INTERNATIONAL STANDARD

ISO 21264

First edition
2019-07

Surface active agents — Detergents — Determination of alkylphenol ethoxylates

Agents de surface — Détergents — Dosage des alkylphénols éthoxylés

Reference number
ISO 21264:2019(E)

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Published in Switzerland

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Foreword

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Surface active agents — Detergents — Determination of alkylphenol ethoxylates

1 Scope

This document provides a method for the determination of alkylphenol ethoxylates (APEOs) in surfactants using high performance liquid chromatography (HPLC) and detected with diode array detector (DAD) or fluorescence detector (FLD).

This method is appropriate for the detection and quantification of APEOs in surfactants.

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*

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 Principle

The sample is weighed into a vial and extracted in methanol using an ultrasonic bath. Subsequently the extract is filtered and analysed using high performance chromatography system with diode array detector (DAD) or fluorescence detector (FLD). Quantification is achieved by external quantification method and using the relationship chromatographic peak through its unique its retention time.

5 Reagents and materials

During the analysis, use only reagents of recognized analytical grade and the water of quality for HPLC analysis.

5.1 Methanol, HPLC Grade.

5.2 Octylphenol ethoxylates (OP_nEO, $2 \leq n \leq 12$), for example, Triton X-100[1)], CAS no. 9002-93-1, Sigma-Aldrich Part Number T9284.

1) Triton X-1000 ®, CAS no. 9002-93-1, Sigma Aldrich Part Number T9284 is an example of products. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.3 Nonylphenol ethoxylates (NP_nEO, $3 \leq n \leq 18$), for example, IGEPAL CO-630[2], CAS no. 9016-45-9, Sigma-Aldrich Part Number 542334.

NOTE There is a possibility that reagents of the grade of OPEO and NPEO are not available.

6 Apparatus

Normal laboratory apparatus and, in particular, the following:

6.1 Analytical balance, weighing to an accuracy of 0,1 mg.

6.2 Ultrasonic bath, 40 kHz, with thermostat.

6.3 Membrane filter, polyamide, 0,45 µm.

6.4 Volumetric flasks, 10 ml, 50 and 100 ml capacity.

6.5 High-performance Liquid Chromatography (HPLC) with DAD or FLD detector.

6.6 Reverse phase column with guard column, a stainless-steel column 150 mm and 2,1 mm in internal diameter filled with 3,5 µm ODS (C_{18}) as stationary phases.

7 Procedure

7.1 Standard preparation

7.1.1 Prepare 1 000 mg/l stock solutions of each APEOs from standard materials or certified solutions in methanol. 100 mg of the OP_nEO (5.2) and NP_nEO (5.3) are dissolved in different 100 ml volumetric flasks with methanol and filled up to the mark respectively.

Depending on the stock concentrations prepared, the solubility at that concentration will have to be ensured.

7.1.2 Prepare calibration curve for samples of each APEO stock standard solution at required concentrations by adding aliquots of stock solutions to a 100 ml volumetric flask and then diluted with methanol to the desired calibration level. If lower reporting limit is required, lower concentration of calibration standard shall be prepared to cover the reporting limit. Each calibration curve is prepared using the respective stock solution. The preparation of the Level 5 standard can be accomplished using different volumes and concentrations of stock solutions as is accustomed in the individual laboratory.

The calibration vials shall be used within 24 h to ensure optimum results. Stock calibration standards are routinely replaced every 14 days if not previously discarded for quality control failure.

7.2 Sample preparation

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

The test samples can be in liquid, paste or solid form. Liquefaction and homogenization can be necessary for paste and solid substances. Liquefaction can be accelerated by gentle warming (below 50 °C).

2) IGEPAL CO-630 ®, CAS no. 9016-45-9, Sigma-Aldrich Part Number 542334 is an example of products. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.