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Wood-based panels — Determination of formaldehyde release —

Part 2:
Small-scale chamber method

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

Partie 2: Méthode à la petite chambre

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Contents

Page

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

A list of all the parts in the ISO 12460 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.

Wood-based panels — Determination of formaldehyde release —

Part 2:
Small-scale chamber method

1 Scope

This document specifies a test method to measure the formaldehyde concentrations in air from wood products under defined test conditions of temperature and relative humidity. Results obtained from this small-scale chamber test method are often used for quality assurance and can be comparable to, or can provide useful correlations to, results obtained from testing larger product samples in larger chamber test methods for wood products, such as the 1 m^3 chamber method specified in ISO 12460-1.

NOTE 1 This document is based on the general principles and methods of ASTM D 6007.

NOTE 2 ISO 12460-1 for the 1 m^3 chamber is the reference method for the determination of formaldehyde release. For factory production control the derived test methods according to this document, ISO 12460-3, ISO 12460-4 and ISO 12460-5 are used.

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 12460-1, *Wood-based panels — Determination of formaldehyde release — Part 1: Formaldehyde emission by the 1-cubic-metre chamber method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
air change rate
N
ratio of conditioned and filtered air, Q, that enters or is replaced in the small chamber in one hour divided by the interior volume of the small chamber, V, air changes per hour (ACH)

3.2
loading ratio
L
total exposed surface area, A, excluding panel edges, of the product being tested divided by the test chamber's interior volume, V, in m^2/m^3

Note 1 to entry: L is equal to A/V.

3.3
make-up airflow
Q
quantity of conditioned and filtered air fed into the chamber per unit time, in m^3/h

3.4
***Q*/*A* ratio**
ratio of air flow through the chamber (*Q*) to sample surface area *(A)*, in m/h

3.5
sample surface area
A
total area of all sample faces exposed in the chamber, in m^2

3.6
measured concentration
C
formaldehyde concentration (expressed in mg/m^3) under the defined environmental test parameters of this method

3.7
volume of closed system
V
interior volume of the test chamber, in m^3

4 General

This method measures the quantity of formaldehyde in an air sample from a small chamber as determined by a modification of the National Institute for Occupational Safety and Health (NIOSH) 3500 chromotropic acid test procedure (see Bibliography). Other analytical procedures may be used to determine the quantity of formaldehyde in the air sample provided that such methods give comparable results. The test report shall include full description of the analytical procedure employed.

This test method specifies testing at the loading rate and air exchange rate used in ISO 12460-1. The test results and test report shall be properly qualified and shall specify the make-up air flow, sample surface area, and chamber volume.

Ideal candidates for small-scale chamber testing are products relatively homogeneous in the formaldehyde release characteristics. Still, product inhomogeneities shall be considered when selecting and preparing samples for small-scale chamber testing.

The values stated in SI units are the standard values. Any values given in parentheses are for information only.

NOTE This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

5 Significance and use

5.1 Various national and regional regulations on formaldehyde emission levels have been established for wood panels. This international test method was adapted from the small chamber test method specified in ASTM D6007 where its use has been well established as part of monitoring mill compliance to formaldehyde regulations of the U.S. Department of Housing and Urban development (HUD)[1]. This test method provides a means of testing smaller samples and reduces the time required for testing.

5.2 Formaldehyde concentration levels obtained by this small-scale method can differ from expected in full-scale indoor environments. Variations in product loading, temperature, relative humidity, and air exchange will affect formaldehyde emission rates and thus likely indoor air formaldehyde concentrations.

5.3 This test method requires the use of a chamber of 0,02 m^3 to 1 m^3 in volume to evaluate the formaldehyde concentration in air using the following controlled conditions which are defined within this standard method:

a) conditioning of specimens prior to testing;

b) exposed surface area of the specimens in the test chamber;

c) test chamber temperature and relative humidity;

d) the Q/A ratio;

e) air circulation within the chamber.

