SLOVENSKI STANDARD
SIST EN 14981:2007

01-april-2007

Površinsko aktivne snovi - Določanje vsebnosti topil z visokim vreliščem v tekočih detergentih s plinsko tekočinsko kromatografijo (GLC)

Surface active agents - Determination of content of high boiling solvents in liquid detergents by GLC

Grenzflächenaktive Stoffe - Bestimmung des Gehaltes an hochsiedenden Lösemitteln in flüssigen Reinigungsmitteln durch GLC

Agents de surface - Détermination de la teneur en solvants a point d'ébullition élevé dans les détergents liquides par chromatographie en phase gazeuse

Ta slovenski standard je istoveten z: **EN 14981:2006**

ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

SIST EN 14981:2007 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14981

October 2006

ICS 71.100.40

English Version

Surface active agents - Determination of content of high boiling solvents in liquid detergents by GLC

Agents de surface - Détermination de la teneur en solvants à point d'ébullition élevé dans les détergents liquides par chromatographie en phase gazeuse

Grenzflächenaktive Stoffe - Bestimmung des Gehaltes an hochsiedenden Lösemitteln in flüssigen Reinigungsmitteln durch GLC

This European Standard was approved by CEN on 28 August 2006.

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.

CEN members are the national standards bodies of Austria, Belgium, 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: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 14981:2006: E

Contents

Page

Foreword

This document (EN 14981:2006) 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 April 2007, and conflicting national standards shall be withdrawn at the latest by April 2007.

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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard specifies a method for the identifying and quantifying of high boiling point solvents in finished liquid detergents and raw materials.

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.

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

3 Term and Definition

For the purposes of this European Standard, the following term and definition applies.

high boiling solvent
solvent, mainly a glycol and glycol ether product, with a boiling point significantly higher than water (100° C)

4 Principle

The organic solvents are determined by gas chromatography. The sample is dissolved in ethanol and injected into a polar phase capillary column and the unknown solvent is identified by its retention time. After qualitative determination, the solvent is quantified using (-) Carvone (2-Methyl-5-(1-methylethenyl)-2-cyclohexene-1-one) as internal standard.

5 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade that have been checked in advance so as not to interfere with the analytical results.

WARNING Some reagents used throughout this procedure are toxic. Care should be taken not to inhale the vapours. Contact with the skin should also be avoided. Safety glasses and gloves should be worn when handling the reagents. Waste solvent disposal should be carried out in accordance with safety and environmental regulations.

5.1 Ethanol (CAS number: 64-17-5).

5.2 (-) Carvone (2-Methyl-5-(1-methylethenyl)-2-cyclohexene-1-one), minimum purity 99,5 %
(CAS number : 6485-40-1)

5.3 Solvents to be determined (see example in Annex A)

WARNING Some solvents may exist as different isomers.

5.4 Carrier gas for gas chromatography

5.5 Auxiliary gas for gas chromatography

6 Apparatus

Ordinary laboratory apparatus and the following:

6.1 Gas chromatograph equipped with split/splitless injection port and flame ionization detector (FID).

6.2 Electronic integrator or, preferably, a suitable data acquisition system.

6.3 Capillary column, capable of the separation characteristics shown in Figures A.1 and A.2.

6.4 Glass tube, with a capacity of at least 40 ml.

NOTE A 30 m x 0,25 mm internal diameter, fused silica capillary column (film thickness 0,25 µm) with 100 % polyethylene glycol stationary phase is advisable.

7 Sampling and preparation of the sample

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

8 Procedure

8.1 Gas chromatographic conditions

The following GC conditions have been found to be suitable. At least the quality of separation shown in Figures A.1 and A.2 shall be achieved.

— Oven temperature programs:

  — qualitative analysis: 60°C (5 min) to 240°C (5 min) at 5°C/min;

  — quantitative analysis: 60°C to 240°C (5 min) at 20°C/min;

— Injection: split ratio at 100:1 and temperature at 225°C;

— Detection : flame ionization detector (FID) at 275°C with nitrogen as make up gas at 25 ml/min;

— Carrier gas: hydrogen at 50 kPa head pressure.

