INTERNATIONAL STANDARD

**ISO 14184-2**

Second edition
2011-08-15

# Textiles — Determination of formaldehyde —

## Part 2: Released formaldehyde (vapour absorption method)



*Textiles — Dosage du formaldéhyde —*

*Partie 2: Formaldéhyde dégagé (méthode par absorption de vapeur)*



Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14184-2 was prepared by Technical Committee ISO/TC 38, *Textiles*.

This second edition cancels and replaces the first edition (ISO 14184-2:1998), of which it constitutes a minor revision.



ISO 14184 consists of the following parts, under the general title *Textiles — Determination of formaldehyde*:



— *Part 1: Free and hydrolysed formaldehyde (water extraction method)*

— *Part 2: Released formaldehyde (vapour absorption method)*

# Textiles — Determination of formaldehyde —

## Part 2: Released formaldehyde (vapour absorption method)

**WARNING — This part of ISO 14184 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 part of ISO 14184 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 formaldehyde released under the conditions of accelerated storage from textiles in any form by means of a vapour absorption method.



The procedure is intended for use in the range of releasable formaldehyde on the fabric between 20 mg/kg and 3 500 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 free formaldehyde and formaldehyde extracted partly through hydrolysis in aqueous solution is given in ISO 14184-1.



## 2 Normative references

The following referenced documents are indispensable for the application 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 139:2005, *Textiles — Standard atmospheres for conditioning and testing*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

## 3 Principle

A weighed fabric specimen is suspended over water in a sealed jar. The jar is placed in an incubator at a controlled temperature for a specified length of time. The amount of formaldehyde absorbed by the water is then determined colorimetrically.

## 4 Reagents

All reagents shall be of 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 1 000 ml volumetric flask and make up to the mark with water (4.1). Store in a brown bottle.

The reagent darkens in colour slightly on standing over the first 12 h. For this reason, the reagent should be held for 12 h before use. Otherwise, the reagent is usable over a considerable period of time, at least 6 weeks. However, 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. As an alternative, the chromotropic acid method described in Annex B may be used.

**4.3 Formaldehyde solution**, approximately 37 % ($M/V$ or $M/m$).

## 5 Apparatus

**5.1 Glass preserving jars**, 0,95 l to 1,0 l with gas-tight sealing caps (see Figure 1).

**5.2 Small wire-mesh baskets** (or other suitable means for suspending the test specimen above the water level inside the jars. As an alternative to the wire-mesh baskets, a double strand of sewing thread may be used to make a loop in the test specimen that has been folded in half twice, suspended above the water level. The two double-thread ends are draped over the top of the jar and held securely by the jar cap.

NOTE A simple support for insertion in the preserving jars can be constructed as follows. A piece of aluminium wire screening 15,2 cm × 14,0 cm is bent around a length of wood 3,8 cm square and fastened together to form a rectangular, open-ended cage. One side is cut at the corners about halfway up the side and the cut section is folded inward and fastened. This folded piece forms the bottom of the wire basket while the other three sides form the support legs. Fastening can be accomplished by twisting short lengths of wire through or around the appropriate part.



**5.3 Incubator**, thermostatically controlled at (49 ± 2) °C.



**5.4 Stoppered volumetric flasks**, 50 ml, 250 ml, 500 ml and 1 000 ml.

**5.5 Pipettes**, 1 ml, 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 30 ml and 50 ml and graduated at intervals of 5 ml.

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

**5.6 Burettes**, 10 ml and 50 ml.

**5.7 Spectrophotometer**, capable of reading absorbance to a minimum of 3 decimal places at a wavelength of 412 nm.

**5.8 Test tubes** or **spectrophotometer tubes**.

**5.9 Water bath**, capable of maintaining a temperature of (40 ± 2) °C.

**5.10 Balance**, accurate to 0,2 mg.

## 6 Preparation of standard solution and calibration

### 6.1 Preparation

Prepare an approximately 1 500 mg/l stock solution of formaldehyde by diluting 3,8 ml of formaldehyde solution (4.3) to 1 litre with water (4.1). Determine the concentration of formaldehyde in the stock solution by the method given in Annex A.

Record the accurate concentration of this standardized stock solution. This stock solution will keep for up to 4 weeks and is used to prepare standard dilutions.

## 6.2 Dilution

The equivalent concentrations of the formaldehyde in the test specimen, based on a mass of 1 g of the test specimen and 50 ml of water, will be 50 times the accurate concentration 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

Dilute 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 of S2 to 500 ml, containing 0,15 µg $CH_2O$/ml = 7,5 mg/kg $CH_2O$ on the fabric

— 2 ml of S2 to 500 ml, containing 0,30 µg $CH_2O$/ml = 15 mg/kg $CH_2O$ on the fabric

— 5 ml of S2 to 500 ml, containing 0,75 µg $CH_2O$/ml = 37,5 mg/kg $CH_2O$ on the fabric

— 10 ml of S2 to 500 ml, containing 1,50 µg $CH_2O$/ml = 75 mg/kg $CH_2O$ on the fabric

— 15 ml of S2 to 500 ml, containing 2,25 µg $CH_2O$/ml = 112,5 mg/kg $CH_2O$ on the fabric

— 20 ml of S2 to 500 ml, containing 3,00 µg $CH_2O$/ml = 150 mg/kg $CH_2O$ on the fabric

— 30 ml of S2 to 500 ml, containing 4,50 µg $CH_2O$/ml = 225 mg/kg $CH_2O$ on the fabric

— 40 ml of S2 to 500 ml, containing 6,00 µg $CH_2O$/ml = 300 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 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 of formaldehyde, a 1,00 g specimen is extracted with 50 ml of water; the solution contains 20 µg of formaldehyde and from this it follows that 1 ml of the test solution contains 0,4 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 testing, store the sample sealed in a container.

