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Textiles — Determination of formaldehyde —

Part 3: Free and hydrolysed formaldehyde (extraction method) by liquid chromatography

Textiles — Dosage du formaldéhyde —
Partie 3: Formaldéhyde libre et hydrolysé (méthode par extraction) par chromatographie liquide

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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 document 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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This document was prepared by Technical Committee ISO/TC 38, *Textiles*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 248, *Textiles and textile products,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

A list of all parts in the ISO 14184 series can be found on the ISO website.

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.

Textiles — Determination of formaldehyde —

Part 3: Free and hydrolysed formaldehyde (extraction method) by liquid chromatography

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1 Scope

This document specifies a method for determining the amount of free formaldehyde and formaldehyde extracted partly through hydrolysis by means of an extraction method. The method can be applied for the testing of textile fibres, fabrics or yarns.

NOTE This method, based on liquid chromatography (LC), is selective and not sensitive to coloured extracts and is intended to be used for precise quantification of formaldehyde.

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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology 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/

4 Conformity

Compared with ISO 14184-1, the two analytical methods should give similar trends but not necessarily the same absolute result. Therefore, in cases of dispute, the method in this document shall be used in preference to ISO 14184-1 (see Note in Clause 1).

5 Principle

The sample is extracted with extraction solution at 40 °C. The eluate is mixed with 2,4-dinitrophenylhydrazine (DNPH), whereby formaldehyde reacts to give the respective hydrazone. It is separated by liquid chromatography with ultraviolet detector (LC-UV) or liquid chromatography with

diode array detector (LC-DAD) or liquid chromotagraphy with single quadrupole mass detector (LC-MS) or liquid chromatography with triple quadrupole mass detector (LC-MSMS) and the amount is quantified.

The process is selective. Formaldehyde is separated and quantified as a derivative from other aldehydes and ketones by LC. Free formaldehyde and formaldehyde which is hydrolysed during extraction to yield free formaldehyde is quantified.

6 Reagents

All reagents shall be of analytical reagent grade, unless otherwise stated.

6.1 Grade 3 water, in accordance with ISO 3696.

6.2 Acetonitrile (CAS Registry Number[1)] 75-05-8), LC -MS grade.

6.3 Formaldehyde solution CH_2O (CAS Registry Number[1)] 50-00-0), approximately 37 % (mass fraction).

6.4 Formaldehyde-2,4-DNPH certified reference material (CRM47177)[2)], 100 µg/ml.

Certified solutions of formaldehyde-2,4-DNPH, which are commercially available should be used. When these solutions are used, the procedure in 9.1 is not required.

6.5 Dinitrophenylhydrazine (DNPH) (CAS Registry Number[1)] 119-26-6) solution consisting of 0,3 g DNPH (2,4 dinitrophenylhydrazine) dissolved in 100 ml acetonitrile (6.2). DNPH commercially available reagent should be ≥ 97 % purity.

6.6 Iodine solution (CAS Registry Number[1)] 7553-56-2), 0,05 mol/l.

6.7 Sodium hydroxide solution (CAS Registry Number[1)] 1310-73-2), 2,0 mol/l.

6.8 Sulfuric acid solution (CAS Registry Number[1)] 7664-93-9), 2,0 mol/l.

6.9 Sodium thiosulfate solution (CAS Registry Number[1)] 10102-17-7), 0,1 mol/l.

6.10 Starch solution (CAS Registry Number[1)] 9005-84-9), 1 %., for example, 1 g in 100 ml water (6.1).

6.11 Sodium acetate (CAS Registry Number[1)] 127-09-3) ≥ 97 % purity.

6.12 Acetic acid (CAS Registry Number[1)] 64-19-7) ≥ 97 % purity.

6.13 Extraction solution. Acetic acid/sodium acetate buffer solution 0,1 mol/l (pH = 5,0).

Prepare 800 ml of distilled water (6.1) in a 1 000 ml flask. Add 9,53 g of Sodium acetate (6.11) and 2,7 g of Acetic acid (6.12). If necessary, adjust the pH with HCl or NaOH, then make up to volume with distilled water (6.1).

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