INTERNATIONAL STANDARD

ISO 12460-4

Second edition
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Wood-based panels — Determination of formaldehyde release —

Part 4:
Desiccator method

Panneaux à base de bois — Détermination du dégagement de formaldéhyde —

Partie 4: Méthode au dessiccateur

Reference number
ISO 12460-4:2016(E)

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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 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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 89, *Wood-based panels.*

This second edition cancels and replaces the first edition (ISO 12460-4:2008), which has been technically revised with the following changes:

a) introduction was deleted;

b) reference to JANS 16 was deleted in the scope;

c) provisions for low emitting boards were added in 5.6.

It also incorporates the Amendment ISO 12460-4:2008/Amd 1:2011.

ISO 12460 consists of the following parts, under the general title *Wood-based panels — Determination of formaldehyde release*:

— *Part 1: Formaldehyde emission by the 1-cubic-metre chamber method*

— *Part 3: Gas analysis method*

— *Part 4: Desiccator method*

— *Part 5: Extraction method (called the perforator method)*

Wood-based panels — Determination of formaldehyde release —

Part 4:
Desiccator method

1 Scope

This part of ISO 12460 specifies a desiccator method for the determination of the quantity of formaldehyde emitted from particleboard, fibreboard, plywood, oriented strand board (OSB) and wooden laminated flooring.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16999, *Wood-based panels — Sampling and cutting of test pieces*

3 Principle

Emission of formaldehyde is determined by placing test pieces of known surface area in a desiccator at a controlled temperature and measuring the quantity of emitted formaldehyde absorbed in a specified volume of water during 24 h.

4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity.

4.1 Acetylacetone-ammonium acetate solution.

Dissolve 150 g ammonium acetate ($C_2H_3O_2NH_4$) in 800 ml water in a 1 000 ml one-mark volumetric flask (5.9). Add 3 ml glacial acetic acid ($C_2H_4O_2$) and 2 ml acetylacetone (pentane-2,4-dione, $C_5H_8O_2$) and mix thoroughly into the solution. Make up to the mark with water. During storage, protect the solution from light. Discard the solution 3 days after preparation.

4.2 Iodine standard solution, $c(I_2)$ = 0,05 mol/l.

Standardize the solution before use.

4.3 Sodium thiosulfate standard solution, $c(Na_2S_2O_3)$ = 0,1 mol/l.

Standardize the solution before use.

4.4 Sodium hydroxide standard solution, $c(NaOH)$ = 1 mol/l.

Standardize the solution before use.

4.5 Sulfuric acid standard solution, $c(H_2SO_4) = 1$ mol/l.

Standardize the solution before use.

4.6 Starch solution, 1 % mass fraction.

5 Apparatus

The usual laboratory apparatus and, in particular, the following:

5.1 Glass desiccators, with an enclosed volume of (11 ± 2) l capable of enclosing a support (5.2).

5.2 Wire grid or **support**, of diameter (240 ± 15) mm of stainless steel wire such that the distance between parallel pieces of wire is not less than 15 mm (see Figure 1).

5.3 Glass crystallizing dish, circular of inside diameter (115 ± 1) mm and depth (60 ± 2) mm.

5.4 Sample holder, of stainless steel wire, to hold the test pieces upright in the desiccator (see Figure 2).

5.5 Temperature-measuring device, e.g. a thermocouple, capable of measuring temperature with an error limit of ±0,1 °C, placed inside a desiccator (5.1) located adjacent to the desiccator(s) containing the test pieces.

5.6 Spectrophotometer, capable of measuring absorbance at 412 nm. The use of cells of pathlength at least 50 mm is recommended and is required for low emitting boards. Fluorometric determination can also be used to gain more sensitivity.

5.7 Water bath, capable of maintaining a temperature of (65 ± 2) °C.

5.8 Volumetric flasks, six, of capacity 100 ml.

5.9 Volumetric flasks, two, of capacity 1 000 ml.

5.10 Bulb pipettes, of capacities 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml, and 100 ml or suitable auto pipette.

5.11 Microburette or **auto dispenser**.

5.12 Flasks with stoppers, of capacity 100 ml.

5.13 Balance, capable of measuring to 0,001 g.

6 Test pieces

6.1 Sampling

Sampling and cutting of the test pieces shall be carried out in accordance with ISO 16999.

