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Surface active agents — Determination of free propylene oxide content in propylene oxide adduct surfactants — GC method

Agents de surface tensioactifs — Détermination de la teneur en oxyde de propylène libre dans les produits d'addition à base d'oxyde de propylène — Méthode de chromatographie en phase gazeuse (GC)

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Foreword

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This document was prepared by Technical Committee ISO/TC 91, *Surface acting agents*.

Surface active agents — Determination of free propylene oxide content in propylene oxide adduct surfactants — GC method

WARNING — The use of this document can involve hazardous materials, operations and equipment. It does not purport to address all of the safety or environmental problems associated with its use. It is the responsibility of users of this standard to take appropriate measures to ensure the safety and health of personnel and the environment prior to application of the standard, and fulfil statutory and regulatory requirements for this purpose.

1 Scope

This document specifies an analytical procedure for the determination of free propylene oxide in surfactants which are synthesized from propylene oxide copolymers.

The method is appropriate for the qualitative and quantitative determination of propylene oxide groups in propylene oxide adducts, polyethers and polyglycol esters by headspace gas chromatography (HS-GC) with a flame ionization detector (FID) based on external procedure. Gas chromatography-mass spectrometry (GC-MS) is used for the confirmatory purposes.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principles

The sample is weighed into a headspace vial and the sealed vial is placed in a head space sampling instrument and allowed to reach thermal equilibrium. A portion of the vapour phase is then analysed by temperature programmed gas chromatography (GC). Qualitative detection is determined by retention time of target compounds with FID or by relative abundance ratios of the fragment ions with MSD. Quantification is achieved by external quantification method for GC-FID.

5 Reagents

During the analysis, use only reagents of recognized analytical grade.

5.1 Water, complying with grade 3 in accordance with ISO 3696.

5.2 Certain concentration of propylene oxide stock solution, prepared by either of the following two methods:

a) Propylene oxide, C_3H_6O, with a purity of mass fraction ≥99,7 %, dissolved in acetonitrile, vials containing 1,0 mg of propylene oxide dissolved in 1 ml of acetonitrile.

b) Commercial propylene oxide stock solutions of appropriate concentration if available may also be used to simplify procedure and maintain the accuracy of solution concentration, e.g. 1 000 µg/mL dissolved suitable solvent, or equivalent.

5.3 N, N-dimetylacetamide (DMAC), C_3H_7NO, with a purity ≥99,9 %.

DMAC may be substituted with other solvent which offers affordable safety and convenience. Applicability of the solvent should be evaluated by appropriate validation. Possible substitutes are including, but not limited to the following:

— Dimethyl formamide (DMF);

— Diethyl formamide (DEF);

— Dimethyl sulfoxide (DMSO);

— Diluted water, grade 3 in accordance with ISO 3696

— Sodium chloride, or other salt.

5.4 Nitrogen, to operate the gas chromatograph, with a purity ≥99,998 %.

5.5 Helium, to operate the gas chromatograph-mass spectrometer, with a purity ≥99,999 %.

6 Apparatus

NOTE An instrument or item of apparatus is listed only where it is special or made to a particular specification, usual laboratory glassware and equipment being assumed to be available.

Ordinary laboratory apparatus and the following:

6.1 Analytical balance, with an accuracy of 0,1 mg.

6.2 Capillary gas chromatography system, equipped with flame ionization detector (FID) or mass spectrometry (MSD) and fitted with an automatic headspace sampler.

6.3 Headspace sampling unit.

6.4 Crimped top glass vials, with a capacity of 20 ml or of another size suitable for the particular auto sampler employed, with PTFE-coated butyl rubber septa and crimp-closures.

6.5 Septum and crimped tops, lined with aluminium or PTFE, for closing the vials.

6.6 Sealing pliers, for the vials.

6.7 Vortex mixer, with a fixed speed mode.

6.8 Volumetric flasks, with a capacity of 10 ml, 50 ml and 100 ml.