

Surface active agents — Determination of the total polyethylene glycol content of non-ionic surface active agents (EO adducts) by HPLC/GPC

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British Standard

ICS 71.100.40

Confirmed June 2009

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 10, an inside back cover and a back cover.

This British Standard, having been prepared under the direction of the Sector Committee for Materials and Chemicals, was published under the authority of the Standards Committee and comes into effect on 15 June 1999

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ISBN 0 580 32268 8

Amendments issued since publication

Amd. No.	Date	Comments

ICS 71.100.40

Descriptors: surfactants, non-ionic surfactants, chemical analysis, determination of content, polyethylene, glycol, condensates, chromatography, high performance liquid chromatography

English version

Surface active agents — Determination of the total polyethylene glycol content of non-ionic surface active agents (EO adducts) by HPLC/GPC

Agents de surface — Détermination de la teneur totale en polyéthylène glycol des agents de surface non ioniques (condensats OE) par CLHP/CPG

Grenzflächenaktive Stoffe — Bestimmung des Gesamtgehaltes an Polyethylenglycol von nichtionischen grenzflächenaktiven Stoffen (EO-Addukten) mit HPLC/GPC

This European Standard was approved by CEN on 25 January 1999.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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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of an identical text or by endorsement, at the latest by August 1999, and conflicting national standards shall be withdrawn at the latest by August 1999.

Annexes A, B, C and D are 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.

1 Scope

This European Standard specifies a method for the determination of the total polyethylene glycol (PEG) content of aromatic and aliphatic non-ionic surface active agents of the type $R-(O-C_2H_4)_{\bar{p}}-OH$, where $\bar{p}$ is the mean ethylene oxide (EO) value. It applies for concentrations of PEG higher than approximately 0,1 g/100 g of the laboratory sample.

The method is applicable up to a degree of ethoxylation of at least 25 for products which are soluble in an 80/20 (V/V) mixture of methanol and water. Long-chain products with a low degree of ethoxylation, such as tallow alcohol¹⁾ 5 EO, are not soluble, and require an amended preliminary treatment.

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.

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

ISO 607:1980, *Surface active agents and detergents — Methods of sample division*.

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series*.

ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series*.

3 Principle

Polyethylene glycol is separated from its monoalkyl ethers by a reverse phase chromatography column. The use of an additional gel chromatography column (GPC) enables the determination of the molecular mass distribution of the polyethylene glycols in the surface active agent raw material by comparison with PEG calibration PEG standards.

Small amounts of low carboxylic acids, which are often used in industrial processes to neutralize the ethoxylation catalyst, and which interfere with the determination, are removed together with long-chain alkyl polyglycol ethers by means of a special cartridge method. This eliminates potential interference factors and decreases the analysis times.

4 Reagents and materials

During the analysis, unless otherwise stated, use only reagents specified in ISO 6353-2:1983 and ISO 6353-3:1987 if listed there, if not, reagents of recognized analytical grade.

4.1 *Methanol*, HPLC grade.

4.2 *Deionized water*, filtered, conforming to the requirements of grade 1 in EN ISO 3696.

4.3 *Polyethylene glycol 800*, GPC grade.

4.4 *Analytical grade anion exchange resin*, 100 mesh to 200 mesh, chloride form, total capacity 3,5 milliequivalents/g dry resin.

4.5 *Reversed phase C18, silica gel*, bulk material, particle diameter about 40 μm to 60 μm .

4.6 *Sodium hydroxide solution*, $c(\text{NaOH}) = 1 \text{ mol/l}$.

4.7 *Nitric acid*, $c(\text{HNO}_3) = 1 \text{ mol/l}$.

4.8 *Silver nitrate solution*, $c(\text{AgNO}_3) = 1 \text{ mol/l}$.

4.9 *Mobile phase*, either

- 80/20 (V/V) mixture of methanol/water for samples of the type alkylphenol (e.g. i-nonyl phenol 7 EO) or fatty alcohols (e.g. C12/C14 7 EO) with a low degree of ethoxylation; or
- 75/25 (V/V) mixture of methanol/water for samples of the type of alkylphenol (e.g. i-octyl phenol 25 EO) or fatty alcohols (e.g. C12/C14 25 EO) with a high degree of ethoxylation.

5 Apparatus

Ordinary laboratory apparatus and the following.

5.1 *Isocratic HPLC unit*, with refractive index detector and column oven.

5.2 *Data systems*, with reintegration facilities for the chromatograms.

5.3 *Sample preparation unit*, for solid phase extraction, such as a vacuum chamber.

5.4 *Disposable syringes*, 10 ml.

5.5 *One-mark volumetric flasks*, 10 ml and 50 ml.

5.6 *One-mark pipette*, 2 ml.

5.7 *Piston-type graduated pipette*, 10 ml.

5.8 *Preparative glass chromatography column*.

5.9 *Special cartridge*, prepared as follows.

Convert the ion exchanger (4.4) from chloride to hydroxide form by filling a chromatography column with it and washing it with sodium hydroxide solution (4.6) until no more chloride ions are detected. Wash the exchanger with water and remove from the column.

NOTE The absence of chloride is confirmed when the eluant does not form a precipitate with nitric acid/silver nitrate solution.

¹⁾ CAS Registry Number 99561-04-3.