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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 de gaz*

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 89, *Wood based panels,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 112, *Wood based panels,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 12460-3:2020), which has been technically revised.

The main changes are as follows:

- laser spectroscopy introduced as a further analytical procedure;
- ready to use formaldehyde standards considered in 8.4.4.3.

A list of all 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 3:
## Gas analysis method

## 1 Scope

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

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

## 3 Terms and definitions

No terms and definitions are listed in this document.

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

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

## 4 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 ($mg/m^2 \cdot h$).

## 5 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.

**5.1 4 ml acetylacetone solution** are added to a 1 000 ml volumetric flask and made up to the mark with distilled water.

**5.2 200 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.

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

**5.4 Standard iodine solution,** $c(I_2)$ = 0,05 mol/l.

**5.5 Standard sodium thiosulphate solution,** $c(Na_2S_2O_3)$ = 0,1 mol/l.

**5.6 Standard sodium hydroxide solution,** $c(NaOH)$ = 1 mol/l.

**5.7 Standard sulphuric acid solution,** $c(H_2SO_4)$ = 1 mol/l.

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

## 6 Apparatus

**6.1 Main composites of test apparatus** (see Figure 1).

**6.1.1 Dust or particle filter**.

**6.1.2 Formaldehyde filter** (bottle filled with water, silicagel or other formaldehyde absorber).

**6.1.3 Desiccator**, 500 ml, containing silica gel.

**6.1.4 Air pump**.

**6.1.5 Needle valve**.

**6.1.6 Equipment for measuring rate of air flow through apparatus**.

NOTE Needle valve and equipment for measuring rate of air flow can be replaced by a mass flow controller.

**6.1.7 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.

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

**6.1.9 Thermostat**.

**6.1.10 Magnetic valves**.

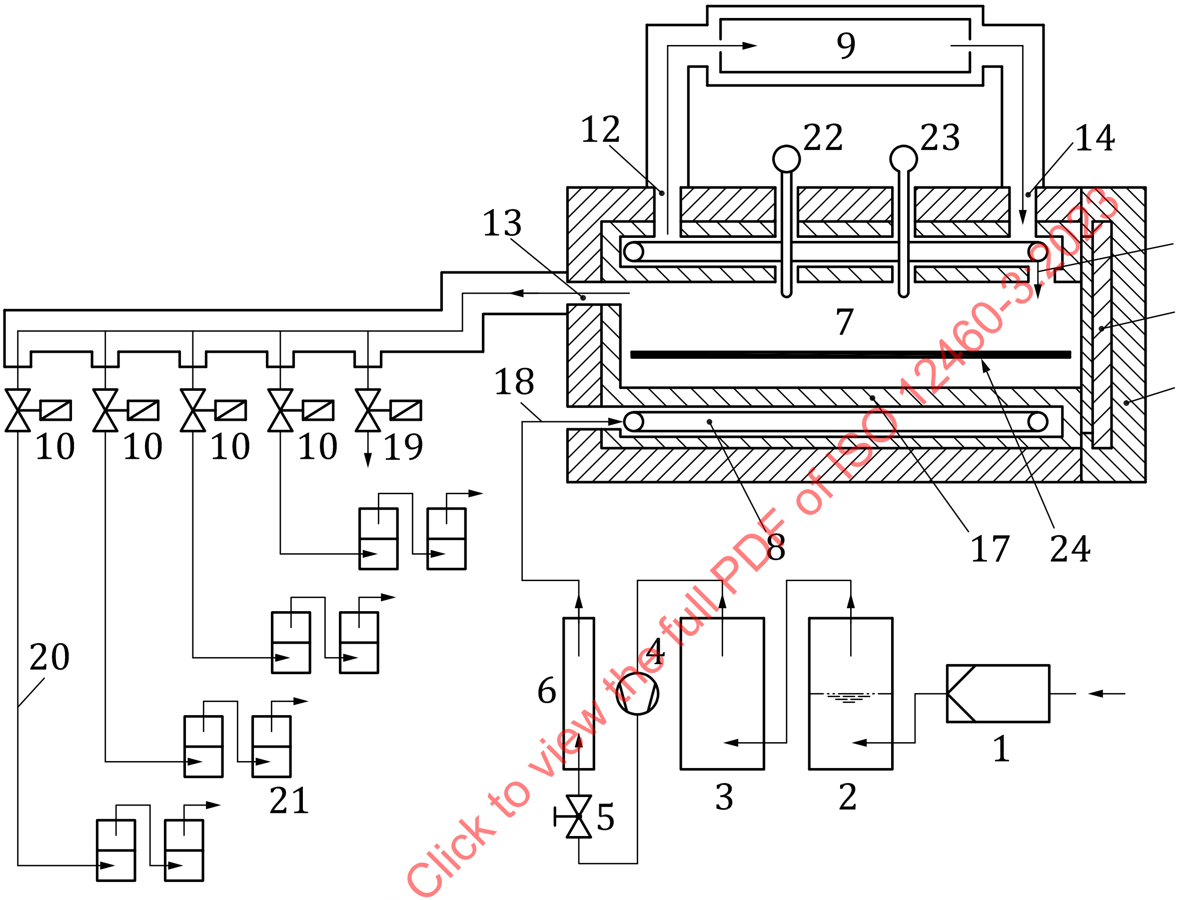
**6.1.11 Pairs of gas wash bottles**, 100 ml or optionally, pairs of gas wash bottles, 30 ml.

