SLOVENSKI STANDARD
SIST EN ISO 14184-1:1999

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Tekstilije - Določevanje formaldehida - 1.del: Prosti in hidrolizirani formaldehid (vodna ekstrakcija) (ISO 14184-2:1998)

Textiles - Determination of formaldehyde - Part 1: Free and hydrolyzed formaldehyde (water extraction method) (ISO 14184-1:1998)

Textilien - Bestimmung des Gehaltes an Formaldehyd - Teil 1: Freier und hydrolisierter Formaldehyd (Wasser-Extraktions-Verfahren) (ISO 14184-1:1998)

Textiles - Dosage du formaldéhyde - Partie 1: Formaldéhyde libre et hydrolysé (méthode par extraction d'eau) (ISO 14184-1:1998)

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Textiles - Determination of formaldehyde - Part 1: Free and hydrolyzed formaldehyde (water extraction method) (ISO 14184-1:1998)

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Textilien - Bestimmung des Gehaltes an Formaldehyd - Teil 1: Freier und hydrolisierter Formaldehyd (Wasser-Extraktions-Verfahren) (ISO 14184-1:1998)

This European Standard was approved by CEN on 30 November 1997.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Foreword

The text of EN ISO 14184-1:1998 has been prepared by Technical Committee CEN/TC 248 "Textiles and textile products", the secretariat of which is held by BSI, in collaboration with Technical Committee ISO/TC 38 "Textiles".

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 1999, and conflicting national standards shall be withdrawn at the latest by June 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

0 WARNING. This standard calls for the use of substances and/or procedures that may 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 standard that the execution of its provisions is entrusted to appropriately qualified and experienced people.

1 Scope

This Part of ISO 14184 specifies a method for determining the amount of free formaldehyde and formaldehyde extracted partly through hydrolysis by means of a water extraction method. The method can be applied to the testing of textile samples in any form.

The procedure is intended for use in the range of free and hydrolysed formaldehyde on the fabric between 20 mg/kg and 3500 mg/kg when determined by this method. The lower limit is 20 mg/kg. Below this limit the result is reported as 'not detectable'.

A method for determination of released formaldehyde is given in ISO 14184-2.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Part of ISO 14184. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this Part of ISO 14184 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid international standards.

ISO 139-1973	Textiles - Standard atmospheres for conditioning and testing
ISO 3696-1987	Specification for water for laboratory use
ISO 4793-1980	Laboratory sintered(fritted) filters - porosity grading, classification and designation.

3 Principle

Formaldehyde is extracted from a textile sample with water at 40°C. The amount of formaldehyde is then determined colorimetrically.

4 Reagents

All reagents shall be analytical reagent quality.

4.1 Distilled water or grade 3 water complying with ISO 3696.

4.2 Acetylacetone reagent (Nash reagent)

Dissolve 150 g of ammonium acetate in about 800 ml of water (4.1), add 3 ml of glacial acetic acid and 2 ml of acetylacetone, transfer into a 1000 ml volumetric flask and make up to the mark with water (4.1). Store in a brown bottle.

NOTE 1 The reagent darkens in colour slightly on standing over the first 12 h. For this reason the reagent should be held 12 h before use. Otherwise, the reagent is useable over a considerable period of time, at least 6 weeks. Since the sensitivity may change slightly over a long period of time, it is good practice to run a calibration curve weekly to correct for slight changes in the standard curve.

4.3 Formaldehyde solution, approximately 37% (W/V or W/W).

4.4 Ethanolic solution of dimedone.

Prepare by dissolving 1 g of dimedone (dimethyl-dihydro resorcinol or 5, 5-dimethyl-cyclohexanedione) in ethanol and by diluting the solution with ethanol to make 100 ml. Prepare immediately before use.

5 Apparatus

5.1 Stoppered volumetric flasks, 50 ml, 250 ml, 500 ml and 1000 ml.

5.2 Flask, 250 ml, with stopper.

5.3 Pipettes, 1 ml, 5 ml, 10 ml and 25 ml volumetric and 5 ml graduated.

NOTE 2 An automatic pipette system of the same accuracy as manual pipettes may be used.

5.4 Burettes, 10 ml and 50 ml.

5.5 Photoelectric colorimeter or spectrometer, (wavelength, 412 nm).

5.6 Test tubes, colorimeter tubes or spectrometer tubes.

5.7 Water bath, at (40 ± 2) °C.

5.8 **Filters,** made from heat resistant glass having a pore size between 40 µm and 100 µm (pore symbol P100 in accordance with ISO 4793).

5.9 Balance, accurate to 0,2 mg.

6 Preparation of standard solution and calibration

6.1 Preparation

Prepare an approximately 1500 mg/l stock solution of formaldehyde by diluting 3.8 ml of formaldehyde solution (4.3) to one litre with water(4.1). Determine the concentration of formaldehyde in the stock solution by the standard method given in annex A.

Record the accurate concentration of this standardized stock solution. This stock solution will keep for up to four weeks and is used to prepare standard dilutions.

6.2 Dilution

The equivalent concentrations of the formaldehyde in the test specimen, based on the mass of 1 g of the test specimen and 100 ml of water, will be 100 times the accurate concentrations of the standard solutions.

6.2.1 Preparation of the standard-solution (S2)

Dilute 10 ml of the titrated standard solution (containing 1,5 mg/ml of formaldehyde), prepared in 6.1, with water (4.1) to 200 ml in a volumetric flask. This solution contains 75 mg/l of formaldehyde.

