



SLOVENSKI STANDARD

oSIST prEN 120:2012

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**Lesne plošče - Določevanje prostega formaldehida - Ekstrakcijska metoda
(imenovana perforatorska metoda)**

Wood-based panels - Determination of formaldehyde release - Extraction method (called the perforator method)

Holzwerkstoffe - Bestimmung des Formaldehydgehaltes - Extraktions-verfahren (genannt Perforatormethode)

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Panneaux à base de bois - Détermination du dégagement de formaldéhyde - Méthode d'extraction (dite méthode au perforateur)

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ICS:

79.060.01	Lesne plošče na splošno	Wood-based panels in general
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EUROPEAN STANDARD
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DRAFT
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Will supersede EN 120:1992

English Version

Wood-based panels - Determination of formaldehyde release - Extraction method (called the perforator method)

Panneaux à base de bois - Détermination du dégagement
de formaldéhyde - Méthode d'extraction (dite méthode au
perforateur)

Holzwerkstoffe - Bestimmung des Formaldehydgehaltes -
Extraktions-verfahren (genannt Perforatormethode)

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 112.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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Foreword

This document (prEN 120:2011) has been prepared by Technical Committee CEN/TC 112 "Wood-based panels", the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 120:1992.

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

Compared to EN 120:1992 the following modifications have been made:

- a) in 5.3 recommendation of cell length for low emitting samples added;
- b) in 6.4 relative humidity for conditioning climate increased to (50 ± 5) %;
- c) in 7.1 and 8.2 procedure and evaluation of third extraction modified;
- d) in 7.3 accuracy of weighing of test pieces enhanced and higher mass of the test pieces added as an option for low emitting boards;
- e) in 7.4 precision of blank test improved;
- f) in 7.5.1 fluorimetric determination of formaldehyde added an option;
- g) in 7.5.4 temperature of water bath increased to 60 °C;
- h) in 8.2 modification factors for moisture content included;
- i) in Figure 4 calibration curve modified;
- j) Annex A for calibration and quality assurance added.

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Introduction

The 'perforator value' as determined by the method laid down in this standard is considered to be the 'formaldehyde content' of the tested board.

The test results shall be considered in relation to the specific board conditions at the time of testing.

The emission of formaldehyde from wood-based panels (e.g. particleboard, plywood, fibreboard) is a complex process. For a given board, the test result depends upon the age, ageing conditions, moisture content, etc. at the time of testing.

In addition, the correlation between the Perforator value and the formaldehyde emission of the board depends on the type of board.

1 Scope

This European Standard specifies an extraction method, known as the 'Perforator method'. It is used for the determination of the formaldehyde content of unlaminated and uncoated wood-based panels.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

<https://standards.iteh.ai/catalog/standards/sist/05e7588b-fad0-4882-bb88-3add7607938f/prEN-120-2012>

EN 326-1, *Wood-based panels — Sampling and cutting of test pieces*

3 Principle

The formaldehyde is extracted from test pieces by means of boiling toluene and then transferred into distilled or demineralized water. The formaldehyde content of this aqueous solution is determined photometrically by the acetylacetone method.

4 Reagents

For the analysis, only reagents of analytical quality and distilled or demineralized water shall be used.

4.1 Toluene, which is free from water and from impurities which may interfere with the test.

4.2 Acetylacetone, of analytical grade.

4.3 Ammonium acetate, of analytical grade.

4.4 Formaldehyde solution

Commercially available formaldehyde solution (concentration typically between 35 to 40 %).

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

5 Apparatus

5.1 Precision balance, with 0,001 g scale intervals.

5.2 Well-ventilated oven, capable of maintaining a temperature of $(103 \pm 2) ^\circ\text{C}$.

5.3 Spectrophotometer, capable of measuring absorbance at 412 nm. A cell of 50 mm path length is recommended, especially for low emitting samples.