Dimensions in millimetre

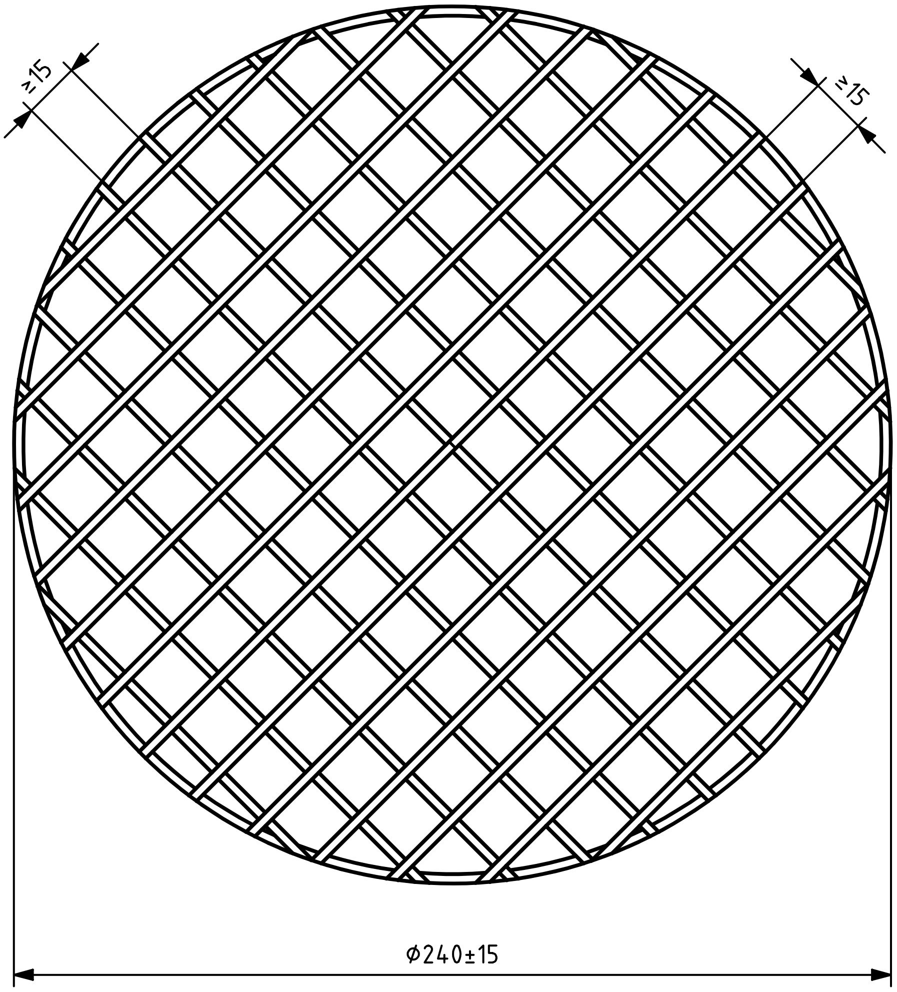
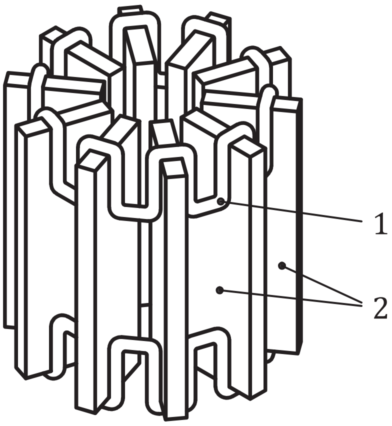


Figure 1 — Stainless steel wire grid for supporting the test pieces in the desiccator



Key

1 supporting metal

2 test pieces

Figure 2 — Example of wire sample holder to hold the test pieces in the desiccator

6.2 Dimensions

The test pieces, of thickness, δ, in millimetres, shall be of length (150 ± 1,0) mm and of width (50 ± 1,0) mm.

6.3 Number of test pieces

The number of test pieces shall be determined by their total surface area. The sum of the areas of the ends, sides and faces shall be as close as possible to 1 800 cm^2.

6.4 Number of emission tests

The emission tests shall be carried out in duplicate.

NOTE For internal routine control, a single emission test might be sufficient.

The differences between emissions of the two tests shall be within 20 % of their arithmetic average, otherwise a third emission test shall be carried out.

6.5 Conditioning

Condition the test pieces for 7 days or to constant mass in an atmosphere with a mean relative humidity of (65 ± 5) % and a temperature of (20 ± 2) °C.

