INTERNATIONAL STANDARD

ISO 12460-3

Second edition
2015-11-01

# Wood-based panels — Determination of formaldehyde release —

## Part 3:
## Gas analysis method

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

*Partie 3: Méthode d'analyse de gaz*

Reference number
ISO 12460-3:2015(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

# Contents

Page

# 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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 12460-3:2008), which has been technically revised.

The objective of the revision was to improve the detection limit and the reproducibility of the method with regard to boards with low formaldehyde content.

Compared to ISO 12460-3:2008, the following modifications have been made:

a) in 6.1 preparation of test pieces is described more in detail;

b) in 6.2 recommendation of maximum time 72 h after sampling for formaldehyde determination is added;

c) in 6.4 conditioning for sampling and testing in case of dispute is added;

d) in 7.1 and 8.2.2 procedure and evaluation of third determination are modified;

e) in 7.3 use of smaller gas wash bottles and volumetric flasks to improve the sensitivity is included as an option;

f) in 7.4.3 the use of a mixed reagent is included as an option to reduce the amounts of aqueous solution and hence improve the sensitivity;

g) in 7.4.3 temperature of water bath increased to 60 °C and cooling procedure is modified;

h) in 7.4.4.1 minimum interval of check of the calibration curve is extended to once a month;

i) in Clause 9 age and treatment of the sample are included in the test report;

j) in Figure 2 calibration curve is modified.

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)*

Additional parts dealing with small-scale chamber method is planned.

# Wood-based panels — Determination of formaldehyde release —

## Part 3:
## Gas analysis method

## 1 Scope

This part of ISO 12460 specifies a procedure for determination of accelerated formaldehyde release from uncoated and coated wood-based panels using the gas analysis method. The procedure is also suitable for the testing of other materials (e.g. edge bands, floor coverings, foams, foils, laminated wood products, veneered wood products, coated wood products).

## 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 16979, *Wood-based panels — Determination of moisture content*

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

## 3 Principle

A test piece of known surface area is placed in a closed chamber in which the temperature, humidity, airflow, and pressure are controlled to defined values. Formaldehyde released from the test pieces mixes with the air in the chamber. This air is continually drawn from the chamber and passes through gas wash bottles, containing water, which absorbs the released formaldehyde. At the end of the test, the formaldehyde concentration is determined photometrically or fluorimetrically. The formaldehyde release is calculated from this concentration, the sampling time, and the exposed area of the test pieces and is expressed in milligrams per square meter and hour (mg/m$^2$h).

## 4 Reagents

Reagents of recognized analytical purity and distilled or demineralised water (referred throughout the following text as distilled water) shall be used for the analysis.

**4.1 4 ml Acetylacetone solution** are added to a 1 000 ml volumetric flask and made up to the mark with distilled water.

**4.2 200 g ammonium acetate solution** are dissolved with distilled water in a 1 000 ml volumetric flask and made up to the mark.

Optionally, a premixed reagent of acetylacetone and ammonium acetate as described in ISO 12460-4 can be used.

**4.3 Formaldehyde solution** commercially available (concentration typically between 35 % mass fraction to 40 % mass fraction).

### 4.4 Standard iodine solution $c(I_2) = 0{,}05$ mol/l

### 4.5 Standard sodium thiosulphate solution $c(Na_2S_2O_3) = 0{,}1$ mol/l

### 4.6 Standard sodium hydroxide solution $c(NaOH) = 1$ mol/l

### 4.7 Standard sulphuric acid solution $c(H_2SO_4) = 1$ mol/l

### 4.8 Starch solution 1 % by mass

## 5 Apparatus

### 5.1 Main composites of test apparatus (see Figure 1)

**5.1.1 Air filter** (1).

**5.1.2 Wash bottle**, 500 ml, containing ca. 400 ml distilled water (2).

**5.1.3 Desiccator**, 500 ml, containing silica gel (3).

**5.1.4 Air pump** (4).

**5.1.5 Needle valve** (5).

**5.1.6 Equipment for measuring rate of air flow through apparatus** (6).

**5.1.7 Test chamber** (diameter: 90 mm to 100 mm with a length which gives an internal volume of (4 000 ± 200) ml with double casing of stainless steel or glass (7).

