International Standard

ISO 12460-2

Second edition
2024-02

# 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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# Foreword

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

This second edition cancels and replaces the first edition (ISO 12460-2:2018), which has been technically revised.

The main changes are as follows:

— implementation of different chamber sizes, analytical procedures, re-calculation of results to other standard parameters and establish a correlation between reference chamber method and the method used for factory production control.

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 2: Small-scale chamber method

## 1 Scope

This document specifies a procedure for a chamber test with different options of chamber sizes to measure the formaldehyde concentrations in air from wood products under defined test conditions of temperature, relative humidity, loading and air exchange rate.

Results obtained from this small-scale chamber test method can be used for quality control (factory production control – 'FPC') based on correlation established by reference chamber test methods according to ISO, EN or ASTM standards. The establishment of a correlation is described in Annex D.

## 2 Normative references

There are no normative references in this document.

## 3 Terms and definitions

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

ISO and IEC maintain terminology 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/

**3.1**
**air exchange rate**
***N***
quotient of air volume $Q$ passing through the chamber per hour ($m^3/h$) and the chamber volume ($m^3$) expressed in ($h^{-1}$)

**3.2**
**loading ratio**
***L***
total exposed surface area, excluding panel edges, of the product being tested divided by the test chamber's volume ($m^2/m^3$)

**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 $m^3/h\ m^2$

**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 $mg/m^3$ and/or ppm rounded to 2 decimal places) under the defined environmental test parameters of this method. In the case of a establishing a correlation, it can be advantageous to round the results to 3 decimal places

**3.7**
**chamber volume**
***V***
interior volume of the test chamber, in $m^3$

## 4 General

This document specifies the measurement to quantify the amount of formaldehyde in an air sample from a test chamber accepted in a range of sizes (examples are described in Annex A) and as determined by different analytical methods as specified in Annex C. Other analytical procedures may be used to determine the quantity of formaldehyde in the air sample provided that such methods give equivalent results. The test report shall include full description of the analytical procedure employed.

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 health and safety 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 chamber test methods specified in different EN, ISO and ASTM standards. This test method provides a means of testing smaller samples and reduces the time required for testing compared with a reference chamber method.

**5.2** Formaldehyde concentration levels obtained by this small-scale chamber 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 is applicable for the use of a chambers from 0,004 $m^3$ to 1 $m^3$ in volume (see examples in Annex A) 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) edge sealing;

d) test chamber temperature and relative humidity;

e) the *Q*/*A* ratio;

f) air exchange rate within the chamber.

# 6 Interferences

Interferences of the used analytical methods should be determined by reference to other applicable standard test methods.

# 7 Apparatus

## 7.1 Test chamber

### 7.1.1 General

The interior volume of the small chamber shall be between 0,004 $m^3$ and 1 $m^3$ (examples see Annex A). 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. For example, 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.

### 7.1.2 Air exchange rate

The clean and conditioned air supply to the chamber shall either be monitored continuously or frequently during testing.

The air exchange rate shall not vary by more than ±5 %.

The effective air exchange shall be regularly checked, by using e.g. either a calibrated gas meter, or the tracer gas procedure (see Clause 11).

### 7.1.3 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. If the air exchange is higher than 10/h mixing fans are not necessary.

### 7.1.4 Make-up air

The make-up air should come from a filtered dust-free environment and contain no more than 0,006 $mg/m^3$ of formaldehyde. Make-up air for the chamber shall pass through a calibrated air flow measuring device. If the make-up air is taken from a conditioning environment it should contain no more than 0,012 $mg/m^3$.

### 7.1.5 Equipment for monitoring of test conditions

Measuring equipment and recording facilities capable of continuous or frequent monitoring of the specified test conditions with an error limit as follows:

— Temperature: 0,1 K;

— Relative humidity: 2 %;

— Air exchange rate: 0,03/h.

### 7.1.6 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 or stainless steel), and the system should be maintained at the same temperature as the test chambers.

## 7.2 Air sampling system

### 7.2.1 Sampling system for wet-chemistry analysis

#### 7.2.1.1 General

Figure 1 shows the principle of a sampling system for the determination of the formaldehyde concentration in the chamber air. The sampling tube shall be placed either in the air outlet, or inside the chamber, close to the air outlet.

Other sampling systems may be used based on the requirements of the analytical procedure used.

The numbers in brackets refer to the numbers in Figure 1:

**7.2.1.2 sampling tube** (1).

**7.2.1.3 one or two 30 ml up to 100 ml gas washing bottle(s)** (2), with inserts like impinger or Muenke or frits, containing between 8 ml to 40 ml absorber solution, or DNPH cartridges for absorption and subsequent determination of formaldehyde.

**7.2.1.4 silica absorber for drying the air** (3).

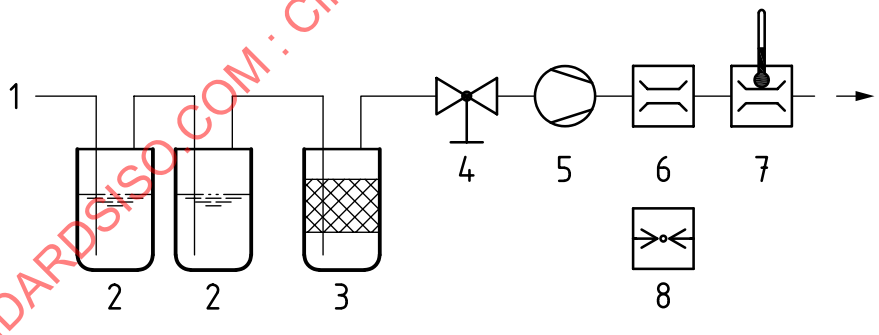
**7.2.1.5 gas flow valve** (4).

**7.2.1.6 gas sampling pump** (5).

**7.2.1.7 gas flow meter** (6).

**7.2.1.8 gas meter (including a thermometer) for measuring the volume of air** (7).

**7.2.1.9 air pressure meter** (8).




**Key**

| | | | |
|---|---|---|---|
| 1 | sampling tube | 5 | gas sampling pump |
| 2 | gas washing bottle | 6 | gas flow meter |
| 3 | silica absorber | 7 | gas meter with thermometer |
| 4 | gas flow valve | 8 | air pressure meter |

**Figure 1 — Example of a sampling system for the determination of formaldehyde concentration in air**

### 7.2.2 Direct sampling

Formaldehyde in the chamber air is determined by direct sampling analysis (e.g. optical or chemical sensors see Annex C). The measuring cell has to be connected with non-absorbent formaldehyde tubes. The length of tube can have an impact on results and has to be considered.

## 8 Sample material handling and specimen conditioning

### 8.1 Handling

Materials selected for testing shall e.g. be wrapped in polyethylene plastic having a minimum thickness of 0,15 mm (0,006 in) 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 described e.g. in the quality manual handbook.

### 8.2 Test specimen

Chambers are operated at a fixed sample size by varying the make-up airflow ($Q$), or at fixed $Q$ by varying the product sample size by product type. Either mode is acceptable as long as the appropriate $Q/A$ ratios for the product type are met (see Table 1 and Table 2). The minimum requirement of $Q/A$ ratio is 1 $m^3/h\ m^2$ with a range of ±2 %

Products can have different surface coverings on front and back side. Therefore, significantly different formaldehyde release characteristics for each surface can occur. In those cases, panels may be tested back-to-back or face-to-face with edges taped together depending on which surface is to be tested.

**Table 1 — Examples of calculated $Q/A$ ratio referring to chamber size used to consider the dimensions specified in the standards (here: EN 717-1[8], DMC[12], ISO 12460-3[3])**

| Type of chamber | Type of WBP[a] | Flow rate adjusted | | $Q$ | Air exchange | Sample size[b] height | Sample size[b] width | Number of samples | Sample surface area $A$ | $Q/A$ ratio |
|---|---|---|---|---|---|---|---|---|---|---|
| | | [l/min] | [l/h] | [$m^3/h$] | rate $N$ [$h^{-1}$] | [mm] | [mm] | | [$m^2$] | [$m^3/h\ m^2$] |
| 1 $m^3$ | PB/PLY | 16,67 | 1 000,2 | 1,000 2 | 1 | 500 | 500 | 2 | 1,000 0 | 1,000 |
| 1 $m^3$ | MDF | 16,67 | 1 000,2 | 1,000 2 | 1 | 500 | 500 | 2 | 1,000 0 | 1,000 |
| 0,225 $m^3$ | PB/PLY | 3,75 | 225,0 | 0,225 0 | 1 | 200 | 280 | 2 | 0,224 0 | 1,004 |
| 0,225 $m^3$ | MDF | 3,75 | 225,0 | 0,225 0 | 1 | 200 | 280 | 2 | 0,224 0 | 1,004 |
| 0,045 $m^3$ | PB/PLY | 8,91 | 534,6 | 0,534 6 | 12,15 | 200 | 381 | 3 | 0,457 2 | 1,169 |
| 0,045 $m^3$ | MDF | 14,51 | 870,6 | 0,870 6 | 19,79 | 200 | 381 | 3 | 0,457 2 | 1,904 |
| 0,004 $m^3$ | PB/PLY | 1,00 | 60,0 | 0,060 0 | 15 | 400 | 50 | 1 | 0,040 0 | 1,500 |
| 0,004 $m^3$ | MDF | 1,00 | 60,0 | 0,060 0 | 15 | 400 | 50 | 1 | 0,040 0 | 1,500 |

[a] WBP: wood-based panel; PB: particleboard, MDF: medium density fibreboard; PLY: plywood.

[b] Tolerance of sample size for samples used for chamber volume of 1 $m^3$, 0,225 $m^3$ and 0,045 $m^3$: ±2 mm; 0,004 $m^3$: ±1 mm

**Table 2 — Examples of calculated $Q/A$ ratio referring to chamber size considering the requirements for different types of wood-based panels (here: ASTM D 6007[11])**

| Type of chamber | Type of WBP[a] | Flow rate adjusted | | $Q$ | Air exchange | Sample size[b] height | Sample size[b] width | Number | Sample surface area $A$ | $Q/A$ ratio |
|---|---|---|---|---|---|---|---|---|---|---|
| | | [l/min] | [l/h] | [$m^3/h$] | rate N [$h^{-1}$] | [mm] | [mm] | of samples | [$m^2$] | [$m^3/h\ m^2$] |
| 1 $m^3$ | PB/PLY | 8,33 | 499,8 | 0,499 8 | 0,5 | 143 | 500 | 3 | 0,429 0 | 1,165 |
| 1 $m^3$ | MDF | 8,33 | 499,8 | 0,499 8 | 0,5 | 85 | 500 | 3 | 0,255 0 | 1,960 |
| 0,225 $m^3$ | PB/PLY | 1,87 | 112,2 | 0,112 2 | 0,5 | 80 | 200 | 3 | 0,096 0 | 1,169 |
| 0,225 $m^3$ | MDF | 1,87 | 112,2 | 0,112 2 | 0,5 | 49 | 200 | 3 | 0,058 8 | 1,908 |

[a] WBP: wood-based panel; PB: particleboard, MDF: medium density fibreboard; PLY: plywood.

[b] tolerance of sample size for samples used for chamber volume of 1 $m^3$, 0,225 $m^3$ and 0,045 $m^3$: ±2 mm; 0,004 $m^3$: ±1 mm

NOTE Table 1 and Table 2 show only examples for some wood-based panel products. Any other wood-based panel or other formaldehyde emitting products (coated or uncoated) can be tested as well, e.g. OSB (Oriented Strand Boards), solid wood panels, cement bonded particleboards, wet process fiber boards, LVL, etc.

## 8.3 Conditioning

The procedure for pre-conditioning of samples prior to testing shall be specified and standardized on an individual basis i.e. at a factory or laboratory. The conditioning and testing parameters shall be specified and kept consistent.

An established procedure for sample conditioning is for example as described:

Condition test specimens with a minimum distance of 0,15 m (6 inch) between each specimen for minimum of 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 inch) of where panels are conditioned shall not be more than the lowest emission limit of the product(s) to be tested during the conditioning period. Alternative conditioning intervals can give better correlation to larger chamber test methods, e.g. 7 days ± 3 h conditioning or 15 days conditioning.

## 8.4 Sealing of test piece edges

Edges shall be sealed completely air-tight by using self-adhesive aluminium tape or wax.

# 9 Procedure

## 9.1 Test conditions

The following conditions shall be maintained in the chamber throughout the test:

— Temperature (25 ± 1) °C [(77 ± 2) °F];

— Relative humidity (50 ± 4) %;

— $Q/A$ ratio minimum of 1 $m^3/h\ m^2$ ± 2 %.

The conditions can be reached by storing the chamber in a well-conditioned surrounding or by using a self-climatisation system.

## 9.2 Test procedure for materials

### 9.2.1 General

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 should not exceed 0,006 mg/m$^3$. Clean chamber surfaces with water or suitable solvent if formaldehyde background concentrations approach 0,006 mg/m$^3$. If the make-up air is taken from a conditioning environment it should contain no more than 0,012 mg/m$^3$.

**9.2.2** Locate the specimens in the chamber so that the conditioned air stream circulates over all panel surfaces.

**9.2.3** 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 $Q/A$ ratio and record this ratio in the report.

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

## 9.3 Air sampling

The sampling shall be carried out as specified in Annex C. The length of sampling tube can have an impact on results and shall be considered.

## 9.4 Analysis of air samples

### 9.4.1 General

Analytical methods for formaldehyde quantification are provided in Annex C. Of the analytical methods provided, the wet-chemistry methods (C.1: Acetylacetone method, C.2: Chromotropic acid method and C.3: DNPH method) are considered reference analytical methods and are the methods that the direct analytical methods (e.g. C.4: Laser Absorption Spectroscopy (LAS), C.5: Chemical sensor) are measured against and have to show equivalent results.

### 9.4.2 Equivalence of analytical procedures – General requirements

#### 9.4.2.1 General

Where an analytical method other than the wet-chemistry reference analytical methods (C.1 to C.3) is used to determine formaldehyde in the air, equivalence to at least one of this reference methods shall be shown.

#### 9.4.2.2 Demonstration of equivalence – Device manufacturer

Prior to use in a factory's laboratory, the device manufacturer shall carry out at least 15 tests using samples of different types of wood-based panels with varying composition (e.g. glue composition, additives, raw material) per product type (e.g. MDF, particleboard, plywood, OSB) evenly distributed in a wide emission range (at least 0,012 mg/m$^3$ up to 0,25 mg/m$^3$). A linear regression shall be calculated and equivalence is shown if the statistical evaluation complies with the requirements of a slope with 1 ± 0,05, $R^2$ ≥ 0,98 and $r$ value ≥ 0,99.

#### 9.4.2.3 Demonstration of equivalence – Factory laboratory

The factory's laboratory shall validate the device manufacturer's data for the specific product(s) intended for FPC testing. To demonstrate equivalence at least five samples of the type of wood-based material respectively products are tested by using one of the reference wet-chemistry methods and the test procedure to be

evaluated. Equivalence is demonstrated if the absolute mean deviation from the wet-chemistry reference method is ≤ 10 %.

## 10 Calculation

**10.1** Convert the volume of air sampled to the volume of air at standard conditions by Formula (1):

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

where

$V_S$ is the volume of air at standard conditions (101 kPa and 298 K), in cubic metres;

$V$ is the volume of air sampled, in cubic metres;

$P$ is the barometric pressure, in kPa;

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

**10.2** Calculate total milligrams of formaldehyde collected in each washing bottle by Formula (2):

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

where

$C_t$ is the total mass of formaldehyde in the sample, in milligrams;

$C_a$ is the total quantity of formaldehyde in the sample aliquots taken from the washing bottle (as determined from the calibration curve in C.1 to C.3), in milligrams;

$F_a$ is the aliquot factor:

$$F_a = \frac{V_{s,sol}}{V_a} \quad (3)$$

where

$V_{s,sol}$ is the sampling solution volume, in ml;

$V_a$ is the. aliquot used, in ml.

**10.3** Calculate the concentration of formaldehyde in air in the small chamber as follows:

$$C_s = C_t / V_s \quad (4)$$

where

$C_s$ is formaldehyde concentration in air in mg/m$^3$;

$V_s$ is the volume of air at standard conditions (101 kPa and 298 K), in cubic metres;

Round calculated formaldehyde concentrations to the nearest 0,01 mg/m$^3$.

NOTE At 25 °C (77°F) and 1 013 hPa the following relationship exists for formaldehyde:

1,23 mg/m$^3$ = 1 ppm (parts per million);

1 mg/m$^3$ = 0,81 ppm (parts per million).

**10.4** When the chamber temperature as described in the selected options of 9.1 differs from the standard parameter, adjust the formaldehyde concentrations obtained to a standard temperature of 25 °C (77 °F) using an equation developed by Berge et al. or other verified calculation models Annex B 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 rounded to one decimal place.

**10.5** The measured chamber formaldehyde concentration in air shall be adjusted to a concentration at 50 % relative humidity and shall to be re-calculated when it differs from 50 % (see Annex B). For re-calculation the measured relative humidity should be rounded to one decimal place.

## 11 Determination of air exchange rate

The determination of the air exchange rate (n) in the unloaded test chamber is based on the method for measuring the contrition dynamics of an indicator gas (tracer gas) which is introduced into the chamber. The indicator gas concentration will decrease over time depending on the air exchange rate. Under ideal air mixing conditions in the chamber, the concentration will follow the Formula (5) and calculated with Formula (6).

$$c_t = c_0\, e^{-nl} \tag{5}$$

$$n = \left(\frac{1}{t}\right) \ln\left(c_0 / c_t\right) \tag{6}$$

where

$c_0$ is the initial concentration of indicator gas, in milligrams per cubic metre;

$c_t$ is the concentration of indicator gas, in milligrams per cubic metre at time $t$ in hours;

$n$ is the air exchange rate per hour (1/h);

$t$ is the time, in hours.

