INTERNATIONAL STANDARD

ISO 17293-2

First edition
2014-04-01

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

Part 2:
Ionic chromatographic method

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

Partie 2: Méthode par chromatographie ionique

Reference number
ISO 17293-2:2014(E)

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

Published in Switzerland

Contents

Page

Foreword iv
1 Scope 1
2 Normative references 1
3 Principle 1
4 Reagents 1
  4.1 General 1
5 Apparatus 2
6 Procedure 3
  6.1 General 3
  6.2 Ion chromatographic condition 3
  6.3 Calibration 3
  6.4 Sampling and analysis 3
7 Results and calculation 4
  7.1 General 4
  7.2 Content of monochloroacetic acid 4
  7.3 Content of dichloroacetic acid 4
8 Precision 4
  8.1 Repeatability limit 4
  8.2 Reproducible limit 5
9 Test report 5
Annex A (informative) **Typical chromatograms** 6

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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 2:
Ionic chromatographic 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 ion chromatographic 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 ≤0,01 µg/ml for monochloroacetic acid and ≤0,05 µg/ml for dichloroacetic acid; and the limit of quantification (LOQ) is ≤0,1 µg/ml for each standard (using a standard solution).

The LOD, at 1 g of sample weight, is ≤1 mg/kg for monochloroacetic acid and ≤5 mg/kg for dichloroacetic acid; and the LOQ is ≤10 mg/kg for each 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 solution is diluted to a proper concentration and flows through an anionic ion exchange column. The monochloroacetic acid (monochloroacetate) and dichloroacetic acid (dichloroacetate) are adsorbed in the column then eluted by the mobile phase and detected by an electrical conductivity detector.

The contents of monochloroacetic acid and dichloroacetic acid in the sample are achieved by the 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 Sulfuric acid (H_2SO_4)

4.5 Sodium carbonate (Na_2CO_3)

4.6 Sodium hydrogen carbonate ($NaHCO_3$)

4.7 Sodium hydroxide (NaOH)

4.8 Sodium carbonate/sodium hydrogen carbonate eluent stock solution, containing 0,32 mol/l sodium carbonate and 0,1 mol/l sodium hydrogen carbonate.

Dissolve 3,39 g of sodium carbonate (4.5) and 0,84 g of sodium hydrogen carbonate (4.6) in 30 ml of water and swirl to mix. Quantitatively transfer the solution to a 100 ml volumetric flask (5.8), make up to the mark with water, stopper, and mix thoroughly.

4.9 Sodium carbonate/sodium hydrogen carbonate eluent solution, containing 3,2 mmol/l sodium carbonate and 1 mmol/l sodium hydrogen carbonate.

Quantitatively transfer 10 ml of sodium carbonate/sodium hydrogen carbonate stock solution (4.8) to a 1 000 ml volumetric flask (5.8), dilute to the mark with water, stopper, and mix thoroughly.

4.10 Sulfuric acid solution, 50 mmol/l

Dilute 3,0 ml of sulfuric acid (4.4) in 1 000 ml of water, mix thoroughly.

4.11 Sodium hydroxide solution, 15 mmol/l

Dissolve 0,6 g of sodium hydroxide (4.7) in 30 ml of water and swirl to mix. Quantitatively transfer the solution to a 1 000 ml volumetric flask (5.8), dilute to the mark with water, stopper, and mix thoroughly.

5 Apparatus

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

5.1 Ion chromatography instrument, equipped with pump, separator column, guard column, appropriate suppressor, data process system, and electrical conductivity detector.

5.2 Separator column, anionic ion exchange column, suitable for resolving monochloroacetic acid and dichloroacetic acid from sample, 4 mm × 250 mm, with appropriate guard column.

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

5.7 Vacuum pump.

5.8 Volumetric flasks, of capacity 100 ml and 1 000 ml.

5.9 Glass beakers, of capacity 25 ml and 100 ml.

6 Procedure

6.1 General

The choice of chromatographic 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.

6.2 Ion chromatographic condition

Two kinds of ion chromatographic conditions can be used in this part of ISO 17293, depending on the apparatus selected.

6.2.1 Ion chromatographic condition 1

a) Eluent solution (4.9)

b) Flow rate: 0,70 ml/min

c) Column temperature: room temperature

d) Injection volume: 20 µl

e) Regeneration solution (4.10)

Based on the above conditions, a typical chromatogram can be found in Figure A.1.

6.2.2 Ion chromatographic condition 2

a) Eluent solution (4.11)

b) Flow rate: 0,85 ml/min

c) Column temperature: room temperature

d) Injection volume: 20 µl

Based on the above conditions, a typical chromatogram can be found in Figure A.2.

6.3 Calibration

1) Dissolve 0,1 g of monochloroacetic acid (4.2) and 0,1 g of dichloroacetic acid (4.3), accurate to 0,1 mg, in 20 ml of water. Transfer quantitatively to a 1 000 ml volumetric flask(5.8), make up to the mark with water and completely homogenize.

Store this solution in a refrigerator at (4 ± 2) °C and renew it every 7 d.

Quantitatively dilute 0,1 ml, 0,5 ml, 1,0 ml, 5,0 ml, and 10,0 ml of this solution to 100 ml with water, respectively. These standards shall be freshly prepared before analysis.

2) Filter (5.6) the diluted solutions and inject 20 µl each to equipment for analysis. The analysis must be done at least twice in accordance with the chromatographic conditions given in 6.2.1 or 6.2.2. The obtained linear correlation coefficient (R) shall be 0,999 or above.

6.4 Sampling and analysis

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

Add approximately 1 g of sample, accurate to 0,1 mg, in 30 ml of water, and stir until the sample is dissolved completely. Transfer into a 100 ml volumetric flask, make up to the mark with water. Filter (5.6) the solution and inject 20 µl for analysis on the appropriate chromatographic condition.

If necessary, pretreatment should be done to remove surfactant in order to protect the ion exchange column. Reversed phase ODS can be used before injection.

7 Results and calculation

7.1 General

Determine the monochloroacetic acid and dichloroacetic acid contents in the test sample using the procedure according to 6.4. Calculate the results in milligrams per kilogram (mg/kg) according to Formula (1) and Formula (2). Under given experimental conditions, no hydrolysis of chloroacetic acid has been observed.

7.2 Content of monochloroacetic acid

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

where

X_1 is the content of monochloroacetic acid, in milligrams per kilogram;

A is the calculated concentration of monochloroacetic acid in the 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 Content of dichloroacetic acid

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

where

X_2 is the content of dichloroacetic acid, in milligrams per kilogram;

B is the calculated concentration of dichloroacetic acid in the 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, 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;

— 15 % for contents less than 50 mg/kg.

8.2 Reproducible 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, shall not exceed the reproducibility limit (*R*) in more than 5 % of cases. *R* can be expected to be 35 %.

9 Test report

The test report shall include the following information:

a) the test method used, with a reference to this part of ISO 17293 (i.e. part of ISO 17293-2);

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 may have influenced the test result(s).