Constant mass is considered to have been reached when the results of two successive weighing operations, carried out at an interval of 24 h, do not differ by more than 0,1 % of the mass of the test piece.

The test pieces to be conditioned shall be separated by at least 25 mm and positioned so that air can circulate freely over all surfaces.

Test pieces containing low levels of formaldehyde will absorb formaldehyde from the atmosphere when background levels of formaldehyde are high. Care should be taken to avoid such conditions during storage and conditioning by use of a formaldehyde removal system or by maintaining low volumes of test pieces in the room. The background level is measured by exposing a glass crystallizing dish (5.3) containing 300 ml water to the conditioning atmosphere for 24 h, and analysing the resulting solution. The maximum background level shall be less than the nominal emission level of the test pieces (e.g. for test pieces from a sample with expected emissions of 0,3 mg/l, the background levels should be less than 0,3 mg/l).

7 Procedure

7.1 Desiccator preparation

7.1.1 New desiccators

Thoroughly clean new desiccators and those previously used for purposes other than the determination of formaldehyde.

7.1.2 Before each determination

Rinse the desiccator (5.1) and the glass dish (5.3) with water and dry them before each determination. Place (300 ± 1) ml of water at (20 ± 1) °C in the glass dish (5.3), and locate it centrally at the bottom of the desiccator. Position the wire mesh support (5.2) above the glass dish within the desiccator.

7.2 Test conditions

Place the desiccator on a vibration-free surface in an environment capable of maintaining the air temperature inside the desiccator at (20 ± 0,5) °C.

7.3 Positioning of the test pieces

Insert the test pieces, free of any loose particles, into the wire sample holder (5.4) before placing it inside the desiccator. Position the sample holder containing the test pieces inside the desiccator at the centre of the wire mesh support so that it is located directly above the glass dish.

7.4 Monitoring of test conditions

7.4.1 Temperature

Prepare a control desiccator (5.1) as specified in 7.1.2 but containing no test pieces, and fit it with a temperature-measuring device (5.5). Monitor the temperature inside the desiccator continuously, or at intervals not exceeding 15 min, and record the mean temperature during the test period.

Alternatively, the temperature may be monitored by locating the temperature-measuring device (5.5) in the test environment adjacent to the desiccator.

7.4.2 Background formaldehyde

Measure the background formaldehyde in the testing environment using the control desiccator (7.4.1). The maximum accepted background level is 0,05 mg/l.

7.5 Test duration

The duration of the test shall be 24 h ± 10 min.

7.6 Sample collection

Thoroughly mix the formaldehyde solution contained in the glass dish. Rinse a 100 ml one-mark volumetric flask (5.8) with the formaldehyde solution and then fill it to the mark with the solution. Use a glass stopper to seal the flask. If the sample is not to be analysed immediately, store it between 0 °C and 5 °C for a maximum of 30 h.

Follow the same procedure for the measurement of background formaldehyde.

8 Determination of formaldehyde

8.1 General

Determine the formaldehyde content of the aqueous solutions photometrically by the acetylacetone method.

8.2 Principle

The determination is based on the Hantzsch reaction in which the formaldehyde reacts with ammonium ions and acetylacetone to yield diacetyldihydrotoluidine (DDL). DDL has an absorption maximum at 412 nm. The reaction is specific to formaldehyde (see Reference [1]).

8.3 Procedure

Pipette (5.10) 25 ml of the formaldehyde solution to be analysed into a 100 ml flask (5.12), add 25 ml of the acetylacetone-ammonium acetate solution (4.1) and seal the flask with a stopper.

Heat the stoppered flask for 10 min in a water bath (5.7) at (65 ± 2) °C. Then store the solution at 20 °C for (60 ± 5) min, protected from light, before determining the absorbance of the solution at a wavelength of 412 nm against water, using a spectrophotometer (5.6). Determine background formaldehyde (7.4.2) using the same procedure.

If maximum absorbance occurs at a wavelength other than 412 nm, all measurements, including calibration, may be conducted at this wavelength.

8.4 Calibration curve

8.4.1 General

The calibration curve is produced from a standard formaldehyde solution, the concentration of which has been determined by iodometric titration. Check the calibration curve at least once a month. It is determined using the method in 8.4.2 or a suitable equivalent.

If monthly calibration show less than 2% variation on the mean values, the calibration curve checking interval may be increased. Records should be kept to justify any increased checking interval.

