 3rd November 1998 on the quality of water intended for human consumption

[3] Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006 (REACH)

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