**5.1.8 Heating equipment for air** (e.g. copper coil inside the double casing) (8).

**5.1.9 Thermostat** (9).

**5.1.10 Magnetic valves** (10).

**5.1.11 4 pairs of gas wash bottles**, 100 ml or optionally, 4 pairs of gas wash bottles, 30 ml (21).

**5.1.12 Pressure monitor** (22).

**5.1.13 Temperature monitor** (23).

**5.1.14 Test piece holder**, constructed as a shelf with three rods made from stainless steel or another inert material (24).

NOTE The test apparatus described in Figure 1 is based on a waterborne heating system. A test apparatus with an electrical heating system can be used optionally.

### 5.2 Laboratory equipment

**5.2.1 Ventilated oven**, as described in ISO 16979 for determination of moisture content (if requested).

**5.2.2 Spectrophotometer**, with cells of 50 mm optical path length and capable of measuring absorbance at 412 mm.

**5.2.3 Water bath**, capable of maintaining a temperature of (60 ± 1) °C.

**5.2.4 Water bath**, capable of maintaining a temperature in the range of 20 °C to 25 °C.

**5.2.5 Six volumetric flasks**, 100 ml (calibrated at 20 °C).

**5.2.6 Four volumetric flasks**, 250 ml or optionally, four volumetric flasks, 100 ml (calibrated at 20 °C).

**5.2.7 two volumetric flasks**, 1 000 ml (calibrated at 20 °C).

**5.2.8 Volumetric pipettes** (calibrated at 20 °C), 1 ml, 2 ml, 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml, 100 ml.

**5.2.9 Six flasks**, 50 ml (with stoppers).

**5.2.10 Microburette**.

**5.2.11 Burette**, 50 ml, graduated (calibrated at 20 °C).

**5.2.12 Balance**, capable of measuring to 0,00 1 g.

# 6 Sampling and preparation of test pieces

## 6.1 Preparation of test pieces

Three test pieces, each with the dimensions of (400 ± 1) mm × (50 ± 1) mm × board thickness, shall be prepared for the determination of formaldehyde release giving a total emitting surface area of 0,04 $m^2$.

If the sample available does not allow the preparation of test pieces of the specified dimension, then the combined emitting surface area of the test pieces(s) should be as close as possible to 0,04 $m^2$.

For testing layer glued materials (e.g. plywood, veneered particleboard), specimens shall be cut from the respective panel with the fibre direction of the faces perpendicular to the longitudinal axis of the specimen.

Each test piece has to be hermetically wrapped immediately after cutting and stored at ambient temperature.

Before testing, each test piece shall be stored hermetically wrapped at least one day at ambient temperature in order to improve the repeatability. For factory production control with hot test pieces, a valid correlation has to be established.

For testing, the test pieces shall be edge sealed with temperature resistant (i.e. ≥60 °C) self-adhesive aluminium tape or an alternative sealing method if equivalence has been demonstrated. The emitting (unsealed) surface area of the sealed test piece has to be measured and calculated in square metres ($m^2$).

## 6.2 Selection of test pieces for factory production control

Sampling and cutting of the test pieces shall be performed according to the principles of ISO 16999.

Test pieces are taken, uniformly distributed over the width of the (cooled) board, but excluding a 250 mm wide strip from the end of each board.

The formaldehyde determination should be carried out not more than 72 h after sampling.

### 6.3 Selection of test pieces for other purposes

The procedure of sampling, preparation of the test pieces, and conditioning (e.g. from boards already installed) shall be noted and described in the test report. The number and dimensions of the test pieces shall be as given in 6.1.

### 6.4 Selection of test pieces in case of dispute

If this method is used in case of dispute, e.g. linked to disagreement about compliance and not otherwise agreed upon, the conditioning of the test pieces shall be carried out according to the following reference conditions.

