# INTERNATIONAL STANDARD

# ISO 12460-3

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# 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 des gaz*

Reference number
ISO 12460-3:2008(E)

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# Foreword

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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 12460-3 was prepared by Technical Committee ISO/TC 89, *Wood-based panels*.

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 2: Small-scale chamber method*
- *Part 3: Gas analysis method*
- *Part 4: Desiccator method*

The extraction (or perforation) method will form the subject of a future Part 5.

# Introduction

ISO 12460-1 specifies the 1-cubic-metre chamber reference method for the determination of formaldehyde release. Derived test methods specified in ISO 12460-2, ISO 12460-3 and ISO 12460-4 are intended for factory production control.

# 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 wood-based panels using the gas analysis method.

## 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 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, air flow and pressure are controlled to defined values. Formaldehyde released from the test piece 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. The formaldehyde release is calculated from this concentration, the sampling time and the exposed area of the test piece, and is expressed in milligrams per square meter hour.

## 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 solution.

Add 4 ml acetylacetone (pentane-2,4-dione, $C_5H_8O_2$) to a 1 000 ml volumetric flask (5.2.6) and make up to the mark with water.

### 4.2 Ammonium acetate solution.

Dissolve 200 g ammonium acetate ($C_2H_3O_2NH_4$) in water in a 1 000 ml volumetric flask (5.2.6) and make up to the mark with water.

Commercially prepared solutions may be used, provided they can be shown to give results equivalent to those obtained with solutions prepared in the laboratory.

**4.3 Iodine standard solution**, $c(I_2) = 0{,}05$ mol/l.

Standardize the solution before use.

**4.4 Sodium thiosulfate standard solution**, $c(Na_2S_2O_3) = 0{,}1$ mol/l.

Standardize the solution before use.

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

Standardize the solution before use.

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

Standardize the solution before use.

**4.7 Starch solution**, 1 % mass fraction.

## 5 Apparatus

### 5.1 Test apparatus

The set-up (see Figure 1) comprises the following main components.

**5.1.1 Air filter**.

**5.1.2 Wash bottle**, of capacity 500 ml, containing about 400 ml distilled water.

**5.1.3 Desiccator**, of capacity 500 ml, containing silica gel.

**5.1.4 Air pump**.

**5.1.5 Needle valve**.

**5.1.6 Air flow meter**.

**5.1.7 Test chamber**, of diameter 90 mm to 100 mm and of such a length as to give an internal volume of (4 000 ± 200) ml, with double casing of stainless steel, glass or other suitable inert material.

