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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

Reference number
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Foreword

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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-dioxan 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*

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-dimetylacetamide(DMA);

— Dimethyl sulfoxide (DMSO);

— Diethyl formamide (DEF);

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