NOTE Dinitrogen monoxide ($N_2O$) is a suitable tracer gas and can be determined using an infrared (IR) detector. Dinitrogen monoxide has a molecular mass of 44,01 g/mol and a density of 1,53 relatives to air.

## 12 Test report

**12.1** Test number.

**12.2** The manner in which materials where shipped or stored, or both: wrapped separately, wrapped collectively 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.

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

**12.4** 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).

**12.5** Specimen condition details to include average rounded to one decimal place the temperature and range, average relative humidity and range, and time to the nearest appropriate measure, e.g. hour or minute.

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

**12.7** Chamber volume: nominal length, width, and height.

**12.8** Chamber *Q*/*A* ratio.

**12.9** Description of specimens (size, edge sealing technique) as loaded into chamber including number of specimens in charge and number of surfaces exposed.

**12.10** Average temperature, average relative humidity, and time to the nearest minute during the sampling period.

**12.11** Chamber formaldehyde concentration in air at test conditions; chamber formaldehyde concentration in air corrected 25 °C (77 °F), 50 % relative humidity, rounded to the nearest 0,01 mg/m$^3$ (or ppm). In the case of a establishing a correlation, it can be advantageous to round the results to three decimal places.

**12.12** The analytical method used to determine formaldehyde in air.

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

**12.14** Air-sampling rate and length of sample time.

**12.15** Date of conditioning start and date of emission test. Additional dates can be mentioned if other standards are considered.

# Annex A
(informative)

# Test chambers

## A.1 General

This document applies to different test chambers for formaldehyde emission testing with a chamber volume of 0,004 $m^3$ to 1 $m^3$. Examples of chambers are described in Annex A.

General specifications and requirements which apply to all types of test chambers included in this document are given in Clause 7.

Materials used for the inner walls and ducts of test chambers shall have a smooth surface, which, prior to testing, can be effectively cleaned with water. The surface shall be as inert and non-absorptive as possible to formaldehyde.

NOTE Proven materials are stainless steel or aluminium (sandblasted or polished), glass and some types of plastics (PVC, PMMA).

## A.2 Example 1: 1 $m^3$ test chamber

### A.2.1 Chamber volume and operation

Chambers of this type have a total interior volume of 1 $m^3$. 1 $m^3$ test chambers are operated with intensive circular air flow (see Figure A.1, Figure A.2 and Figure A.3).

The climatic test conditions (temperature, relative humidity) can be established within the chamber either by special conditioning devices (e.g. air heater, steam injector), or by using preconditioned inlet air. Chambers operated in this way need effective thermal wall insulation.

The temperature within the test chamber can also be established by placing the chamber in a larger compartment with controlled temperature. Test chambers operated in this way shall have no insulation.

The volume of the inlet air is measured and adjusted by a gas pump or a compressed air system in connection with gas flow meters (see Figure A.4) and can be measured by a calibrated gas meter in the outlet. The effective air exchange rate shall be regularly checked (see 7.1.2).

### A.2.2 Construction principle

The test apparatus comprises the following components, see Figure A.1, Figure A.2 and Figure A.3 (the numbers in brackets refer to the numbers in the figures):

#### A.2.2.1 Chamber with a total interior volume of (1 ± 0,01) $m^3$ (1).

The 1 $m^3$ test chambers shown in the figures as examples are constructed from metal, glass or plastic plates.

#### A.2.2.2 Air inlet (2).

The volume of the air flow through the test chamber is measured by a gas meter.

#### A.2.2.3 Ventilation fan (at least one) (3).

The ventilation fan mixes the air in the chamber by intensive circulation.

#### A.2.2.4 **Inlet for monitoring equipment/sensors** (4).

To introduce sensors for continuous or frequent monitoring of temperature and air relative humidity.

#### A.2.2.5 **Air outlet** (5).

The air outlet openings are placed far from the inlet openings. Tubes for air sampling are inserted through the outlet pipes and sampling is made close to the outlets.

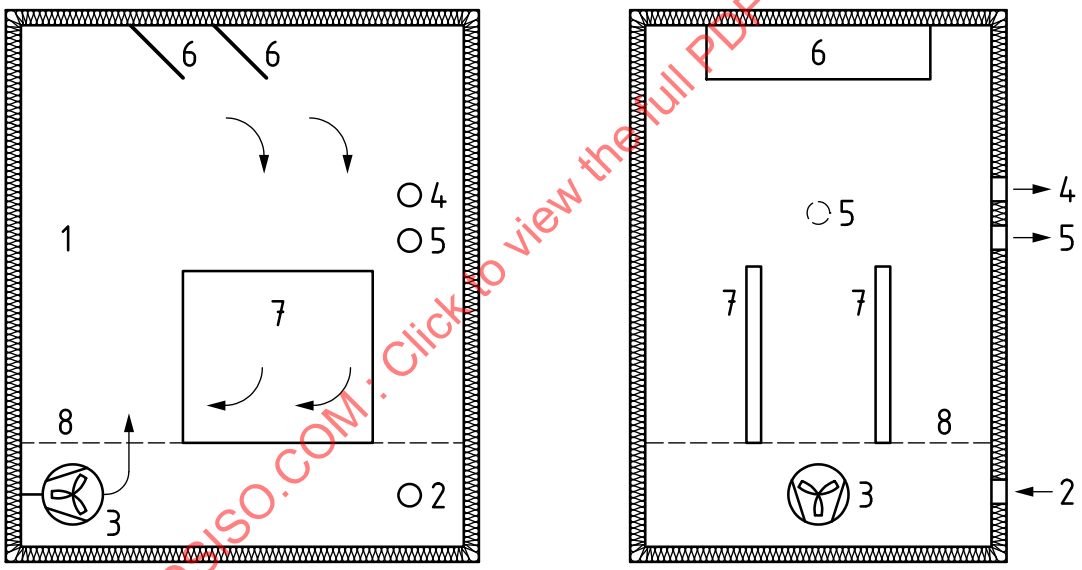
#### A.2.2.6 **Baffle plates or partition**, or perforated bottom (6).

#### A.2.2.7 **Test pieces** (7).

#### A.2.2.8 **Perforated bottom** (8).

### A.2.3 Test pieces

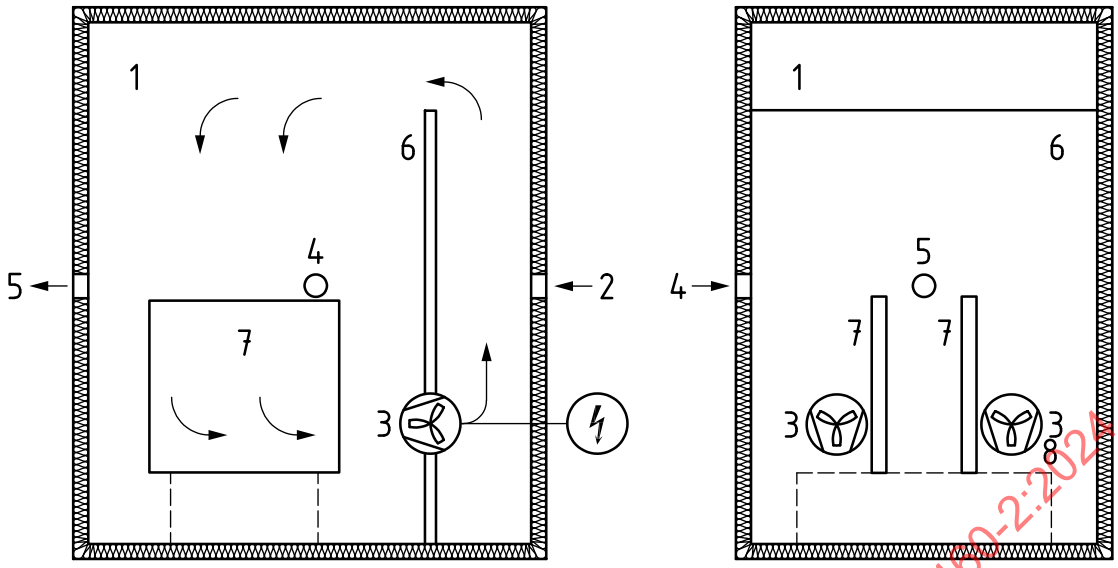
Produce and prepare test pieces in accordance with Clause 8. The size of the test pieces should be (500 ± 2) mm × (500 ± 2) mm × board thickness to reach a $Q/A$ ratio of 1 (see examples in Table 1). Other examples of sample sizes considering different loading and flow rates are given in Table 2. The chamber is loaded with two test pieces of this size. The test pieces are positioned near to the centre of the chamber, parallel to the air flow, with their faces separated by a minimum distance of 200 mm.




**Key**

| | | | |
|---|---|---|---|
| 1 | 1 m$^3$ test chamber | 5 | air outlet |
| 2 | air inlet | 6 | baffle plates |
| 3 | ventilation fan | 7 | test pieces |
| 4 | inlet for monitoring equipment/sensors | 8 | perforated bottom |

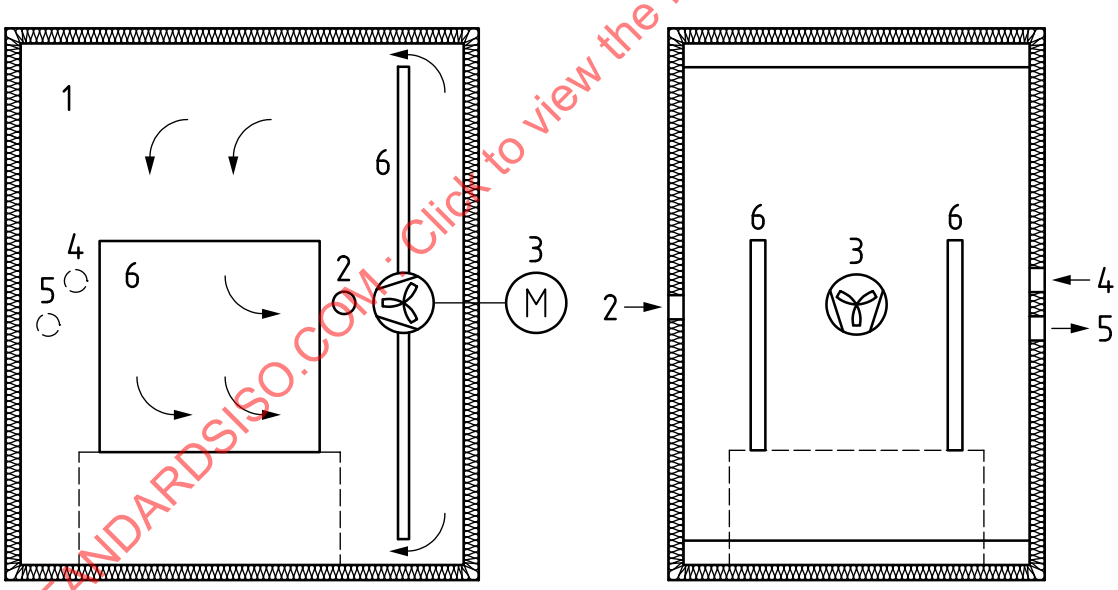
**Figure A.1 — Example 1 of a construction scheme for a 1 m$^3$ test chamber**




**Key**

1 1 m$^3$ test chamber
2 air inlet
3 ventilation fan with electric power supply
4 inlet for monitoring equipment/sensors
5 air outlet
6 partition
7 test pieces

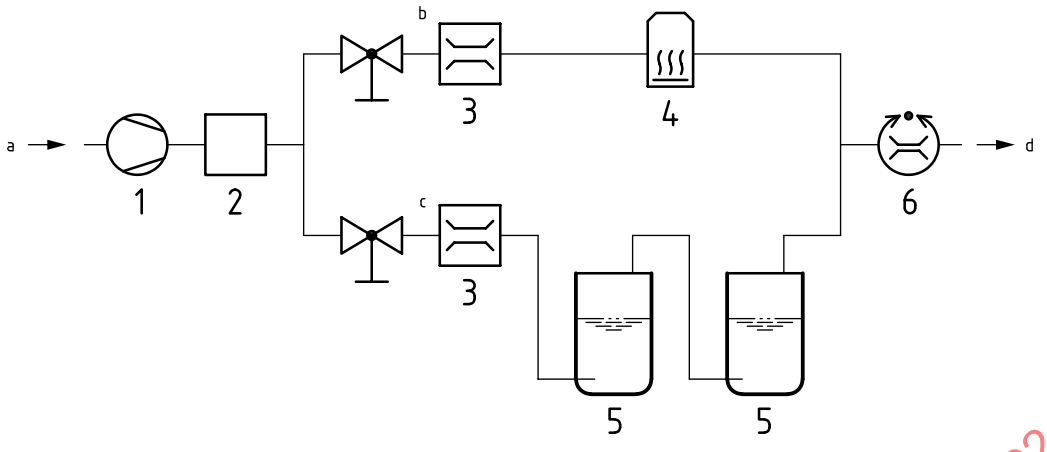
**Figure A.2 — Example 2 of a construction scheme for a 1 m$^3$ test chamber**




**Key**

1 1 m$^3$ test chamber
2 air inlet
3 ventilation fan with external electric power
4 inlet for monitoring equipment/sensors
5 air outlet
6 test pieces

**Figure A.3 — Example 3 of a construction scheme for a 1 m$^3$ test chamber**




**Key**

| | | | |
|---|---|---|---|
| 1 | gas pump | 4 | silica gel filter |
| 2 | charcoal filter | 5 | washing bottle(s) (humidification) with a minimum volume of 1 000 ml |
| 3 | gas flow meter and gas flow control | 6 | flow controller or gas meter |
| a | Air inlet. | | |
| b | Dry air, ~50 % of total air flow. | | |
| c | Wet air, ~50 % of total air flow. | | |
| d | Air outlet, to the chamber. | | |

**Figure A.4 — Example of a device for establishing a controlled air flow with a relative humidity of 50 %**



## A.3 Example 2: 0,225 m³ test chamber

### A.3.1 Chamber volume and operation

This test chamber has a nominal interior volume of 0,225 $m^3$. The chamber is operated with circular air flow (see Figure A.5).

The climatic test conditions (temperature, relative humidity) can be established within the chamber either by special conditioning devices (e.g. air heater, steam injector), or by using preconditioned inlet air. Chambers operated in this way need effective thermal wall insulation.

The temperature within the test chamber can also be established by placing the chamber in a larger compartment with controlled temperature. Test chambers operated in this way shall have no insulation.

The volume of the inlet air is measured and adjusted by a gas pump or a compressed air system in connection with gas flow meters (see Figure A.6) and can be measured by a calibrated gas meter in the outlet. The effective air exchange rate shall be regularly checked (see 7.1.2).

### A.3.2 Construction principle

The test apparatus comprises the following components, see Figure A.6 (the numbers in brackets refer to the numbers in Figure A.6):

#### A.3.2.1 Test chamber with a volume of (0,225 ± 0,004 5) m³ (1).

The dimensions of the chamber are 0,7 m × 0,5 m × 0,65 m (example).

**A.3.2.2 Air inlet** (2).

The air inlet of the chamber is placed close to the inlet port of the fan.

**A.3.2.3 Ventilation fan with external electric motor** (3).

The fan ensures a symmetric, circular air flow around the test pieces.

**A.3.2.4 Inlet for monitoring equipment/sensors** (4).

To introduce sensors for continuous or frequent monitoring of temperature and air relative humidity.

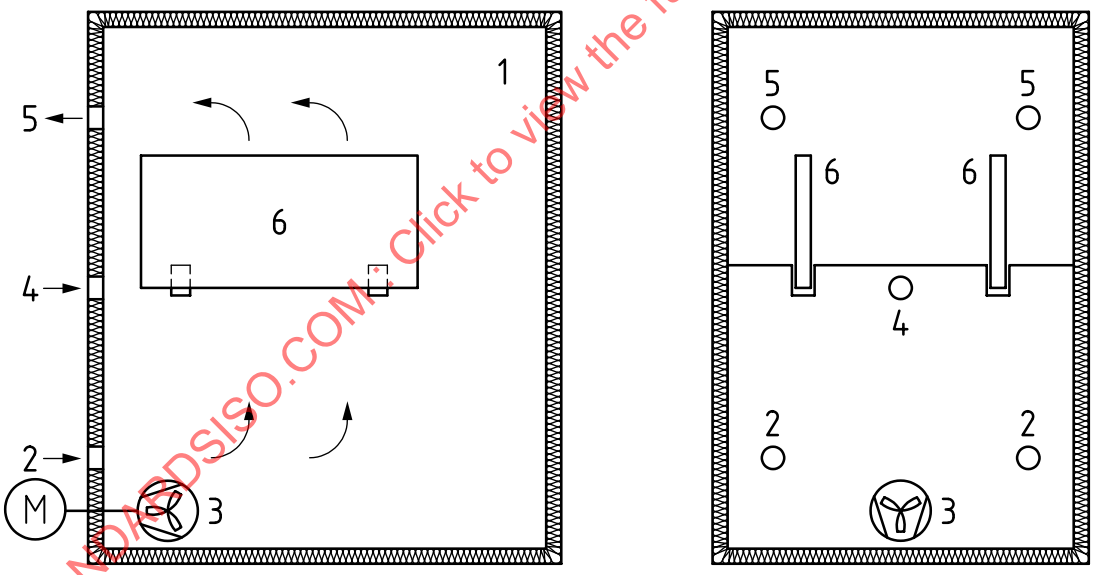
**A.3.2.5 Air outlet** (5).

The air outlet openings are placed far from the inlet openings. Tubes for air sampling are inserted through the outlet pipes and sampling is made close to the outlets.