The test pieces shall be conditioned to constant mass at a temperature of (20 ± 2) °C and a relative humidity of (65 ± 5) %.

Constant mass is considered to have been reached when the results of two successive weighings, carried out at intervals of not less than 24 h, do not differ by more than 0,1 % of the mass of the test pieces. Alternative two weeks of conditioning can be used.

Contamination of test pieces from other sources of formaldehyde during conditioning shall be avoided.

## 7 Procedure

### 7.1 Number of determinations

Determination shall always be made in duplicate using two different test pieces prepared according to 6.1. A third determination shall be carried out

- if the average emission value of the two determinations is >1,0 mg/m$^2$h and the two replicates deviate more than 20 % of the average value, or
- if the average emission value of the determinations is ≤1,0 mg/m$^2$h and the two replicates deviate more than 0,2 mg/m$^2$h from the average value.

For factory production control, a single determination can be sufficient.

### 7.2 Determination of moisture content

Moisture content shall be determined according to ISO 16979 using a separate sample (see 6.1).

### 7.3 Determination of formaldehyde release

Seal the edges of the test pieces in accordance with 6.1.

Close chamber (5.1.7) and preheat it to (60 ± 0,5) °C.

Connect two gas wash bottles (see 5.1.11), each containing between 20 ml and 40 ml distilled water, in series to the outlet of each magnetic valve (see 5.1.10) using flexible tubing.

To improve the sensitivity of the analytical procedure, 30 ml gas wash bottles with a reduced volume of water may be used optionally

The water volume is chosen to maintain an overpressure of (1 100 ± 100) Pa in the test chamber. The pressure in the test chamber is monitored during the entire test period (see 5.1.12).

Place a test piece in the preheated test chamber. After closing the test chamber and starting the test, the test piece is uniformly exposed to practically formaldehyde free, heated air (60 ± 0,5) °C with a relative humidity of ≤3 %. Immediately set the airflow into the chamber to (60 ± 3) l/h using the needle valve

(5.1.5) and the air volume meter (5.1.6). This air is led into one of a series of pairs of gas wash bottles via a magnetic valve (5.1.10).

Instead of leading dry and clean air through gas wash bottles, dry and clean compressed air may be used optionally.

As the formaldehyde released from the test piece shall be determined at hourly intervals (up to 4 h from starting the test), a new series of gas wash bottles has to be connected every hour. This exchange should be automatic.

Transfer the contents of each pair of gas wash bottles to a 250 ml volumetric flask (5.2.6). Rinse the bottles and their associated tubing thoroughly and transfer the rinsings to the flask.

To improve the sensitivity of the analytical procedure, 100 ml volumetric flasks (5.2.5) may be used optionally, possibly in combination with 30 ml gas wash bottles.

Fill to volume with distilled water and determine the formaldehyde content as specified in 7.4.

## 7.4 Determination of formaldehyde content of the aqueous solutions

### 7.4.1 General

The formaldehyde content of the aqueous solution from each one hour sampling period shall be determined photometrically.

A fluorimetric determination may be used optionally. The description of a procedure is given, e.g. in EN 717-1.

### 7.4.2 Principle

The determination is based on the Hantzsch reaction in which aqueous formaldehyde reacts with ammonium ions and acetylacetone to yield diacetyldihydrolutidine (DDL). DDL has an absorption maximum at 412 nm. The reaction is highly specific to formaldehyde.

Other suitable photometric procedure can also be used.

### 7.4.3 Analytical Procedure

10 ml are taken from the aqueous solution (see 7.3) with a pipette (5.2.8) and added to 10 ml acetylacetone solution (4.1) and 10 ml ammonium acetate solution (4.2) in a stoppered flask (5.2.9).

To improve the sensitivity of the analytical procedure, equal amounts of aqueous solution (see 7.3) and mixed reagent (see 4.2) can be added optionally considering that the service life of such a mixture is shorter than the service life of the separate solutions.

