SLOVENSKI STANDARD
SIST EN 13268:2002

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Površinsko aktivne snovi - Določanje etilen oksidnih in propilen oksidnih skupin v etilen oksidnih in propilen oksidnih kondenzatih

Surface active agents - Determination of ethylene oxide and propylene oxide groups in ethylene oxide and propylene oxide adducts

Grenzflächenaktive Stoffe - Bestimmung von Ethylenoxid- und Propylenoxid-Gruppen in Ethylenoxid- und Propylenoxid-Addukten

Agents de surface - Détermination de la teneur en oxyde d'éthylene et en oxyde de propylene dans les condensats a base d'oxyde d'éthylene et d'oxyde de propylene

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English version

Surface active agents - Determination of ethylene oxide and propylene oxide groups in ethylene oxide and propylene oxide adducts

Agents de surface - Détermination de la teneur en oxyde d'éthylène et en oxyde de propylène dans les condensats à base d'oxyde d'éthylène et d'oxyde de propylène

Grenzflächenaktive Stoffe - Bestimmung von Ethylenoxid- und Propylenoxid-Gruppen in Ethylenoxid- und Propylenoxid-Addukten

This European Standard was approved by CEN on 19 January 2001.

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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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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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of an identical text or by endorsement, at the latest by October 2001, and conflicting national standards shall be withdrawn at the latest by October 2001.

Annex A is informative.

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.

Introduction

Under the specified reaction conditions, ethylene oxide groups convert stoichiometrically into ethyl iodide. However, the conversion of propylene oxide groups to isopropyl iodide is not stoichiometric.

1 Scope

This European Standard specifies a method for the qualitative and quantitative determination of ethylene oxide and propylene oxide groups in ethylene oxide (EO) and propylene oxide (PO) adducts, polyethers and polyglycol esters.

NOTE If a suitable calibration is performed, methoxy groups can also be determined.

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 (including amendments).

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*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results – Part 2 : Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

3 Principle

Hydroiodic acid and nonane are added to the sample to be analysed and the mixture is heated in a closed vessel for 1 h at 150 °C. Upon conversion, ethylene oxide groups form stoichiometric amounts of ethyl iodide, and propylene oxide groups form (74 % to 77 % (*m*/*m*)) of propyl iodide (mainly the iso-form, less frequently the n-form). These compounds are extracted by the nonane present and thus removed from the reaction medium.

Under the specified digestion conditions, ethyl iodide is formed from all compounds containing ethoxy groups (ethers and esters) and ethylene glycol derivatives; and isopropyl iodide is formed from all compounds containing isopropoxy groups, propylene glycol and glycerol derivatives.

The alkyl iodides are determined directly from the nonane solution by means of gas chromatography using toluene as internal standard.

Substances that contain isobutyl groups give isobutyl iodide when cleavage takes place. Isobutyl iodide cannot be exactly separated from the internal standard, toluene, in the chromatogram.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and water complying with grade 3 as defined in EN ISO 3696.

4.1 Hydroiodic acid, HI, $w(HI) \geq 6,7$ %.

4.2 Adipic acid, $C_8H_6O_4$, $w(C_8H_6O_4) \geq 98$ %.

4.3 Toluene, C_7H_8, $w(C_7H_8) \geq 99$ %.

4.4 Nonane, C_9H_{20}, $wC_9H_{20}) \geq 99$ %, GC quality.

4.5 Ethyl iodide, C_2H_5I, $w(C_2H_5I) \geq 99$ %, GC quality.

4.6 Propyl iodide, C_3H_7I, $w(C_3H_7I) \geq 98$ %.

4.7 Isopropyl iodide, C_3H_7I, $w(C_3H_7I) \geq 98$ %.

4.8 Internal standard solution. Fill a 1000 ml volumetric flask with about 200 ml nonane (4.4). Add approximately 2 g of toluene (4.3), weighed to nearest 0,01 g. Fill the flask up to the mark with nonane.

4.9 2-phenoxyethanol, $C_8H_{10}O_2$, $w(C_8H_{10}O_2) \geq 98$ %.

4.10 1,2-propanediol, $C_3H_8O_2$, $w(C_3H_8O_2) \geq 99$ %.

5 Apparatus

Ordinary laboratory apparatus and the following:

5.1 Metal heating block, thermostatically controlled, to fit the pressure resistant digestion vial (5.2), capable of maintaining a temperature of (150 ± 5) °C, or **drying cupboard**, capable of maintaining a temperature of (150 ± 5) °C.

5.2 Pressure resistant digestion vial for hydroiodic cleavage, for example as shown in Figure 1, with a screw top lined with a rubber septum onto which a protective polytetrafluorethylene foil is glued.

5.3 Syringes, with capacities of 1 µµl and 50 µµl.

5.4 Gas chromatograph, equipped with on-column injector and flame ionization detector (FID) or thermal conductivity detector (TCD).

5.5 Integrator, or **computer-based chromatography data system**.

5.6 Laboratory shaker, or magnetic stirrer suitable for the metal heating block (5.1).

6 Sampling and preparation of the test sample

6.1 General

The sample shall be taken in accordance with ISO 607 and prepared as specified in 6.2.

6.2 Digestion

Weigh approximately 50 mg of the test sample to the nearest 0,1 mg and transfer it into the pressure resistant digestion vial (5.2). Add approximately 150 mg of adipic acid (4.2), 2 ml of hydroiodic acid (4.1) and 2 ml of internal standard solution (4.8). Add a stirring bar if the laboratory shaker or the magnetic stirrer (5.6) is used.

Immediately seal the pressure resistant digestion vial tightly and either place it into the metal heating block (5.1) set at (150 ± 5) °C ((130 ± 5) °C if the laboratory shaker or the magnetic stirrer (5.6) is used) or in the drying cupboard (5.1) set at (150 ± 5) °C. Maintain the corresponding temperature for 1,5 h ± 5 min (1 h ± 5 min in case of the metal heating block (5.1) without the laboratory shaker or the magnetic stirrer (5.6)).

Cool to room temperature.

Shake vigorously in order to obtain a phase separation.

Inject with the 1 µl syringe (5.3) 0,3 µl of nonane phase into the gas chromatograph for the analysis.

NOTE 1 Strict adherence to the specified conditions is important because the digestion is relatively sensitive to temperature variations.

NOTE 2 The fully digested samples can be kept sealed up to 38 h at 4 °C. Older or tapped test samples give misleading results.

7 Procedure

7.1 Chromatography conditions

Detector :	FID or TCD ;
Injector :	on-column ;
Carrier gas :	helium ;
Column :	fused silica capillary column, coated with a stationary phase of 3 % volume units of cyanopropylpolysiloxane/3 % volume units of phenylpolysiloxane/94 % volume unit of methyl polysiloxane ;
	length : 30 m ;
	internal diameter : 0,25 mm ;
	film thickness : 1µm
Temperature programme :	60 °C, 3 min isothermal ;
	60 °C to 130 °C, 6 °C/min ;
	130 °C to 280 °C , 15 °C/min ;
	280 °C, 20 min isothermal.
Injector temperature :	130 °C
Detector temperature :	200 °C

NOTE If the separation is comparable to that demonstrated by the chromatogram as shown in Figure 2, different instruments, columns and conditions can also be used for the GC analysis.

7.2 Calibration

Add the following substances in sequence to the pressure resistant digestion vial (5.2) :

— about 50 mg of adipic acid (4.2) ;

— 2,00 ml of internal standard solution (4.8) ;

— 2 ml of hydroiodic acid (4.1).