**A.3.2.6 Test pieces** (6).

### A.3.3 Test pieces

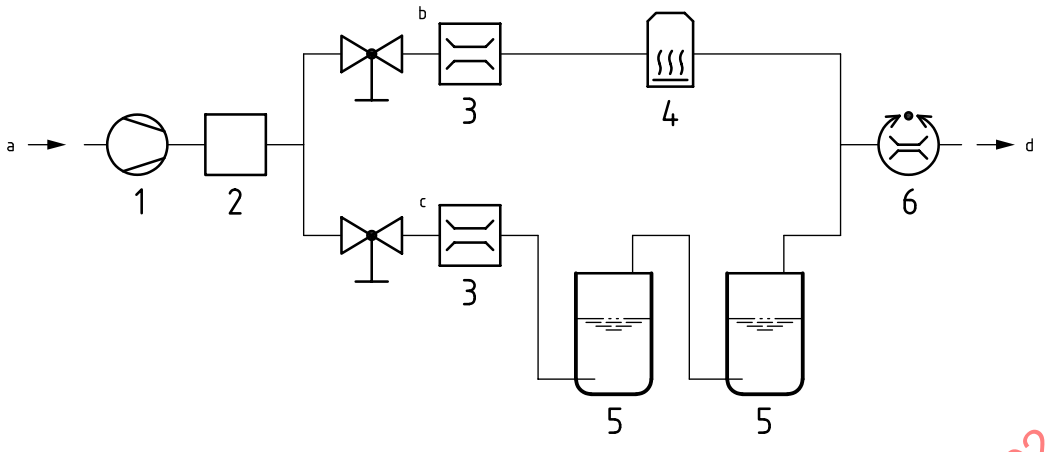
Produce and prepare test pieces in accordance with Clause 8. The size of the test pieces should be (200 ± 2) mm × (280 ± 2) mm × board thickness. The chamber is loaded with two test pieces of this size to reach a $Q/A$ ratio of 1 (see examples in Table 1). Other examples of sample sizes considering different loading and flow rates are given in Table 2. The test pieces are positioned near to the centre of the chamber, parallel to the air flow, with their faces separated by a minimum distance of 200 mm.




**Key**

| | | | |
|---|---|---|---|
| 1 | 0,225 m³ test chamber | 4 | inlet for monitoring equipment/sensors |
| 2 | air inlet | 5 | air outlet |
| 3 | ventilation fan with external electric motor | 6 | test pieces |

**Figure A.5 — Example of a construction scheme for a 0,225 m³ test chamber**




**Key**

| | | | |
|---|---|---|---|
| 1 | gas pump | 4 | silica gel filter |
| 2 | charcoal filter | 5 | washing bottle(s) (humidification) with a minimum volume of 1 000 ml |
| 3 | gas flow meter and gas flow control | 6 | flow controller or gas meter |
| a | Air inlet. | | |
| b | Dry air, ~50 % of total air flow. | | |
| c | Wet air, ~50 % of total air flow. | | |
| d | Air outlet, to the chamber. | | |

**Figure A.6 — Example of a device for establishing a controlled air flow with a relative humidity of 50 %**



## A.4 Example 3: 0,045 m³ test chamber

### A.4.1 Chamber volume and operation

This test chamber has a nominal interior volume of 0,045 $m^3$ and is operated with intensive circular air flow (see Figure A.7 and Figure A.8).

The climatic test conditions (temperature, relative humidity) can be established within the chamber either by special conditioning devices (e.g. air heater, steam injector), or by using preconditioned inlet air. Chambers operated in this way need effective thermal wall insulation.

The temperature within the test chamber can also be established by placing the chamber in a larger compartment with controlled temperature. Test chambers operated in this way shall have no insulation.

The volume of the inlet air is measured and adjusted by a gas pump or a compressed air system in connection with gas flow meters (see Figure A.9) and can be measured by a calibrated gas meter in the outlet. The effective air exchange rate shall be regularly checked (see 7.1.2).

### A.4.2 Construction principle

The test apparatus comprises the following components, see Figure A.7 and Figure A.8 (the numbers in brackets refer to the numbers in the figures):

**A.4.2.1 Stainless steel test chamber with a volume of 0,045 m³ (±0,005 m³),** corresponding e.g. the dimensions of (349 ± 2) mm × (622 ± 2) mm × (205 ± 2) mm (width × length × high) with stainless steel ports (1).

**A.4.2.2 Air Inlet** (2).

**A.4.2.3 Sample air outlet** (3).

**A.4.2.4 Air circulation,** minimum 0,2 m/s at blower exit (4).

**A.4.2.5 Relative humidity measurement,** accurate to ±2 %.

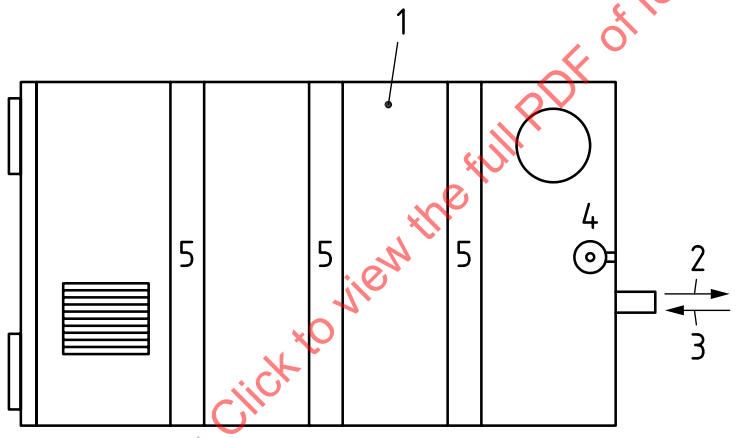
Temperature measurement, accurate to ±0,1 K (5)

### A.4.3 Test pieces

Produce and prepare test pieces in accordance with Clause 8. The size of the test pieces should be (200 ± 2) mm × (381 ± 2) mm × board thickness. The chamber is loaded with three test pieces of this size to reach the required *Q*/*A* ratios (see examples in Table 1). The test pieces are positioned in the chamber as shown in the Figure A.8 to create a serpentine airflow pattern.

Test pieces are secured in an upright position using a shim plate or similar fixture which does not impede air circulation or block sample surface area.

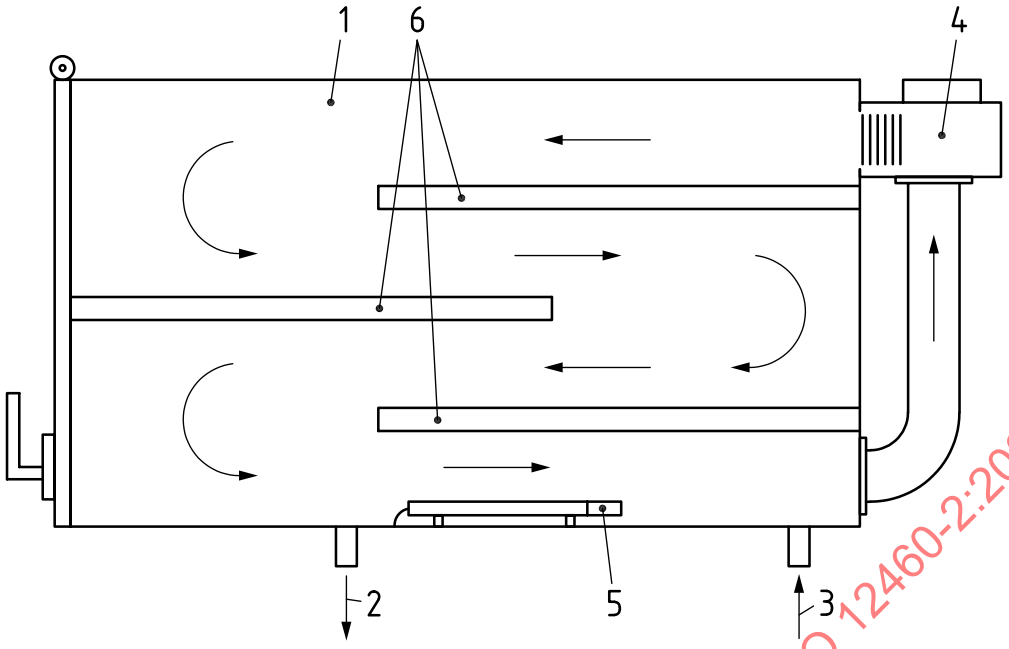
The samples shall be positioned (clamped) between the floor and the ceiling of the chamber to avoid spaces and to ensure air circulation. In case of a slight deviation in chamber size or dimension, the samples shall be cut correspondingly.




**Key**

1 0,045 $m^3$ test chamber
2 air outlet
3 air inlet
4 temperature & humidity sensor
5 test pieces

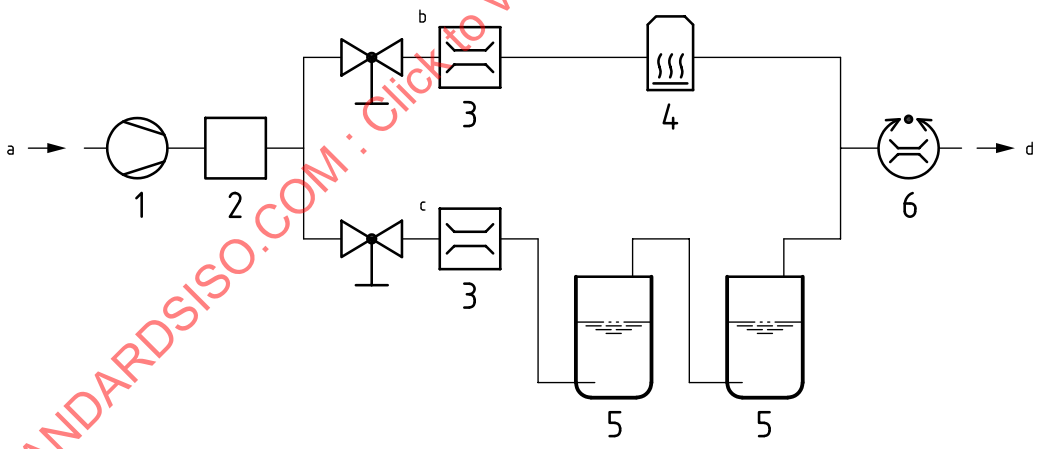
**Figure A.7 — Example of a construction scheme for a 0,045 $m^3$ test chamber; front view of the (open) chamber**




**Key**

| | | | |
|---|---|---|---|
| 1 | 0,045 $m^3$ test chamber | 4 | ventilation fan |
| 2 | air outlet | 5 | temperature & humidity sensor |
| 3 | air inlet | 6 | test pieces |

**Figure A.8 — Example of a construction scheme for a 0,045 $m^3$ test chamber; Top down, internal view of the chamber. The sample panels are placed in specific positions, which creates a circulating, serpentine airflow pattern**




**Key**

| | | | |
|---|---|---|---|
| 1 | gas pump | 4 | silica gel filter |
| 2 | charcoal filter | 5 | washing bottle(s) (humidification) with a minimum volume of 1 000 ml |
| 3 | gas flow meter and gas flow control | 6 | flow controller or gas meter |
| a | Air inlet. | | |
| b | Dry air, ~50 % of total air flow. | | |
| c | Wet air, ~50 % of total air flow. | | |
| d | Air outlet, to the chamber. | | |

**Figure A.9 — Example of a device for establishing a controlled air flow with a relative humidity of 50 %**

## A.5 Example 4: 0,004 $m^3$ test chamber

### A.5.1 Chamber volume and operation

Chambers of this type have a total nominal interior volume of 0,004 $m^3$. This type of test chamber is operated with intensive air flow.

The climatic test conditions (temperature, relative humidity) can be established within the chamber either by special conditioning devices (air heater, steam injector), or by using preconditioned inlet air, as shown in Figure A.10. Chambers operated in this way need effective wall insulation or can alternatively be placed in a larger compartment with controlled temperature.

The volume of the inlet air is measured and adjusted by a gas pump or a compressed air system in connection with gas flow meters (see Figure A.10) and can be measured by a calibrated gas meter in the outlet. The effective air exchange rate shall be regularly checked.

NOTE For air sampling no additional pump is needed, as by using the gas analysis apparatus, the sample air gets directly transferred into the connected gas wash bottles by slight overpressure.

### A.5.2 Construction principle

#### A.5.2.1 General

The test apparatus comprises the following main components, if operated with pre-humidified (50 % ± 4 % RH) inlet-air (the numbers in brackets (A.5.2.2 to A.5.2.15) refer to the numbers in Figure A.10).

NOTE If the apparatus is not operated in a preconditioned environment, humidity can be adjusted by either mixing of clean wet and dry air in desired ratios (see Figure A.11), or by employing a humidifier/evaporator

**A.5.2.2 Particle filter** (1).

**A.5.2.3 Formaldehyde filter** (2).

**A.5.2.4 Air pump** (3).

**A.5.2.5 Needle valve** (4).

**A.5.2.6 Mass flow measurement device** (5).

NOTE Needle valve and mass flow measurement device can be replaced with a mass flow controller.

**A.5.2.7 Humidity sensor** (6).

The humidity sensor shall be positioned directly in the chamber. Alternatively, the makeup air stream can be measured.

**A.5.2.8 Test chamber** (7); 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.

**A.5.2.9 Heating equipment for air** (e.g. copper coil inside the double casing) (9).

**A.5.2.10 Thermostat** (9).

NOTE The heating equipment can be omitted if the chamber is operated in an air-conditioned environment at the required temperature and humidity conditions.

**A.5.2.11 Magnetic valves** for sampling (10) and purging (19).

**A.5.2.12 One pair of gas wash bottles,** 100 ml or optionally, one pair of gas wash bottles, 30 ml (20).

**A.5.2.13 Pressure monitor** (22).

**A.5.2.14 Temperature monitor** (23).

**A.5.2.15 Test piece holder,** made from stainless steel or another inert material (24).

NOTE The test apparatus described in Figure A.10 is based on a waterborne heating system. A test apparatus with an electrical heating system can be used optionally.

## A.5.3 Test pieces

### A.5.3.1 Preparation of test pieces

Test pieces, 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 $m^2$ to reach the required *Q/A* ratio (see example in Table 1).

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.

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 shall be established.

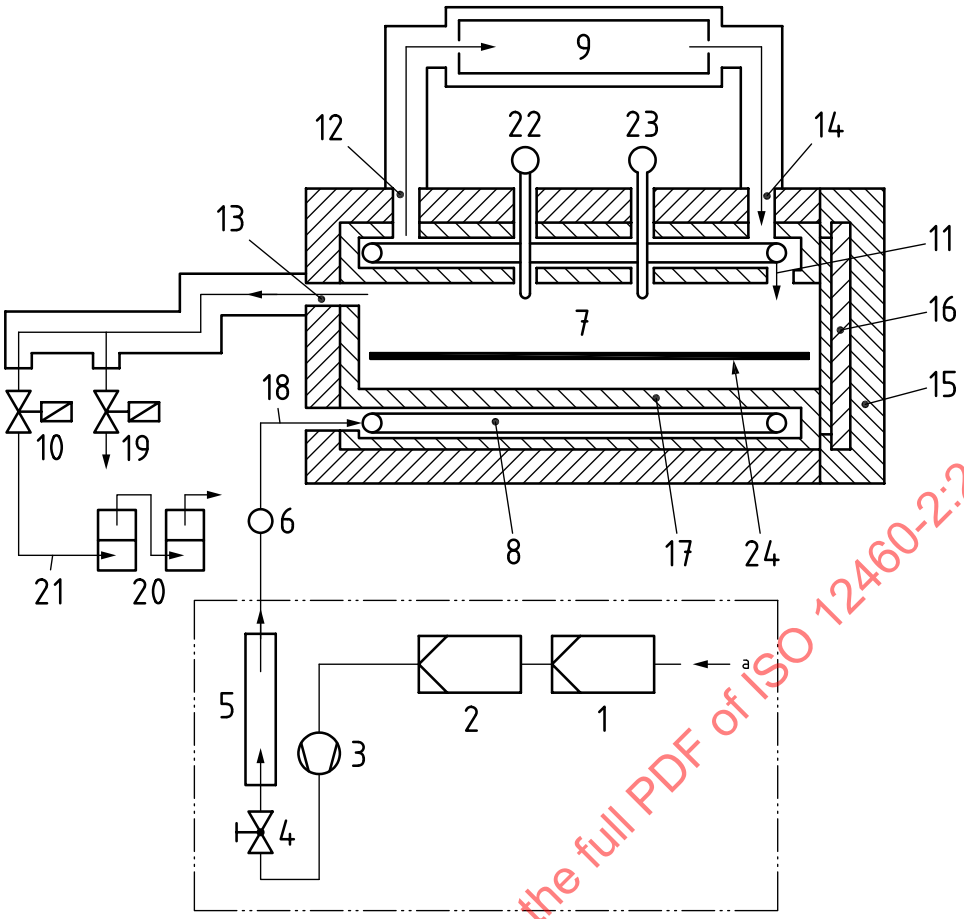
For testing, the test pieces shall be edge sealed with self-adhesive aluminium tape or an alternative sealing method if equivalence has been demonstrated. The emitting (unsealed) surface area of the sealed test piece has to be measured and calculated in square metres ($m^2$).

The test pieces shall be conditioned as described in Clause 8.