From the sample, cut at least two specimens into small pieces and weigh approximately 1 g of the pieces to an accuracy of 10 mg.

NOTE 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.

## 8 Procedure

Pour 50 ml of water (4.1) into the bottom of each jar. Suspend one specimen above the water in each jar, using a wire-mesh basket or other means. Seal the jars and place them in the incubator (5.3) at (49 ± 2) °C for 20 h ± 15 min. Remove and cool the jars for (30 ± 5) min and remove the specimen and baskets, or other support, from the jars. Recap the jars and shake them to mix any condensation formed on the jar sides.

Pipette 5 ml of acetylacetone reagent (4.2) into a suitable number of tubes (5.8), and pipette 5 ml of the acetylacetone reagent into at least one additional tube for a reagent blank. Add 5 ml aliquots from each of the sample-preserving jars to the tubes and 5 ml of water (4.1) to the tube which is used as a reagent blank.

Mix and place the tubes in a water bath (5.9) at (40 ± 2) °C for (30 ± 5) min. Cool and read the absorbance in the colorimeter or spectrophotometer (5.7) against the reagent blank using a wavelength of 412 nm in a 10 mm absorption cell. Determine the formaldehyde concentration, in µg/ml, in the sample solutions using the prepared calibration curve.

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

**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 a considerable time (at least overnight) and reading may be delayed, if desired.**



## 9 Calculation



Calculate the amount of formaldehyde released for each specimen ($w_F$), to the nearest mg/kg, using the following equation:

$$w_F = \frac{\rho \times 50}{m}$$

where

$\rho$ is the concentration of formaldehyde in solution, in mg/l, as read from the calibration graph;

$m$ is the mass of test specimen, in grams.

Calculate the arithmetic mean of the two values.

If the result is less than 20 mg/kg, report it as “not detectable”.

## 10 Test report

The test report shall include the following information:

a) a reference to this part of ISO 14184, i.e. ISO 14184-2:2011;

b) the date the sample was received, the means in which it was stored prior to testing and the date tested;

c) description of the sample tested and how packaged;

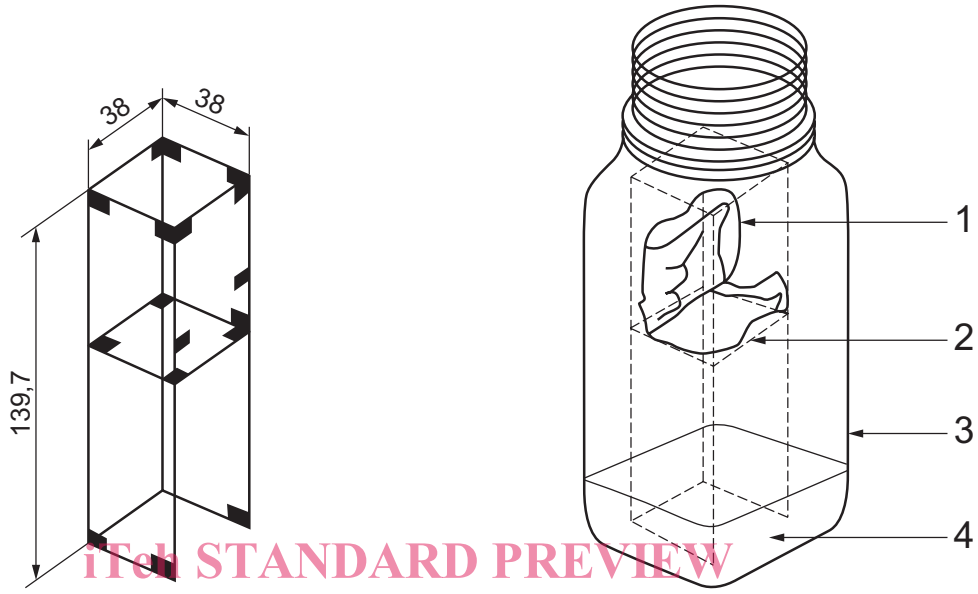
d) the mass of the test specimens;

e) the range of the calibration graph;

f) the amount of formaldehyde released from the sample, expressed as in Clause 9;

g) any deviation, by agreement or otherwise, from the procedure specified.

Dimensions in millimetres






**a) Wire-mesh basket** **b) Sealed jar containing one specimen and a basket**



**Key**

1 fabric specimen
2 basket
3 jar
4 water

**Figure 1 — Wire-mesh basket (aluminium) which is suspended in a sealed jar with one specimen**