**6.1.12 Pressure monitor**.

**6.1.13 Temperature monitor**.

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

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.



**Key**

| | | | |
|---|---|---|---|
| 1 | dust or particle filter | 13 | outlet of test air |
| 2 | formaldehyde filter | 14 | heating medium (inlet) |
| 3 | desiccator | 15 | insulation |
| 4 | air pump | 16 | test chamber door |
| 5 | needle valve | 17 | double casing |
| 6 | equipment for measurement of air flow | 18 | inlet for air (heating coil) |
| 7 | test chamber | 19 | magnetic valve for purging |
| 8 | heating coil | 20 | connection tube |
| 9 | thermostat | 21 | pairs of wash bottles |
| 10 | magnetic valves | 22 | pressure monitor |
| 11 | inlet of air (test chamber) | 23 | temperature monitor |
| 12 | heating medium (outlet) | 24 | test piece holder |

**Figure 1 — Gas analysis test apparatus**

## 6.2 Laboratory equipment

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

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

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

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

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

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

**6.2.7 Two volumetric flasks**, 1 000 ml (calibrated at 20 °C).

**6.2.8 Volumetric pipettes** (calibrated at 20 °C), 1 ml, 2 ml, 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 30 ml, 50 ml, 100 ml.

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

**6.2.10 Microburette**.

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

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

## 7 Sampling and preparation of test pieces

### 7.1 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), test pieces shall be cut from the respective panel with the fibre direction of the faces perpendicular to the longitudinal axis of the test piece.

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.

Test pieces shall be representative for the tested boards.

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 ($m^2$).

For decor finish foils it is recommended to pre-condition the material as follows in order to take into account inhomogeneity as to improve precision: 7 days at (20 ± 2) °C and (65 ± 5) % relative humidity.

### 7.2 Selection of test pieces for factory production control

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

### 7.3 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 7.1.

### 7.4 Preparation 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.

## 8 Procedure

### 8.1 Number of determinations

Determination shall always be made in duplicate using two different test pieces prepared according to 7.1. 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.

### 8.2 Determination of moisture content

Moisture content shall be determined in accordance with ISO 16979 using a separate sample (see 7.1).

### 8.3 Determination of formaldehyde release

#### 8.3.1 General

The determination of formaldehyde release can be done by using different procedures, which are described as standard procedure (see 8.3.2), to improve the sensitivity (see 8.3.3 and 8.3.4) and to simplify the procedure (see 8.3.5).

Seal the edges of the test pieces in accordance with 7.1.

Close chamber (6.1.7) and preheat it to (60 ± 0,5) °C.