**5.1.8 Air-heating equipment**, e.g. copper coil inside double casing.

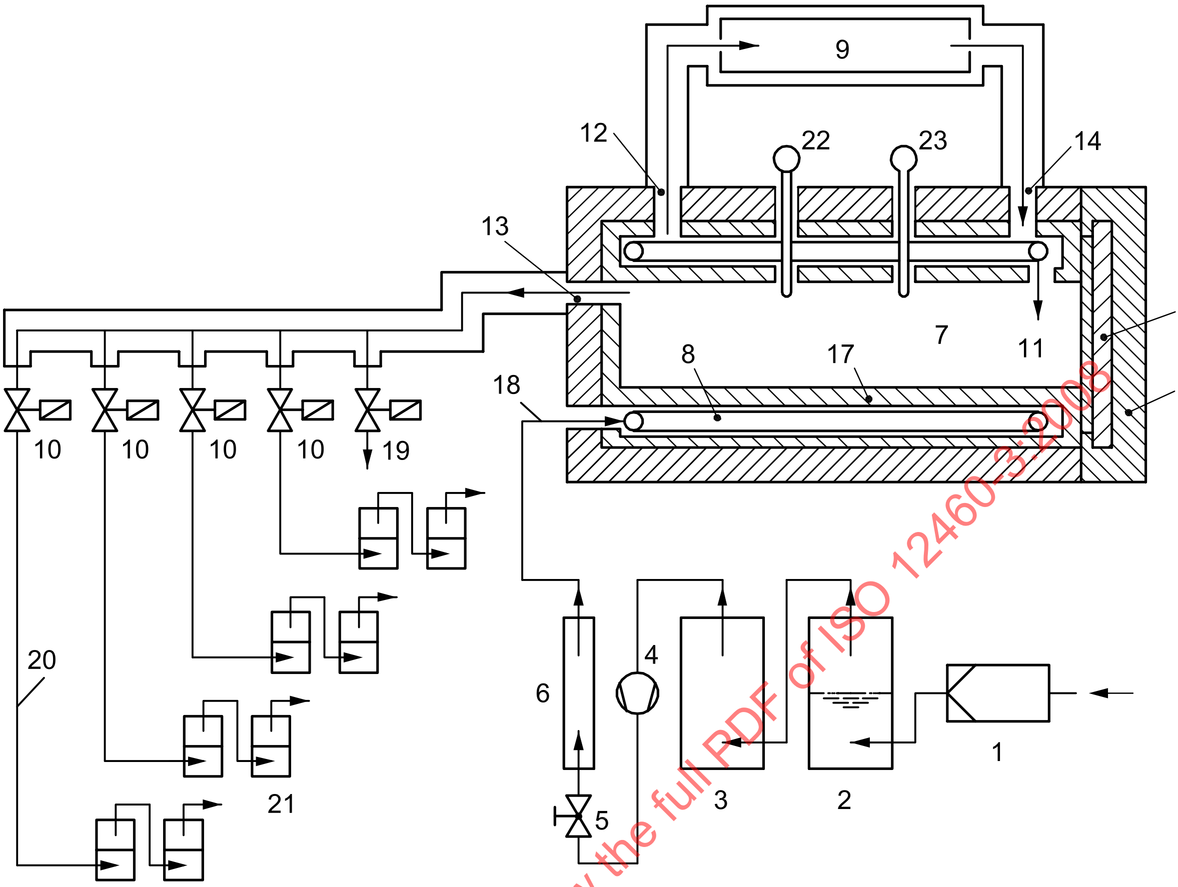
**5.1.9 Thermostat**.

**5.1.10 Magnetic valves**.

**5.1.11 Wash bottles**, each of capacity 100 ml, four pairs.

**5.1.12 Pressure gauge**.

**5.1.13 Thermometer**.



**Key**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | air filter | 7 | test chamber | 13 | test air outlet | 19 | magnetic purging valve |
| 2 | wash bottle | 8 | air-heating coil | 14 | heating medium inlet | 20 | connection tube |
| 3 | desiccator | 9 | thermostat | 15 | insulation | 21 | wash bottles (pair) |
| 4 | air pump | 10 | magnetic valves | 16 | test chamber door | 22 | pressure gauge |
| 5 | needle valve | 11 | test chamber air inlet | 17 | double casing | 23 | thermometer |
| 6 | air flow meter | 12 | heating medium outlet | 18 | heating coil air inlet | | |

**Figure 1 — Gas analysis test apparatus**

### 5.2 Laboratory equipment

Usual laboratory equipment and in particular the following.

**5.2.1 Ventilated oven**, as specified in ISO 16979.

**5.2.2 Spectrophotometer**, with cells of optical pathlength 50 mm, capable of measuring absorbance at 412 nm.

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

**5.2.4 Volumetric flasks**, six, each of capacity 100 ml.

**5.2.5 Volumetric flasks**, four, each of capacity 250 ml.

**5.2.6 Volumetric flasks**, two, each of capacity 1 000 ml.

**5.2.7** **Wash bottles**, eight, each of capacity 100 ml.

**5.2.8** **Bulb pipettes**, of capacities 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml, and 100 ml.

**5.2.9** **Flasks with stoppers**, six, each of capacity 50 ml.

**5.2.10** **Microburette**.

**5.2.11** **Burette**, of capacity 50 ml.

**5.2.12** **Balance**, capable of measuring to 0,001 g.

## 6 Test pieces

### 6.1 Selection of test pieces for production control

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

All test pieces have to be placed in a hermetically sealed container immediately after cutting and stored at room temperature.

Test pieces are taken uniformly distributed over the width of the (cooled) board, but excluding a 500 mm wide strip at each end of the board.