6 Interferences

The NIOSH 3500 analytical method lists phenols as a negative interference when present at an 8:1 excess over formaldehyde. Modification in the analytical procedure shall be made when relatively high phenol to formaldehyde concentrations (8:1) are anticipated (see Hakes paper[8] and Tech Bulletin 415[9]). Interferences of other analytical methods should be determined by reference to other applicable standard test methods.

7 Apparatus

7.1 Test chamber

The interior volume of the small chamber shall be from 0,02 m^3 to 1 m^3. The interior of the test chamber shall be free of refrigeration coils that condense water and items such as humidifiers with water reservoirs since water has the potential for collecting formaldehyde and thus influencing test results. The interior surfaces of the small chamber, including any sample support system, shall be a non-absorbent material. Stainless steel, aluminium, and polytetrafluoroethylene (PTFE) have been found appropriate as chamber lining materials. All joints except for doors used for loading and unloading specimens should be sealed. Doors shall be self-sealing.

7.2 Make-up air

7.2.1 General

The make-up air shall come from a filtered dust-free environment and contain no more than 0,006 mg/m^3 of formaldehyde. This can be accomplished by passing make-up air through a filter bed of activated carbon, activated alumina impregnated with potassium permanganate, or other materials capable of absorbing, or oxidizing formaldehyde.

Make-up air for the chamber shall pass through a calibrated air flow measuring device.

7.2.2 Air circulation

Low speed mixing fans or multi-port inlet and outlet diffusers are two techniques that have been used successfully to ensure mixing of the chamber air over all sample surfaces.

7.2.3 Air sampling port

The exhaust flow (that is, chamber outlet) is normally used as the sampling point, although separate sampling ports in the chamber can be used. The sampling system shall be constructed of a material to minimize absorption (for example, glass stainless steel), and the system should be maintained at the same temperature as the test chambers.

8 Hazards

8.1 Chromotropic acid reagent treatment

During this hazardous operation, the operator must wear rubber gloves, apron, and a full face mask or be protected from splashing by a transparent shield such as a hood window. The solution becomes extremely hot during addition of sulfuric acid. If acid is not added slowly, some loss of sample could occur due to splattering (see 11.3.4 and 11.3.5).

8.2 Cleaning chemicals for glassware

Use appropriate precautions if cleaning chemicals are considered to be hazardous.

9 Test specimens

9.1 Standard loading ratio

(*L* or *A*/*V*) is defined as the total exposed specimen surface area, excluding edge area, divided by the chamber volume. The loading ratio, L, shall be (1,0 ± 0,02) m^2/m^3.

9.2 Number of test pieces

Two square test pieces, sized to meet the loading ratio specified in 9.1, shall be cut.

9.3 Nonstandard sample configuration testing products with single surface exposed

Some products have significantly different formaldehyde release characteristics for each surface. In those cases, panels may be tested back-to-back with edges taped together. The panels shall be identified as tested in the back-to-back mode.

10 Sample material handling and specimen conditioning

10.1 Handling

Materials selected for testing shall be wrapped in polyethylene plastic having a minimum thickness of 0,15 mm (6 mil) until sample conditioning is initiated. When testing wood products that are not newly manufactured such as after original application, installation or use, the method of packaging and shipping the products for testing shall be fully described. Information on the age and history of the product shall be detailed in the test report.

10.2 Conditioning

Condition test specimens with a minimum distance of 0,15 m (6 in.) between each specimen for 2 h ± 15 min at the conditions of (24 ± 3) °C (75 ± 5) °F and (50 ± 5) % relative humidity. The formaldehyde concentration in the air within 0,3 m (12 in.) of where panels are conditioned shall be not more than 0,01 mg/m^3 during the conditioning period. Alternative conditioning intervals can give better correlation to larger chamber test methods, such as 7 days ± 3 h conditioning or 15 days conditioning that parallels ISO 12460-1.

The air exchange ratio (*Q*/*V* ratio) shall be (1,0 ± 0,05)/h.

10.3 Sealing of test piece edges

After conditioning, test piece edges shall be partly sealed using self-adhesive aluminium tape to give a constant ratio of the length, *U*, of the open (unsealed) edges to the surface area, *A*, so that $U/A = 1{,}5\ \text{m/m}^2$.