For each test hour connect a pair of wash bottles (see 6.1.11), each bottle containing between 8 ml to 40 ml absorber solution, in series to the outlet of each magnetic valve (see 6.1.10) using flexible tubing by using four different options to improve the sensitivity of the analytical procedure and simplify the test method (8.3.2 to 8.3.5). The flexible tube shall be isolated in case of using the procedure of 8.3.3 or 8.3.5 in order to avoid condensation which requires the rinsing of the tube.

The absorber 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 6.1.12).

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 (6.1.5) and the air volume meter (6.1.6). This air is led into one of a series of pairs of gas wash bottles via a magnetic valve (6.1.10).

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 3 h or 4 h from starting the test), a new series of gas wash bottles has to be connected and analysed every hour according to the analytical procedures given in 8.3.2 to 8.3.5. This exchange should be automatic.

For uncoated and coated particleboards and MDF with a thickness of at least 6 mm the test period may be reduced to 3 h. For all other material, a test period of 4 h is required. Tests can be carried out for 3 h provided that there is no significant difference with the results obtained after 4 h.

An overview of the 4 options for the determination of formaldehyde release is given in Table 1.

**Table 1 — Options for the analytical procedure**

| Option | Volume of wash bottle<br>ml | Number of wash bottles connected to gas analysis apparatus | Number of wash bottles used for analysis | Type of absorber | Volume of absorber solution filled in wash bottles<br>ml | Procedure for analysis | Volume of solution resp. volumetric flask after rinsing<br>ml |
|---|---|---|---|---|---|---|---|
| 1 | 100 | 2 | 2 | distilled water | 20 to 40 | transfer and rinsing to defined volume of volumetric flask | 250 |
| 2 | 30 | 2 | 2 | distilled water | 8 to 10 | transfer and rinsing to defined volume of volumetric flask | 100 |
| 3 | 100 | 2 | 2 | distilled water | 30[c] | 10 ml distilled water used for analysis (8.4.3) | not applicable |

[a] Mixed reagent: 10 ml distilled water/10 ml acetylacetone solution/10 ml ammonium acetate solution.

[b] 2nd wash bottle can be filled with water for pressure adjustment only.

[c] Other volumes may be used but the exact volume has to be determined and recorded for further calculation.

[d] Provided equivalence of test results was shown for each individual type of wood-based panel.

**Table 1** *(continued)*

| Option | Volume of wash bottle<br>ml | Number of wash bottles connected to gas analysis apparatus | Number of wash bottles used for analysis | Type of absorber | Volume of absorber solution filled in wash bottles<br>ml | Procedure for analysis | Volume of solution resp. volumetric flask after rinsing<br>ml |
|---|---|---|---|---|---|---|---|
| 4 | 100 | 2 | 1<br>only 1st wash bottle[b] | mixed acetylacetone reagent[a] | 30 | to be analysed directly (8.4.3) | not applicable |
| 5 | 100 | 2 | none | air | wash bottles only used to maintain the pressure | to be analysed by laser spectroscopy[d] | not applicable |

[a] Mixed reagent: 10 ml distilled water/10 ml acetylacetone solution/10 ml ammonium acetate solution.

[b] 2nd wash bottle can be filled with water for pressure adjustment only.

[c] Other volumes may be used but the exact volume has to be determined and recorded for further calculation.

[d] Provided equivalence of test results was shown for each individual type of wood-based panel.

### 8.3.2 Determination of formaldehyde release — Option 1

Use two washing bottles with volume of 100 ml (see 6.1.11), each containing between 20 ml and 40 ml distilled water. Transfer the contents of each pair of gas wash bottles to a 250 ml volumetric flask (6.2.6). Rinse the bottles and their associated tubing thoroughly and transfer the rinsings to the flask.

### 8.3.3 Determination of formaldehyde release — Option 2

Use two gas wash bottles with volume of 30 ml (see 6.1.11), each containing between 8 ml and 10 ml distilled water. To improve the sensitivity of the analytical procedure, 100 ml volumetric flasks (6.2.5) 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 8.4.

