INTERNATIONAL STANDARD

ISO 16560

First edition
2015-04-15

Surface active agents — Determination of polyethylene glycol content in nonionic ethoxylated surfactants — HPLC method

Agents de surface tensioactifs — Dosage de la teneur en polyéthylène glycol dans les surfactants éthoxylés non ioniques — Méthode par CLHP

Reference number
ISO 16560:2015(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 91, *Surface active agents*.

Introduction

This International Standard was developed based on EN 12582.

Surface active agents — Determination of polyethylene glycol content in nonionic ethoxylated surfactants — HPLC method

1 Scope

This International Standard specifies a method for the determination of the polyethylene glycol (PEG) content in aromatic and aliphatic non-ionic surface active agents of the type R-$(O\text{-}C_2H_4)_n$ OH; where n is the mean ethylene oxide (EO) value. It is applicable to all ethoxylated products soluble in methanol or methanol/water mixture. This method applies to PEG concentrations as mass fraction greater than or equal to 0,1 %. This International Standard is not applicable to PEG whose molar mass is lower than 400 g/mol. Monomeric ethylene glycol, diethylene glycol, triethylene glycol, and glycerol are not detected.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
polyethylene glycol content
amount of polyethylene glycol, expressed as a percentage by mass, calculated from the calibration curve in accordance with this International Standard

4 Principle

Polyethylene glycol is separated from the polyethoxylated surface active agents by means of reversed phase liquid chromatography. In this process PEG is eluted in the first minutes while the non-ionic surface active agents are retarded. Evaporative light scattering detector (ELSD) or charged aerosol detector (CAD) does not detect volatile materials such as the sample solvent; interferences with the PEG peak are limited. The sample is dissolved in an 80/20 (V/V) mixture of methanol/water or in another methanol/water mixture to obtain a clear solution. A portion of the sample solution is then analysed by high performance liquid chromatography (HPLC). Quantification of PEG content is achieved by external calibration with PEG molar mass equal to 1 000 g/mol.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and the water used shall conform to grade 3 in accordance with ISO 3696.

5.1 Polyethylene glycol, with molar mass of 1 000 g/mol, gel permeation chromatography (GPC) grade.

5.2 Methanol, HPLC grade, filtered before use with filter unit (6.5).

5.3 Water, HPLC grade, filtered before use with filter unit (6.5).

5.4 Helium gas, chromatography grade, for degassing eluent.

5.5 Nitrogen or air, dry, and without dust.

5.6 Mobile phase, either of the following:

a) 80/20 (V/V) mixture of methanol and water;

b) methanol.

6 Apparatus

Ordinary laboratory apparatus and glassware with the following.

6.1 HPLC unit, equipped with gradient pump.

6.2 Evaporative light scattering detector (ELSD), or charged aerosol detector (CAD).

6.3 Chromatography column, octadecyl C18 bonded phase silica gel; 5 µm; 250 mm length and 4,6 mm internal diameter.

6.4 Data logger/plotter, capable of recording and displaying the chromatographic peak area.

6.5 Filter unit, for solvent (0,45 µm).

7 Sampling

7.1 Preparation of the test sample

Prepare and store the test sample in accordance with ISO 607.

7.2 Preparation of test solutions

Weigh, to the nearest 0,1 mg, the test sample mass given in Table 1 for the expected PEG content into a 100 ml volumetric flask. Fill to the mark with the mobile phase [5.6 a)] or other suitable mixture of methanol/water and dissolve to obtain a clear solution. If necessary, filter through 0,45 µm filter unit.

Table 1

Expected PEG content, %	Sample mass, g[a]
<0,1	>1
0,1 to 2	1
2 to 5	0,5
5 to 10	0,25
10 to 25	0,1
[a] Sample mass can be adjusted depending on the detector sensitivity.	

8 Procedure

8.1 Apparatus settings

Set the HPLC unit according to the following conditions.

8.1.1 Gradient

a) t = 0 min 0 % methanol [5.6 b)];

b) t = 6 min 0 % methanol [5.6 b)];

c) t = 7 min 100 % methanol [5.6 b)];

d) t = 30 min 100 % methanol [5.6 b)];

e) t = 35 min 0 % methanol [5.6 b)].

NOTE Going from mobile phase [5.6 a)] to mobile phase [5.6 b)] is done in order to elute the ethoxylated products more rapidly.

8.1.2 Flow rate: 1,0 ml/min.

8.1.3 Temperature: room temperature.

8.1.4 Injection volume: 20 µl.

8.1.5 Detector: evaporative light scattering detector (ELSD), or charged aerosol detector (CAD).

Optimize the working conditions, depending on the apparatus in use and the physical parameters.

8.2 Calibration

8.2.1 Preparation of calibration solutions

Weigh, to the nearest 0,1 mg, 0,1 g of polyethylene glycol (PEG 1 000) (5.1) into a 100 ml volumetric flask, dissolve with the mobile phase [5.6 a)] and make up to the mark. Quantitatively transfer 1,0 ml, 5,0 ml, 10 ml, 25 ml of this solution each into 100 ml volumetric flasks and make up to the volume with the mobile phase. The concentrations of PEG in these solutions respectively are 0,01 g/l, 0,05 g/l, 0,1 g/l, 0,25 g/l. Mix the solution thoroughly. If necessary, filter through a 0,45 µm filter unit.

NOTE Mass of polyethylene glycol can be adjusted depending on the detector sensitivity.