SLOVENSKI STANDARD
SIST EN 12829:2011

01-junij-2011

Nadomešča:
SIST EN 12829:1997

Površinsko aktivne snovi - Priprava vode z znano kalcijevo in magnezijevo trdoto

Surface active agents - Preparation of water with known calcium and magnesium hardness

Grenzflächenaktive Stoffe - Herstellung von Wasser mit bekannter Calcium- und Magnesiumhärte

Agents de surface - Préparation d'une eau de dureté calcique et magnésienne déterminée

Ta slovenski standard je istoveten z: **EN 12829:2011**

ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

SIST EN 12829:2011 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 12829

April 2011

ICS 71.100.40

Supersedes EN 12829:1997

English Version

Surface active agents - Preparation of water with known calcium and magnesium hardness

Agents de surface - Préparation d'une eau de dureté calcique et magnésienne déterminée

Grenzflächenaktive Stoffe - Herstellung von Wasser mit bekannter Calcium- und Magnesiumhärte

This European Standard was approved by CEN on 26 February 2011.

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-CENELEC 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-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, 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.

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 12829:2011: E

Contents

Page

Foreword

This document (EN 12829:2011) has been prepared by Technical Committee CEN/TC 276 “Surface active agents”, 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 October 2011, and conflicting national standards shall be withdrawn at the latest by October 2011.

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 12829:1997.

The new edition includes the preparation of water with known magnesium hardness.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method of preparing water of known calcium and magnesium hardness for use in testing surface active agents and products containing them.

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 385, *Laboratory glassware – Burettes (ISO 385:2005)*

EN ISO 648, *Laboratory glassware – Single-volume pipettes (ISO 648:2008)*

EN ISO 1042, *Laboratory glassware – One-mark volumetric flasks (ISO 1042:1998)*

EN ISO 3696:1995, *Water for analytical laboratory use – Specification and test methods*

ISO 1773, *Laboratory glassware – Narrow-necked boiling flasks*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
water hardness

Measure for the content of dissolved calcium and magnesium salts and, in special cases, salts of strontium and/or barium

NOTE 1 The unit of measurement of water hardness is the millimole per litre (mmol/l). 1 mmol/l of calcium hardness corresponds to 40,08 mg/l of calcium ions. 1 mmol/l of magnesium hardness corresponds to 24,31 mg/l of magnesium ions.

NOTE 2 The equivalents for other degrees of hardness of water, as well as other units used for measuring water hardness and the relationships between them, are given for information in Annex A.

NOTE .3 Originally, the hardness of a sample of water was determined by measuring its power to destroy the foam formed by soap. This property is primarily due to the presence of calcium and magnesium, but salts of other metals, such as iron, aluminium and manganese, behave in a similar manner, although these seldom occur in natural waters.

4 Principle

Preparation of a stock solution by dissolving an appropriate quantity of calcium chloride or magnesium chloride in water. Determination of the calcium or magnesium ions in this stock solution by complexometric titration with the disodium salt of (ethylenedinitrilo)tetraacetic acid (EDTA) using a mixture of Mordant Black 11 (C.I. 14645) and methyl red as indicator.

Preparation of dilute solutions, of the hardness required, by dilution of appropriate volumes of the stock solution.

5 Reagents

5.1 General

WARNING — Your attention is drawn to the regulations covering the handling of hazardous substances. Technical, organisational and personal protection measures should be observed.

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade and have been checked in advance as to not interfere with the analytical results and water complying with grade 3 as defined in EN ISO 3696:1995.

5.2 Calcium chloride dihydrate ($CaCl_2.2H_2O$), (CAS number: 10035-04-8)

If the dihydrate is not available, use an equivalent quantity of another hydrate or the anhydrous salt.

5.3 Magnesium chloride hexahydrate ($MgCl_2.6H_2O$), (CAS number: 7791-18-6)

If the hexahydrate is not available, use an equivalent quantity of another hydrate or the anhydrous salt.

5.4 Ammonia solution

Dilute 57 ml of ammonia solution (ρ_{20} 0,90 g/ml) and 1 g of potassium cyanide[1)] with water to 100 ml.

5.5 Disodium salt of EDTA (Na_2EDTA), standard volumetric solution, c($Na_2EDTA.2H_2O$) = 0,05 mol/l, (CAS number : 60-00-4)

Dissolve 18,612 g of the disodium salt of (ethylenedinitrilo)tetraacetate (EDTA) dihydrate in water in a 1 000 ml one-mark volumetric flask (see 6.1), and make up to the mark with water.

1 ml of this solution, which is stable, is equivalent to 0,05 mmol, i.e. 2,004 mg, of calcium ions or 1,216 mg, of magnesium ions.

5.6 Mixed indicator

5.6.1 Preparation of magnesium-disodium salt of EDTA ($MgNa_2EDTA$), hexahydrate

Dissolve 18,6 g of disodium (ethylenedinitrilo)tetraacetate dihydrate in 75 ml of very hot water (> 80 °C).

