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EN 717-3

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Descriptors: Wood products, wooden boards, determination, emission, formaldehyde, analysis methods

English version

Wood-based panels — Determination of formaldehyde release — Part 3: Formaldehyde release by the flask method

Panneaux à base de bois — Détermination du dégagement de formaldéhyde —
Partie 3: Dégagement de formaldéhyde par la méthode au bocal

Holzwerkstoffe — Bestimmung der Formaldehydabgabe —
Teil 3: Formaldehydabgabe nach der Flaschen-Methode

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 112, Wood-based panels, the Secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 1996, and conflicting national standards shall be withdrawn at the latest by September 1996.

This standard is one of a series which specifies methods for determining formaldehyde potential in or formaldehyde release from wood-based panels.

The other standards in this series are:

EN 120	*Wood-based panels — Determination of formaldehyde content — Extraction method called the perforator method*
EN 717-1	*Wood-based panels — Determination of formaldehyde release — Part 1: Formaldehyde release by the chamber method*[1)]
EN 717-2	*Wood-based panels — Determination of formaldehyde release — Part 2: Formaldehyde release by the gas analysis method*

No existing European Standard is superseded.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Contents

[1)] At present at the draft stage

Introduction

The principle of the flask method for measuring the formaldehyde release has been published by Wilhelm-Klauditz-Institute (Roffael 1975). Thereafter, the method has been modified in different ways by many authors in several countries. After consideration of published work focusing on the method and its reliability, a temperature of 40 °C and a period of 3 h have been selected for this standard.

1 Scope

This European Standard specifies a method, known as the flask method, for determination of formaldehyde release from uncoated wood-based panels.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard, only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 322 *Wood-based panels — Determination of moisture content*

EN 323 *Wood-based panels — Determination of density*

EN 326-1 *Wood-based panels — Sampling, cutting and inspection — Part 1: Sampling and cutting of test pieces and expression of test results*

3 Principle

Formaldehyde release is determined by suspending test pieces of known mass over water in a closed container, maintained at a constant temperature. Formaldehyde released from the test pieces during a defined period of time is absorbed by the water. The formaldehyde content of the water is determined photometrically by the acetylacetone method, and the result is expressed in milligrams formaldehyde per kilogram of dry board.

4 Reagents

4.1 General

Reagents of recognized analytical purity and distilled or demineralized water (referred to below as distilled water) shall be used for the analysis.

4.2 *Acetylacetone*, of analytical grade.

4.3 *Ammonium acetate*, of analytical grade.

NOTE: Commercially prepared reagents may be used, provided it can be shown that they give an equivalent result.

5 Apparatus

5.1 Test apparatus

Test apparatus consisting of the following main components:

5.1.1 Polypropylene, or polyethylene flask-container of type 1 or 2 *(see figures 1 and 2)*, of 500 ml volume with tightly fitting lid of the same material.

5.1.2 *Metal test piece holder or rubber band and hook.* Metal parts shall be of stainless steel (see figures 1 and 2).

5.2 Laboratory equipment

5.2.1 *Waterbath*, capable of maintaining a temperature of (40 ± 1) °C.

5.2.2 *Cells*, with a suitable path length for the spectrophotometer.

5.2.3 *Volumetric flasks 1000 ml* (calibrated at 20 °C).

5.2.4 *Volumetric flasks 100 ml* (calibrated at 20 °C).

5.2.5 *Flasks 50 ml* (with stoppers).

5.2.6 *Bulb pipettes* (calibrated at 20 °C), 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml, 100 ml.

5.2.7 *Erlenmeyer flask 250 ml.*

5.2.8 *Microburette.*

5.2.9 *Burette 50 ml*, graduated in 0,05 ml (calibrated at 20 °C).