NOTE As a result of the constant ratio $U/A = 1{,}5\ \text{m/m}^2$, the percentage of open edges area related to the surface area depends on the thickness of the test piece, as shown in the following examples.

Panel thickness	Percentage of open edges area
10 mm	1,5 %
19 mm	2,8 %
32 mm	4,8 %

11 Procedure

11.1 Test procedure for materials

11.1.1 Purge the chamber by running empty or with the use of filters designed to reduce the formaldehyde background concentration in air, or both. The formaldehyde background concentration in air of the empty operating chamber shall not exceed $0{,}006\ \text{mg/m}^3$. Clean chamber surfaces with water or suitable solvent if formaldehyde background concentrations approach $0{,}005\ \text{mg/}^3$.

11.1.2 Load specimens to obtain a loading rate, *L*, of $1, \text{m}^2/\text{m}^3$. Locate the specimens in the chamber so that the conditioned air stream circulates over all panel surfaces.

11.1.3 Operate the chamber at (25 ± 1) °C (77 ± 2) °F and (50 ± 4) % relative humidity. Record the temperature, relative humidity, and barometric pressure during the testing period. Conduct the chamber test at the specified Q/V ratio and record this ratio in the report. Conduct the chamber test at the specified Q/V ratio of (1,0 ± 0,05)/h and record this ratio in the report. The air exchange and loading ratios result in a Q/A ratio of 1,0.

11.1.4 After placing the specimens in the chamber, allow time for no less than two full air changes before beginning the air sampling.

11.2 Air sampling

Purge air sampling lines for 1 min. At the sampling station, bubble air through a single impinger containing 20 ml of a 1 % sodium bisulfite ($NaHSO_3$) solution. A filter trap may be placed between the impinger and the flowmeter. Set a calibrated flowmeter to maintain an average airflow of (1 ± 0,05) l/min for 30 min to 60 min with time measured accurately to within 5 s resulting in a minimum air sampling of 30 l to 60 l. For products with very low emission rates, it may be necessary to sample the air for 60 min. Following air sampling, analyse the collection solution.

11.3 Analysis of air samples

The following analysis is based on the chromatropic acid method as described in the ASTM D6007 method from which this standard was derived. Other analytical methods, such as the acetyl acetone method described in ISO 12460-1:2007, Clause 9 is a permitted alternate analytical method. Other methods are acceptable if they provide similar results.

11.3.1 Pipet 4 ml of the $NaHSO_3$ solution from the impinger into each of three 16 mm by 150 mm screwcap test tubes for triplicate analysis of each impinger sample.

11.3.2 Pipet 4 ml of 1 % $NaHSO_3$ into a 16 mm by 150 mm screwcap test tube to act as a reagent blank.

11.3.3 Add 0,1 ml of 1 % chromotropic acid reagent to each test tube. Shake tube after addition.

11.3.4 Slowly and carefully pipet 6,0 ml concentrated sulfuric acid (H_2S0_4) into each test tube (Precaution – see 8.1) and allow to flow down the side of test tube. Allow the volumetric pipet to drain. Do not blow out. Before placing caps on test tubes, check the condition of the polytetrafluoroethylene (PTFE) cap liners to make sure they are clean and not deteriorated.

11.3.5 Slowly and gently agitate test tubes to affect mixing. Mixing is complete when there is no sign of stratification. Caution needs to be taken due to the exothermic chemical reaction. Rapid mixing will cause heating and a pressure increase which may break the test tube. Vent test tubes to release pressure.

11.3.6 If absorbance readings exceed 1,0 or if spectrophotometric analysis is performed within 2 h, heat capped test tubes to 95 °C or place capped test tubes in a boiling water bath for (15 ± 2) min. to ensure that the chemical reaction is completed. Remove tubes from water bath and allow to cool to room temperature.

