
**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

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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).

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).

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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 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 $\leq 0,01$ $\mu\text{g}/\text{ml}$ for monochloroacetic acid and $\leq 0,05$ $\mu\text{g}/\text{ml}$ for dichloroacetic acid; and the limit of quantification (LOQ) is $\leq 0,1$ $\mu\text{g}/\text{ml}$ 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 (ClCH₂COOH), purity > 99 % (w/w)**

4.3 **Dichloroacetic acid (Cl₂CHCOOH), purity > 99 % (w/w)**

4.4 **Sulfuric acid (H₂SO₄)**

4.5 **Sodium carbonate (Na₂CO₃)**

4.6 **Sodium hydrogen carbonate (NaHCO₃)**

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.