**6.1.1** For the determination of formaldehyde release, three test pieces of dimensions 400 mm × 50 mm × $d$, where $d$ is the board thickness.

**6.1.2** For the determination of moisture content, five or six test pieces of dimensions 25 mm × 25 mm × $d$.

### 6.2 Selection of test pieces for other purposes

The procedure of taking test pieces (e.g. from boards already installed) shall be recorded and included in the test report. The number and dimensions of the test pieces shall comply with 6.1.

### 6.3 Preparation of test pieces

The test pieces shall be edge sealed.

NOTE Three coats of polyurethane lacquer or self-adhesive aluminium tape have proved to be suitable.

## 7 Procedure

### 7.1 Number of determinations

Two determinations shall be made, each using different test pieces (6.1.1). If the individual mean values of a duplicate determination differ from each other by more than 0,5 mg/m$^2$ h, then a third determination shall be carried out.

NOTE For internal inspection, a single determination can be sufficient.

### 7.2 Determination of moisture content

Moisture content can be determined according to ISO 16979 using different test pieces (see 6.1.2).

## 7.3 Determination of formaldehyde release

Seal the edges of the test pieces in accordance with 6.3.

Close the chamber (5.1.7) and pre-heat it to (60 ± 0,5) °C.

Connect two wash bottles (5.1.11), each containing between 20 ml and 30 ml of water, in series to the outlet of each magnetic valve (5.1.10) using flexible tubing.

NOTE The water volume is chosen to maintain the test chamber at 1,0 kPa to 1,2 kPa above atmospheric pressure.

Place a test piece (6.1.1) in the pre-heated test chamber. After closing the test chamber and starting the test, the test piece is uniformly exposed to practically formaldehyde-free air, heated to (60 ± 0,5) °C, with a relative humidity $\leqslant 3$ %. Immediately set the air flow rate into the chamber at (60 ± 3) l/h, by starting the pump (5.1.4) and adjusting the needle valve (5.1.5) as necessary after reading the air flow meter (5.1.6). This air is led into one of a series of pairs of wash bottles (5.1.2) via a series of magnetic valves (5.1.10).

Determine the formaldehyde released from the test piece at hourly intervals for up to 4 h from starting the test. Connect up a new series of wash bottles every hour. This exchange should be automatic.

Over the whole test period, monitor (Figure 1, label 22) the pressure in the test chamber and maintain it at 1,0 kPa to 1,2 kPa above that of the ambient atmosphere.

Transfer the contents of each pair of wash bottles to a 250 ml volumetric flask (5.2.5). Rinse the bottles and their associated tubing thoroughly with water and transfer the rinsings to the flask.

**CAUTION — Care should be taken that the combined contents of the bottles and rinsings do not exceed a volume of 250 ml.**

Make the flask up to the mark with 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

Determine photometrically the formaldehyde content of the aqueous solution from each sampling period of 1 h.

### 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 (see Reference [4]).

NOTE Other suitable photometric procedures can also be used.

### 7.4.3 Procedure

Pipette (5.2.8) 10 ml of aqueous solution (7.3) into a 50 ml flask (5.2.9) and add 10 ml acetylacetone solution (4.1) and 10 ml ammonium acetate solution (4.2). Stopper the flask, shake and warm it for 10 min in a water bath (5.2.3) at 60 °C. Store the solution at room temperature for about 1 h protected from light. Then determine the absorbance of the solution 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 month.

#### 7.4.4.2 Formaldehyde standard solution

Dilute about 1 ml of formaldehyde solution (mass fraction 35 % to 40 %) in a 1 000 ml volumetric flask (5.2.6) 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 (4.3) and 10 ml sodium hydroxide solution (4.5). Allow to stand for 15 min, protected from light, then add 15 ml of sulfuric acid solution (4.6). Back-titrate the surplus iodine with the thiosulfate solution (4.4). Near the end of the titration, add a few drops of starch solution (4.7) 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 Equation (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 (4.4) concentration, in moles per litre;

$V$ is the volume, in millilitres, of thiosulfate solution (4.4) consumed in the test titration;

$V_0$ is the volume, in millilitres, of thiosulfate titration solution (4.4) consumed in the blank titration.