8.2 Preparation of solutions

8.2.1 Solvent reference solution 1

Into a glass tube (6.4), weigh approximately 20 mg of each solvent as listed on chromatogram 1. Add 30 ml of ethanol and mix well.

8.2.2 Solvent reference solution 2

Into a glass tube (6.4), weigh approximately 20 mg of each solvent as listed on chromatogram 2. Add 30 ml of ethanol and mix well.

Inject 1 µl of the solvent reference solutions 1 and 2 into the gas chromatograph. Refer to the annexed chromatograms 1 and 2 for peak identities.

8.2.3 Stock solution for calibrations

Weigh to the nearest 0,1 mg, approximately 250 mg of each of the solvent(s) of interest into a 100 ml volumetric flask. Dilute to volume with ethanol, and mix well.

8.2.4 Carvone internal standard solution

Weigh to the nearest 0,1 mg, approximately 500 mg of carvone into a 100 ml volumetric flask. Dilute to volume with ethanol and mix thoroughly (5,0 mg/ml of carvone).

8.3 Calibration

Using precision glass pipettes, transfer 5,0 ml, 7,0 ml, 10,0 ml, 12,0 ml, and respectively 15,0 ml of the stock solution for calibrations into a series of glass tubes (6.4).

Using a volumetric glass pipette, add 5,0 ml of the carvone internal standard solution, make up to 30 ml with ethanol and mix well.

Inject 1 µl of each solution into the gas chromatograph.

Record the peak areas of the peaks of interest and carry out a regression analysis of (Area of solvent/Area of internal standard) versus (mass of solvent/mass of internal standard), for each solvent(s).

The response factor, K_i, for each solvent i, is the slope of the calculated regression curve.

If a correlation factor of less than 0,98 is obtained prepare new calibration solution and re-inject.

If a data acquisition system is not available, calculate the response factor for all the standard solutions according to the following formula:

$$K_{\mathrm{i}} = \frac{m_{\mathrm{s}} \times f_s \times A_{\mathrm{i}}}{m_{\mathrm{i}} \times f_i \times A_{\mathrm{s}}} \qquad (1)$$

where

m_s is the mass, in milligrams, of the internal standard ;

f_{s} is the purity of the internal standard, in % (m/m);

fi is the purity of the solvent i, in % (m/m);

A_{i} is the peak area of the solvent i;

m_{i} is the mass, in milligrams, of the solvent i;

A_{s} peak area of the internal standard.

Calculate the mean response factor of all K_i for each solvent and the relative standard deviation. If the relative standard deviation (RSD) of K_{i} is > 3 %, repeat the calibration procedure.

8.4 Sample analysis

8.4.1 Solvent identification

Into a glass tube (6.4), weigh 1g to 2 g of sample.

Add 30 ml of ethanol and mix thoroughly. Inject 1 µl of the obtained solution into the GC and allow the chromatogram to develop. Identify the solvent(s) present in the sample by comparing the retention time(s) with the reference chromatogram.

Spike the sample with 20 mg of each of the identified solvent(s). Mix the solution thoroughly and inject 1 µl of this new solution into the GC. Allow the chromatogram to develop.

Confirm the identification of the solvent(s).

8.4.2 Solvent quantification

Into a glass tube (6.4) accurately weigh sufficient sample to contain approximately 25 mg of solvent.

Using a volumetric glass pipette, add 5,0 ml of the carvone internal standard solution followed by 25 ml of ethanol and mix well.

Inject 1 µl of the ethanolic solution into the gas chromatograph.

Record the peak areas of the peaks of interest and calculate the results according to clause 9.

9 Calculation and expression of results

The content of solvent i, w_i, expressed in grams per 100 grams is calculated according to the following equation:

$$w_{\mathrm{i}} = \frac{m_0 \times f_o \times A_{\mathrm{i}}}{m \times A_0 \times K_{\mathrm{i}}} \qquad (2)$$

where

m_0 is the mass, in milligrams, of the internal standard;

f_0 is the purity of the internal standard, expressed in % (m/m);

A_{i} is the peak area of the solvent i in the sample analysis;

m is the mass, in milligrams, of the sample;

A_0 is the peak area of the internal standard in the sample analysis;

K_{i} is the response factor for solvent i, calculated in 8.3.