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ISO/TC 38~~/WG 22~~

Secretariat: SAC

Date: 2023-08-17

Textiles — Determination of formaldehyde — —

Part 3:

Free and ~~hydrolyzed~~hydrolysed formaldehyde (~~water~~ 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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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.

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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.

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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.

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

~~ISO 14184-1, *Textile — Determination of formaldehyde — Part 1: Free and hydrolysed formaldehyde (water extraction method)*~~

3 Terms and definitions

No terms and definitions are listed in this document.

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

— ISO Online browsing platform: available at ~~https://www.iso.org/obp~~https://www.iso.org/obp

— IEC Electropedia: available at ~~https://www.electropedia.org/~~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).~~Clause 1).

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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~~liquid chromatography with ~~Ultraviolet Detector (~~ultraviolet detector (LC-UV) or ~~Liquid Chromatography~~liquid chromatography with ~~Diode Array Detector~~diode array detector (LC-DAD) or ~~Liquid Chromotagraphy~~liquid chromotagraphy with ~~Single Quadrupole Mass~~single quadrupole mass detector (-LC-MS-) or ~~Liquid Chromatography~~liquid chromatography with ~~Triple Quadrupole Mass~~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), 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~~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)~~(6.2). DNPH commercially available reagent should be ≥ 97 %.

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 (C CAS Registry Number[1] 10102-17-7), 0,1 mol/l.

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

[1] Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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Prepare 800 ml of distilled water ~~(6.1)~~(6.1) in a ~~1000~~ 1 000 ml flask. Add 9,53 g of Sodium acetate ~~(6.11)~~(6.11) and 2,7 g of Acetic acid ~~(6.12)~~.(6.12). If necessary, adjust the pH with HCl or NaOH, then make up to volume with distilled water ~~(6.1)~~.(6.1).

7 Apparatus

The usual laboratory apparatus and laboratory glassware shall be used, and in particular the following:

7.1 Stoppered volumetric flasks, for example,10 ml, 25 ml, 500 ml and 1 000 ml.

7.2 Conical flasks with stopper or screw cap, 250 ml.

7.3 Micro pipettes, for example, 10 µl to 100 µl, 100 µl to 1 000 µl and 1 ml to 5 ml.

7.4 Burettes, for example, 10 ml and 50 ml.

7.5 Water bath, thermostatically controlled to (40 ~~±~~ ± 2) °C, fitted with a flask shaker, frequency (50 ± 10) min^{-1}.

7.6 Water bath or oven, thermostatically controlled to (50 ~~±~~ ± 2) °C.

7.7 Strainer with glass fibre filter, GF8 (or glass filter strainer G3, diameter 70 mm to 100 mm).

7.8 Analytical balance, with the resolution of 0,1 mg.

7.9 HPLC system with UV, DAD, MS or MSMS detection

7.10 Membrane filter, for example polyamide, 0,45 µm.

8 Preparation of test specimen

Do not condition the sample because the pre-drying and humidity in connection with the conditioning may cause changes in the formaldehyde content of the sample. Prior to testing, store the sample in a container.

Storage can be in a polyethylene bag and wrapped in aluminium foil. The reason for the storage precaution is that formaldehyde might diffuse through the pores of the bag. In addition, catalysts, or other compounds present in a finished, unwashed fabric, can react with the foil if in direct contact.

From the sample, cut the textile component into pieces of about 0,3 cm to 0,5 cm edge length.

9 Procedure

9.1 Formaldehyde stock solution

9.1.1 Preparation of formaldehyde stock solution

The use of commercially available Certified Reference Material solutions ~~(6.4)~~(6.4) is recommended. In-house prepared stock solutions may be used only upon verification of precision data with the formaldehyde-2,4-DNPH certified reference material solution.