11.4 Absorbance readings

11.4.1 Standardize the spectrophotometer using distilled water at 580 nm in accordance with the instrument's operating instructions. The reagent blank shall be read against distilled water because an absorbance above 0,100 for the reagent blank indicates contamination of reagent blank or improper solution preparation. If absorbance for the reagent blank compared to distilled water is greater than 0,100, repeat the entire standardization procedure.

11.4.2 Zero the instrument using the reagent blank if the absorbance is not greater than 0,100 (compared to distilled water as zero). Alternatively, the instrument may be left zeroed on distilled water, and the absorbance of the reagent blank subtracted from the absorbance of the standard solutions.

11.4.3 Read and record absorbance at 580 nm for each test tube prepared (see A.4.6 to A.4.9). If the absorbance of the specimen solution is found to fall outside the preferred absorbance range (>1,0), steps 11.3.1 to 11.3.4 may be repeated using an appropriate dilution of each impinger solution.

12 Calculation

12.1 Convert the volume of air sampled to the volume of air at standard conditions as per Formula (1):

$$V_s = \frac{V \times P \times 298}{101 \times (T + 273)} \tag{1}$$

where

V_1 is the volume of air at standard conditions (101 kPa and 298 K), in litres l;

V is the volume of air sampled, in litres l;

P is the barometric pressure, in kPa;

T is the temperature of sample air, in °C.

12.2 Calculate total micrograms of formaldehyde collected in each impinger sample as per Formula (2):

$$C_t = C_a \times F_a \quad (2)$$

where

C_t is the total mass of formaldehyde in the sample, in µg;

C_a is the total quantity of formaldehyde in the sample aliquots taken from the impinger (as determined from the calibration curve in A.4), in µg;

F_a is the aliquot factor:

$$F_a = \frac{\text{sampling solution volume, mL}}{\text{aliquot used, mL}} \quad (3)$$

12.3 Calculate the concentration of formaldehyde in air in the small chamber as per Formula (4):

$$C = C_t / V_S \quad (4)$$

where C is the formaldehyde in mg/m³.

Round calculated formaldehyde concentrations to the nearest 0,01 mg/m³.

NOTE At 23 °C and 1 013 hPa the following relationship exists for formaldehyde:

— 1,24 mg/m³ = 1 ppm (parts per million);

— 1 mg/m³ = 0,81 ppm (parts per million).

12.4 When the chamber temperature differs from 25 by 0,25 °C (77 by 0,50 °F) or more, adjust the formaldehyde concentrations obtained to a standard temperature of 25 °C (77 °F) using a formula developed by Berge et al. Annex A contains a table of conversion factors for use at different observed test temperatures as calculated using this formula. The observed test temperature is the average temperature for the total period of 15 min prior to air sampling plus the time of air sampling.

12.5 The small chamber formaldehyde concentration in air shall be adjusted to a concentration at 50 % relative humidity when the difference in relative humidity from 50 % is greater than or equal to 1 % (see Annex A).

13 Report

Report the following information.

Test number.

13.1 Title of report shall state if standard face and back configuration testing (see 9.1) or if nonstandard configuration testing (see 9.2) was performed.

13.2 The manner in which materials where shipped or stored, or both: wrapped separately in vapour retarder, wrapped collectively in vapour retarder or in original box or container. If materials were shipped unwrapped, or no in the original box or container, it shall be noted in the test report. Information on age and product history, if known, shall be described in the test report.

13.3 Name or product manufacturer or name of company submitting material, or both, date of manufacture, and sampling date (if known).

13.4 Description of test material or product shall include generic product name, thickness, size, if surface is finished or sealed (both surfaces should be described), and special treatment (if known).

13.5 Specimen condition details to include average temperature and range (nearest 0,25 °C), average relative humidity and range (nearest 1 %), and time to the nearest minute.

13.6 Formaldehyde background concentration in the air in the area where specimens are conditioned (rounded to the nearest 0,01 mg/m^3).

13.7 Chamber volume: nominal length, width, and height.

13.8 Chamber Q/L ratio.

13.9 Description of specimens as loaded into chamber including number of specimens in charge and number of surfaces exposed.