### A.5.3.2 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 and edge of each board.

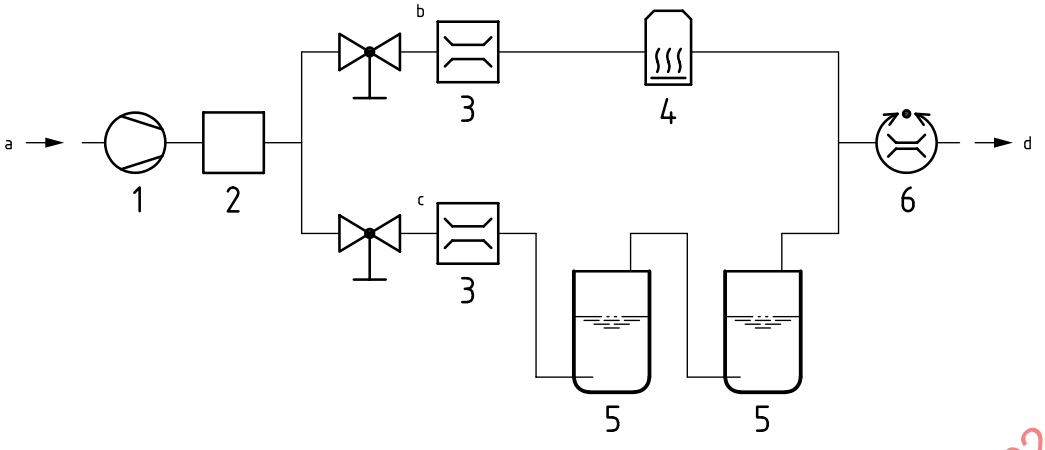



For operation without pre-humidification adjust as shown in Figure A.1

**Key**

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

**Figure A.10 — 0,004 m$^3$ test chamber construction scheme for operation with pre-humidified air**




**Key**

| | | | |
|---|---|---|---|
| 1 | gas pump | 4 | silica gel filter |
| 2 | charcoal filter | 5 | washing bottle(s) (humidification) with a minimum volume of 1 000 ml |
| 3 | gas flow meter and gas flow control | 6 | flow controller or gas meter |

a Air inlet.

b Dry air, ~50 % of total air flow.

c Wet air, ~50 % of total air flow.

d Air outlet, to the chamber.

**Figure A.11 — Example of a device for establishing a controlled air flow with a relative humidity of 50 %**

# Annex B
(informative)

# Conversion of formaldehyde test results

## B.1 General

The climatic parameters have to be monitored (see 7.1.5 and 9.2.3) during the entire period of the chamber test. If the measured values of temperature and/or relative humidity (calculated as averages) differ from the required standard parameter, the measured formaldehyde concentration in the chamber air shall be re-calculated using temperature and relative humidity values converted to the required standard parameters and given as 'corrected' values.

This annex provides three methods of performing this conversion.

NOTE 1 Berge et al.[15] equation is provided for re-calculation of different temperature and/or relative humidity, the Andersen equation has an additional option to include the air exchange and loading ratio. The WKI calculation model is based on formaldehyde test results received by EN 717-1 and is able to convert this result to different chamber variants by consideration of all chamber test parameters.

NOTE 2 At 25 °C (77 °F) and 1 013 hPa the following relationship exists for formaldehyde:

1,23 mg/m$^3$ = 1 ppm (parts per million);

1 mg/m$^3$ = 0,81 ppm (parts per million).

## B.2 Re-calculation of temperature using Berge et al. equation

Table B.1 is based on the Berge et al.[15] equation to correct formaldehyde concentrations in air for temperature:

$$C = C_0 \times e^{-R(1/t - 1/t_0)} \quad \text{(B.1)}$$

or

$$C_0 = C_e^{-R(1/t - 1/t_0)} \quad \text{(B.2)}$$

where

$C$ is the measured formaldehyde concentration level in mg/m$^3$;

$C_0$ is the corrected formaldehyde concentration level in mg/m$^3$;

$E$ is the natural log base;

$R$ is the coefficient of temperature (9 799);

$t$ is the measured temperature in Kelvin, K;

$t_0$ is the corrected temperature in Kelvin, K.

NOTE The Berge et al. equation 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 B.1 — Relative humidity conversion factors for formaldehyde**

| 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 |
| 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 | | | |

## B.3 Re-calculation of relative humidity using Berge et al. equation

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

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

or

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

where

$C$ is measured formaldehyde concentration level in mg/m$^3$;

$C_0$ is corrected formaldehyde concentration level in mg/m$^3$;

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

$H$ is measured relative humidity in %;

$H_0$ is corrected relative humidity in %.

**Table B.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 | ... | ... |

## B.4 Re-calculation of temperature, relative humidity, loading ratio and air exchange rate using Andersen Equation

To calculate the indoor air concentration $C_M$ measured under certain conditions ($T_M$, $H_M$, $L_M$, $N_M$) into another indoor air concentration $C_R$ with corrected (reference) conditions ($T_R$, $H_R$, $L_R$, $N_R$) the "Andersen equation"[14] (B.5) has to be transformed to Formula (B.6):

$$C = (0{,}08\,T - 0{,}764)\cdot(0{,}143\,H + 0{,}048)\,\frac{1}{1 + 0{,}304\cdot N / L} \tag{B.5}$$

where

$C$ is formaldehyde concentration in mg/m$^3$;

$T$ is temperature in K;

$H$ is relative humidity in %;

$N$ is air exchange in h$^{-1}$;

$L$ is loading ratio in m$^2$/m$^3$.

$$C_R = C_M \cdot \frac{(0{,}08\,T_R - 0{,}764)\cdot(0{,}143\,H_R + 0{,}048)\cdot(1 + 0{,}304\,N_M / L_M)}{(0{,}08\,T_M - 0{,}764)\cdot(0{,}143\,H_M + 0{,}048)\cdot(1 + 0{,}304\,N_R / L_R)} \tag{B.6}$$

where

$C_M$ is measured formaldehyde concentration in mg/m$^3$;

$T_M$ is measured temperature in K;

$H_M$ is measured relative humidity in %;

$N_M$ is measured air exchange in h$^{-1}$;

$L_M$ is measured loading ratio in m$^2$/m$^3$;

$C_R$ is corrected formaldehyde concentration in mg/m$^3$;

$T_R$ is corrected temperature in K;

$H_R$ is corrected relative humidity in %;

$N_R$ is corrected air exchange in h$^{-1}$;

$L_R$ is corrected loading ratio in m$^2$/m$^3$.



## B.5 Calculation of formaldehyde release – WKI calculation models

### B.5.1 General

The calculation models[8][10] are based on the determination of formaldehyde in a climate of 23 °C and 45 % relative humidity, an air exchange of 1/h and a loading ratio of 1 m$^2$/m$^3$ (here: $C_{ref}$). The determined formaldehyde emission value ($C_{ref}$) can be re-calculated to other chamber parameters[17][18].

### B.5.2 Linear model

The linear model according to Formula (B.7) is applicable to a range of relative humidity from 30 % to 50 %.

$$C = 0{,}00555 \cdot (C_{\text{ref}} + 0{,}008) \cdot (T - 12{,}7) \cdot (\text{RH} - 1{,}2) \frac{1}{1 + 1{,}75 \cdot N / L} \tag{B.7}$$

where

$C$ is corrected formaldehyde concentration in mg/m$^3$;

$C_{\text{ref}}$ is measured formaldehyde concentration (at reference parameter: 23 °C, 45 %, $N$ = 1, $L$ = 1) in mg/m$^3$;

$T$ is temperature in °C;

RH is relative humidity in %;

$N$ is air exchange per h;

$L$ is loading ratio in m$^2$/m$^3$.

### B.5.3 Exponential model – 2014

The exponential model according to Formula (B.8) is applicable to a range of relative humidity from 30 % to 80 %.

$$C = 0{,}0366 \cdot C_{\text{ref}} \cdot (T - 13{,}15) \cdot \left[ e^{(0{,}0403 \cdot \text{RH})} + 2{,}073 \right] \frac{1}{1 + 2{,}07 \cdot N / L} \tag{B.8}$$

### B.5.4 Exponential model – 2022

The exponential model according to Formula (B.9) is applicable to a range of relative humidity from 30 % to 80 %.

$$C = 0{,}28 \cdot C_{\text{ref}} \cdot \left( \left[ e^{(0{,}045 \cdot T)} - 1{,}24 \right] \cdot \left[ e^{(0{,}039 \cdot \text{RH})} + 1{,}16 \right] + 1{,}16 \right) \frac{1}{1 + 1{,}99 \cdot N / L} \tag{B.9}$$

where

$C$ is corrected chamber value in mg/m$^3$;

$C_{\text{ref}}$ is measured formaldehyde concentration (at reference parameter: 23 °C, 45 %, $N$ = 1, $L$ = 1) in mg/m$^3$;

$T$ is temperature in °C;

RH is relative humidity in %;

$N$ is air exchange per h;

$L$ is loading ratio in m$^2$/m$^3$.

# Annex C
(normative)

# Analytical procedure of formaldehyde determination

## C.1 General

This annex comprises different analytical procedures which can be used to determine formaldehyde in air. Known analytical procedures in which formaldehyde is analysed in a liquid phase are commonly referred to as 'wet-chemistry' and are described in C.1 to C.3. Other methods that can determine formaldehyde in air directly are described in C.4 and C.5.

## C.2 Acetylacetone method - Calibration curve and sampling

### C.2.1 General

The determination is based on the Hantzsch reaction in which aqueous formaldehyde reacts with ammonium ions and acetylacetone to yield diacetyldihydrolutidine (DDL), see Figure C.1. The reaction is highly specific to formaldehyde. DDL has an absorption maximum at 412 nm. The fluorescence intensity is determined at a wavelength of excitation $\lambda_{ex}$ = 410 nm and a wavelength of emission $\lambda_{em}$ = 510 nm to 515 nm (maximum wavelength has to be verified).

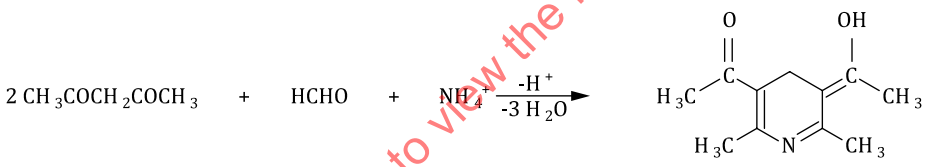



**Figure C.1 — Reaction scheme of the acetylacetone method**

### C.2.2 Hazards

Please refer to Safety Data Sheet of following substances:

— Formaldehyde solution (35 % to 40 %);

— Acetylacetone;

— Ammonium acetate.

### C.2.3 Equipment for chemical analysis

**C.2.3.1 Spectrophotometer**, suitable for use with cells with a path-length of at least 50 mm and capable of measuring absorbance at 412 nm.

**C.2.3.2 Water bath**, capable of maintaining a temperature of (60 ± 1) °C (140 °F).

**C.2.3.3 Water bath**, capable of maintaining a temperature in the range of 20 °C to 25 °C (68 °F to 77 °F).

**C.2.3.4 Six flasks**, 50 ml, with stoppers.

**C.2.3.5 Seven volumetric flasks**, 100 ml (calibrated at 20 °C/68 °F).

**C.2.3.6 Four (at least two) volumetric flasks**, 1 000 ml (calibrated at 20 °C/68 °F).

**C.2.3.7** Volumetric) pipettes, 1 ml, 2 ml, 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml, and 100 ml (calibrated at 20 °C/68 °F).

**C.2.3.8 Micro burette.**

**C.2.3.9 Erlenmeyer flasks**, 250 ml and beakers.

**C.2.3.10 Balance**, scale interval 0,001 g.

**C.2.3.11 Air sampling system** as described in 7.2 of the general part of this document.

### C.2.4 Reagents

Reagents and water of recognized analytical purity are used for the analysis.

#### C.2.4.1 Acetylacetone solution.

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

#### C.2.4.2 Ammonium acetate solution.

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

Commercially prepared solutions may be used.

### C.2.5 Calibration curve.

#### C.2.5.1 General

The calibration curve (see Figure C.2) is produced from a standard formaldehyde solution, the concentration of which has been determined by iodometric titration. This calibration curve shall be checked at least once a week. It is possible to modify this frequency if it is proven 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.

#### C.2.5.2 Formaldehyde standard solution

##### C.2.5.2.1 Reagents

**C.2.5.2.1.1 Standard iodine solution**, $c(I_2)$ = 0,05 mol/l.

**C.2.5.2.1.2 Standard sodium thiosulphate solution**, $c(Na_2S_2O_3)$ = 0,1 mol/l.

**C.2.5.2.1.3 Standard sodium hydroxide solution**, $c(NaOH)$ = 1 mol/l.

**C.2.5.2.1.4 Standard sulfuric acid solution**, $c(H_2SO_4)$ = 1 mol/l.

The above solutions shall be standardized before use starch solution 1 % by mass

For using photometric detection with path length 50 mm, dilute 1 g of formaldehyde solution (concentration 35 % to 40 %) with water in a 1 000 ml volumetric flask and make up to the mark.

#### C.2.5.2.2 Determination of formaldehyde concentration

Mix 20 ml of the formaldehyde standard solution with 25 ml iodine solution and 10 ml sodium hydroxide solution. After 15 min standing protected from light add 15 ml of sulfuric acid solution. Titrate back the excess iodine with the sodium thiosulphate solution. Near the end of the titration add some drops of the starch solution as an indicator. Carry out in parallel a blank test with 20 ml of water.

Ready to use formaldehyde standards with defined formaldehyde content may be used. The determination of formaldehyde content by titration of the standard solution is not required. Where ready to use formaldehyde standard solutions are used, the formaldehyde solution has to be only diluted in order to prepare a solution containing 3 mg of formaldehyde in 1 000 ml volumetric flask (C.2.3.6).

NOTE 1 Other methods, like using spectrophotometers with pre-calibrated curves or similar, can be used after demonstration that they provide equivalent results.

The formaldehyde content is calculated as follows:

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

where

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

$c(\mathrm{Na_2S_2O_3})$ is the thiosulphate concentration, in mols per litre (mol/l);

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

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

NOTE 2 1 ml of 0,1 mol/l thiosulphate corresponds to 1 ml of 0,05 mol/l iodine solution and 1,5 mg formaldehyde.

NOTE 3 With digital/automatic pipettes the volumes of the solutions can be reduced provided they give the same results.

### C.2.5.3 Formaldehyde calibration solution

Using the concentration value determined in C.2.5.2.2, calculate the volume of the formaldehyde solution which will contain about 3 mg formaldehyde. Transfer this volume, using a micro burette, to a 1 000 ml volumetric flask and make up to the mark with water. 1 ml of this calibration solution contains about 3 µg formaldehyde.

NOTE For using fluorescence detection the standard solution is prepared from a formaldehyde sodium bisulfite solution. 4,470 3 g formaldehyde sodium bisulfite are dissolved in water in a 1 000 ml volumetric flask and made up to the mark with water. 1 ml of this solution contains 1 mg formaldehyde and is transferred to a 1 000 ml volumetric flask and made up to the mark with water. 1 ml of this calibration solution contains 1 µg formaldehyde and is used to continue as described in C.2.5.3.

### C.2.5.4 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 (C.2.5.2) into a 100 ml volumetric flask and make up to the mark with water. 10 ml of each dilution are analyzed photometrically by the same procedure as described in C.2.7. The absorbance values are plotted against the formaldehyde concentrations $c$ (between 0 and 0,003 mg/ml, see example in Figure C.2). The slope $f$ of the graph is either determined graphically or calculated.

## C.2.6 Sampling

Add at least 25 ml of water to each of the two gas washing bottles and connect them to the air sampling system (C.2.3.11). Sample the air from the chamber by passing at a rate of maximum 2 l/min through the gas washing bottles.

The sampling procedure is a minimum of 15 min by repeated sampling or determinations.

The options for sampling considering the chamber size and analytical procedure are mentioned in Table C.1.

The volume of air to be sampled depends also on its formaldehyde concentration. With photometric determination the procedure described above is suitable for concentrations higher than 0,005 mg/m$^3$. For determination of lower concentrations, the volume of the sampled air should be increased and/or the volume of the air sample solution reduced. The sensitivity of the analysis can also be increased by using a fluorometric determination of the reaction product (diacetyldihydrolutidine) instead of a photometric determination.

**Table C.1 — Examples for sampling considering the chamber size and analytical procedure**

| Chamber size | Option | Air sampling volume | Volume air flow | Number of wash bottles used for analysis | Absorber solution | | Volume of absorber solution filled in wash bottles | Procedure for analysis | | |
|---|---|---|---|---|---|---|---|---|---|---|
| $m^3$ | | $m^3$ | L/min | | distilled water[a] | acety-lacetone reagent[b] | ml | | photom-etry | fluores-cence |
| 1<br>0,225<br>0,045 | 1 | 0,12 | 2 | 2 | x | | 25 or 30 | 10 ml used for analysis | x | x |
| | 2 | 0,12 | 2 | 1 | | x | 30 | to be analysed directly | x | x |
| | 3 | 0,06 | 2 | 2 | x | | 25 or 30 | 10 ml used for analysis | x | x |
| | 4 | 0,06 | 2 | 1 | | x | 30 | to be analysed directly | x | x |
| | 5 | 0,03 | 2 | 2 | x | | 25 or 30 | 10 ml used for analysis | / | x |
| | 6 | 0,03 | 2 | 1 | | x | 30 | to be analysed directly | / | x |
| 1<br>0,225<br>0,045<br>0,004 | 7 | 0,015 | 1 | 2 | x | | 25 or 30 | 10 ml used for analysis | / | x |
| | 8 | 0,015 | 1 | 1 | | x | 30 | to be analysed directly | / | x |

[a] Exact volume shall be recorded for further calculation.

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

NOTE For total air sampling volume the chamber air inlet and chamber volume have to be considered.

## C.2.7 Analytical procedure

### C.2.7.1 General

The solution prepared for analysis (formaldehyde containing solution and reagents) shall be shaken and heated for 10 min in a water bath (C.2.3.2) at 60 °C (140 °F). The heated stopper flask or wash bottle is then cooled in the absence of sunlight, e.g. in a water bath (C.2.3.3) operated in a temperature range between 20 °C and 25 °C (68 and 77 °F) for at least 15 min or cool it to room temperature for at least 60 min.

### C.2.7.2 Absorption solution: distilled water

Pipette 10 ml of each of the absorption solutions into a 50 ml flask, add 10 ml acetylacetone solution (C.2.4.2) and 10 ml of ammonium acetate solution (C.2.4.3), stopper the flask and shake it. Follow the analytical procedure as described in C.2.7.1.