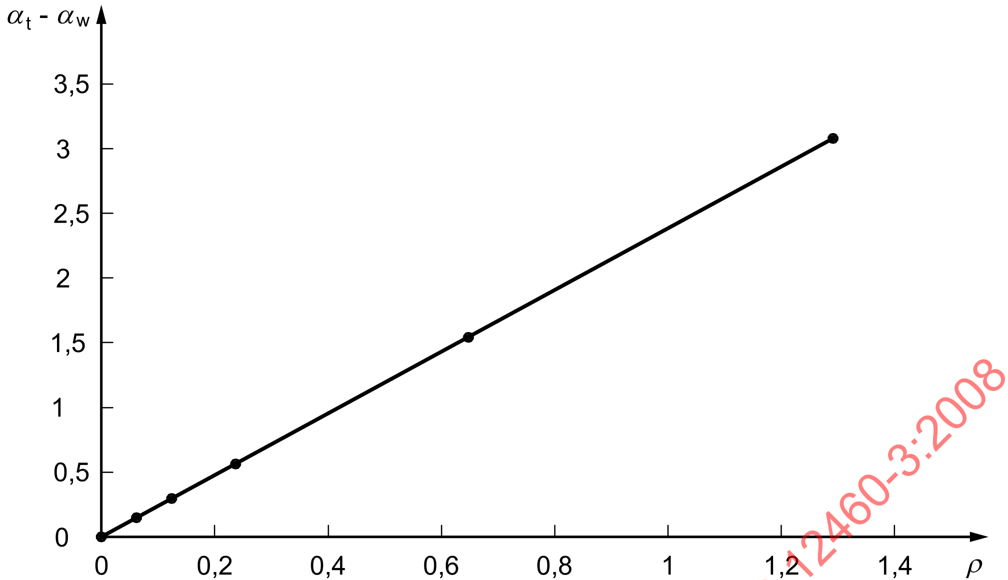
NOTE 1 ml 0,1 mol/l thiosulfate solution (4.4) corresponds to 1 ml 0,05 mol/l iodine solution (4.3) and 1,5 mg formaldehyde.

#### 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 (5.2.10), to a 1 000 ml volumetric flask (5.2.6) and make up to the mark with water; 1 ml of this calibration solution contains 3 µg formaldehyde.

#### 7.4.4.4 Determination of the calibration curve

Pipette (5.2.8) 0 ml, 5 ml, 10 ml, 20 ml, 50 ml and 100 ml of formaldehyde calibration solution (7.4.4.3) into separate 100 ml volumetric flasks (5.2.4) and make up to the mark with water. Analyse 10 ml of each dilution photometrically by a procedure similar to that in 7.4.3. Plot the absorbance values against formaldehyde concentrations, $\rho$ (between 0 and 0,003 mg/ml), either manually or by using software (see Figure 2). Determine the slope, $f$, from the graph generated.



$\rho = f(\alpha_t - \alpha_w)$

**Key**

| | |
|---|---|
| $\alpha_t - \alpha_w$ | absorbance |
| $\rho$ | calibration solution concentration, µg/ml |

**Figure 2 — Example of a calibration curve for formaldehyde determined by the acetylacetone method (pathlength, 50 mm)**

## 8 Expression of results

### 8.1 Gas analysis value

For each solution from each sampling period of 1 h, determine the formaldehyde areal yield, $G_i$ , where $i$ is 1, 2, 3 or 4, in milligrams per square metre, from Equation (2):

$$G_i = \frac{(\alpha_t - \alpha_w) f V}{A} \tag{2}$$

where:

$\alpha_t$ is the absorbance of the solution from the wash bottles;

$\alpha_w$ is the absorbance of water;

$A$ is the combined area, in square metres, of the emitting (unsealed) surface;

$f$ is the slope, in milligrams per millilitre, of the formaldehyde calibration curve (7.4.4.4);

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

### 8.2 Calculation of results

As a rule, the formaldehyde content in the liquid absorbent accumulated during hour 1 is lower than that of hour 2, as the temperature of the test piece does not reach 60 °C immediately. In this case, the formaldehyde areal yield is calculated from the sum of the contents accumulated for hours 2 to 4 and is related to the