13.10 Average temperature and range (nearest 0,25 °C), average relative humidity and range (nearest 1 %), and time to the nearest minute during the sampling period.

13.11 Chamber formaldehyde concentration in air at test conditions; chamber formaldehyde concentration in air corrected to 24 °C, 50 % relative humidity, rounded to the nearest 0,01 mg/m^3.

13.12 The analytical method employed if different from the NIOSH 3500 chromotropic acid test procedure described in this standard.

13.13 Formaldehyde background concentration of air in chamber prior to test and formaldehyde concentration of make-up (rounded to the nearest 0,01 mg/m^3).

13.14 Air-sampling rate and length of sample time.

13.15 Date of test.

Annex A
(normative)

Conversion factors for formaldehyde, standard solutions, calibration curve

A.1 General

Table A.1 is based on the Berge et al[10] formula to correct formaldehyde concentrations in air for temperature:

$$C = C_0 \times e^{-R(1/t-1/t_0)} \tag{A.1}$$

or

$$C_0 = C_e^{-R(1/t-1/t_0)} \tag{A.2}$$

where

C is the test formaldehyde concentration level;

C_0 is the corrected formaldehyde concentration level;

e is the natural log base;

R is the coefficient of temperature (9 799);

t is the actual temperature, in Kelvin, K;

t_0 is the corrected temperature, in Kelvin, K.

Table A.1 — Temperature conversion

Actual		To convert to 25 °C (77 °F) multiple by	Actual		To convert to 25 °C (77 °F) multiply by
°C	(°F)		°C	(°F)	
22,2	(72)	1,36	25,3	(77,5)	0,97
22,5	(72,5)	1,32	25,6	(78)	0,94
22,8	(73)	1,28	25,8	(78,5)	0,91
23,1	(73,5)	1,24	26,1	(79)	0,89
23,3	(74)	1,20	26,4	(79,5)	0,86
23,6	(74,5)	1,17	26,7	(80)	0,83
23,9	(75)	1,13	26,9	(80,5)	0,81
24,2	(75,5)	1,10	27,2	(81)	0,78

NOTE The Berge et al. formula is an exponential function. The greater the variance between actual and corrected temperature, the greater the potential error. Two horizontal lines within the table delineate the specified test temperature ranges (25 ± 1) °C (77 ± 2) °F.

Table A.1 *(continued)*

Actual		To convert to 25 °C (77 °F) multiple by	Actual		To convert to 25 °C (77 °F) multiply by
°C	(°F)		°C	(°F)	
24,4	(76)	1,06	27,5	(81,5)	0,76
24,7	(76,5)	1,03	27,8	(82)	0,74
25,0	(77)	1,00			

NOTE The Berge et al. formula is an exponential function. The greater the variance between actual and corrected temperature, the greater the potential error. Two horizontal lines within the table delineate the specified test temperature ranges (25 ± 1) °C (77 ± 2) °F.

A.2 Relative humidity conversion factors for formaldehyde

A.2.1 Berge et al. formula

Table A.2 is based on the Berge et al. formula to correct formaldehyde concentrations in air for relative humidity:

$$C = C_0\left[1 + A\left(H - H_0\right)\right] \tag{A.3}$$

or

$$C_0 = \frac{C}{1 + A\left(H - H_0\right)} \tag{A.4}$$

where

C is the test formaldehyde concentration level;

C_0 is the corrected formaldehyde concentration level;

A is the coefficient of humidity (0,017 5);

H is the actual relative humidity in %;

H_0 is the relative humidity, in %.

Table A.2 — Relative humidity conversion table for formaldehyde

Actual relative humidity %	To convert to 50 % relative humidity multiply by	Actual relative humidity %	To convert to 50 % relative humidity multiply by
46	1,08	51	0,98
47	1,06	52	0,97
48	1,04	53	0,95
49	1,02	54	0,93
50	1,00		

A.2.2 WKI calculation models

A.2.2.1 General

The calculation models are based on a reference climate of 23 °C and 45 % relative humidity, an air exchange of 1/h and a loading rate of 1 m^2/m^3.