### C.2.7.3 Absorption solution: mixed reagent

Close the wash bottle with suitable closures or transfer the solution into a 50 ml flask with stopper, shake it, Follow the analytical procedure as described in C.2.7.1.

NOTE Combination of reagents can be used as described in ISO 12460-3 [option 4], especially for reduced sampling time (e.g. 15 min): Using two gas wash bottles with volume of 100 ml (C.2.3.1), each containing 10 ml distilled water, 10 ml acetylacetone solution (C.2.4.2) and 10 ml ammonium acetate solution (C.2.4.3) as mixed reagent.

## C.2.8 Calculation of the amount of absorbed formaldehyde

The amount of formaldehyde absorbed in the water of the gas washing bottles is calculated by the following equation:

$$G = (A_s - A_b) \times f \times V_{sol} \quad \text{(C.2)}$$

where

$G$ is the amount of formaldehyde in each of the trapping solutions, in milligrams;

$A_s$ is the absorbance of the solution from the gas washing bottle;

$A_b$ is the absorbance of the blank value (refer to 0 ml point of calibration curve in C.2.5.3);

$f$ is the slope of the calibration curve for the standard formaldehyde solution, in milligrams per millilitre;

$V_{sol}$ is the volume of the trapping solution, in millilitres.

The values of $G$ from both trapping solutions are added to give the total amount of formaldehyde $G_{tot}$.

## C.2.9 Calculation of the formaldehyde emission

The formaldehyde emission from the wood-based panel being tested is expressed as the concentration in the air of the test chamber and is calculated by the following Formula (C.3):

$$c = G_{tot} / V_{air} \quad \text{(C.3)}$$

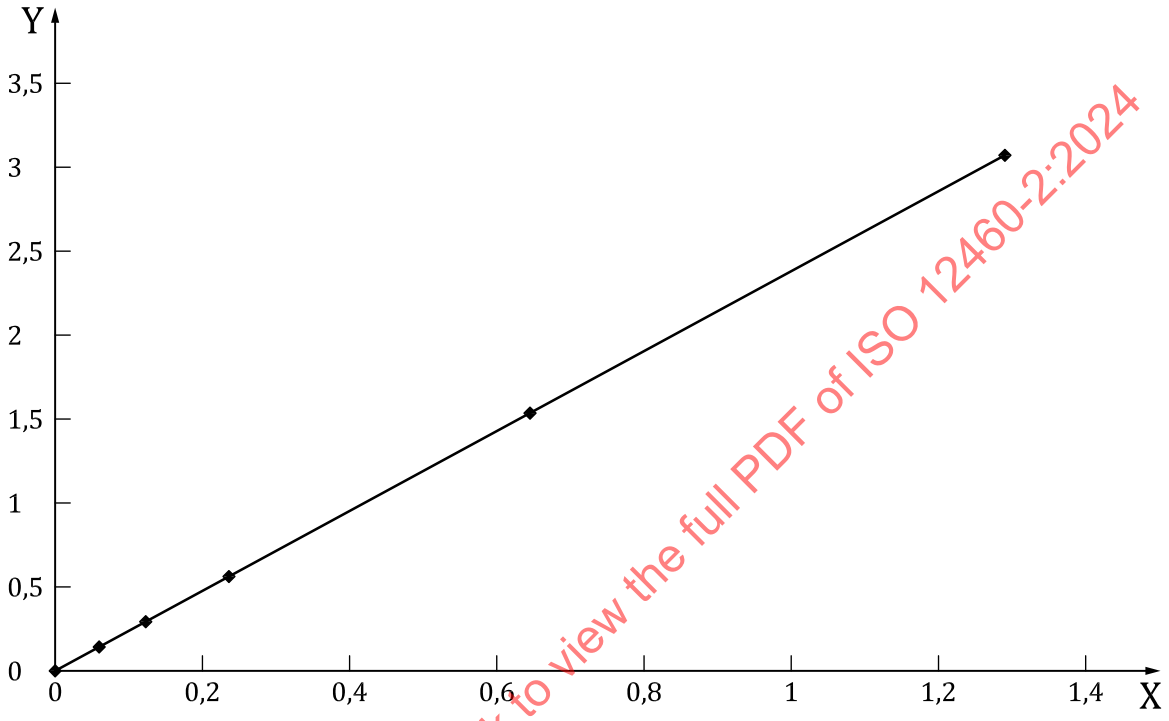
where

$c$ is the formaldehyde concentration, in milligrams per cubic metres;

$G_{tot}$ is the total amount of formaldehyde trapped, in milligrams;

$V_{air}$ is the volume of the air sample, in cubic metres.

The volume of the sampled air should be corrected to a standard temperature of 25 °C (77 °F) and a standard air pressure of 1 013 hPa.




**Key**

Y absorbance $(A_s - A_b)$

X concentration of the diluted calibration solution $c$ [$10^{-3}$ mg/ml]

$c = f \cdot (A_s - A_b)$

**Figure C.2 — Example of a calibration curve for formaldehyde determined by acetylacetone method (path length 50 mm)**



### C.2.10 Performance characteristic

The reaction of formaldehyde with acetylacetone and ammonium acetate is specific.

The limits of detection using a 50 mm cuvette and 10 ml of absorption solution and 10 ml of each of reagent solutions I and II are as follows[13]:

| | | | |
|---|---|---|---|
| absolute: | $C_{LD,\,abs.}$ | = | 0,000 3 mg per sample |
| relative: | $C_{LD,\,rel.}$ | = | 0,005 mg/m$^3$ for a 0,06 m$^3$ sample volume |
| | $C_{LD,\,rel.}$ | = | 0,003 mg/m$^3$ for 0,10 m$^3$ sample volume |

The repeatability standard deviation from duplicate determinations (50 mm cuvette) is 0,001 mg/m$^3$ to 0,002 mg/m$^3$ at formaldehyde concentrations between 0,05 mg/m$^3$ and 0,2 mg/m$^3$.

### C.2.11 Interferences

In the concentration range from 8 µg formaldehyde per 10 ml of absorption solution, the presence of acetaldehyde up to 50 times the amount of formaldehyde does not interfere with the formaldehyde determination.

Higher concentrations of sulfur dioxide (greater than 0,3 mg of $SO_2/m^3$) and oxidizing substances (for example ozone) which weaken the color reaction, can, however, prove interfering. These substances, however, are generally not present in interfering amounts in indoor air.

## C.3 Chromotropic acid method – Calibration curve and sampling

### C.3.1 General

Samples of wood product are placed in an environmentally controlled chamber with air pumped through the chamber and then purged through an aqueous solution. The formaldehyde that is emitted from the wood product is in the air and then transferred to the aqueous solution. The solution is analysed for formaldehyde content using the chromotropic acid procedure. There is a reaction between the chromotropic acid and formaldehyde with the formaldehyde in the presence of a strong acid, see Figure C.3.

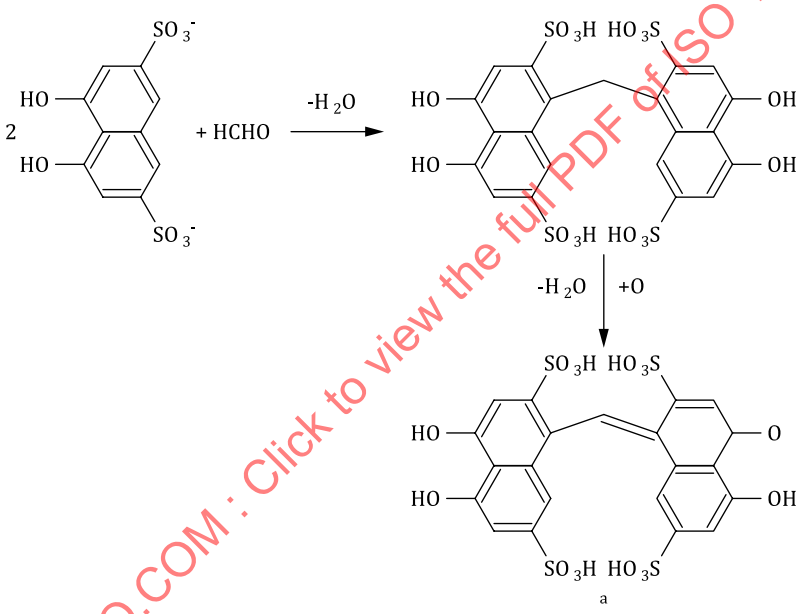



**Figure C.3 — Reaction scheme of the chromotropic acid method**

Two molecules of chromotropic acid react with one molecule of formaldehyde in the presence of concentrated sulfuric acid to form a chromogen which is violet/purple in colour. The coloured liquid is analysed by a spectrophotometer at 580 nm for absorbance. The resulting absorbance reading is used in a calibration curve calculation to determine the formaldehyde content in the solution.

### C.3.2 Hazards

Please refer to Safety Data Sheet of following substances:

— chromotropic acid;

— sulfuric acid;

— formaldehyde.

### C.3.3 Equipment for chemical analysis

**C.3.3.1 Spectrophotometer,** suitable for use with cells with a path-length of at least 10 mm and capable of measuring absorbance at 580 nm.

**C.3.3.2 Beaker**, 150 ml, low form.

**C.3.3.3 Volumetric flasks,** (calibrated at 20 °C/68 °F).

**C.3.3.4 pH meter.**

**C.3.3.5 Magnetic stirrer.**

**C.3.3.6 Pipets,** calibrated.

**C.3.3.7 Test tubes,** 16 by 150 mm, with PTFE lined screw cap.

**C.3.3.8 Water bath,** capable to maintain 95 °C (203 °F) ± 1 K.

**C.3.3.9 Balance,** scale interval 0,001 g.

**C.3.3.10 Air sampling system** as described in 7.2 of the general part of this document.

### C.3.4 Reagents

Reagents and water of recognized analytical purity are used for the analysis.

#### C.3.4.1 Chromotropic acid reagent

Dissolve 0,10 g of chromotropic acid (4,5-dihydroxy-2,7-naphthalene-disulfonic acid disodium salt) in freshly distilled water and dilute to 10 ml.

This solution is to be made up daily.

**C.3.4.2** Sulfuric Acid ($H_2SO_4$), concentrated, reagent grade. Nitrate concentration shall be less than 10 µg/g.

**C.3.4.3** Sodium bisulfite 1 % ($NaHSO_3$), reagent grade.

### C.3.5 Calibration curve

#### C.3.5.1 General

A calibration curve shall be conducted on a routine basis in duplicate and verified when there are reagent changes, equipment changes or other reasons to question the validity of the curve. The final calibration curve is expressed as a composite of the duplicate values. The calibration curve is expressed as absorbance of the prepared samples versus formaldehyde concentration of the samples and is calculated using a linear least squares fitting technique. The calibration curve may not be linear at high formaldehyde concentrations (high absorbance readings). If the plot shows the last few points deviating from linearity, omit the points from calculations or repeat entire procedure.

The calibration curve as described in this annex is provided as an example. In use, the calibration curve concentrations can be adjusted to reflect the needs of the analyte. If absorbance readings of samples are outside of the range of the calibration curve, dilute the solution with distilled water to a concentration that is within the calibration curve.

#### C.3.5.2 Formaldehyde standard solution

##### C.3.5.2.1 Reagents

**C.3.5.2.1.1 Formaldehyde solution**, mass 37 %.

**C.3.5.2.1.2 Buffer solution**, pH 9,0.

**C.3.5.2.1.3 Hydrochloric acid**, (HCl) 0,100 N, standard.

**C.3.5.2.1.4 Sodium sulfite solution**, 1,0 M.

Dissolve 12,67 g anhydrous sodium sulfite ($Na_2SO_3$) (ACS assay 99,5 %) in a 100 ml volumetric flask and dilute to the mark with freshly distilled water. The correct amount to be dissolved should be 12.6/ACS assay of the anhydrous sodium sulfite actually being used (read assay from bottle label).

##### C.3.5.2.2 Determination of formaldehyde concentration

**C.3.5.2.2.1 Formaldehyde standard solution A (content: 1,0 mg/ml)**

Pipet 2,70 ml of 37,0 % (should be adjusted for actual formaldehyde amount) formaldehyde solution into a 1 000 ml volumetric flask. Dilute to mark with freshly distilled water and mix well. This solution is stable for at least one month.

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 each standard solution A aliquot.

NOTE Calibrate the pH meter according to manufacturer's recommendations.

Place solution on magnetic stirrer. Immerse pH electrodes into the solution and carefully titrate with 0,100 N hydrochloric acid (HCl) to past pH 9,5. Record volume of HCl and corresponding pH intermittently. Make a graph of pH versus volume of HCl use this graph to calculate the volume ($V$) of 0,1 N HCl used to reach pH 9,5. Use this value to calculate the concentration of $C_A$.

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

$$C_A = \frac{V \times 0,1 \times 30,03}{50} \quad \text{(C.4)}$$

where

$C_A$ is the concentration of formaldehyde standard solution A (mg/ml);

$V$ is 0,100 N HCl required at pH of 9,5 from the graph prepared in C.3.5.2.2.1, ml, and normality of HCl;

$N$ is the concentration of standard Solution A will be the average of the two analyses conducted.

**C.3.5.2.2.2 Formaldehyde standard solution B (content 0,01 mg/ml)**

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

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

$$C_B = \frac{C_A \times 1\,000 \times 1}{100} \quad \text{(C.5)}$$

Record the value.

### C.3.5.3 Determination of the calibration curve

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.

Label six 16 mm by 150 mm screw capped test tubes with 1, 2, 3, 4, 5, and 6.

Pipet the volumes of 1 % sodium bisulfite solution and then standard solution B (C.3.5.2.2.2) into the labelled test tubes as specified in Table C.2:

**Table C.2 — Volumes of sodium bisulfite solution and standard solution B**

| Tube | Volume ml | | Target concentration | Target content of HCHO |
|---|---|---|---|---|
| No. | $NaHSO_3$ | Solution B | µg/ml | µg |
| 1 | 4,0 | 0 | 0 | 0 |
| 2 | 3,9 | 0,10 | 0,025 | 0,10 |
| 3 | 3,7 | 0,30 | 0,075 | 0,30 |
| 4 | 3,5 | 0,50 | 0,125 | 0,50 |
| 5 | 3,3 | 0,70 | 0,175 | 0,70 |
| 6 | 3,0 | 1,00 | 0,250 | 1,00 |

NOTE 1 No Solution B is added to Test Tube 1. Test Tube 1 will be the reagent blank.

NOTE 2 Concentrations used in calibration curve can be adjusted to adequately span the expected formaldehyde concentration in routine analysis.

Plot absorbance against micrograms of formaldehyde in the test solutions. Note the amount of formaldehyde in micrograms is based upon the concentration of formaldehyde in standard Solution B, which is dependent upon the standardization carried out on standard solution A in C.3.5.2.2.1.

Read and record absorbance at 580 nm for each standard prepared (tubes 2 to 6).

The absorbance of each tube should be plotted against the total micrograms of formaldehyde in each tube.

## C.3.6 Analytical procedure

Pipet 4 ml of the $NaHSO_3$ solution from the gas wash bottle into each of three 16 by 150 mm screw-cap test tubes for triplicate analysis of each gas wash bottle sample.

Pipet 4 ml of 1 % $NaHSO_3$ into a 16 by 150 mm screw-cap test tube to act as a reagent blank and add 0,1 ml of 1 % chromotropic acid reagent to each test tube. Shake tube after addition.

Slowly and carefully pipet 6,0 ml concentrated sulfuric acid ($H_2SO_4$) into each test tube and allow to flow down the side of test tube. Before placing caps on test tubes, check the condition of the polytetrafluoroethylene (PTFE) cap liners to make sure they are clean and not deteriorated.

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.

If absorbance readings exceed 1,0 or if spectrophotometric analysis is performed within 2 h, heat capped test tubes to 95 °C (203 °F) or place capped test tubes in heat block or boiling water bath for (15 ± 2) min to ensure that the chemical reaction is completed. Remove tubes from heat source and allow to cool to room temperature.

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. A high absorbance 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 0,040 (using a 12 mm cell path length) or above 0,030 (using a 10 mm cell path length), repeat the entire standardization procedure.

Zero the instrument using the reagent blank, or the instrument may be left zeroed on distilled water, and the absorbance of the reagent blank subtracted from the absorbance of the standard solutions.

Read and record absorbance at 580 nm for each test tube prepared. If the absorbance of the specimen solution is found to fall outside the preferred absorbance range (>1,0), analysis may be repeated using an appropriate dilution of each gas wash bottle solution.

### C.3.7 Sampling

Add 20 ml of a 1 % sodium bisulfite ($NaHSO_3$) solution to each of the two gas washing bottles and connect them to the air sampling system (C.2.3.11). Sample the air from the chamber periodically by passing, for example, for a 1 $m^3$ chamber a minimum of 60 l, at a rate of approximately (1 ± 0,05) l/min, through the gas washing bottles.

The volume of air to be sampled depends also on its formaldehyde concentration. For determination of lower concentrations, the volume of the sampled air should be increased and/or the volume of the air sample solution reduced.

### C.3.8 Calculation of the amount of absorbed formaldehyde

The absorbance of each chamber gas wash bottles aliquot specimen determined in C.3.7 is compared to the calibration curve, and the total micrograms of formaldehyde in the aliquot is represented as $C_a$ in Formula (C.6).

The amount of formaldehyde absorbed in the each of the used gas washing bottles is calculated by the following equation using the calibration curve Formula (C.6):

$$C_a = mx + b \tag{C.6}$$

where

$C_a$ is the total amount of formaldehyde, in microgram;

$m$ is the slope of the calibration curve;

$x$ is the absorbance of analyte;

$b$ is the Y axis intercept.

$$C_t = C_a \times F_a \tag{C.7}$$

where

$C_t$ is the total amount of formaldehyde in each of the trapping solutions, in microgram;

$C_a$ is the total quantity of formaldehyde in the sample aliquots taken form the gas washing bottle.

$$F_a = \frac{V_{s,sol}}{V_a} \quad (C.8)$$

The values of $C_t$ from both trapping solutions are added to give the total amount of formaldehyde $C_{tot}$.

