SLOVENSKI STANDARD
SIST EN 12829:1997

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Površinsko aktivne snovi - Priprava vode z znano kalcijevo trdoto (ISO 2174:1990 spremenjen)

Surface active agents - Preparation of water with known calcium hardness (ISO 2174:1990 modified)

Grenzflächenaktive Stoffe - Herstellung von Wasser mit bekannter Calciumhärte (ISO 2174:1990 modifiziert)

Agents de surface - Préparation d'une eau de dureté calcique détermination (ISO 2174:1990 modifiée)

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71.100.40 Površinsko aktivna sredstva Surface active agents

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English version

Surface active agents - Preparation of water with known calcium hardness (ISO 2174:1990 modified)

Agents de surface - Préparation d'une eau de dureté calcique déterminée (ISO 2174:1990 modifiée)

Grenzflächenaktive Stoffe - Herstellung von Wasser mit bekannter Calciumhärte (ISO 2174:1990 modifiziert)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This European Standard 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of an identical text or by endorsement, at the latest by March 1998, and conflicting national standards shall be withdrawn at the latest by March 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 2174:1990 has been approved by CEN as European Standard with agreed common modifications as given below :

- Deletion of the subclause 1.2 of ISO 2174:1990.

1 Scope

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

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 385-1	Laboratory glassware - Burettes - Part 1 : General requirements
ISO 648	Laboratory glassware - One-mark pipettes
ISO 1042	Laboratory glassware - One-mark volumetric flasks
ISO 1773	Laboratory glassware - Boiling flasks (narrow-necked)
ISO 3696	Water for analytical laboratory use - Specification and test methods

3 Definition

For the purposes of this standard, the following definition applies :

water hardness : The property resulting from the presence of calcium and magnesium salts and, in special cases, salts of strontium and/or barium.

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 of calcium ions per litre.

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 : 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 in water. Determination of the calcium 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

All reagents shall be of recognized analytical grade and the water used shall be grade 3 as defined in ISO 3696.

5.1 Calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$)

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

5.2 Ammonia solution

WARNING : Comply with safety instructions for the handling of poisonous substances.

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

5.3 Disodium salt of EDTA (Na_2EDTA), standard volumetric solution, $c(Na_2EDTA \cdot 2H_2O)$ = 0,05 mol/l.

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.

5.4 Mixed indicator

5.4.1 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.

To this solution add 12,3 g of magnesium sulfate heptahydrate dissolved in 25 ml of very hot water. 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.

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

5.4.2 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 5.4.1), and continue grinding until a homogeneous mixture is obtained. Store the mixed indicator in a glass bottle with a ground-glass stopper.

5.4.3 Notes

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

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

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

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

6.2 One-mark pipettes, capacity 25 ml and 50 ml, complying with 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 ISO 385-1.

6.6 Analytical balance.

7 Procedure

7.1 Preparation of stock solution

Dissolve 220,5 g of calcium chloride dihydrate (see 5.1) in water, dilute to 5 litres and store in the bottle (see 6.3).

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2) For example Eriochrome Black T.

From this solution, which contains about 300 mmol of calcium ions per litre, water of the required calcium hardness can be prepared by dilution.

7.2 Determination of calcium content of stock solution

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

Using a 25 ml pipette (see 6.2), take 25 ml of this solution and transfer it to the conical flask (see 6.4). Dilute with about 100 ml of water, and add 4 ml of ammonia solution (see 5.2) from a measuring cylinder and 0,3 g of the mixed indicator (see 5.4). Heat the mixture to about 40 °C and titrate it with Na_2EDTA solution (see 5.3) to the end-point colour change to green.

Calculate the calcium content c_0 of the stock solution, expressed in millimoles of calcium 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 $Na_2EDTA \cdot 2H_2O$ per litre, of this solution.

7.3 Preparation of water of known calcium hardness

Calculate the volume V_0, expressed in millilitres, of stock solution required to prepare a given volume of solution of known calcium 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 millimoles of calcium ions per litre, of the stock solution calculated in 7.2 ;

c_1 is the required hardness, in millimoles of calcium ions per litre, 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.