



SLOVENSKI STANDARD

SIST ISO 17280:2017

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Površinsko aktivne snovi - Določevanje ostankov 1,4-dioksana v površinsko aktivnih snoveh, pridobljenih iz epoksietana, s plinsko kromatografijo

Surface active agents - Determination of 1,4-dioxan residues in surfactants obtained from epoxyethane by gas chromatography

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Agents de surface - Détermination de la teneur en résidus de 1,4-dioxane dans les agents tensioactifs obtenus à partir d'époxyéthane par chromatographie en phase gazeuse

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INTERNATIONAL
STANDARD

ISO
17280

First edition
2015-05-15

**Surface active agents — Determination
of 1,4-dioxan residues in surfactants
obtained from epoxyethane by gas
chromatography**

*Agents de surface — Détermination de la teneur en résidus
de 1,4-dioxane dans les agents tensioactifs obtenus à partir
d'époxyéthane par chromatographie en phase gazeuse*

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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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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*.

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Surface active agents — Determination of 1,4-dioxane residues in surfactants obtained from epoxyethane by gas chromatography

1 Scope

This International Standard provides a method for the determination of 1,4-dioxane residues in surfactants which are synthesized from epoxyethane, such as alkyl ether sulfates and alcohol ethoxylates.

The method is applicable for samples containing 1,4-dioxane more than 5 mg/kg. For samples containing 1,4-dioxane more than 100 mg/kg, the sample solutions should be diluted to appropriate concentration.

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*

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3 Principle

The sample is weighed into a head space vial and a calibration solution is added. The sealed vial is placed in a head space sampling instrument and allowed to reach thermal equilibrium. A portion of the vapour phase is then analysed by temperature programmed GC with flame ionization detection. Either an internal standard or standard addition method of quantification are employed.

4 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.

4.1 1,4-Dioxane, purity 99,5 % minimum.

4.2 4-Methyl-1,3-dioxane, purity 98 % minimum.

4.3 N, N-dimethylformamide (DMF).

DMF may be substituted with other solvent. Applicability of the solvent should be evaluated by appropriate validation. Possible substitutes are including, but not limited to following:

- N, N-dimethylacetamide(DMA);
- Dimethyl sulfoxide (DMSO);
- Diethyl formamide (DEF);

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— Formamide.

Diluent shall be free from peaks which interfere 1,4-dioxane and 4-methyl-1,3-dioxane.

4.4 Carrier gas: nitrogen or helium, purity 99,99 % minimum.

5 Apparatus

Ordinary laboratory apparatus and the following.

5.1 Analytical balance, 0,1 mg readability.

5.2 Gas chromatography system, attached with temperature programmed device and flame ionization detector (FID).

5.3 Head space sampling unit

5.4 20 ml crimped top glass vials, sealing pliers and sealing clamp suitable for head space unit.

5.5 Septum and crimped tops, lined with aluminium or PTFE, for closing the vials.

5.6 Sealing pliers, for the vials.

5.7 Capillary column, capable of the separation characteristics shown in [Figure 2](#).

NOTE A 30 m × 0,32 mm (id) fused silica capillary column (film thickness: 0,25 µm) with nitroterephthalic acid modified polyethylene glycol stationary phase is advisable.

5.8 Volumetric flasks, 50 ml and 100 ml capacity.

5.9 Gas tight syringes or glass pipettes, 1 ml ± 0,01 ml capacity.

6 Preparation of calibration solutions

6.1 Calibration solution A

Add approximately 40 ml of DMF ([4.3](#)) to a 50 ml volumetric flask. Weigh 0,2 g of 1,4-dioxane ([4.1](#)) to record the exact mass to obtain the accurate concentration in each calibration solution I. Make up to volume with DMF and thoroughly homogenize. The solution is A.

Prepare each calibration solution I according to [Table 1](#).