### C.3.9 Calculation of the formaldehyde emission

The formaldehyde emission from the wood-based panels being tested is expressed as the concentration in the air of the test chamber and is calculated by the following Formula (C.9):

$$c = C_{tot} / V_{air} \quad (C.9)$$

where

$c$ is the formaldehyde concentration, in micrograms per cubic metre;

$C_{tot}$ is the total amount of formaldehyde trapped, in micrograms;

$V_{air}$ is the volume of the air sample, in cubic metres.

The volume of the sampled air should be corrected to a standard temperature of 25 °C (77 °F) and a standard air pressure of 1 013 hPa.

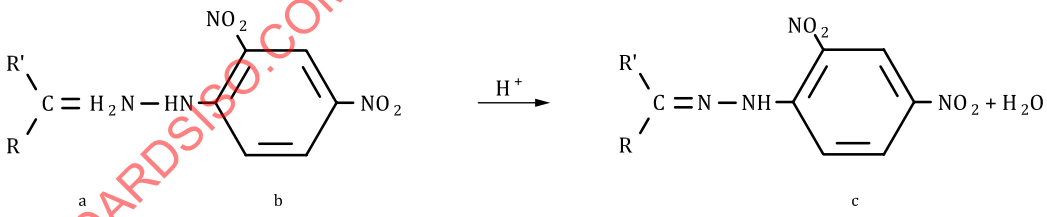
### C.3.10 Interferences

The NIOSH 3500 analytical method lists phenols as a negative interference when present at an 8:1 excess over formaldehyde. Modifications in the analytical procedure shall be made when relatively high phenol to formaldehyde concentrations (8:1) are anticipated.

## C.4 DNPH method - Calibration curve and sampling

### C.4.1 General

Formaldehyde reacts with 2,4-dinitrophenylhydrazine to the corresponding hydrazone, which can be determined qualitatively and quantitatively by HPLC, detection at 360 nm.




**Key**

R, R′ H, alkyl group, aromatic group

a Carbonyl compund (aldehyde or ketone).

b 2,4-Dinitrophenylydrazin (DNPH).

c DNPH derivative.

**Figure C.4 — Reaction of carbonyl compounds to form 2,4-dinitrophenylhydrazones**

The sampling flow rate specified has been validated for sampling rates of 1,0 l/min to 2,0 l/min. The solid-sorbent sampling procedure is specific for sampling and analysis of formaldehyde.

Exposure of the DNPH-coated sampling cartridges to direct sunlight may produce artefacts and should be avoided[16].

NOTE In principle, it is also possible to determine formaldehyde with DNPH using self-impregnated filters or with absorption in DNPH solution. This were described in other standards and can be used after proof of equivalence if necessary (further description see ISO 16000-3[6]).

### C.4.2 Hazards

Please refer to Safety Data Sheet of following substances:

— formaldehyde 2,4-DNPH standard (solid);

— acetonitrile;

— methanol.

2,4-Dinitrophenylhydrazine is explosive in the dry state and shall be handled with extreme care. It is also toxic (in the rat, LD50 = 654 mg/kg), has been shown to be mutagenic in some tests, and is irritating to the eyes and skin.

### C.4.3 Equipment for chemical analysis

Usual laboratory apparatus and in particular the following (C.4.3.1 to C.4.3.6).

**C.4.3.1 Commercially sampling cartridge,** packed with silica gel and coated with DNPH.

The ratio of the silica gel bed diameter to bed length shall not exceed 1:1. The capacity of the cartridge for formaldehyde shall be at least 75 µg and the collection efficiency at least 95 % at a sampling rate of 1,5 l/min. Sampling cartridges with very low blank levels and high performance are commercially available.

NOTE Some commercially available pre-coated cartridges exhibit lower pressure-drops, which permit the use of battery-operated personal sampling pumps.

**C.4.3.2 Air sampling pump,** capable of accurately and precisely sampling at a flow rate of 1,0 l/min to 2,0 l/min.

**C.4.3.3 Flow controller,** mass flow meters and mass flow controllers, or other suitable device for metering and setting air flow rates of 1,0 l/min to 2,0 l/min through the sample cartridge.

**C.4.3.4 Flow calibrator,** such as a rotameter, soap-bubble meter or wet test meter.

NOTE If the analyses will not take place in the same lab for transport: Cartridge containers, e.g. borosilicate glass culture tubes (20 mm ¥ 125 mm) with polypropylene screw caps, or other suitable containers, to transport coated cartridges. To keep the cartridges air tight, cool (< 7 °C) and protected from sunlight.

**C.4.3.5 HPLC system,** consisting of (C.4.3.5.1 to C.4.3.5.7)

**C.4.3.5.1 Mobile phase reservoir with an outgassing device**, (e.g. membrane under reduced pressure).

**C.4.3.5.2 High-pressure pump.**

**C.4.3.5.3 Injection valve** (automatic sampler with a 25 µl or other convenient loop volume).

**C.4.3.5.4 C-18 reverse phase (RP) column**, (e.g. 25 cm ¥ 4,6 mm inside diameter, 5 µm particle size).

**C.4.3.5.5 UV detector** or **diode array detector**, operating at 360 nm.

#### C.4.3.5.6 Data system.

#### C.4.3.5.7 **Column oven** (optional).

The DNPH-formaldehyde derivative is determined using isocratic reverse phase HPLC, equipped with an ultraviolet (UV) absorption detector operated at 360 nm.

NOTE 1 Most commercial HPLC analytical systems are adequate for this application.

NOTE 2 A column oven can be used to assure constant column operating temperature and improve reproducibility.

### C.4.3.6 Syringes and pipettes

HPLC injection syringes, with capacity at least four times the loop volume

Syringes, volume 10 ml, used to prepare DNPH-coated cartridges (polypropylene syringes are adequate). Syringe fittings and plugs, to connect cartridges to the sampling system and to cap prepared cartridges.

Pipettes, positive-displacement, repetitive-dispensing type, with capacities in the 0 ml to 10 ml range, ISO 8655-2[1].

## C.4.4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, e.g. best quality grade, grade for chemical analysis or grade for HPLC analysis, and distilled or demineralized water or water of equivalent purity.

#### C.4.4.1 2,4-Dinitrophenylhydrazine.

#### C.4.4.2 Acetonitrile, UV grade.

#### C.4.4.3 **Formaldehyde DNPH sandard** (ready for use).

#### C.4.4.4 Methanol.

### C.4.4.5 Preparation of reagents

#### C.4.4.5.1 Preparation of DNPH-formaldehyde derivative

Most common is a commercially available standard.

NOTE DNPH-formaldehyde derivate can be prepared in the labs see ISO 16000-9.

#### C.4.4.5.2 Preparation of DNPH-formaldehyde standards

Prepare a standard stock solution of the DNPH-formaldehyde derivative by dissolving accurately weighed amounts in acetonitrile. Prepare a working calibration standard from the standard stock solution. The concentration of the DNPH-formaldehyde derivative in the standard solutions should be adjusted to reflect the range of concentrations expected in real samples. Individual stock solutions of approximately 50 mg/l can be prepared by dissolving 2,5 mg of the solid derivative in 50 ml of acetonitrile. The individual solution is used to prepare calibration standards containing the derivative of interest at concentrations of 0,07 mg/l to 15 mg/l, that spans the concentration of interest. Store all standard solutions in tightly capped containers in a refrigerator and protected from light. Allow them to equilibrate to room temperature before use. They should be replaced after 4 weeks.

Sampling cartridges have been found to be stable for at least 6 months when stored at 4 °C in the absence of light.

### C.4.5 Calibration curve

#### C.4.5.1 General

To identify and quantify formaldehyde, calibration with external standard is performed.

Prepare calibration standards in acetonitrile from the DNPH-formaldehyde derivative. Individual stock solutions of 50 mg/l are prepared by dissolving 5 mg of solid derivative in 100 ml of mobile phase.

This calibration curve shall be checked weekly at minimum using a one-point calibration standard. It is possible to modify this frequency if it is proven 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.

#### C.4.5.2 Formaldehyde standard calibration solution

Preparation of the standards (see Table C.3 to Table C.7):

Approximately 50 mg/l pure standards are to be prepared. For this purpose, the purity of the solid DNPH derivatives and the aldehyde content (purity) of the derivative shall be considered.

Calibration range per standard in pure substance approximately 0,1 mg/l to 15 mg/l

**Table C.3 — Preparation of stock solutions ~ 50 mg/l (example)**

| Stock solution | Formaldehyde-DNPH |
|---|---|
| Molar mass of derivative [g/mol] | 210,15 |
| Molar mass formaldehyde [g/mol] | 30,03 |
| Nominal mass in of Derivate [mg] | 40 |
| Exact weighed-in derivative [mg/l] (Example) | 40,02 |
| Corresponds to weighed-in quantity formaldehyde [mol] | 0,19 |
| Corresponds to formaldehyde pure substance [mg] | 5,7 |
| Purity [%] | 98,1 % |
| Volume formaldehyde standard solution [ml] | 100 |
| Concentration of ready to use stock solution [mg/l] | 56,10 |

**Table C.4 — Calibration solution prepared from the stock solutions as follows, if the DNPH-formaldehyde derivate as a purity of 100 % (exact 50 mg/l formaldehyde Standard)**

| Calibration solution | Target concentration<br>mg/l | Stock solution<br>ml | Correspond in absolute<br>mg/20 ml |
|---|---|---|---|
| Cal 1 | 7,5 | 3 | 0,150 |
| Cal 2 | 2,5 | 1 | 0,050 |
| Cal 3 | 1,25 | 0,5 | 0,025 |

**Table C.5 — The calibration solutions further diluted as follows**

| Calibration solution | Target concentration<br>mg/l | Stock solution<br>ml | Correspond in absolute<br>mg/20 ml |
|---|---|---|---|
| Cal 4 | 0,75 | Cal 1 solution 1:10 | 0,015 |
| Cal 5 | 0,25 | Cal 2 solution 1:10 | 0,005 |
| Cal 6 | 0,125 | Cal 3 solution 1:10 | 0,002 5 |

**Table C.6 — Example of correct calculated concentration of the calibration standards**

| Calibration solution | | | Volume of stock solution (~50 mg/l) to fill up to 20 ml | Resulting formaldehyde-DNPH standard expressed by formaldehyde concentration |
|---|---|---|---|---|
| | | | ml | mg/l |
| Cal 1 | Stock solution (~ 50 mg/l) | | 3 | **8,42** |
| Cal 2 | Stock solution (~ 50 mg/l) | | 1 | **2,81** |
| Cal 3 | Stock solution (~ 50 mg/l) | | 0,5 | **1,40** |
| Cal 4 | Cal 1 | 01:10 | 1 | **0,84** |
| Cal 5 | Cal 2 | 01:10 | 1 | **0,28** |
| Cal 6 | Cal 3 | 01:10 | 1 | **0,14** |
| Cal 7 | Cal 3 | 01:20 | 0,5 | **0,07** |

**Table C.7 — Examples of sampling | Overview which sampling conditions causes which concentrations values**

| Sampling time | Volume reagent | Sampling rate | Total sampling volume | Formaldehyde result mg/l | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | ppm | 0,005 | 0,01 | 0,025 | 0,05 | 0,10 | 0,15 | 0,20 |
| min | ml | l/min | $m^3$ | $mg/m^3$ | 0,006 3 | 0,013 | 0,031 | 0,063 | 0,125 | 0,188 | 0,25 |
| 60 | 5 | 2 | 0,12 | | 0,15 | 0,30 | 0,75 | 1,50 | 3,00 | 4,50 | 6,00 |
| 60 | 5 | 1 | 0,06 | | 0,08 | 0,15 | 0,38 | 0,75 | 1,50 | 2,25 | 3,00 |
| 15 | 5 | 1,5 | 0,022 5 | | 0,03 | 0,06 | 0,14 | 0,28 | 0,56 | 0,84 | 1,13 |
| 15 | 5 | 2 | 0,03 | | 0,04 | 0,08 | 0,19 | 0,38 | 0,75 | 1,13 | 1,50 |
| 30 | 5 | 1 | 0,03 | | 0,04 | 0,08 | 0,19 | 0,38 | 0,75 | 1,13 | 1,50 |
| 30 | 5 | 2 | 0,06 | | 0,08 | 0,15 | 0,38 | 0,75 | 1,50 | 2,25 | 3,00 |

The highest concentration of the calibration curve should be at least 25 % above the highest suspected concentration.

### C.4.6 Determination of the calibration curve

Analyse each calibration standard (at least five levels) twice and tabulate area response against the mass injected or more conveniently, versus the DNPH-formaldehyde injected, for a fixed loop volume. Perform all calibration runs as specified for sample analysis. To avoid carry-over effects, start with the lower concentration. Using the UV detector or the diode array detector, a linear response range of approximately 0,07 mg/l to 8,4 mg/l should be achieved for e.g. 25 µl injection volumes. The results can be used to prepare a calibration curve. Linear response is indicated where a correlation coefficient of at least 0,999 for a linear least-squares fit of the data (concentration versus area response) is obtained. The retention times for each analyte should agree within 2 %.

**Operating parameters, HPLC**

Column: C-18 reverse phase

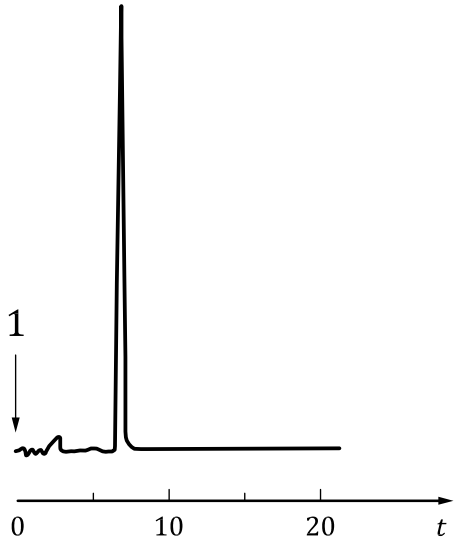
Mobile phase: 60 % volume fraction acetonitrile +40 % volume fraction water

Detector: ultraviolet, at 360 nm

Flow rate: 1 ml/min

Retention time: about 7 min for formaldehyde

Sample injection volume: 25 µl




**Key**

*t* time, in min

1 injection

**Figure C.5 — Example chromatogram of DNPH-formaldehyde derivative**

Once linear response has been documented, an intermediate concentration standard near the anticipated level shall be prepared and measured. This concentration shall be at least 10 times higher than the detection limit.

### C.4.7 HPLC system

Assemble the HPLC system and calibrate as specified. Either a gradient or an isocratic elution program may be employed (see ISO 16000-3). An isocratic mobile phase of a 60 % volume fraction acetonitrile +40 % volume fraction water mixture has been shown to be suitable. The parameters are as follows:

| | |
|---|---|
| Column | C-18 (4,6 mm inside diameter ¥ 25 cm, or equivalent)<br>a column oven may be used<br>for more accurate temperature control, if needed |
| Mobile phase | 60 % volume fraction acetonitrile +40 % volume fraction water, isocratic |
| Detector | Ultraviolet, operating at 360 nm |
| Flow rate | 1,0 ml/min |
| Retention time | 7 min for formaldehyde with one C-18 column, 13 min for formaldehyde with two C-18 columns |
| Sample injection volume | 25 µl |

Before each analysis, check the detector baseline to ensure stable conditions.

Mobile phase:

Prepare the HPLC mobile phase by mixing 600 ml of acetonitrile and 400 ml of water or set the parameters on the gradient elution HPLC appropriately.

Place the mobile phase in the HPLC solvent reservoir and set the pump at a flow rate of 1,0 ml/min.

Several column volumes of 100 % volume fraction acetonitrile. The same protection can be achieved if precolumns are used.

NOTE The chromatographic conditions specified here have been optimized for the detection of formaldehyde. Analysts are advised to experiment with their HPLC system to optimize chromatographic conditions for their particular analytical needs. HPLC systems with automated injection and start of data acquisition can also be used.

### C.4.8 Sampling/test duration

#### C.4.8.1 General

Sample collection: Assemble the sampling system and ensure that the pump is capable of constant flow rate throughout the sampling period. The sampling cartridges can be safely used for sampling air when the temperature is above 10 °C.

Before sample collection, check the system for leaks. Plug the inlet (short end) of the cartridge so no flow is indicated at the outlet end of the pump. The flow meter should not indicate any air flow through the sampling apparatus.

For unattended or extended sampling periods, a mass flow controller or, as appropriate, a compensated personal sampling pump is highly recommended to maintain constant flow. The flow controller should be set at least 20 % below the fixed maximum air flow rate through the cartridge.

Install the entire assembly (including a “dummy” sampling cartridge) and check the flow rate at a value near the desired rate. Flow rates of 1,0 l/min to 2,0 l/min shall be employed. The total number of moles of carbonyl in the volume of air sampled shall not exceed that of the DNPH (e.g. 1 mg DNPH/cartridge for commercially available pre-coated cartridges). In general, a safe estimate of the sample size should be lower than 75 % of the DNPH molar loading of the cartridge.

NOTE EN 1232[10] specifies an appropriate calibration scheme that does not require a sealed flow system downstream of the pump.

Measure and record the sampling flow rate at the beginning and end of the sampling period to determine sample volume. If the sampling period exceeds 2 h, the flow rate should be measured at intermediate points during the sampling period. Include a rotameter to allow observation of the flow rate without interruption of the sampling process. Alternatively, a sampling pump which directly measures and continuously records the flow rate can be used.

With commercial pre-coated DNPH cartridges, follow the manufacturer’s instructions. Let the cartridge warm to room temperature before connecting to the sampling train.