To this solution add 12,3 g of magnesium sulfate heptahydrate dissolved in 25 ml of very hot water (> 80 °C). Mix the two solutions thoroughly, cover the mixture and let it cool overnight. Pour off the supernatant solution and wash the residue three times with cold water, pouring off the washings each time.

Wash the crystals with water in a Buchner funnel and dry them under reduced pressure in a desiccator, or in an oven at a temperature of 85 °C.

5.6.2 Preparation of mixed indicator

Grind 200 mg of Mordant Black 11[2)] (C.I. 14645) and 37 mg of methyl red with 50 g of ammonium chloride. Add 150 g of ammonium chloride and 10 g of $MgNa_2EDTA$ (see 5.5), and continue grinding until a homogeneous mixture is obtained. Store the mixed indicator in a glass bottle with a ground-glass stopper.

---

1) Potassium cyanide solution may be destroyed by treatment with sodium hypochlorite and hydrogen peroxide.

2) For example Eriochrome Black T.

NOTE 1 The mixed indicator is prepared and stored as a dry powder because solutions of Mordant Black 11 are unstable; it is used in the ground state with ammonium chloride and it reacts with magnesium ions.

NOTE 2 The inclusion of $MgNa_2EDTA$ allows the indicator to react with calcium or magnesium ions, whilst the addition of methyl red enhances the colour change at the end-point of the titration.

NOTE 3 It is also possible to use buffered indicator tablets instead of the mixed indicator; the colour change is from red to green, via grey.

6 Apparatus

Ordinary laboratory apparatus and the following :

6.1 One-mark volumetric flasks, capacity 250 ml and 1 000 ml, complying with EN ISO 1042.

6.2 One-mark pipettes, capacity 25 ml and 50 ml, complying with EN ISO 648.

6.3 Bottle, capacity 5 litres, made of dark-brown glass, with a ground-glass stopper.

6.4 Conical flask, capacity 250 ml, complying with ISO 1773.

6.5 Burette, capacity 50 ml, class A, complying with EN ISO 385.

6.6 Analytical balance.

7 Procedure

7.1 Preparation of stock solution

7.1.1 Calcium stock solution

Dissolve 220,5 g of calcium chloride dihydrate (see 5.2) in water, dilute to 5 litres and store in the bottle (see 6.3).

Water of the required calcium hardness can be prepared by dilution from this solution which contains about 300 mmol/l of calcium ions.

7.1.2 Magnesium stock solution

Dissolve 305,0 g of magnesium chloride hexahydrate (see 5.2) in water, dilute to 5 litres and store in the bottle (see 6.3).

Water of the required magnesium hardness can be prepared by dilution from this solution which contains about 300 mmol/l of magnesium ions.

7.2 Determination of calcium or magnesium content of stock solution

Take 50 ml of the stock solution prepared as described in 7.1, using a 50 ml pipette (see 6.2), transfer it to a 250 ml one-mark volumetric flask (see 6.1) and make up to the mark with water.

Take 25 ml of the stock solution, using a 25 ml pipette (see 6.2), and transfer to the conical flask (see 6.4). Dilute with about 100 ml of water, and add 4 ml of ammonia solution (see 5.4) from a measuring cylinder and 0,3 g of the mixed indicator (see 5.6). Heat the mixture to about 40 °C and titrate it with Na_2EDTA solution (see 5.5) to the end-point colour change to green.

Calculate the calcium or magnesium content c_0 of the stock solution, expressed in millimoles of calcium or magnesium ions per litre, using the equation:

$$c_0 = 0,05 \times V \times \frac{250}{25} \times \frac{1000}{50} = 10 \times V$$

where

V is the volume, in millilitres, of Na_2EDTA solution (see 5.3) used for the titration;

0,05 is the actual concentration, expressed in moles of $Na_2EDTA.2H_2O$ per litre, of this solution.

7.3 Preparation of water of known calcium or magnesium hardness

Calculate the volume V_0, expressed in millilitres, of stock solution required to prepare a given volume of solution of known calcium or magnesium hardness from the equation:

$$V_0 = \frac{V_1 c_1}{c_0}$$

where

V_1 is the required volume, in millilitres, of water of known hardness;

c_0 is the hardness, in millimole per litre of calcium or magnesium ions, of the stock solution calculated in 7.2;

c_1 is the required hardness, in millimole per litre of calcium or magnesium ions, of the solution of volume V_1.

Choose the volume V_1 so that it corresponds to the capacity of a one-mark volumetric flask, and in such a way V_0 is more than 10 ml and less than 50 ml.

Fill the burette (see 6.5) with the stock solution (see 7.1).

Transfer the calculated volume V_0, measured to the nearest 0,1 ml, of the stock solution to a one-mark volumetric flask of capacity V_1 and make up to the mark with water.