If in-house stock solution is used, pipette 5 ml of the formaldehyde solution ~~(6.3)~~(6.3) into a ~~1000~~1 000 ml volumetric flask ~~(7.1)~~(7.1) containing approximately 100 ml water ~~(6.1)~~(6.1) and fill the flask with water ~~(6.1)~~(6.1) up to the mark. This solution is the formaldehyde stock solution (S1).

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9.1.2 Determination of the formaldehyde concentration in the stock solution

Pipette 5 ml from the solution prepared as in ~~9.1.1~~9.1.1 into a 250 ml conical flask ~~(7.2)~~(7.2) and mix with 50 ml iodine solution ~~(6.6).~~(6.6). Add sodium hydroxide ~~(6.7)~~(6.7) until it turns yellow. Allow it to react for (15 ± 1) min at 18 °C to 26 °C and then add 15 ml of sulfuric acid ~~(6.8)~~(6.8) while swirling.

After adding 2 ml of starch solution ~~(6.10),~~(6.10), titrate the excess iodine with sodium thiosulfate ~~(6.9)~~(6.9) until the colour changes. Make three individual determinations.

Titrate at least two blank solutions in the same manner.

The concentration of formaldehyde stock solution is calculated according ~~Formula (1):~~Formula (1):

$$\rho_{FA} = \frac{(V_0 - V_1) \times c_1 \times M_{FA}}{2} \quad (1)$$

where

ρ_{FA} is the concentration of the formaldehyde stock solution, in mg/10 ml;

V_0 is the titre of the thiosulfate solution for the blank solution, in ml;

V_1 is the titre of the thiosulfate solution for the sample solution, in ml;

M_{FA} is the relative molecular mass of formaldehyde, 30,02 g/mol;

c_1 is the concentration of the thiosulfate solution, in mol/l.

9.2 Determination of formaldehyde

9.2.1 Calibration of HPLC

9.2.1.1 General

Proposals for suitable HPLC conditions are given in ~~Annex B.~~Annex B.

9.2.1.2 Calibration with formaldehyde stock solution

At least, four calibration solutions shall be used to cover the formaldehyde concentration range to cover the formaldehyde concentration range of 5 mg/kg to 100 mg/kg.

Prepare the standard solution (S2) for calibration purposes, i.e~~.~~, the standard solution is approximately 4 µg/ml in formaldehyde content.

For example, pipette 1 ml of the formaldehyde stock solution (S1) obtained in ~~9.1.1~~9.1.1, with a precisely know formaldehyde content, into a 500 ml volumetric flask ~~(7.1),~~(7.1), pre-filled with approximately 100 ml of extraction solution ~~(6.13).~~(6.13). Mix and fill to the mark with extraction solution ~~(6.13).~~(6.13), then mix again. Add the standard solution (S2) into each of four 25 ml volumetric flasks ~~(7.1),~~(7.1), for sample under the given conditions and fill the flasks ~~(7.1)~~(7.1) up to the mark with extraction solution ~~(6.13)~~(6.13) and mix.

Here is an example:

— ——0,25 ml of S2 to 25 ml, containing 0,05 µg CH_2O/ml = 5 mg/kg CH_2O on the fabric

— ——0,5 ml of S2 to 25 ml, containing 0,10 µg CH_2O/ml = 10 mg/kg CH_2O on the fabric

— ——2,5 ml of S2 to 25 ml, containing 0,50 µg CH_2O/ml = 50 mg/kg CH_2O on the fabric

— ——5,0 ml of S2 to 25 ml, containing 1,00 µg CH_2O/ml = 100 mg/kg CH_2O on the fabric

Pipette 5 ml of each formaldehyde calibration solution, into 10 ml volumetric flasks ~~(7.1),~~(7.1), pre-filled with 4 ml acetonitrile ~~(6.2).~~(6.2). Immediately upon addiction of the calibration solution, mix each flask and add 0,5 ml DNPH solution ~~(6.5).~~(6.5). Fill the flasks up to the mark with extraction solution

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{6.13}(6.13) and mix. Place the flasks in a water bath or oven {7.6}(7.6) preheated at (50 ± 2) °C per (180 ± 2) min. Then analyse the calibration solutions using liquid chromatography (LC-UV or LC-DAD or LC-MS or LC-MSMS).

