



# SLOVENSKI STANDARD

## SIST EN 14669:2005

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Surface active agents - Determination of anionic surface active agents and soaps in  
detergents and cleansers - Potentiometric two-phase titration method

Grenzflächenaktive Stoffe - Bestimmung des Gehaltes an anionischen  
grenzflächenaktiven Stoffen und Seifen in Detergenzien und Reinigern -  
Potentiometrische Zweiphasen-Titration

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Agents de surface - Détermination des agents de surface anioniques et des savons dans  
les détergents et produits de nettoyage - Méthode de titrage potentiométrique dans deux  
phases

Ta slovenski standard je istoveten z: EN 14669:2005

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**ICS:**

71.100.40 Površinsko aktivna sredstva Surface active agents

**SIST EN 14669:2005**

en

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EUROPEAN STANDARD

EN 14669

NORME EUROPÉENNE

EUROPÄISCHE NORM

June 2005

ICS 71.100.40

English version

## Surface active agents - Determination of anionic surface active agents and soaps in detergents and cleansers - Potentiometric two-phase titration method

Agents de surface - Détermination des agents de surface anioniques et des savons dans les détergents et produits de nettoyants - Méthode de titrage potentiométrique dans deux phases

Grenzflächenaktive Stoffe - Bestimmung des Gehaltes an anionischen grenzflächenaktiven Stoffen und Seifen in Detergenzien und Reinigern - Potentiometrische Zweiphasen-Titration

This European Standard was approved by CEN on 19 May 2005.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

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## Contents

	Page
Foreword .....	3
1 Scope .....	4
2 Normative references .....	4
3 Principle.....	4
4 Reagents.....	4
5 Apparatus .....	6
6 Sampling and preparation of the test solution .....	7
7 Procedure .....	7
8 Calculation and expression of results.....	9
9 Precision.....	9
10 Test report .....	10
Annex A (informative) Titration apparatus settings.....	11
Annex B (informative) Potentiometric two-phase titration typical titration curve.....	13
Annex C (informative) Results of inter-laboratory test.....	14
Bibliography.....	16

SIST EN 14669:2005  
<https://standards.iteh.ai/catalog/standards/sist/1f9e2682-9561-48de-b292-a384f5a0dd4a/sist-en-14669-2005>

## Foreword

This European Standard (EN 14669:2005) 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 December 2005, and conflicting national standards shall be withdrawn at the latest by December 2005.

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

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## 1 Scope

This European Standard specifies a method for the determination of the content of anionic surface active agents and soaps in detergents and cleansers, defined as being the amount of anionic surface active agents expressed in millimoles per 100 g of product.

NOTE 1 The applicability in products different that those tested should be checked in each particular case.

NOTE 2 In comparison to usual laboratory two-phase titration with visual endpoint determination (see ISO 2271), potentiometric titration offers the advantage of automation; operator-dependent differences in recognising the equivalence point can be neglected, and a non-critical solvent replaces the toxicologically critical chloroform.

## 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 14670, *Surface active agents – Sodium dodecyl sulfate – Analytical method.*

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

ISO 607, *Surface active agents and detergents – Methods of sample division.*

## 3 Principle

Anionic surface active agents and soaps are combined with cationic surface active agents to form water-insoluble ion pairs which are immediately extracted into a water immiscible organic solvent. This fundamental reaction is the basis for the titration of equivalents of ionic surface active agents with an oppositely charged surface active agent standard volumetric solution in the two-phase titration.

This procedure is supported by intensively stirring the two-phase mixture of aqueous solution and organic phase. The potential, which is formed in the emulsion during the titration, is recorded with the help of a special solvent-resistant surface active agent-sensitive electrode in combination with a silver/silver chloride reference electrode against the amount of titrant added. The equivalence point of the added cationic surface active agent corresponds to that one of the test solution at the inflection point of the titration curve (Annex B).

The titration is carried out twice, once under acidic conditions for the determination of the anionic surface active agents and then under alkaline conditions in order to determine the sum of soaps and anionic surface active agents. The soap concentration is calculated from the difference of the titrant consumptions.

## 4 Reagents

**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 recognised analytical grade that have been checked in advance as to not interfere with the analytical results.

**4.1 Water**, complying with grade 3 as defined in EN ISO 3696.

NOTE If the water is purified via ion-exchange resins, ensure that no cationic or anionic species from the resins cause interference.

**4.2 Sodium dodecyl sulfate**,  $C_{12}H_{25}OSO_3Na$ , % (m/m)( $C_{12}H_{25}SO_4Na$ )  $\geq 99$  as determined following the method EN 14670.

**4.3 Anionic surface active agent** standard volumetric solution,  $c(C_{12}H_{25}OSO_3Na) = 0,005$  mol/l.

Weigh 1,455 g of sodium dodecyl sulfate (4.2) with a known active content to the nearest 1 mg, in a conical flask, and dissolve in about 500 ml water. Transfer quantitatively the solution into a 1000 ml volumetric flask and make up to the mark with water and mix well.

The concentration of the anionic surface active agent standard volumetric solution,  $c_a$ , expressed in millimoles per millilitre is calculated in accordance with the following equation (1):

$$c_a = \frac{m \times w}{M \times 100} \quad (1)$$

where

$m$  is the mass of sodium dodecyl sulfate (4.2) in grams;

$w$  is the active matter content of sodium dodecyl sulfate (4.2) in grams per 100 g;

$M$  is the molar mass of sodium dodecyl sulfate, in grams per mole (288,4 g/mol).