5.4 Extraction apparatus

The apparatus consists of:

- spiral condenser, total length: approximately 400 mm, cone 45/40; socket 29/32 (item reference 2);
- conical adaptor, socket 45/40, cone 71/51 (item reference 3);
- filter insert, porosity P 160 (100 μm to 160 μm), bowl and filter diameters: 60 mm (item reference 4);
- perforator attachment, 1000 ml, with stopcock (4 mm bore), socket 71/51, cone 29/32 (item reference 5);
- conical adaptor, socket 29/32, cone 45/40 (item reference 8);
- 1000 ml round bottom flask, socket 45/40 (item reference 9);
- (double) bulbed tube, cone 29/32 (length: approximately 380 mm), external diameter: approximately 10 mm, bulb diameter: approximately 50 mm, distance between bulb and bottom end of tube: approximately 200 mm (distance between the bulbs approximately 50 mm) (item reference 6);
- absorption bulb (e.g. conical flask 250 ml) (item reference 7).

NOTE 1 The item references are given in Figure 1.

NOTE 2 Permanent PTFE seals should be used rather than paraffin or silicone grease sealants.

5.5 Laboratory equipment

- Volumetric flask, 2000 ml, calibrated at $20 ^\circ\text{C}$;
- Conical flask, 250 ml;
- Precision burette, 50 ml, calibrated at $20 ^\circ\text{C}$;
- Watch glass, with diameter of about 120 mm;
- Two volumetric flasks, 1000 ml, calibrated at $20 ^\circ\text{C}$;
- Six volumetric flasks, 100 ml, calibrated at $20 ^\circ\text{C}$;
- Volumetric pipette or automatical/digital pipette, 100 ml, calibrated at $20 ^\circ\text{C}$;
- Volumetric pipette or automatical/digital pipette, 25 ml, calibrated at $20 ^\circ\text{C}$;
- Volumetric pipettes (calibrated at $20 ^\circ\text{C}$), 1 ml, 2 ml, 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml, 100 ml;
- Three volumetric pipettes or automatical/digital pipettes, 10 ml, calibrated at $20 ^\circ\text{C}$;

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- Stopped flasks, each 50 ml;
- Two volumetric cylinders, 250 ml;
- Water bath;
- Desiccator.

6 Test pieces**6.1 General**

Sampling and cutting are done in accordance with EN 326-1.

6.2 Sampling

6.2.1 The test pieces are to be taken, evenly distributed, over the width of the (cooled) board, but excluding a 500 mm wide strip at either end of the board.

6.2.2 Take 12 test pieces of 25 mm x 25 mm x thickness of the board for the determination of the moisture content and a sufficient number of test pieces of the same dimensions to obtain approximately 500 g of the board for the extraction by perforator.

6.3 For production control

If this method is used for production control, the board selected for sampling is immediately cut up after cooling. The test pieces taken from the board are to be stored hermetically sealed at room temperature.

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

6.4 For other purposes

If this method is used for other purposes, e.g. for boards already installed, the method chosen for sampling, preparation of test pieces and conditioning, all which influence the final result, shall be agreed between the parties and indicated in the test report.

Unless otherwise agreed, the test pieces shall be conditioned to constant mass at a temperature of (23 ± 1) °C and a relative humidity of (50 ± 5) %.

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

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

7 Procedure**7.1 Number of extractions**

The extractions are to be carried out in duplicate.

NOTE For internal routine control, a single extraction may be sufficient.

The individual values of a duplicate extraction may not deviate by more than 0,5 mg/100 g; otherwise, a third extraction is to be carried out.

7.2 Determination of moisture content

Determine the moisture content in accordance with EN 322.

Determine the moisture content in duplicate on a sample of at least 4 test pieces (25 mm x 25 mm).

Weigh the sample (5.1) to an accuracy of 0,1 g onto the watch glass (5.5) and dry in the oven (5.2) at a temperature of (103 ± 2) °C to constant mass (about 12 h).