The flask is stoppered, shaken, and heated for 10 min in a water bath (5.2.3) at 60 °C. The heated flask is then cooled in a water bath (5.2.4) operated in a temperature range between 20 °C and 25 °C for at least 15 min protected from sunlight or is cooled to room temperature protected against the influence of light (for about 1 h).

The absorbance of this solution is determined at a wavelength of 412 nm against distilled water using a spectrophotometer (5.2.2). A blank value is determined in parallel with distilled water and taken into consideration in the determination of the gas analysis value.

### 7.4.4 Calibration curve

#### 7.4.4.1 General

The calibration curve is produced from a standard formaldehyde solution, the concentration of which has been determined by iodometric titration. The calibration curve shall be checked at least once a week. It is possible to modify this frequency if it is proved that the slope of the standard curve does not deviate. In this case, the checking shall be made at least once a month and for each change of reagents.

### 7.4.4.2 Formaldehyde standard solution

Reagents:

| | | |
|---|---|---|
| — Standard iodine solution | $c(I_2)$ | = 0,05 mol/l |
| — Standard sodium thiosulphate solution | $c(Na_2S_2O_3)$ | = 0,1 mol/l |
| — Standard sodium hydroxide solution | $c(NaOH)$ | = 1 mol/l |
| — Standard sulphuric acid solution | $c(H_2SO_4)$ | = 1 mol/l |
| — Starch solution | | = 1 % |

The solutions shall be standardized before use.

About 1 ml formaldehyde solution (concentration 35 % to 40 %) is diluted in a 1 000 ml volumetric flask (5.2.6) with distilled water and made up to the mark. The exact formaldehyde concentration of this solution is determined as follows.

20 ml of the formaldehyde standard solution are mixed with 25 ml iodine solution and 10 ml sodium hydroxide solution. After 15 min standing protected from light, 15 ml of sulphuric acid solution is added. The surplus iodine is back-titrated with the thiosulphate solution. At the end of the titration, some drops of starch solution are added as an indicator. A blank test with 20 ml distilled water is carried out in parallel.

The formaldehyde content is calculated using Formula (1):

$$c(\mathrm{HCHO}) = \left(V_0 - V\right) \times 15 \times c\left(\mathrm{NA_2S_2O_3}\right) \times 1\,000 / 20 \qquad (1)$$

where

$c(\mathrm{HCHO})$ is the formaldehyde concentration, in milligrams per litre (mg/l);

$c(Na_2S_2O_3)$ is the thiosulfate concentration, in mols per litre (mol/l);

$V_0$ is the volume of thiosulphate titration solution for the blank test, in millilitres (ml);

$V$ is the volume of thiosulphate titration solution for the test, in millilitres (ml).

NOTE 1 ml 0,1 mol/l thiosulphate solution corresponds to 1 ml 0,05 mol/l iodine solution and 1,5 mg formaldehyde.

Other titration procedures such as the method described by ISO 2227 or ISO 14184-1 can be used for the determination of the formaldehyde concentration.

### 7.4.4.3 Formaldehyde calibration solution

Using the concentration determined in 7.4.4.2, calculate the volume which will contain 3 mg formaldehyde. Transfer this volume using a microburette to a 1 000 ml volumetric flask and make up to the mark with distilled water. 1 ml of this calibration solution contains 3 µg formaldehyde.

### 7.4.4.4 Determination of the calibration curve

Pipette 0 ml, 1 ml, 2 ml, 5 ml, 10 ml, 20 ml, 50 ml, and 100 ml of formaldehyde calibration solution (see 7.4.4.3) in 100 ml volumetric flasks and fill to volume with distilled water. 10 ml of each dilution are analysed photometrically by the same procedure as described above (see 7.4.3). The absorbance values are plotted against the formaldehyde concentrations, *c* (between 0 mg/ml and 0,003 mg/ml), and the slope, *f*, is calculated (see example in Figure 2).