Calculate the first-order regression curve of the type y = a + bx. This regression curve will be used for all measurements. If the test specimens contain a higher amount of formaldehyde than 500 mg/kg, dilute the sample solution.

9.2.1.3 Calibration with derivatized DNPH-formaldehyde

At least, four calibration solutions shall be used. Add the formaldehyde-2,4-DNPH {6.4}(6.4) into each of four 25 ml volumetric flasks {7.1}(7.1) pre-filled with 4 ml acetonitrile {6.2},(6.2), in order to cover the formaldehyde concentration range of 5 mg/kg to 100 mg/kg on sample, under the given conditions, and fill the flasks {7.1}(7.1) up to the mark with demineralized water {6.1}(6.1) and mix.

Here is an example:

— 0,01 ml of formaldehyde-2,4-DNPH {6.4}(6.4) to 25 ml, containing 0,05 µg CH_2O/ml = 5 mg/kg CH_2O on the fabric

— 0,02 ml of formaldehyde-2,4-DNPH {6.4}(6.4) to 25 ml, containing 0,10 µg CH_2O/ml = 10 mg/kg CH_2O on the fabric

— 0,1 ml of formaldehyde-2,4-DNPH {6.4}(6.4) to 25 ml, containing 0,50 µg CH_2O/ml = 50 mg/kg CH_2O on the fabric

— 0,2 ml of formaldehyde-2,4-DNPH {6.4}(6.4) to 25 ml, containing 1,00 µg CH_2O/ml = 100 mg/kg CH_2O on the fabric

Plot the concentrations in micrograms per ml in a calibration graph against the measured formaldehyde derivative peak area. X-axis: concentration in micrograms per ml, y-axis: peak area.

9.2.2 Extraction of the test specimen

For each test specimen, put (2,0 ± 0,1) g of test pieces (8) into a 250 ml flask with a stopper {7.2}(7.2) and record the mass to the nearest of 10 mg. Add 100 ml of extraction solution {6.13},(6.13), Stopper tightly and place in a water bath {7.5}(7.5) at (40 ± 2) °C for (60 ± 5) min. Shake the flask at least every 5 min, ensuring that the test specimens are entirely wet. Then filter the solution into another flask through a filter {7.7},(7.7).

If it is difficult ~~to obtain completely wet~~for the test specimens to be completely wet, a mechanical-shaking water bath should be used.

Immediately after the extraction of the test specimen, proceed with the reaction with DNPH as reported below.

9.2.3 Derivatization with DNPH and analysis

9.2.3.1 Pipette 4,0 ml of acetonitrile {6.2},(6.2), 5,0 ml aliquot of the filtered eluate {9.2.1}(9.2.1) and 0,5 ml of DNPH solution {6.5}(6.5) into a 10 ml volumetric flask {7.1},(7.1). Place the flask in the water bath or oven {7.6}(7.6) preheated at (50 ± 2) °C for (180 ± 2) min. Cool the flask down to room temperature (18 °C to 26 °C). Fill the volumetric flask with extraction solution {6.13}(6.13) up to the mark and shake it briefly by hand to mix the components. If necessary, filter through a membrane filter {7.10}(7.10) and then analyse the solution with liquid chromatography {7.9}(7.9) (LC-UV or LC-DAD or LC-MS or LC-MSMS).

Examples of chromatographic and spectroscopic conditions are given in ~~Annex B.~~Annex B.

9.2.3.2 For a high content of formaldehyde (> 500 mg/kg), make aliquots smaller than 5 ml up to 5 ml with extraction solution {6.13},(6.13). Example of the procedure when formaldehyde content is

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