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Footwear — Critical substances potentially present in footwear and footwear components — Determination of certain organic solvents

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 216, *Footwear*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

This document covers a test method for the determination of the certain organic solvents in footwear products.

Industrial organic solvents are often used in or ingredients of adhesive, treating agents and coating agents for footwear.

Some organic solvents have been proved to be toxic. Harmful solvents left in footwear materials may penetrate into human body through contact with human skin during wearing and through human breath after being released from footwear, which harms the health of consumers. Both glycol ethers solvents and amide solvents will form highly toxic compounds after metabolism in the body, which cause permanent damage to the blood circuit and the nervous system. Long-term exposure to high concentration may cause cancer. In addition, ethylene glycol ether solvents may cause permanent damage to the reproductive system of the female.

Due to this potential effect, the listed organic solvents are based on those which have been restricted in some regulations (e.g. in the European Union (regulation (EU) No 1907/2006^[1] and U.S. Environmental Protection Agency).

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Footwear — Critical substances potentially present in footwear and footwear components — Determination of certain organic solvents

WARNING — This document calls for the use of substances and/or procedures that might be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. It has been assumed in the drafting of this document that the execution of its provisions is entrusted to appropriately qualified and experienced operators.

1 Scope

This document specifies a method for the quantification of certain organic solvents (see [Annex A](#)) residues in footwear materials with gas chromatography-mass spectrometry (GC-MS).

This document is applicable to footwear materials where there is a risk of the presence of certain solvents residues (for example, solvents present in glues, leather finishing, coated textiles, etc...).

NOTE ISO/TR 16178 describes which materials are concerned by this determination.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

ISO 21061, *Footwear — Chemical tests — General principles on the preparation of samples*

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3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

certain organic solvents

solvents used in the footwear industry and listed in the [annex A](#) of this document, as glycol ether, amides and pyrrolidone solvents which are normally used in textile, leather and polymer industry

4 Principle

The sample is cut in small pieces and extracted with methanol in a sealed vial at 40 °C in an ultrasonic bath for 60 min. After evaporation and dissolution in methanol, an aliquot is then analysed using a gas chromatograph with a mass selective detector.

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5 Reagents

5.1 Chemicals

Unless otherwise specified, use only reagents of recognized analytical grade.

5.1.1 Methanol, Chemical Abstract Service Registry Number® (CAS RN®): 67-56-1.

5.1.2 Acetone, CAS RN®: 67-64-1.

5.1.3 Organic solvents, see [Annex A](#).

5.1.4 Internal standards.

— **Ethylene glycol methyl ether acetate**(EGMEA) , CAS RN®: 110-49-6.

— **Ethylene glycol monobutyl ether**(EGME), CAS RN®: 111-76-2.

— **N-Methylformamide**(MF), CAS RN®: 123-39-7.

The use of internal standard is defined in [Annex C, table C.1](#).

5.2 Standard solutions

5.2.1 Target organic solvents — Stock solution

Based on its tasks a laboratory shall decide which -organic solvents from [Table A.1](#) need to be determined.

Based on this decision, standard stock solution for each organic solvent shall be available either as commercially available certified mixes, individual components in solution or self-prepared individual -standard stock solutions of each organic solvent in methanol ([5.1.1](#)).

Prepare, for example, standard stock solutions with concentrations of 1 000 µg/ml. Weigh 50,0 mg of each organic solvent ([Table A.1](#)) into 50 ml volumetric flasks, fill the volumetric flasks up to the mark with methanol ([5.1.1](#)) and mix thoroughly to dissolve completely the substance.

5.2.2 Internal standard — Stock solution

Prepare an internal standard stock solution that is in the same concentration range as the standard stock solutions ([5.2.1](#)) by dissolving the internal standard ([5.1.4](#)) in methanol ([5.1.1](#)), according to [5.2.1](#).

5.2.3 Calibration solutions

Prepare at least five appropriate organic solvent calibration solutions, each containing an equal amount of the target organic solvents ([5.1.3](#)) and an amount of internal standard ([5.1.4](#)) in methanol ([5.1.1](#)).

An examples of calibration solutions are shown in [Table 1](#).

Table 1 — Example of calibration solutions extraction

| Standard | L1 | L2 | L3 | L4 | L5 |
|---|----|-----|-----|-------|-------|
| Concentration of organic solvents (µg/ml) | 5 | 10 | 50 | 200 | 500 |
| Volume of the organic solvent stock solution (µl) (5.2.1) | 50 | 100 | 500 | 2 000 | 5 000 |