## 8 Expression of results

### 8.1 Gas analysis value

For each 1 h sampling period, the gas analysis value, $G_i$, in milligrams per square metre and hour (mg/m²h) is determined and calculated using Formula (2):

$$G_i = \frac{\left(A_S - A_B\right) \times f \times V}{F} \qquad (2)$$

where

$G_i$ is the formaldehyde content of the solution for each hourly sample in milligrams (mg) divided by the area of the exposed, unsealed surface in square metres (m²);

$i$ is the first, second, third, or fourth hour;

$A_S$ is the absorbance of the solution from the wash bottles;

$A_B$ is the absorbance of distilled water;

$f$ is the slope of the calibration curve for standard formaldehyde solution, in milligrams per millilitre (mg/ml);

$F$ is the combined area of the emitting (unsealed) surface, in square metres (m²) (see 6.1);

$V$ is the volume of the volumetric flask, in millilitres (ml).

### 8.2 Calculation of results

#### 8.2.1 Calculation of results of test pieces

If the formaldehyde content of the liquid absorbent accumulated during the 1st hour is lower than that of the 2nd hour, the formaldehyde yield shall be calculated from the sum of the contents collected during the 2nd to the 4th hour and is related to the surface area, *F*, of the test pieces. If the formaldehyde accumulated in the 1st hour is equal to or greater than that of the 2nd hour, the sum of all four hourly samples shall be used in the calculation.

Consequently, the arithmetic average formaldehyde gas analysis value, $G_m$, in milligrams per square metre and hour (mg/m²h) of a test piece is calculated according to either Formula (3) or Formula (4):

$$G_m = \frac{G_2 + G_3 + G_4}{3} \qquad (3)$$

or

$$G_m = \frac{G_1 + G_2 + G_3 + G_4}{4} \qquad (4)$$

where

$G_m$ is the average gas analysis value of a test piece, in milligrams formaldehyde per square metre and hour (mg/m²h).

As a rule, the formaldehyde content of the liquid absorbent accumulated during the 1st hour is lower than that of the 2nd hour as the temperature of the test piece does not reach 60 °C immediately.

### 8.2.2 Calculation of emission report value

Calculate the gas analysis value of the sample as the mean value of the, $G_m$, values of the two test pieces.

If three replicates are carried out (see 7.1), calculate the gas analysis value as the mean value of all three, $G_m$, values of the test pieces unless

— the average, $G_m$, value is >1,0 mg/m²h and one replicate deviates more than 20 % of the closest value. Then, calculate the gas analysis value as the mean value of the remaining two, $G_m$, values, or

— the average $G_m$ value is ≤1,0 mg/m²h and one replicate deviates more than 0,2 mg/m²h of the closest value. Then, calculate the gas analysis value as the mean value of the remaining two, $G_m$, values.

Results shall be expressed to one decimal place.

## 8.3 Moisture content

See ISO 16979.

## 9 Test report

The test report shall be in accordance with ISO 16999 and shall contain the following additional information:

a) a reference to this part of ISO 12460, i.e. ISO 12460-3;

b) origin of the board;

c) place, location[1], and state[2] of the board at the time of sampling;

d) type of board;

e) thickness of board (mm);

f) density of board (kg/m³);

g) date of manufacture of the board;

h) date of sampling;

i) information referring to the board regarding finishing, sanding, etc.;

j) date of formaldehyde determination;

k) age and treatment of the sample before analysis (e.g. plywood);

l) the average gas analysis value of the sample (see 8.2.2) together with each individual, $G_m$, value (see 8.2.1), in milligrams formaldehyde per square metre per hour (mg/m²h);

m) deviations from the procedures described in this part of ISO 12460 (e.g. dimension of test piece);

n) if requested, moisture content of sample at the time of testing (see ISO 16979);

o) limit of quantification, in milligrams formaldehyde per square metre per hour (mg/m²h) and the estimated uncertainty of measurement, in percent (%) or in in milligrams formaldehyde per square metre per hour (mg/m²h).

---

[1] For example, factory, etc. in the case of installed boards, ceiling, floor, wall, etc.

[2] For example, moisture content, surface coating, finishing.