INTERNATIONAL STANDARD

**ISO 17293-1**

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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*

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# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 $< 2 \times 10^{-5}$ AU/s (blank cell) and at 254 nm $< 1 \times 10^{-3}$ 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.

**6.1.5** Injection volume: 20 µl.

Based on the above conditions, a typical chromatogram is shown in Figure B.1 in Annex B.

## 6.2 Calibration

a) Weigh 0,1 g of monochloroacetic acid (4.2) and 0,1 g of dichloroacetic 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 ± 2) °C and renew it every 7 d.

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

b) Filter (5.7) the diluted solutions and inject 20 µl to the HLPC for analysis. The analysis shall 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), and stir until the sample is dissolved completely. Measure the pH with a pH meter (5.9). Adjust the pH to the same as that of the mobile phase with HCl solution (4.7). Quantitatively dilute the sample 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 monochloroacetic acid or dichloroacetic acid in the betaine chromatogram was found during the test of the 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 monochloroacetic acid and dichloroacetic acid contents in the test sample using the procedure according to 6.3. Calculate the results in milligrams per kilogram (mg/kg) according to Formula (1) and Formula (2).

### 7.2 The content of monochloroacetic acid

The content of monochloroacetic acid in the test sample is calculated using Formula (1):

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

where

$X_1$ is the content of monochloroacetic acid, in mg/kg;

$A$ is the calculated concentration of monochloroacetic acid in the test sample solution, in µg/ml;

$V$ is the volume of the test sample, in ml;

$m$ is the mass of the test sample, in g.

### 7.3 The content of dichloroacetic acid

The content of dichloroacetic acid in the test sample is calculated using Formula (2):

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

where

$X_2$ is the content of dichloroacetic acid, in mg/kg;

$B$ is the calculated concentration of dichloroacetic acid in the test sample solution, in µg/ml.

## 8 Precision

### 8.1 Repeatability limit

The absolute difference between two independent single test results, obtained using the same method on identical test materials in the same laboratory by the same operator using the same equipment within a short interval of time, shall 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, and

— 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 materials in different laboratories by different operators using different equipment, shall not exceed the reproducibility limit ($R$) in more than 5 % of cases. $R$ can be expected to be 40 %.

## 9 Test report

The test report shall contain at least the following information:

a) the test method used, with reference to this part of ISO 17293 (i.e. ISO 17293-1);

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 part of ISO 17293, or regarded as optional, together with details of any incidents which might have influenced the test result(s).