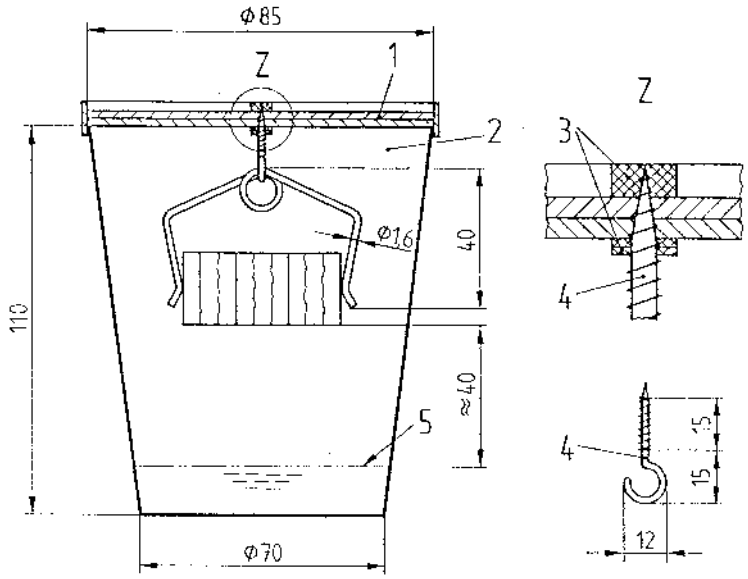
5.2.10 *Stop watch.*

5.2.11 *Balance*, scale interval 0,001 g.

5.2.12 *Ventilated drying oven*, capable of maintaining a temperature of (103 ± 2) °C.

5.2.13 *Ventilated oven* (maximum volume: 60 l), capable of maintaining a temperature of (40 ± 1) °C at any position in the oven.

5.2.14 *Spectrophotometer*, capable of measuring absorbance at 412 nm.

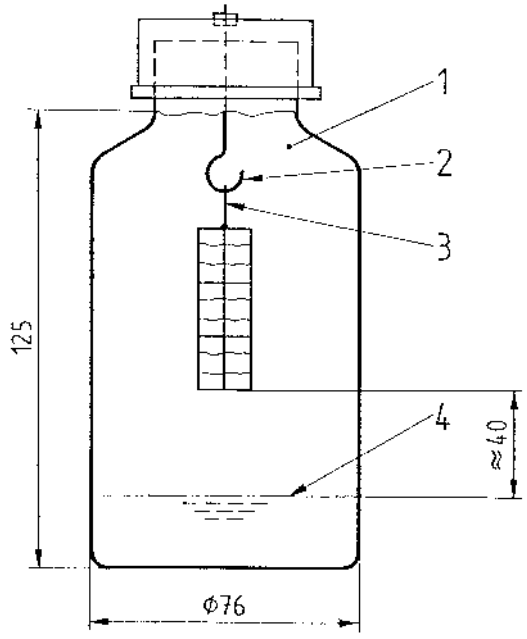


1 Lid
2 500 ml container
3 Sealing joints
4 Hook with thread diameter of 3 mm to 4 mm
5 Surface of water

Dimensions in millimetres

NOTE. The dimensions of the clip and of the hook are only examples

Figure 1. Test apparatus type 1 for the flask method



1 500 ml polyethylene bottle with bottle top
2 Hook out of stainless steel
3 Elastic rubber band
4 Surface of water

Dimensions in millimetres

Figure 2. Test apparatus type 2 for the flask method

6 Test pieces

6.1 Sampling

Test pieces of 25 mm × 25 mm × board thickness shall be taken according to EN 326-1, evenly distributed over the width of the (cooled) board, but excluding a 500 mm wide strip at either end of the board.

6.2 Test pieces for the determination of the moisture content

Take 12 test pieces for the determination of the moisture content.

6.3 Test pieces for the determination of the flask value

Take a sufficient number of test pieces to obtain approximately 100 g of board for determining the flask value (F_v). Determine the mass of the test pieces to the nearest 0,01 g. The number of test pieces for each set has to be chosen so that their total mass is as near as possible to 20 g. In case of doubt the total mass should be preferably below 20 g.

The test pieces shall be cut immediately after the board has been cooled. After cutting, the test pieces shall be stored, hermetically sealed, at room temperature. For comparative reasons the time elapsing between cutting and testing should be kept as constant as possible, but shall not exceed 72 h.

7 Procedure

7.1 Determination of moisture content

Determine the moisture content in accordance with EN 322.

Determine the moisture content in duplicate on two samples of at least four test pieces.

7.2 Determination of formaldehyde release

Determinations shall always be made on duplicate sets of test pieces. The individual values shall only differ from each other by a maximum of 20 % related to the higher of two single values. Otherwise a third determination shall be carried out.

Fix the test pieces face to face in the clip and attach the clip to the lid of the container, using the hook as shown in figure 1 or fix the test pieces with a rubber band as shown in figure 2. Add 50 ml of distilled water at 20 °C to the container using a bulb pipette, attach the lid with the suspended test pieces and close the container so that it is completely airtight. The bottom surfaces of the test pieces should be approximately 40 mm above the surface of the water. A second container shall be prepared in the same way.