Connect the cartridge to the sampling train so that the short end becomes the sample inlet.

Turn the sampler on and adjust the flow to the desired rate. A typical flow rate through one cartridge is between 1,0 l/min and 2,0 l/min. Operate the sampler for the desired period, with periodic recording of the sampling variables.

At the end of the sampling period, check the flow rate just before stopping the flow. If the flow rates at the beginning and end of the sampling period differ by more than 10 %, the sample should be marked as suspect.

If there is the need to convert the concentrations to standard conditions (temperature and pressure) by calculation, temperature and pressure have to be measured during sampling.

Immediately after sampling, remove the cartridge from the sampling system, cap with the original end plugs, Refrigerate the exposed sample cartridge until analysis. The refrigeration period prior to analysis should not exceed 30 days.

If samples are to be transported to a central laboratory for analysis, the duration of the non-refrigerated period should be kept to a minimum, preferably less than 2 days.

Calculate the average sample flow rate using Formula (C.10):

$$\bar{q}_V = \frac{q_1 + q_2 + \ldots + q_n}{n} \quad \text{(C.10)}$$

where

$\bar{q}_V$ is the average flow rate, in millilitres per minute;

$q_1 + q_2 + \ldots + q_n$ is are the flow rates determined at beginning, end and intermediate points during sampling;

$n$ is the number of points averaged.

The total flow is then calculated using Formula (C.11):

$$V_m = \frac{(t_2 - t_1) \times \bar{q}_V}{1\,000} \quad \text{(C.11)}$$

where

$V_m$ is the total volume, in litres, sampled at the measured temperature and pressure;

$t_2$ is the stop time;

$t_1$ is the start time;

$t_2 - t_1$ is the total sampling duration, in minutes;

$\bar{q}_V$ is the average flow rate, in millilitres per minute.

#### C.4.8.2 Calculation of the formaldehyde emission

Calculate the mass of formaldehyde in the blank cartridge using Formula (C.12):

$$m_b = A_b \frac{\gamma_{std}}{A_{std}} V_b \, d_b \quad \text{(C.12)}$$

where

$m_b$ is the analyte mass, in micrograms, in the blank cartridge;

$A_b$ is the number of area counts, eluate from blank cartridge;

$A_{std}$ is the number of area counts, standard;

$\gamma_{std}$ is the concentration, in micrograms per millilitre, of analyte in the daily calibration standard;

$V_b$ is the total volume, in millilitres, of the blank cartridge eluate;

$d_b$ is the dilution factor for the blank cartridge eluate = 1,0.

Calculate the uncorrected mass of the DNPH derivate using Formula (C.13):

$$m_s = A_s \frac{\gamma_{std}}{A_{std}} V_s \, d_s \quad \text{(C.13)}$$

where

$m_s$ is the uncorrected mass of the DNPH derivative, in micrograms, on the sample cartridge;

$A_s$ is the number of area counts, eluate from sample cartridge;

$\gamma_{std}$ is the concentration, in micrograms per millilitre, of analyte in the daily calibration standard;

$A_{std}$ is the number of area counts, standard;

$V_s$ is the total volume, in millilitres, of the sample cartridge eluate;

$d_s$ is the dilution factor for the sample cartridge eluate:
= 1 if sample was not rediluted
= $V_d/V_a$ if sample was rediluted to bring the detector response within linear rang;

where

$V_d$ is the redilution volume, in millilitres,

$V_a$ is the aliquot used for redilution, in millilitres;

$d_b$ is the dilution factor for the blank cartridge eluate = 1,0.

Calculate the corrected total mass, in micrograms, of analyte (DNPH derivative) extracted from the cartridge, $m_d$, for each sample using Formula (C.14):

$$m_d = m_s - m_b \quad \text{(C.14)}$$

where

$m_s$ is the uncorrected mass of the DNPH derivative, in micrograms, on the sample cartridge;

$m_b$ is the analyte mass, in micrograms, in the blank cartridge.

Calculate the concentration, in nanograms per litre, of the carbonyl compound in the original sample, $y_A$, from Formula (C.15)

$$y_A = m_d \frac{M_c}{M_{der}} \times \frac{1\,000}{V_m} \quad \text{(C.15)}$$

where

$m_d$ is corrected total mass, in micrograms, of analyte (DNPH derivative) extracted from the cartridge;

$V_m$ is the total air sample volume, in litres, under sampling conditions;

$M_c$ is the molar mass of carbonyl compound (for formaldehyde = 30 g/mol);

$M_{der}$ is the molar mass of the DNPH derivative (for formaldehyde = 210 g/mol).

NOTE The volume of the sampled air should be corrected to a standard temperature of 25 °C (77 °F) and a standard air pressure of 1 013 hPa.

### C.4.8.3 Process blanks

At least one blank shall be analysed with each new batch of cartridges.

#### C.4.8.4 Sample desorption

Connect the sample cartridge (inlet or short end during sampling) to a clean syringe.

The sampling and extraction shall be performed according to the cartridge manufacturer's recommendation.

Desorb the DNPH derivatives of the carbonyls and the unreacted DNPH from the cartridge (gravity feed) by passing e.g. 5 ml of acetonitrile (C.4.4.2) from the syringe through the cartridge to a graduated test tube or to a 5 ml volumetric flask. Other volumes of acetonitrile may be appropriate, depending on the sampling cartridge used.

Make up to the 5 ml mark with acetonitrile. Pipette an aliquot into a sample vial with a PTFE-lined septum. Analyze the aliquot for the carbonyl derivatives by HPLC. As a backup, a second aliquot may be taken and stored under refrigeration until the results of the analysis of the first aliquot are complete and validated. The second aliquot can be used for confirmatory analysis, if necessary.

It shall be checked whether residual DNPH peak is retained in the cartridge. Otherwise all DNPH was used for reaction and no DNPH is remaining, this means the derivation of formaldehyde is probably not complete. In this case a re-sampling should be conducted.

### C.4.9 Precision and uncertainty

As is the case for other compounds, the precision and uncertainty of the determination of formaldehyde in air is influenced by two parameters, the reproducibility of the analytical procedure and the variation over time of the analyte concentration in the air.

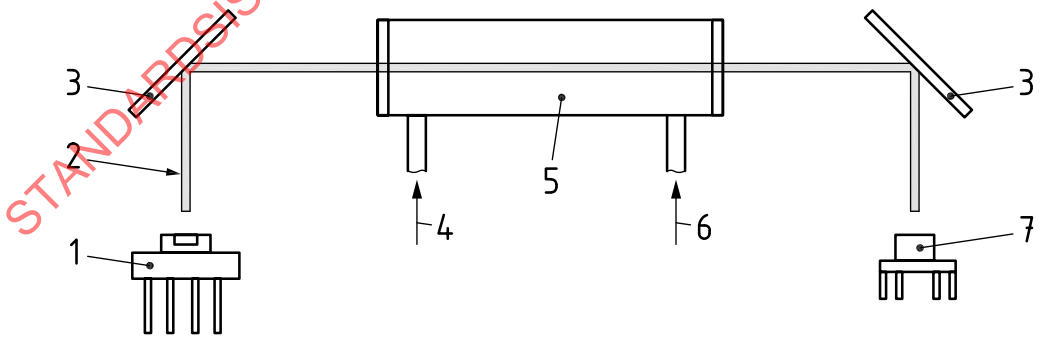
Analytical precision should be ±10 % or less for analyte concentrations of 1 µg/ml or greater. At the 0,5 µg/ml level and below, precision of replicate analysis could vary up to 25 % for some carbonyl compounds.

NOTE Additional information concerning round-robin tests are given in ISO 16000-3.

## C.5 Laser absorption spectroscopy (LAS)

### C.5.1 Measurement principle

A sketch of the measuring principle is shown in Figure C.6. Monochromatic infrared laser light gets generated in a semiconductor diode and passed through an optical cell, which is purged with the formaldehyde containing sample gas. Due to optical absorption by the formaldehyde gas the intensity of the light beam gets attenuated. The change in light intensity gets detected by a photoelectric sensor and can be recalculated to sample gas concentrations, as the absorbance obeys the Lambert-Beer law.




**Key**

1 laser diode
2 laser beam
3 mirror
4 gas outlet

5 optical cell
6 gas inlet
7 detector

**Figure C.6 — Working principle of the laser absorption formaldehyde analyser**

### C.5.2 Hazards

Operation of the LAS analyser has to be according to the operating manual. Do not open the device during operation, high energetic infrared radiation can cause harm to the eye.

Handling the chemicals used in the calibration process involves additional risks. Please refer to the hazard information in the corresponding sections.

### C.5.3 Equipment for chemical analysis

For the real-time measurement of the formaldehyde concentration in the chamber air, an extractive infrared laser absorption analyser should be used. The LAS analyser shall have a measurement range of 0 mg/m$^3$ to (at least) 6,15 mg/m$^3$ (5 ppm) and a limit of detection (LOD) lower than 0,0025 mg/m$^3$ (2 ppb). The precision should not exceed 1 % of the measured value (or the LOD, higher value) and the accuracy 2 % of the measured value (or the LOD, higher value).

For the calibration additional equipment is required, which is described in the corresponding sections in C.2 to C.4.

### C.5.4 Reagents

No reagents are required for the measurement itself. For the calibration water and reagents of analytical purity are used, as described in C.2 to C.4.

### C.5.5 Calibration

#### C.5.5.1 General

It is recommended to perform a calibration at least once every month or whenever the experimental setup is modified. The analyzer can either be calibrated utilizing standardized calibration gases or by referencing against the analytical standard procedures described in C.2 to C.4. Referencing is done against one set of wood-based panels, showing an emission level, in the regime of the test specimen to be investigated. The wet-chemistry sampling is done according to the procedure described in 7.2.1.

Pure nitrogen is used to set the zero level of the formaldehyde analyser. The purity should not be less than 99,999 %. The standard should be 100 % to 150 % of the anticipated production level emissions. This value could be derived from an established Quality Control Limit (QCL).

#### C.5.5.2 Sample based calibration procedure

**C.5.5.2.1** Prepare a sample (set) as described in 8.

**C.5.5.2.2** Insert the sample set into the test chamber for conditioning for at least 15 min. 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 calibration at the specified *Q*/*A* ratio and record this ratio in the report.

**C.5.5.2.3** The wet-chemistry sampling is done according to the procedure described in 7.2.1.

**C.5.5.2.4** For analytical quantification of the absorbed formaldehyde follow the procedures described in C.2 to C.4.

**C.5.5.3** Calculation of the calibration factor

The calibration factor of the LAS analyser is calculated by relating the spectroscopically determined formaldehyde concentration with the concentration determined by wet-chemistry (in mg/m$^3$ or ppm). This is achieved by following the steps below (Formula (C.16) to Formula (C.20)).

The average formaldehyde concentration in the air sample measured via laser absorption spectroscopy (LAS) is determined by Formula (C.16):

$$c_{ø(LAS)} = \frac{1}{n}\sum_{i=1}^{n} c_{(LAS),i} \quad \text{(C.16)}$$

where

$c_{ø(LAS)}$ is the average formaldehyde concentration in the calibration gas stream, measured by the LAS analyser (mg/m$^3$ or ppm);

$c_{(LAS),i}$ is the formaldehyde concentration, measured by the LAS analyser (mg/m$^3$ or ppm);

$n$ is the number of collected data points.

The average formaldehyde concentration in the sampled air, measured by photometry, is determined by Formula (C.17):

$$c_{ø(\text{Photo})} = \frac{c_{(\text{Photo})} \cdot V \cdot R \cdot T}{0,1 \cdot \dot{V} \cdot t \cdot M \cdot p} \quad \text{(C.17)}$$

where

$c_{ø(\text{Photo})}$ is the average formaldehyde concentration in the sampled air, measured in the absorber solution by photometry (mg/m$^3$ or ppm);

$c_{(\text{Photo})}$ is the formaldehyde concentration in the adsorber solution, measured by photometry (mg/m$^3$);

$V$ is the total volume of the absorber solution (m$^3$);

$R$ is the molar gas constant (8,314 J mol$^{-1}$ K$^{-1}$);

$T$ is the sample gas temperature (K);

$\dot{V}$ is the air flow of the calibration gas sampling (m$^3$/min);

$t$ is the sampling time in minutes (min);

$M$ is the molar mass of formaldehyde (30,031 g/mol);

$p$ is the pressure (mbar).

For standard test conditions ($p$ = 1 013,25 mbar, $T$ = 298,15 K) Formula (C.17) can be simplified to Formula (C.18):

$$c_{ø(\text{Photo})} = 0,815 \cdot \frac{c_{(\text{Photo})} \cdot V}{\dot{V} \cdot t} \quad \text{(C.18)}$$

The calibration factor (f) of the LAS analyser is determined by Formula (C.19):

$$\text{f} = \frac{c_{ø(\text{Photo})}}{c_{ø(\text{LAS})}} \quad \text{(C.19)}$$

The measured concentration of the LAS analyser should subsequently be corrected, according to Formula (C.20):

$$c_{\text{LAS(corrected)}} = \text{f} \cdot c_{\text{LAS(uncorrected)}} \quad \text{(C.20)}$$

NOTE It is recommended to have the LAS analyser calibrated if the overall readjustment of the measured concentration is exceeding 10 %.

Maintenance and calibration should be carried out according to the laser manufacturer recommendations.

### C.5.6 Sampling

The exhaust flow (that is, chamber outlet) is normally used as the sampling point, although separate sampling ports in the chamber can be used. Gas carrying parts (i.e. tubing) should be constructed of a material that minimizes absorption (for example steel, PTFE). The sampling is done at room temperature. Heating of connecting tubing and gas-carrying analyser components can be useful to prevent adsorption and condensation. The applied sampling flow may be chosen freely, however, it needs to be smaller than the air exchange flow of the test chamber. A typical air sampling flow is 0,5 l/min.

### C.5.7 Test duration

The sampling procedure is a minimum of 15 min due to repeated sampling or determinations.

NOTE Sampling time can be elongated if adsorptive effects are observed.

### C.5.8 Calculation of the formaldehyde emission

The formaldehyde emission from the wood-based panels being tested are expressed as the concentration in the air of the test chamber sampled at the chamber exhaust. The test result is the mean value of the recorded concentration values (min. 3) of the last sample run minute.

### C.5.9 Performance characteristic

Performance characteristics are strongly influenced by the individual design (i.e. analytical wavelength and quality of optical compounds) of the LAS analyser. For a system fulfilling the requirements described in C.5.3 the detection limits ($C_{\text{LD}}$) are typically below 0,002 5 mg/m$^3$ (<2 ppb) and the repeatability standard deviation is 2,5 % (0,001 25 mg/m$^3$) at formaldehyde concentration of 0,05 mg/m$^3$.

### C.5.10 Interferences

Measurements based on laser infrared absorption spectroscopy are highly sensitive to formaldehyde, however, there can be cross-interferences with other organic volatile organic compounds (VOCs) which need to be considered. Typical VOCs e.g. terpenes ($\alpha$-pinen, $\beta$-pinen, carene), aldehydes (benzaldehyde, decanal, furfural), carboxylic acids (acetic acid, formic acid) and other VOCs may originate from the wood or the resin system.

### C.5.11 Equivalence

The LAS analyzer needs to show equivalent test results for formaldehyde determination to the reference wet-chemistry method as described in 9.4.2.2 and 9.4.2.3 and no significant cross-interferences with other organic VOCs.

## C.6 Chemical sensor

### C.6.1 Measurement principle

Electrochemical sensing of formaldehyde is accomplished via oxidation reaction of formaldehyde adsorbed onto a precious metal working anode in the presence of an electrolyte in a potentiostatic circuit. Equal and opposite reduction occurs on the reference/counter cathode. The resultant change in current is proportional to the gaseous formaldehyde concentration.

The proposed electrochemical reaction of formaldehyde occurs as follows (see Figures C.7 to C.8):

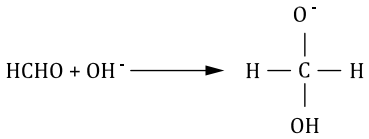



**Figure C.7 — Reaction scheme of formaldehyde oxidation to a geminal-diol anion**

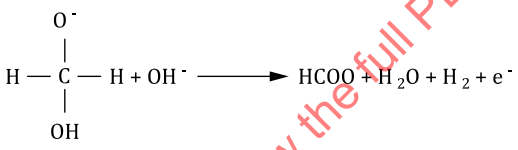



**Figure C.8 — Reaction scheme of resultant anion reacts with a hydroxyl ion at the working electrode to form formate, water, and hydrogen ion (see Figure C.7)**

With sufficient diameter working electrode surface area the lower detection limit of formaldehyde is $3{,}0 \times 10^{-7}$ M. See construction of a sensor:

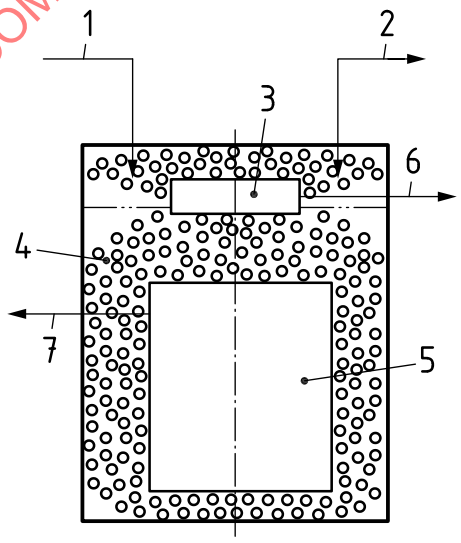



**Figure C.9 — Schematic diagram of the sensor**

In a potentiostatic circuit, the electrical potential across electrodes (commonly called bias voltage) is maintained by external electronics. The working electrode, electrolyte, and counter electrode create an

electrical path. The current flowing through this path is modulated by oxidation of formaldehyde at the working electrode according to the above reaction (see Figures C.7 and C.8).