6.2.2 Preparation of the calibration-solutions

Prepare calibration solutions from the standard solution (S2), by diluting with water (4.1) in 500 ml volumetric flasks, using a minimum of five solutions from the following:

1 ml S2 to 500 ml, containing 0,15 µg CH_2O / ml ≡ 15 mg/kg CH_2O on the fabric
2 ml S2 to 500 ml, containing 0,30 µg CH_2O / ml ≡ 30 mg/kg CH_2O on the fabric
5 ml S2 to 500 ml, containing 0,75 µg CH_2O / ml ≡ 75 mg/kg CH_2O on the fabric
10 ml S2 to 500 ml, containing 1,50 µg CH_2O / ml ≡ 150 mg/kg CH_2O on the fabric
15 ml S2 to 500 ml, containing 2,25 µg CH_2O / ml ≡ 225 mg/kg CH_2O on the fabric
20 ml S2 to 500 ml, containing 3,00 µg CH_2O / ml ≡ 300 mg/kg CH_2O on the fabric
30 ml S2 to 500 ml, containing 4,50 µg CH_2O / ml ≡ 450 mg/kg CH_2O on the fabric
40 ml S2 to 500 ml, containing 6,00 µg CH_2O / ml ≡ 600 mg/kg CH_2O on the fabric

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.

NOTE 3. This double-dilution is necessary to have the same formaldehyde concentrations in the calibration solutions as in the test solutions of the fabrics. If the fabric contains 20 mg/kg formaldehyde, a 1,00 g specimen is extracted with 100 ml water; the solution contains 20 µg formaldehyde and from this follows, 1 ml of the test solution contains 0,2 µg of formaldehyde.

7 Preparation and conditioning of test specimens

Do not condition the test specimen because the predrying and humidity in connection with the conditioning may cause changes in the formaldehyde content of the sample. Prior to test store the sample in a container.

NOTE 4 Storage may be in a polyethylene bag and wrapped in aluminium foil. The reason for the storage precaution is that formaldehyde may diffuse through the pores of the bag. In addition, catalysts, or other compounds present in a finished, unwashed fabric may react with the foil if in direct contact.

From the sample, cut two test specimens into small pieces, and weigh approximately 1 g of the pieces to an accuracy of 10 mg. If the formaldehyde content is low, increase the test specimen weight to 2,5 g in order to achieve a sufficient accuracy.

For each test specimen, put the weighed pieces into a 250 ml flask with stopper(5.2) and add 100 ml of water (4.1). Stopper tightly and place in a water bath at (40 ± 2)°C for (60 ± 5) min. Shake the flask at least every 5 min. Then filter the solution into another flask through a filter (5.8).

In cases of dispute use a conditioned parallel specimen to calculate a correction coefficient to be used in correcting the mass of the test specimen to be used for the test.
Cut the test specimen from the sample, weigh it immediately and again after conditioning (in accordance with ISO 139). Use these values to calculate the correction coefficient to two integers and use the coefficient to calculate the conditioned weight of the test specimen used for the sample solution.

8 Procedure

8.1 Put 5 ml of the filtered test specimen solution into a tube (5.6) and 5 ml of the standard formaldehyde solutions into further tubes(5.6). Add 5 ml of acetylacetone reagent (4.2) into each tube and shake them.

8.2 Keep the test tubes first in a water bath at (40 ± 2)°C for (30 ± 5) min and then at ambient temperature for (30 ± 5) min. Taking the solution of 5 ml of acetylacetone reagent solution in 5 ml of water having been treated in the same way as the blank reagent and using a

spectrometer, measure the absorbances in a 10 mm absorption cell at a wavelength of 412 nm against water (4.1).

8.3 If it is anticipated that the fabrics have formaldehyde extraction levels of more that 500 mg/kg, or if the calculated levels from the test using the 5 : 5 ratio are more that 500 mg/kg dilute the extract to give absorbance in the range of the calibration curve (the dilution factor shall be taken into account when calculating the results).

8.4 To account for the effect of any impurities or discoloration in the test specimen solution, put 5 ml of the sample solution in a separate test tube, add 5 ml of water (4.1) instead of acetylacetone and treat in the same way as above. Determine the absorbance of this solution, in the same way as above, but using water (4.1) as the control.

8.5 Make at least two parallel tests.

CAUTION. Exposure of the developed yellow colour to direct sunlight for a period of time will cause some fading. If there is appreciable delay (e.g. 1 h) in reading the tubes after colour development and strong sunlight is present, care should be exercised to protect the tubes such as by covering them with a formaldehyde free enclosure. Otherwise the colour is stable for considerable time (at least overnight) and reading may be delayed, if desired.

8.6 If there is a doubt that the absorption may not be due to formaldehyde but for example to an extracted colouring agent, carry out a confirmation test, with dimedone(see 8.7).

NOTE 5 Dimedone reacts with formaldehyde, and thus no colour resulting from formaldehyde reaction will be observed.

8.7 For dimedone confirmation, put 5 ml of the sample solution in a test tube (diluted where necessary, see clause 7), add 1 ml of ethanol solution of dimedone and shake.

Warm the solution in a water bath at (40 ± 2)°C for (10 ± 1) min, then add 5 ml of acetylacetone reagent, shake and continue to warm the solution in a water bath at (40 ±2)°C for (30 ± 5) min. Leave the solution still at room temperature for (30 ± 5) min. Determine the absorbance of the solution using a control solution prepared in the same way as above, but with water instead of the sample solution. The absorbance from formaldehyde at 412 nm (4.1) disappears.