8.4.2 Formaldehyde standard solution

Dilute about 1 ml formaldehyde solution (mass fraction 35 % to 40 %) in a 1 000 ml volumetric flask (5.9) with water, and make up to the mark with water.

To determine the exact formaldehyde concentration of this solution, mix a 20 ml volume with 25 ml iodine solution (4.2) and 10 ml sodium hydroxide solution (4.4). Allow to stand for 15 min, protected from light, then add 15 ml of sulfuric acid solution (4.5). Back-titrate the surplus iodine with the sodium thiosulfate solution (4.3). Near the end of the titration, add a few drops of starch solution (4.6) as indicator. Perform a blank test on 20 ml water in parallel.

The formaldehyde content, as a concentration in milligrams per litre, $\rho(\mathrm{HCHO})$, is calculated from Formula (1):

$$\rho(\mathrm{HCHO}) = (V_0 - V) \times 15 \times c(\mathrm{Na_2S_2O_3}) \times 1\ 000 / 20 \qquad (1)$$

where

$c(\mathrm{Na_2S_2O_3})$ is the thiosulfate solution (4.3) concentration, in moles per litre;

V is the volume, in millilitres, of thiosulfate solution (4.3) consumed in the test titration;

V_0 is the volume, in millilitres, of thiosulfate solution (4.3) consumed in the blank titration.

NOTE 1 ml 0,1 mol/l thiosulfate solution (4.3) corresponds to 1 ml 0,05 mol/l iodine solution (4.2) and 1,5 mg formaldehyde.

8.4.3 Formaldehyde calibration solution

Using the concentration value determined in 8.4.2, calculate the volume which will contain 3 mg formaldehyde. Transfer this volume, using a microburette (5.11), to a 1 000 ml volumetric flask (5.9) and make up to the mark with water; 1 ml of this calibration solution contains 3 µg formaldehyde.

8.4.4 Determination of the calibration curve

Pipette (5.10) 0 ml, 5 ml, 10 ml, 20 ml, 50 ml and 100 ml of formaldehyde calibration solution (8.4.3) into separate 100 ml volumetric flasks (5.8) and make up to the mark with water. Analyse 25 ml of each dilution photometrically by a procedure similar to that in 8.3. Determine a blank value in parallel, using a solution made up of 25 ml water and 25 ml acetylacetone-ammonium acetate solution (4.1); this is taken into consideration when determining the absorbance values of the calibration solutions. Plot the absorbance values against the formaldehyde concentration, ρ (between 0 mg/l and 3 mg/l). Determine the slope, f, from the graph generated, either manually or by using software.

8.5 Calculation of the concentration of formaldehyde in the glass crystallizing dish in the desiccator

The formaldehyde concentration, G, in milligrams per litre, in the glass crystallizing dish in the desiccator due to the test pieces is calculated using Formula (2):

$$G = f \times (\alpha_{\mathrm{d}} - \alpha_{\mathrm{b}}) \times 1\,800 / A \qquad (2)$$

where

α_{b} is the absorbance of the background formaldehyde solution (7.4.2);

α_{d} is the absorbance of the solution from the desiccator containing the test pieces (8.3);

A is the combined area, in square centimetres, of the test pieces (6.3);

f is the slope, in milligrams per litre, of the formaldehyde calibration curve (8.4.4).

9 Expression of results

The emission of formaldehyde from the samples is measured as the quantity of formaldehyde collected in the glass dish, expressed in milligrams per litre, rounded to the nearest 0,01 mg/l.

Both the arithmetic average and the two (or three) individual values shall be given in the test report.

10 Test report

The test report shall contain at least the following information:

a) a reference to this part of ISO 12460, i.e. ISO 12460-4:2016;

b) the name and address of test laboratory;

c) the sampling report according to ISO 16999;

d) the date of the test report;

e) the panel type, thickness (δ) and density;

f) the date of sampling;

g) the date of formaldehyde determination;

h) the relevant product specification;

i) the surface treatment, if relevant;

j) the specific apparatus used, in case of different possibilities allowed in this part of ISO 12460;

k) the test results expressed as stated in Clause 9;

l) the background level as calculated in 8.5;

m) all deviations from this part of ISO 12460.

Bibliography

[1] Belman S. The fluorimetric determination of formaldehyde. Anal. Chim. Acta. 1963, **29** pp. 120–126

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