**4.4 1,3-didecyl-2-methylimidazolium chloride** (e.g. TEGO Trant A100<sup>1)</sup>).

**4.5 Cationic surface active agent**, standard volumetric solution,  $c = 0,005$  mol/l:

Weigh 2,00 g of 1,3-didecyl-2-methylimidazolium chloride (4.4), to the nearest 1 mg, in a conical flask and dissolve it in about 500 ml water. Transfer quantitatively the solution into a 1000 ml volumetric flask, make up to the mark with water and mix well. Standardize the solution as specified in 7.1.

Standardize the cationic surface active agent solution only when the solution has reached its equilibrium, i.e. after standing for at least one day.

NOTE Cationic surface active agents adsorb on glass surfaces like such of the burette and reagent bottle of the titration unit.

**4.6 Potassium chloride** solution,  $c(KCl) = 3$  mol/l.

**4.7 Hydrochloric acid**,  $c(HCl) = 0,5$  mol/l.

**4.8 Sodium hydroxide** solution,  $c(NaOH) = 0,5$  mol/l.

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1) TEGO Trant A100 is the trade name of product supplied by Metrohm Ltd. (CH-9101 Herisau, Switzerland). This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Equivalent product may be used if it can be shown to lead to the same results.

## EN 14669:2005 (E)

**4.9 Emulsifier** (TEGO Add<sup>2</sup>).

NOTE The emulsifier has the task of supporting the formation of a stable emulsion and at the same time of preventing the deposition of the ion associate formed during the titration on the electrode surface.

**4.10 Propan-2-ol (Isopropanol)** ( $C_3H_8O$ )  $\geq 99$  %.**4.11 Ethanol** denatured 96% (V/V).**4.12 Methyl isobutyl ketone** (MIBK) (CAS number: 108.10.1), 4-methyl-2-pentanone, % (m/m) ( $C_6H_{12}O$ )  $\geq 99$ .**4.13 MIBK / propan-2-ol - mixture**

Measure 600 ml MIBK (4.12) and 400 ml propan-2-ol (4.10) using a measuring cylinder, transfer into a 1000 ml flask and mix well.

**5 Apparatus**

Normal laboratory apparatus and the following:

**5.1 Automatic potentiometric titration apparatus**, with drift-controlled data acquisition and dynamic titrimetric dosing equipped with a piston burette delivery system of 20 ml capacity.

**5.2 Propeller stirring system.**

In a potentiometric two-phase titration a thorough blending is required. Hence, a stirring propeller is compulsory. The stirrer should be constructed so that an optimal emulsification of the vessel contents is achieved with a simultaneous low degree of air entrapment. Propeller stirrers shaped like ship screws have proven effective, while magnetic stirrers are not suitable. It is advisable to pay special attention to the geometric arrangement of the immersing parts (electrodes, burette tip, and stirrer). If arranged optimally, no foam is produced, not even with heavy stirring.

**5.3 Combined glass pH-electrode.**

**5.4 Solvent-resistant- surface active agent -sensitive electrode** (Surfactrode Refill or Surfactrode Resistant<sup>3</sup>).

**5.5 Ag/AgCl- double-junction ground joint diaphragm reference electrode**, inner and outer chambers filled with potassium chloride solution (4.6).

2) TEGO Add is the trade name of product supplied by Metrohm Ltd. (CH-9101 Herisau, Switzerland). This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Equivalent product may be used if it can be shown to lead to the same results.

3) Surfactrode Refill and Surfactrode Resistant are trade names of products supplied by Metrohm Ltd. (CH-9101 Herisau, Switzerland). This information is given for the convenience of users of this document and does not constitute an endorsement by CEN. Equivalent products may be used if they can be shown to lead to the same results.



## 6 Sampling and preparation of the test solution

### 6.1 Sampling

The sample shall be taken and stored in accordance with ISO 607.

### 6.2 Preparation of the test solution

Powder detergents and powdery cleansers shall be homogenised by additional milling if amounts lower than 10 g are used.

The sample amount and the concentration of test solution shall be calculated in a way that the consumption of the titrant solution (4.5) used for the titration of 10 ml test solution is approximately 10 ml.

Weigh, to the nearest 0,1 mg, the homogenised sample ( $m_1$ ) in a glass beaker and add approximately 100 ml water. Stir the mixture using a magnetic stirrer until the substance is dissolved and possibly insoluble product ingredients are suspended homogeneously in the solution. For soap raw material heating is necessary to achieve complete dissolution. For fatty acids instead of water, ethanol is used as solvent.

Transfer quantitatively the sample solution into a 200 ml volumetric flask, to minimise foaming add a small quantity of ethanol (4.11). Make up to the mark with water and mix well.

## 7 Procedure

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### 7.1 Standardisation of the cationic surface active agent standard volumetric solution

Accurately transfer 10 ml of the sodium dodecyl sulfate standard volumetric solution (4.3) into the titration vessel and add about 70 ml of water.

Adjust the pH to  $3 \pm 0,2$  with the hydrochloric acid solution (4.7).

Add 10 ml of methyl isobutyl ketone (MIBK) (4.12), 10 ml of ethanol (4.11) and 200  $\mu$ l of emulsifier (4.9). Stir the mixture intensively for 60 s in order to form a stable emulsion.

Carry out the titration with the cationic surface active agent standard volumetric solution (4.5) under intensive stirring (5.2).

Record the reagent consumption,  $V$ , at the inflection point of the titration curve (see Figure B.1).

NOTE Examples for instrument settings are given in Annex A.