Table 1 — preparation of calibration solutions

| Calibration solution I | I ₁ | I ₂ | I ₃ | I ₄ | I ₅ |
|--|----------------|----------------|----------------|----------------|----------------|
| Volume of the solution A/ml | 0,0 | 0,5 | 1,0 | 2,5 | 5,0 |
| Make up to volume with DMF/ml | 100 | 100 | 100 | 100 | 100 |
| The approximate concentration of 1,4-dioxane ^a /(µg/ml) | 0 | 20 | 40 | 100 | 200 |

^a The accurate concentration of 1,4-dioxane in each calibration solution I is obtained by the calculation with the mass of 1,4-dioxane.

6.2 Calibration solution B

The calibration solution B which contains the calibration solution B₁ and the calibration solution B₂ is used to relative response factor procedure.

- a) Add approximately 40 ml of DMF to a 50 ml volumetric flask, weigh 0,4 g of 4-methyl-1,3-dioxane (4.2) to record the exact mass to obtain the accurate concentration in calibration solution C. Make up to volume with DMF and thoroughly homogenize. The solution is C.
- b) Add 0,5 ml ± 0,01 ml of this solution C and 1,0 ml ± 0,01 ml of solution A to a 100 ml volumetric flask, make up to volume with DMF and thoroughly homogenize. The solution is B₁. The accurate concentration of 4-methyl-1,3-dioxane and 1,4-dioxane in the final solution B₁ is obtained by the calculation with the mass of 4-methyl-1,3-dioxane and 1,4-dioxane.
- c) Add 0,5 ml ± 0,01 ml of this solution C to a 100 ml volumetric flask, make up to volume with DMF and thoroughly homogenize. The solution is B₂. The accurate concentration of 4-methyl-1,3-dioxane in the final solution B₂ is obtained by the calculation with the mass of 4-methyl-1,3-dioxane.

7 Procedure

7.1 Preparation of the test sample

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

7.2 Standard addition procedure

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7.2.1 Preparation of test sample solutions

7.2.1.1 Add 2 g ± 0,01 g of test sample (7.1) to each of three separate head space sample vials. For samples where the 1,4-dioxane content is expected to be less than 20 mg/kg, use the procedure described in 7.2.1.2. For samples expected to have a content of 1,4-dioxane from 20 mg/kg to 100 mg/kg, use the procedure described in 7.2.1.3.

7.2.1.2 Using the 1ml gas tight syringe or glass pipette (5.9), add the following to each of the three vials:

- a) 1 ml ± 0,01 ml of calibration solution I₁ (6.1) to the first vial. Seal immediately with septum and crimped top (5.5) and homogenize by shaking vigorously.
- b) 1 ml ± 0,01 ml of calibration solution I₂ (6.1) to the second vial. Seal immediately with septum and crimped top (5.5) and homogenize by shaking vigorously. The added amount of 1,4-dioxane relative to the test sample content is approximate 10 mg/kg.
- c) 1 ml ± 0,01 ml of Calibration solution I₃ (6.1) to the third vial. Seal immediately with septum and crimped top (5.5) and homogenize by shaking vigorously. The added amount of 1,4-dioxane relative to the test sample content is approximate 20 mg/kg.

7.2.1.3 Using the 1 ml gas tight syringe or glass pipette (5.9), add the following to each of the three vials:

- a) 1 ml ± 0,01 ml of calibration solution I₁ (6.1) to the first vial. Seal immediately with septum and crimped top (5.5) and homogenize by shaking vigorously.
- b) 1 ml ± 0,01 ml of calibration solution I₄ (6.1) to the second vial. Seal immediately with septum and crimped top (5.5) and homogenize by shaking vigorously. The added amount of 1,4-dioxane relative to the test sample content is approximate 50 mg/kg.
- c) 1 ml ± 0,01 ml of Calibration solution I₅ (6.1) to the third vial. Seal immediately with septum and crimped top (5.5) and homogenize by shaking vigorously. The added amount of 1,4-dioxane relative to the test sample content is approximate 100 mg/kg.