Insert the closed containers into the oven (see **5.2.13**) at a temperature of (40 ± 1) °C. This temperature shall be maintained throughout the whole test period. The containers shall occupy less than 10 % of the whole volume of the oven, that means not more than one container per 6 l of oven volume in order to avoid fluctuation in the temperature. The containers shall be placed staggered and with a distance of at least 50 mm between each other. After (180 ± 1) min remove the containers from the oven and immediately take off the lids with the test pieces attached. Transfer the solution from the containers to each of the two 50 ml flasks, close them tightly and allow the contents to cool at ambient temperature to approximately 20 °C.

7.3 Determination of formaldehyde concentration of the aqueous solution

7.3.1 *General*

The formaldehyde content of the aqueous solution shall be determined photometrically.

7.3.2 *Principle*

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

NOTE: Other suitable photometric procedures may also be used.

7.3.3 *Reagents*

7.3.3.1 *Acetylacetone solution*

4 ml acetylacetone are added to a 1000 ml volumetric flask and made up to the mark with distilled water.

7.3.3.2 *Ammonium acetate solution*

200 g ammonium acetate are dissolved with distilled water in a 1000 ml volumetric flask and made up to the mark.

7.3.4 *Procedure*

10 ml are taken from the aqueous solution (see **7.2**) with a pipette (**5.2.6**) and added to 10 ml acetylacetone solution (**7.3.3.1**) and 10 ml ammonium acetate solution (**7.3.3.2**) in a 50 ml flask (**5.2.5**). The flask is stoppered, shaken and warmed for 15 min in a water bath (**5.2.1**) of (40 ± 1) °C. The now greenish-yellow solution is cooled to room temperature protected from light (about 1 h). The absorbance of this solution is determined at a wavelength of 412 nm against distilled water using a spectrophotometer (**5.2.14**). A blank test shall be made with distilled water, the blank value shall be taken into consideration in the determinations of the flask value (F_v).

7.3.5 *Calibration curve (see figure 3)*

7.3.5.1 *General*

A calibration curve is produced from a standard formaldehyde solution, the concentration of which has been determined by iodometric titration. The calibration curve shall be checked at least once a week.

7.3.5.2 *Formaldehyde standard solution*

Reagents:

– standard iodine solution c (I_2) = 0,05 mol/l;

– standard sodium thiosulfate solution c ($Na_2S_2O_3$) = 0,1 mol/l;

– standard sodium hydroxide solution c (NaOH) = 1 mol/l;

– standard sulfuric acid solution c (H_2SO_4) = 1 mol/l.

The above solutions shall be standardized before use.

– starch solution 1 % *m/m*.

Dilute about 2,5 g formaldehyde solution (concentration 35 % to 40 %) in a 1000 ml volumetric flask with distilled water and make up to the mark. Determine the exact formaldehyde concentration as follows:

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 thiosulfate 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 distilled water.

Calculate the formaldehyde content as follows:

$$c\,(\mathrm{HCHO}) = (V_o - V) \times 15 \times c(Na_2S_2O_3) \times \times 1000/20 \quad (1)$$

where:

c (HCHO) is the formaldehyde concentration, in milligrams per litre;

V is the volume of the thiosulfate titration solution, in millilitres;

V_o is the volume of thiosulfate titration for the blank, in millilitres;

c ($Na_2S_2O_3$) is the concentration of sodium thiosulfate solution, in moles per litre.

