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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

ICS 71.100.40

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ISO 17293-1 was prepared by Technical Committee ISO/TC 91 Surface active agents.

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Surface active agents - Determination of chloroacetic acids (chloroacetates) in surfactants-Part 1: HPLC method

1 Scope

This International Standard specifies a method for the determination of mono-chloroacetic acid (mono-chloroacetate) and di-chloroacetic acid (di-chloroacetate) 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) should be $\leq 0,3 \mu\text{g/ml}$ for mono-chloroacetic acid and $\leq 0,2 \mu\text{g/ml}$ for di-chloroacetic acid; and the limit of quantification (LOQ) should be $\leq 1,0 \mu\text{g/ml}$ for mono-chloroacetic acid and $\leq 0,75 \mu\text{g/ml}$ for di-chloroacetic acid (using a standard solution), respectively.

The LOD, at 5g of sample weight, should be $\leq 6 \text{ mg/kg}$ for mono-chloroacetic acid and $\leq 4 \text{ mg/kg}$ for di-chloroacetic acid; and the LOQ should be $\leq 20 \text{ mg/kg}$ for mono-chloroacetic acid and $\leq 15 \text{ mg/kg}$ for di-chloroacetic acid, respectively.

2 Normative references

The following referenced documents are indispensable for the application 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 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 Principle

The sample is dissolved in the mobile phase in order to analyze by high performance liquid chromatography (HPLC). After injection, it flows through an C_8 -bonded silicone gel column. The mono-chloroacetic acid (mono-chloroacetate) and di-chloroacetic acid (di-chloroacetate) are separated in the column and detected by an UV detector.

The contents of mono-chloroacetic acid and di-chloroacetic 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 Mono-chloroacetic acid (ClCH_2COOH): purity $> 99 \%$ (w/w)

- 4.3 Di-chloroacetic 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 photo diode array detector, of which the noise and the drift of base line at 254 nm $<2 \times 10^{-5}$ AU/s (blank cell) and at 254 nm $<1 \times 10^{-3}$ AU/h (blank cell, after stabilizing for 60 minutes), respectively.

5.2 HPLC column: C_8 -bonded phase silicone gel (particle size 5 μm) , 250 mm \times 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 Ultra sonic 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 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 wave length: 214 nm

6.1.4 Column temperature : room temperature

6.1.5 Injection volume: 20 µl

Based on above conditions, a typical chromatogram is shown in Fig 1 at annex B

6.2 Calibration

1) Weigh 0,1 g of mono-chloroacetic acid (4.2) and 0,1 g of di-chloroacetic acid (4.3), accurate to 0,1 mg, in a 50 ml beaker, add approximately 20 ml of mobile phase (6.1.1) to it, and then stir to thoroughly dissolve. Transfer quantitatively to a 100 ml volumetric flask, make up to the mark with mobile phase and completely homogenize.

Store this solution in a refrigerator at $(4 \pm 2) ^\circ\text{C}$ and renew it every 7 days.

Quantitatively dilute 0,1ml, 0,5 ml, 1 ml, 1,5 ml, 2,5 ml of this solution to 100 ml with mobile phase (6.1.1), respectively. These standards shall be freshly prepared before analysis.

2) Filter (5.7) the diluted solutions and inject 20 µl to the HPLC for analysis. The analysis must be done at least twice in accordance with the chromatographic conditions given in 6.1. The obtained linear correlation coefficient (R) shall be 0,999 or above.

6.3 Sampling and analysis

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

Add approximately 5 g of test sample, accurate to 0,1 mg, in 30 ml of mobile phase (6.1.1), stir until the sample is dissolved completely. Measure the pH with a pH meter (5.9). Adjust pH to the same as that of mobile phase with HCl solution (4.7). Quantitatively dilute to 100 ml with mobile phase (6.1.1). Filter (5.7) it and inject 20 µl of the diluted solution for analysis on the chromatographic conditions given in 6.1.