### 8.3.4 Determination of formaldehyde release — Option 3

Use two gas wash bottles with volume of 100 ml (see 6.1.11) and add 30 ml distilled water with a pipette (6.2.8) in each wash bottle as absorber solution. 10 ml are taken from the aqueous solution with a pipette (6.2.8) and added to 10 ml acetylacetone solution (5.1) and 10 ml ammonium acetate solution (5.2) in a stoppered flask (6.2.9).

### 8.3.5 Determination of formaldehyde release — Option 4

Using two gas wash bottles with volume of 100 ml (see 6.1.11), each containing 10 ml distilled water, 10 ml acetylacetone solution (5.1) and 10 ml ammonium acetate solution (5.2) as mixed reagent. Close the wash bottle, shake it, and heated for 10 min in a water bath (6.2.3) at 60 °C. The heated wash bottle is then cooled in a water bath (6.2.4) 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).

Only the first wash bottle has to be analysed. The second wash bottle is only used to maintain the overpressure and can be rejected. To obtain the pressure of (1 100 ± 100) Pa for option 4 the volume of the second wash bottle can be 20 ml to 40 ml depending on the device used.

## 8.4 Determination of formaldehyde content of the absorber solutions

### 8.4.1 General

The formaldehyde content of the absorber solution from each 1 h 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. In case of using the analytical procedure described in 8.3.4 or 8.3.5 the photometric determination by using a cell with 10 mm path length can be used as described in ISO 12460-5.

### 8.4.2 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 provided the methods show equivalent test results to acetylacetone analysis for each individual type of wood-based panel.

NOTE The procedure to show equivalent test results is described in ISO 12460-2.

### 8.4.3 Analytical procedure

10 ml are taken from the aqueous solution (see 8.3.2 to 8.3.4) with a pipette (6.2.8) and added to 10 ml acetylacetone solution (5.1) and 10 ml ammonium acetate solution (5.2) in a stoppered flask (6.2.9).

To improve the sensitivity of the analytical procedure, equal amounts of aqueous solution (see 8.3.2 to 8.3.4) and mixed reagent (see 5.2) 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 or wash bottle closed (see 8.3.5), shaken, and heated for 10 min in a water bath (6.2.3) at 60 °C. The heated flask is then cooled in a water bath (6.2.4) 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 (6.2.2). A blank value is determined in parallel with distilled water and taken into consideration in the determination of the gas analysis value.

### 8.4.4 Calibration curve

#### 8.4.4.1 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.

### 8.4.4.2 Formaldehyde standard solutions

- 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 % mass fraction.

The solutions shall be standardized before use.

About 1 g formaldehyde solution (concentration 35 % to 40 %) is diluted in a 1 000 ml volumetric flask (6.2.6) 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):

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

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.

### 8.4.4.3 Formaldehyde calibration solution

Using the concentration determined in 8.4.4.2, 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.

Ready to use formaldehyde standards with defined formaldehyde content may be used as well. The determination of formaldehyde content by titration of standard solution is not required, the ready to use formaldehyde solution has to be only diluted in order to prepare a solution containing 3 mg of formaldehyde in 1 000 ml volumetric flask.

### 8.4.4.4 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 8.4.4.3) 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 8.4.3). 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).

# 9 Expression of results

## 9.1 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):

$$G_i = \frac{(A_\mathrm{S} - A_\mathrm{B}) \times f \times V}{F} \tag{2}$$

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 ($\mathrm{m}^2$);

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

$A_\mathrm{S}$ is the absorbance of the solution from the wash bottles;

$A_\mathrm{B}$ 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 ($\mathrm{m}^2$) (see 7.1);

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

## 9.2 Calculation of results

### 9.2.1 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 3rd or 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 three or four hourly samples shall be used in the calculation.

Consequently, the arithmetic average formaldehyde gas analysis value, $G_\mathrm{m}$, in milligrams per square metre and hour ($\mathrm{mg/m^2h}$) of a test piece is calculated considering the test period used to determine the formaldehyde release (see 8.3.1) according to either Formula (3) or (4) (test period of 3 h) or Formula (5) or (6) (test period of 4 h):