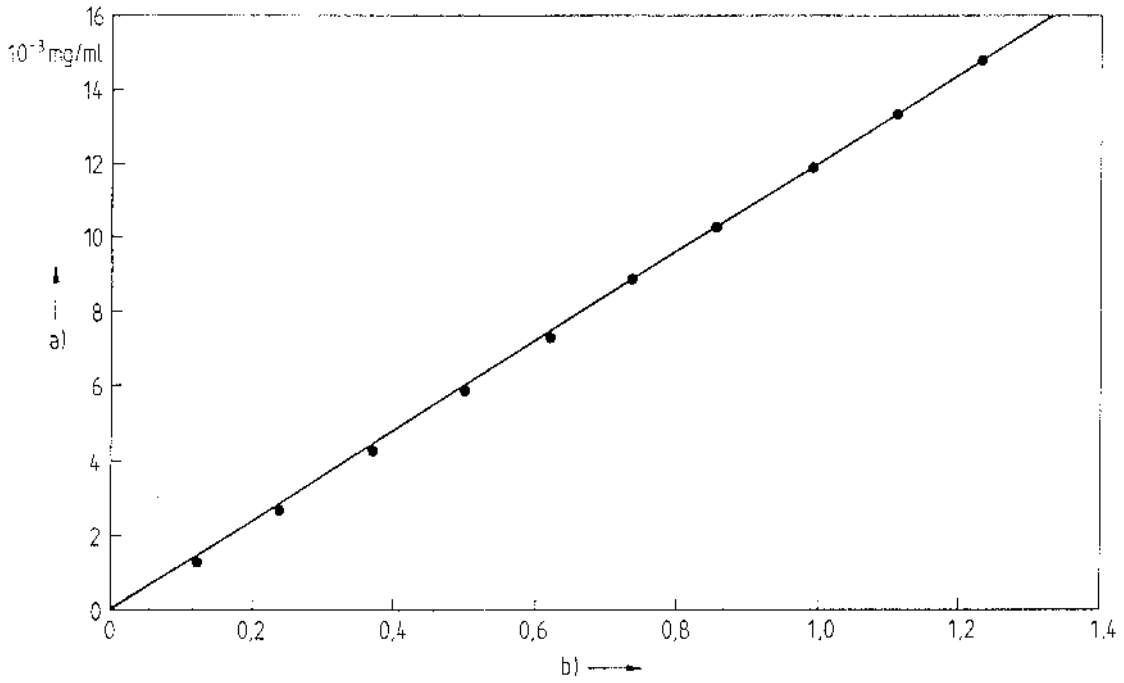
NOTE: 1 ml 0,1 mol/l thiosulfate solution corresponds to 1 ml 0,05 mol/l iodine solution and 1,5 mg formaldehyde.

7.3.5.3 *Formaldehyde calibration solution*

Using the concentration determined in **7.3.5.2**, calculate the volume of the formaldehyde solution which will contain 15 mg formaldehyde. Transfer this volume, using a microburette (**5.2.8**), to a 1000 ml volumetric flask (**5.2.3**) and make up to the mark with distilled water. 1 ml of this calibration solution contains 15 µg formaldehyde.

7.3.5.4 *Determination of the calibration curve*

Pipette either zero, 5, 10, 20, 50 or 100 ml of formaldehyde calibration solution (see **7.3.5.3**) into a 100 ml volumetric flask (**5.2.4**) and make up to the mark with distilled water. 10 ml of each dilution is analysed photometrically by the same procedure as described in (**7.3.4**). The absorbance values are plotted against the formaldehyde concentrations (c) (between 0 mg/ml and 0,015 mg/ml). The slope (f) of the graph is determined either graphically, or calculated (see figure 3).



$c = f \times (A_S - A_B)$

a) Concentration of the diluted calibration solution (c)

b) Absorbance ($A_S - A_B$)

Figure 3. Example of a calibration curve for formaldehyde determined by acetylacetone method (path length 10 mm)

8 Expression of results

8.1 Moisture content

The moisture content H shall be calculated according to EN 322.

8.2 Flask value (F_V)

The amount of formaldehyde absorbed in 10 ml of solution from the containers is determined spectrophotometrically as described under **7.3.4**.

The flask value (F_V) in milligrams per kilogram of oven-dry board is calculated by the following equation:

$$F_V = \frac{(A_S - A_B) \times f \times 50 \times 10\ (100 + H)}{m} \qquad (2)$$

where:

- A_S is the absorbance of the analysed solution from the containers;
- A_B is the absorbance of an analysis with distilled water;
- f is the slope of the calibration curve, in milligrams per millilitre;
- H is the moisture content of the test pieces, in percent;
- m is the mass of the test pieces, in grams.

9 Test report

The test report shall be issued in accordance with EN 326-1. Additionally, the test report shall include the following information:

- type of board;
- density of the board according to EN 323, in kilograms per cubic metre;
- information regarding age, finishing, sanding etc.;
- date of formaldehyde determination;
- moisture content, in percent, at the time of testing;
- flask value (F_V) in milligrams per kilogram expressed to 0,1 mg/kg oven-dry board (individual values of each determination and mean value);
- description of further details.

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