Note: If interference to the peak of mono-chloroacetic acid or di-chloroacetic acid in the betaine chromatogram was found during the test of betaine sample, prepare the sample solution in accordance with the method in annex A, using a cation-exchange column to remove the interference. After preliminary treatment, this solution shall be filtered (5.7) and injected 20 µl for analysis on the chromatographic conditions given in 6.1.

7 Results and calculation

7.1 General

Determine the mono-chloroacetic acid and di-chloroacetic acid contents in test sample using the procedure according to 6.3. Calculate the results in milligrams per kilogram (mg/kg) according to equation (1) and equation (2).

7.2 The content of mono-chloroacetic acid

$$X_1(\text{mg} / \text{kg}) = \frac{A \times V}{m} \dots\dots\dots (1)$$

where

X_1 is the content of mono-chloroacetic acid.

A is the calculated concentration of mono-chloroacetic acid in test sample solution, in micrograms per millilitre.

V is the volume of test sample, in millilitres.

m is the mass of the test sample, in grams.

7.3 The content of di-chloroacetic acid

$$X_2 (mg / kg) = \frac{B \times V}{m} \dots\dots\dots(2)$$

where:

X_2 is the content of di-chloroacetic acid.

B is the calculated concentration of di-chloroacetic acid in test sample solution, in micrograms per millilitre.

8 Precision

8.1 Repeatability limit

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not exceed the repeatability limit (r) in more than 5% of cases.

According to ISO 5725-2, r can be expected to be:

- 10% for contents greater than or equal to 50 mg/kg
- 15% for contents less than 50 mg/kg.

8.2 Reproducibility limit

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will not exceed the reproducibility limit (R) in more than 5% of cases. R can be expected to be 40%.

9 Test report

This test report shall contain at least the following information:

- a) the test method used, with reference to this International Standard
- b) all information necessary for the complete identification of the test compound;
- c) all the data (e.g. in tabular form) obtained and the calibration curve;
- d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result (s).

Annex A

Preliminary treatment of betaine sample

A.1 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, unless otherwise specified.

A.1.1 Cation exchanger (KAT), strongly acidic, 50-100 mesh

A.1.2 Hydrochloric acid, 4 mol/l solution

Add 700 ml of hydrochloric acid to 1400 ml of water, mix well

A.1.3 Acetonitrile (CH₃CN): HPLC grade

A.1.4 Acetonitrile solution, 10% (V/V)

Add 30 ml of acetonitrile (A.1.3) to 270 ml of water, mix well

A.2 Apparatus

A.2.1 Volumetric flask, of capacity 100 ml

A.2.2 Glass beakers, of capacity 200 ml and 3000 ml

A.2.3 Glass exchange column with tap for resin preparation: inner tube 50-60 mm in diameter and 550-600 mm in height

A.2.4 Glass exchange column with tap for sample analysis: inner tube 15 mm in diameter and 400 mm in height

A.3 Procedure

A.3.1 Preparation of cation exchange resin

Place 600 ml of cation-exchange resin (A.1.1) in a 3000 ml beaker (A.2.2) and cover by adding 2000 ml of hydrochloric acid (A.1.2). Allow to stand for at least two hours, with occasional stirring. Decant the acid and transfer the resin into the preparation column (A.2.3) by means of deionised water.

The column should contain a glass-wool plug. Wash the column with water until the eluate is free of chloride.

A.3.2 Cation exchange column

Place 60 ml of cation-exchange resin in the analytical column (A.2.4) with a glass-wool plug in it. Wash with water until the pH of the eluate reaches 7 approximately. Displace the water with 100 ml of acetonitrile solution (A.1.4) at a rate of 1-3 ml/min.

A.3.3 Sample elution

Weigh 5 g of betaine samples, accurate to 0,1 mg, in a 200 ml beaker (A.2.2), add 40 ml of acetonitrile solution (A.1.4) and stir until the sample is dissolved completely. Pass the sample solution quantitatively through the exchanger at a rate of 1 ml/min, add more acetonitrile solution (A.1.4) to elute sample. Receive the eluate in a 100 ml volumetric flask (A.2.1) up to the mark for HPLC analysis.