A.2.2.2 Linear model

The linear model according to Formula (A.5) is applicable to a range of relative humidity from 30 % to 50 %:

$$C = 0{,}005\,55 \cdot (C_{\text{reference}} + 0{,}008) \cdot (T - 12{,}7) \cdot (RH - 1{,}2) \frac{1}{1+1{,}75\ \text{N/L}} \tag{A.5}$$

where

C	is the chamber value in ppm;
$C_{\text{reference}}$	is the chamber value (at 23 °C, 45 %, N = 1, L = 1) in ppm;
T	is the temperature in °C;
RH	is the relative humidity in %;
N	is the air exchange per h;
L	is loading rate in m^2/m^3.

A.2.2.3 Exponential model

The exponential model according to Formula (A.6) is applicable to a range of relative humidity from 30 % to 80 %:

$$C = 0{,}0366 \cdot C_{\text{reference}} \cdot (T - 13{,}15) \cdot \left(e^{(0{,}0403 * RH)} + 2{,}073\right) \frac{1}{1+2{,}07 * \text{N/L}} \tag{A.6}$$

where

C	is the chamber value in ppm;
$C_{\text{reference}}$	is the chamber value (at 23°C, 45%, N=1, L=1) in ppm;
T	is the temperature in °C;
RH	is the relative humidity in %;
N	is the air exchange per h;
L	is loading rate in m^2/m^3.

A.3 Standard solution A and B

A.3.1 Standardization of Formaldehyde Standard Solution A (1,0 mg/mL)

A.3.1.1 Pipet 2,70 mL of 37,0 % formaldehyde solution into a 1 L volumetric flask. Dilute to mark with freshly distilled water and mix well. This solution is stable for at least one month.

A.3.1.2 Calibrate the pH meter with standard buffer solution of pH 9,0.

A.3.1.3 Pipet two 50 mL aliquots of formaldehyde standard Solution A into two 150 mL beakers for duplicate analysis and add 20 mL of 1 M sodium sulfite (Na_2SO_3) to each beaker. Sodium sulfite solution can age, thus the 1 M sodium sulfite solution should be adjusted to a 9,5 pH before adding to standard Solution A aliquots.

A.3.1.4 Place solution on magnetic stirrer. Immerse pH electrodes into the solution and carefully titrate with 0,100 N hydrochloric acid (HCl) to the original pH of the solution. Record volume of HCl and corresponding pH intermittently. Make a graph of pH versus volume of HCl.

A.3.1.5 Calculate the concentration, C_A, of formaldehyde standard Solution A in milligrams per millilitre as follows:

$$C_A = \frac{V \times N \times 30{,}03\left(\text{mg per milliequivalent}\right)}{50\left(\text{ml}\right)} \tag{A.7}$$

where

V 0,100 N HCl required at pH of 9,5 from the graph prepared in A.3.1.4, ml;

N normality of HCl. The concentration of standard Solution A will be the average of the two analyses conducted.

A.3.2 Standard Solution B

A.3.2.1 Prepare formaldehyde standard Solution B by diluting 1 mL of standard solution A and 1 g of sodium bisulfite ($NaHSO_3$) to 100 mL in a volumetric flask using distilled water. This standard is stable for at least one week.

A.3.2.2 Calculate the concentration of formaldehyde C_B in standard Solution B in micrograms per millilitre as follows:

$$C_B = \frac{C_A \times 1\,000 \times 1\ \text{ml}}{100} \tag{A.8}$$

A.3.2.3 Record the value.

A.4 Calibration curve

A.4.1 Prepare a 1 % sodium bisulfite ($NaHSO_3$) solution by dissolving 1 g of $NaHSO_3$ in a 100 ml volumetric flask and diluting to the mark with distilled water. This solution is stable at room temperature and should be prepared on a weekly basis.

A.4.2 Label six 16 mm by 150 mm screwcapped test tubes 1, 2, 3, 4, 5, and 6.