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Surface active agents — Determination of chloroacetic acid (chloroacetate) in surfactants —

Part 1: HPLC method

Agents de surface — Détermination de l'acide chloroacétique (chloroacétate) dans les agents tensioactifs —

Partie 1: Méthode CLHP

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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*.

ISO 17293 consists of the following parts, under the general title *Surface active agents — Determination of chloroacetic acid (chloroacetate) in surfactants*:

— *Part 1: HPLC method*

— *Part 2: Ionic chromatographic method*

Surface active agents — Determination of chloroacetic acid (chloroacetate) in surfactants —

Part 1: HPLC method

1 Scope

This part of ISO 17293 specifies a method for the determination of monochloroacetic acid (monochloroacetate) and dichloroacetic acid (dichloroacetate) in surfactants by HPLC method.

The method applies for anionic surfactants such as alkyl (phenyl) ethoxylated carboxylate (AEC) or amphoteric surfactants such as alkyl imidazoline carboxylate, alkyl dimethyl betaine, and fatty acetyl propyl dimethyl betaine.

The limit of detection (LOD) is ≤0,3 µg/ml for monochloroacetic acid and ≤0,2 µg/ml for dichloroacetic acid; the limit of quantification (LOQ) is ≤1,0 µg/ml for monochloroacetic acid and ≤0,75 µg/ml for dichloroacetic acid (using a standard solution).

The LOD, at 5 g of sample weight, is ≤6 mg/kg for monochloroacetic acid and ≤4 mg/kg for dichloroacetic acid; and the LOQ is ≤20 mg/kg for monochloroacetic acid and ≤15 mg/kg for dichloroacetic acid.

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

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

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

The sample is dissolved in the mobile phase in order to analyse by high performance liquid chromatography (HPLC). After injection, it flows through a C_8-bonded silicone gel column. The monochloroacetic acid (monochloroacetate) and dichloroacetic acid (dichloroacetate) are separated in the column and detected by an UV detector.

The contents of monochloroacetic acid and dichloroacetic acid in the sample are achieved by external calibration method.

4 Reagents

4.1 General

During the analysis, use only reagents of recognized analytical grade and the water used shall conform to grade 1 in accordance with ISO 3696, unless otherwise specified.

4.2 Monochloroacetic acid ($ClCH_2COOH$), purity > 99 % (w/w).

4.3 Dichloroacetic acid ($Cl_2CHCOOH$), purity > 99 % (w/w).

4.4 Acetonitrile (CH_3CN) HPLC grade, filtered before use with filter unit (5.7).

4.5 Phosphoric acid (H_3PO_4).

4.6 Hydrochloric acid (HCl).

4.7 Hydrochloric acid solution, 1:1 (V/V).

Add to about 10 ml of hydrochloric acid (4.6) and 10 ml of water in portions. Mix well.

5 Apparatus

Use usual laboratory apparatus and, in particular, the following.

5.1 HPLC instrument, equipped with pump and a high-resolution UV detector or photodiode array detector, with the noise and the drift of baseline at 254 nm < 2×10^{-5} AU/s (blank cell) and at 254 nm < 1×10^{-3} AU/h (blank cell, after stabilizing for 60 min), respectively.

5.2 HPLC column: C_8-bonded phase silicone gel (particle size 5 µm), 250 mm × 4,6 mm (ID), pH range from 1 to 8, or equivalent.

5.3 Filter syringe, of capacity 2 ml or 5 ml.

5.4 Injection syringe, of capacity 25 µl.

5.5 Analytical balance, accurate to 0,1 mg.

5.6 Ultrasonic device, for the degassing of reagents.

5.7 Filters with suitable porosity (0,2 µm or 0,45 µm), for the filtration of reagents and sample.

5.8 Vacuum pump.

5.9 pH meter, for pH measurement.

5.10 Volumetric flasks, of capacity 50 ml and 100 ml.

5.11 Glass beakers, of capacity 50 ml and 100 ml.

6 Procedures

6.1 HPLC conditions

The choice of HPLC conditions depends on the apparatus in use and can be varied from those given below, provided that suitable separation of the compounds of interest is maintained. The following conditions have been found to be suitable for the HPLC column recommended in 5.2.

6.1.1 Mobile phase: Add 100 ml of acetonitrile (4.4) in 900 ml of water, then pipette 2,0 ml of phosphoric acid (4.5) and mix well. Before using, the mobile phase should be degassed with an ultrasonic device (5.6).

6.1.2 Flow rate: 1,0 ml/min.

6.1.3 Detecting wavelength: 214 nm.

6.1.4 Column temperature: room temperature.