### C.6.2 Hazards

Operation of the electro chemical sensor shall be according to the operating manual. Do not open the sensor. Handling the chemicals used in the calibration process involves additional risks. Please refer to the hazard information in the corresponding sections.

Sensors of this nature rarely have user-serviceable parts. Users should not disassemble the sensors and instead contact the manufacturer if issues arise.

Electrochemical sensors contain strong alkali and electrodes may contain material which is dangerous to health. Refer to the safety data sheet and contact the manufacturer for recycling and disposal.

### C.6.3 Equipment for chemical analysis

For the real-time measurement of the formaldehyde concentration in the chamber air an electrochemical sensor should be used.

Electrolyte replenishment is required at regular intervals to avoid crystallization of electrolyte or asymmetric concentration gradients due to evaporative losses. Replenishment should occur daily after use with deionized water (unless otherwise specified) to a manufacturer-specified concentration.

When not in use, this type of electrochemical sensor should be sealed from external environment and connected to the specified bias voltage.

Sensors may regain responsiveness while not in operation.

### C.6.4 Reagents

No reagents are required for the measurement itself. For the calibration water and reagents of analytical purity are used, as described in C.2 to C.4.

### C.6.5 Calibration

#### C.6.5.1 General

The electrochemical sensor shall be calibrated daily and every four hours of continuous operation using the working standard. The calibration shall result in an acceptable signal strength value from the sensor; as determined by the manufacturer. Adjustments to the calibration may be made based on this signal strength value.

#### C.6.5.2 Sample based calibration procedure

**C.6.5.2.1** Prepare a sample (set) as described in Clause 8.

**C.6.5.2.2** Insert the sample set into the test chamber for conditioning for at least 15 min. 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 calibration at the specified $Q/A$ ratio and record this ratio in the report.

**C.6.5.2.3** Ensure test chamber is on, at steady-state and in nominal conditions. If the working standard was conditioned outside of the test chamber insert the sample working standard set into the test chamber for conditioning for at least 15 min.

**C.6.5.2.4** The wet-chemistry sampling is done according to the procedure described in 7.2.1.

**C.6.5.2.5** For analytical quantification of the absorbed formaldehyde follow the procedures described in C.2 to C.4.

### C.6.5.3 Calculation of the calibration factor

The calibration factor of the sensor is calculated by relating the electrochemically determined formaldehyde concentration with the concentration determined by wet-chemistry (in $mg/m^3$ or ppm).

## C.6.6 Sampling

The exhaust flow (that is, chamber outlet) is normally used as the sampling point, although separate sampling ports in the chamber can be used. Gas carrying parts (i.e. tubing) should be constructed of a material to minimize absorption (for example, steel, PTFE). The sampling is done at room temperature. Heating of connecting tubing and gas-carrying analyser components may be useful to prevent adsorption and condensation. The applied sampling flow can be chosen freely, however, it needs to be smaller than the air exchange flow of the test chamber. A typical air sampling flow is 0,5 l/min to 1 l/min.

## C.6.7 Test duration

The sampling procedure is minimum 15 min.

NOTE Sampling time can be elongated if adsorptive effects are observed or if the response from the sensor is not stable.

## C.6.8 Calculation of the formaldehyde emission

The formaldehyde emission from the wood-based panels being tested is expressed as the concentration in the air of the test chamber sampled at the chamber exhaust at the end of the 15 min sampling period. The test result is the mean value of the recorded concentration values (minimum 5) of the last sample run minute.

## C.6.9 Performance characteristic

Performance characteristics are strongly influenced by the individual sensor design. For a system fulfilling the requirements described in C.6.3 The sensor shall have a measurement range of 0 to (at least) 2,46 $mg/m^3$ (2,0 ppm) and a limit of detection (LOD) lower than 0,0246 $mg/m^3$ (20 ppb). The precision should not exceed 5 % of the measured value (or the LOD, higher value) and the accuracy 10 % of the measured value (or the LOD, higher value).

The durability of the sensor is approximately >200 h discontinuous operation.

The sensor is calibrated at standard conditions (18 °C to 30 °C/64 °F to 86 °F, 0,95 bar to 1,05 bar).

Calibration of the sensor should be assessed for signal strength/responsiveness, consistency, and drift. When sensors lose 60 % or more of their signal strength (ratio of $mA/mg/m^3$ response), or respond too slowly (< 90 % of maximum signal at 600 seconds), the sensor should be replaced.

Drift, or the change in sensor response over time, may occur during normal operation. Drift may be caused by a gradual increase in zero or a decrease in responsiveness.

Sensor drift should be monitored for compensation or correction. Significant drift (>5 % signal loss since last calibration) necessitates recalibration or replacement.

## C.6.10 Interferences

Chemicals that have demonstrated interference for this detection technique include acetaldehyde, acetone and methylethylketone. Care shall be used when developing analysis methods to account for any possible chemical interference.

### C.6.11 Equivalence

The chemical sensor needs to show equivalent test results for formaldehyde determination to the reference wet-chemistry method as described in 9.4.2.2 and 9.4.2.3. and no significant interferences with other chemical compounds (see C.6.10).

# Annex D
(informative)

# Establishing of a correlation

## D.1 General

According to the legal or commercial requirements, wood-based materials shall meet the formaldehyde emission requirements before being placed on the market or sold. In most cases, the legally published requirements are based on chamber methods. To ensure conformity, material tests are continuously carried out in the manufacturer's own laboratory using derived test methods. Material tests carried out according to derived test methods shall correlate with the reference method (chamber method) so that a so-called product-related limit value can be calculated for the derived method used and in turn compliance with the reference method.

## D.2 Terms and definitions

**D.2.1**
**correlation**
describes a relationship between two or more characteristics, states or functions.

**D.2.1.1**
**linear regression**
used to predict the relationship between two variables by applying a linear equation to observed data

**D.2.1.2**
**cluster**
similar test results leading to a grouping (cluster) of data pairs

**D.2.1.3**
**cluster approach**
approach using a data pair added to the data cluster as an additional data point or as an anchor point (e.g. blank value of test methods) to establish a linear regression achieving a minimum statistical requirement (e.g. 'r'-value)

**D.2.1.4**
**threshold approach**
preliminary threshold value on the basis of average value of reported factory production control (FPC) test results that is used if the linear regression derived by the cluster approach does not meet the minimum statistical requirements

**D.2.1.5**
**'r'-value**
pearson's 'r'-value, also known as Pearson's Correlation Coefficient is the measure of the linear correlation between in two variables

**D.2.2**
**Limit values**

**D.2.2.1**
**quality control limit**
**QCL**
means the value from the quality control method test that is the correlative equivalent to the applicable emission standard based on the reference chamber method The QCL is the absolute maximum class limit for the derived test method that corresponds to a concentration of the maximum required formaldehyde value of reference chamber method.

**D.2.2.2**
**excursion limit value**
quality control level (QCL) that shall not be exceeded by a certified lot. This limit value is designed to that when 95 % of all tests fall below QCL, 99,9 % of all values will be below the excursion limit

**D.2.2.3**
**Target Operating Level**
**TOL**
to reasonably assure that at least 95 % of the factory production values (FPC) are at or below the QCL, the average value of FPC should be at the QCL minus a safety factor derived by the manufacturer based on the knowledge of their process

**D.2.2.4**
**shipping quality control limit value**
**SQCL**
based upon the correlation between the FPC test method of a product ready for shipment and the reference test method. Depending on the shipment data fixed by the manufacturer it can be differ or be the same as the QCL

**D.2.3**
**Test methods**

**D.2.3.1**
**reference test method**
procedure for directly measuring the formaldehyde emission of a product under the exact conditions (chamber method) for which the respective limit value has been set.

**D.2.3.2**
**derived test method as factory production control method**
**FPC**
test method used to develop the correlation to the reference test method (chamber) and calculate the QCL

**D.2.4**
**product**
type of composite wood product defined by the manufacturer or a regulation or any other external requirements.

Note 1 to entry: A composite wood product can be defined based on e.g. production line, product recipe, resin system, thickness range.

## D.3 Sampling and sample preparation

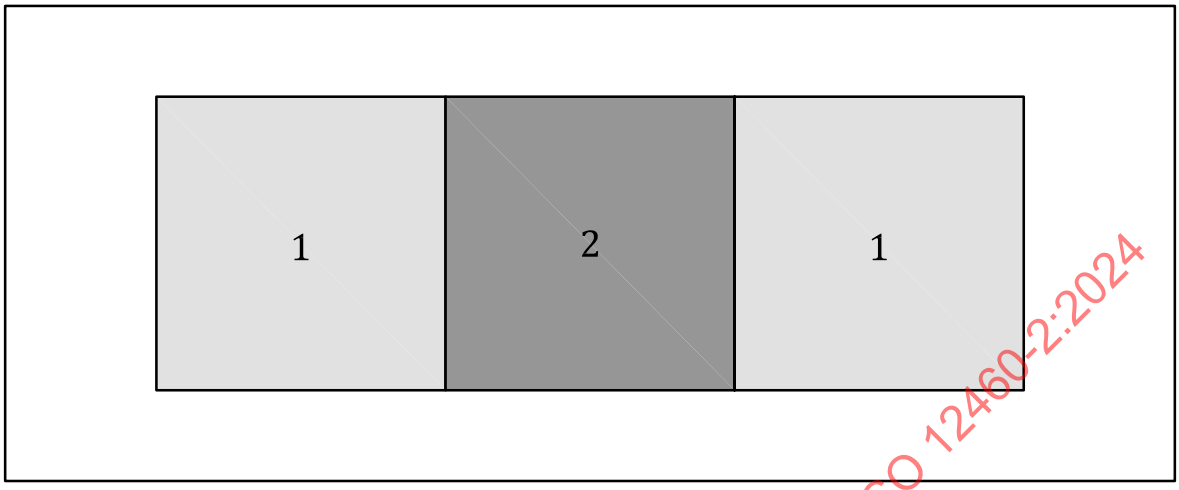
### D.3.1 General

The paired test pieces shall be sampled according to the principles of side matched for comparison between the reference test method and the derived test method.

All sample preparation (e.g. sealing, conditioning etc.) should be carried out in accordance with the chosen test method's requirements.

### D.3.2 Side matching

Sampling of one or more test pieces from the same board, for the derived test method(s), that are directly next to, or as near as practical to the reference test method test piece (see Figure D.1). The test pieces shall be taken preferably at least 250 mm from the edges.




**Key**

1 sample derived test method

2 sample reference test method

**Figure D.1 — Sample cutting principle**



### D.3.3 Product test data

For the purpose of establishing a QCL for a product, only the paired test results shall be used.

### D.3.4 Factory production control procedures

Whenever the formaldehyde content and/or the formaldehyde emission of a wood-based panel is determined shortly after its production (also referred to as “hot testing”), the effects of post-curing, aging and/or off-gassing on emission characteristics shall be considered.

Therefore, clear documented quality control procedures for the timing, strategy and processing of samples in routine FPC testing should be established to ensure consistency of measurement with that carried out during the whole procedure of testing.

## D.4 Test methods

### D.4.1 Reference chamber test method

The test according to reference chamber method can be performed by the manufacturer’s laboratory or by an external laboratory if the manufacturer wishes or as required by regulatory requirements.

In the case of a establishing a correlation, the formaldehyde test results should be rounded to 3 decimal places.

### D.4.2 Derived test methods

For FPC purposes different derived test methods or alternative documented methods may be used depending, among other factors, on the type of the product. Table D.1 provides guidance for the selection a derived test method.

The derived test method should preferably be tested in the factory and not at an outside laboratory to reduce the influence of external variability into the assessment.

Guidance on the application of test methods is provided in Table D.1 based on the current state of the art. A manufacturer may use a combination of test method for specific board types not listed in Table D.1.

**Table D.1 — Guidance for the use of derived test methods for different wood-based panels are given in this table as examples**

| Method | Standard | Type(s) of wood-based panels | Examples of wood-based panels |
|---|---|---|---|
| Chamber | ISO 12460-1 | all kinds of wood-based panels, raw or otherwise covered | |
| Chamber | ISO 12460-2 | all kinds of wood-based panels, raw or otherwise covered | |
| Gas analysis | ISO 12460-3 | all kinds of wood-based panels, raw or otherwise covered | particleboard, fibreboard, OSB, flaxboard, plywood, solid wood panels, LVL, all types of melamine faced boards |
| Desiccator | ISO 12460-4 | raw or otherwise covered boards | particleboard, MDF, Plywood |
| Perforator | ISO 12460-5 | raw particle and fibre based boards | particleboard, fibreboard, OSB, flaxboard |
| Chamber | ASTM D 6007 | raw particle, fibre based boards and raw laminar composites | particleboard, MDF, plywood |
| DMC | DMC Manual | raw particle, fibre based boards and raw laminar composites | particleboard, MDF, plywood |
| Flask | EN 717-3 | all kinds of wood-based panels | |

## D.5 Establishing the correlation

### D.5.1 General context

Correlation is the degree to which two variables are linearly related. This is an important step in bi-variate data analysis. The correlation coefficient is a statistical measure of the strength of the relationship between the relative movements of two variables.

### D.5.2 Selection of data

There are different cases for data selection considering the initial correlation, data of surveillance procedure and so called 'special cases' resulting in additional testing.

The **initial correlation** of a product requires at least 5 data pairs for a product group up to 12 data pairs are to be used.

The **continuous surveillance** is supplemented by a check of the QCL value in a period of 3 years a new measured value is available for each product of a product group. The check of the limit value is done depending on the number of monitoring periods per year.

**Special cases** are the extension of the product scope, and especially the product change as well as voluntary additional testing (e.g. thickness range extension, changes of glue or glue supplier).

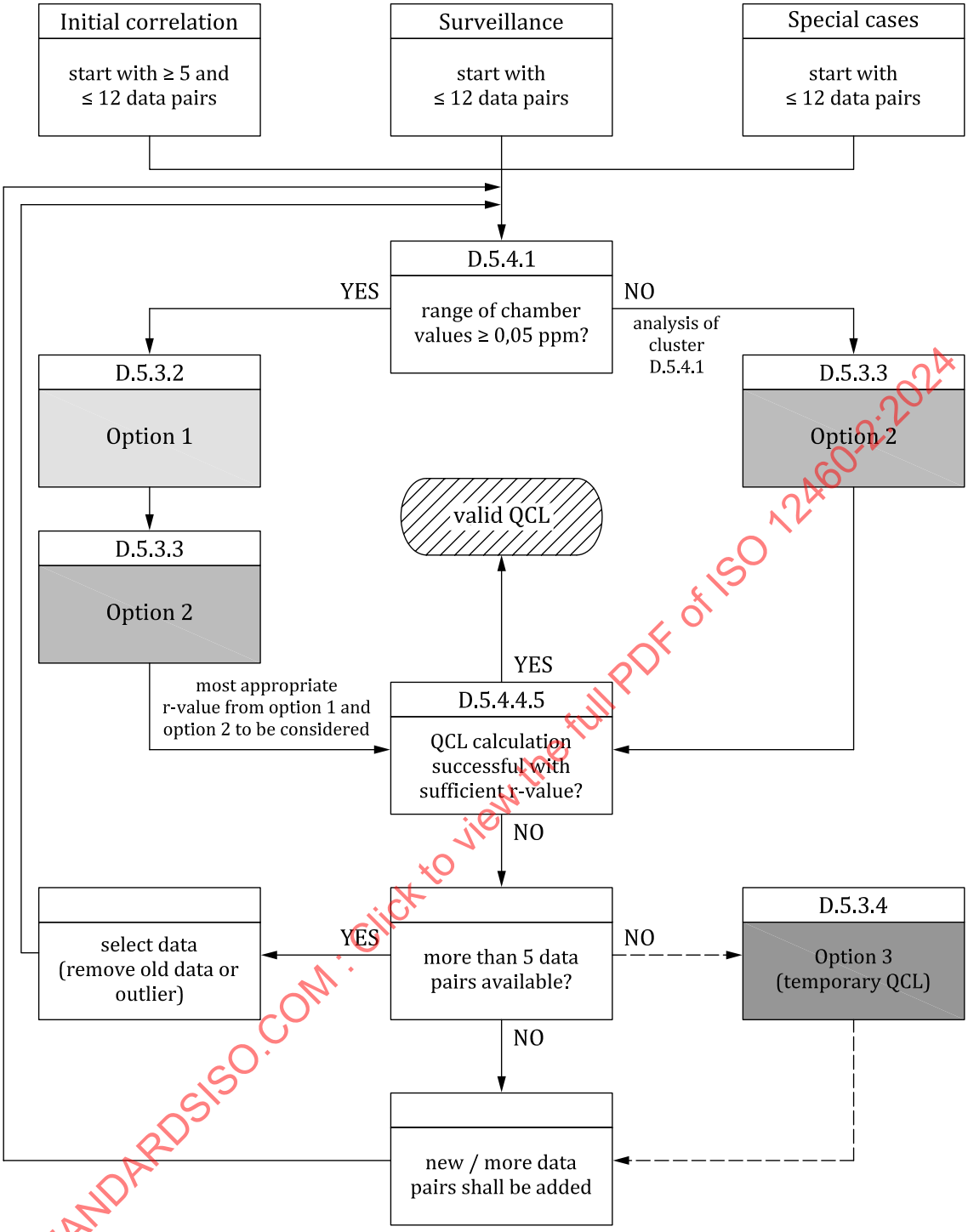



**Figure D.2 — Flow diagram of data selection and options for establishing a correlation**



## D.5.3 Options to establish a correlation

### D.5.3.1 General

The correlation is the means to predict the manufacturer´s value against the reference standard value.

The process to determine the linear regression as exclusively bivariate analysis proceeds uses of raw data (option 1), data augmentation (option 2.1) and fixed origin for the estimator function (option 2.2). The process is understood to be an escalation if the simple regression is not leading to a sufficient result. Thus, the raw data is manipulated and greater requirements are being added to the regression.