Constant mass is considered to have been reached when the results of two successive weightings, carried out at intervals of not less than 6 h, do not differ by more than 0,1 % of the mass of the test pieces.

After the removal from the drying oven, the test pieces shall be allowed to cool in a desiccator before weighing.

7.3 Extraction in the perforator

Before the apparatus is used, the sidearm of the perforator attachment shall be provided with thermal insulation, in order to achieve circulation of the toluene.

About 110 g of test pieces are weighed to 0,01 g and put into the round bottom flask (5.4). 600 ml of toluene (4.1) are added. About 1000 ml of distilled water is poured into the perforator attachment, leaving a space of 20 mm to 30 mm between the surface of the water and the siphon outlet. Subsequently, the round bottom flask is connected to the perforator. The condenser and the gas absorption equipment are then connected. The absorption bulb of the gas absorption equipment (5.4) is filled with about 100 ml of distilled water and connected to the apparatus.

NOTE For boards with a low formaldehyde content, the mass of the test pieces can be extended up to 200 g provided that the density of the samples enables such an extension.

When the apparatus has been assembled, the cooling water and heating are turned on.

Toluene shall flow back regularly throughout the whole period of perforation, with a reflux rate of 70 drops to 90 drops per min.

Care shall be taken that no water flows back from the absorption bulb (item reference 7) into other parts of the equipment during and after the extraction process.

The extraction is carried out for (120 ± 5) min, starting at the moment that the first bubbles pass through the filter insert. The heating shall be such that the onset of bubbling occurs between 20 min and 30 min after turning on the heating device.

After these 120 min the heating is switched off and the gas absorption bulb removed.

The water contained in the perforator is, after cooling to room temperature, transferred into the volumetric flask by means of the stopcock. The perforator is rinsed twice, each time with 200 ml of distilled water. The rinsing water is poured into the volumetric flask and the toluene discarded. The water contained in the absorption bulb of the gas absorption equipment is poured into the flask. The volume of the water contained in the flask is then made up to 2 000 ml with distilled water.

7.4 Blank test

Repeat the test without test pieces using new toluene from the same batch as used for the extraction.

The quality of toluene is checked by this test. If the blank value of formaldehyde is higher than 0,2 mg/600 ml of toluene, the toluene shall be rejected.

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7.5 Determination of formaldehyde in the extract**7.5.1 General**

The formaldehyde content of the aqueous extract is determined photometrically, using the acetylacetone method.

NOTE A fluorimetric determination can be used optionally.

7.5.2 Principle

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

NOTE Other suitable photometric procedures may also be used.

7.5.3 Reagents**7.5.3.1 Acetylacetone solution**

4 ml acetylacetone is transferred to a 1 000 ml volumetric flask, dissolved in distilled water and filled to volume with distilled water.

7.5.3.2 Ammonium acetate solution

200 g ammonium acetate is transferred to a 1 000 ml volumetric flask, dissolved in distilled water in and filled to volume with distilled water.

NOTE Optionally, a premixed reagent of acetylacetone and ammonium acetate as described in ISO 12460-4:2008, 4.1 may be used.

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7.5.4 Analytical Procedure

10 ml is taken from the aqueous solution (7.3) with a pipette and added to 10 ml acetylacetone solution (7.5.3.1) and 10 ml ammonium acetate solution (7.5.3.2) in a 50 ml flask. The flask is stoppered, shaken and warmed for 10 min in a water bath of 60 °C. The greenish-yellow solution is cooled to room temperature, protected against the influence of light (for about 1 h). The absorbance of this solution is determined at a wavelength of 412 nm against distilled water using a spectrophotometer (5.3). A blank value is determined in parallel with distilled water and taken into consideration in the determination of the perforator value (8.2).

NOTE When smaller volumes of solutions with the same ratio of reagents are used, e.g. applying digital/automatic pipettes, it should be ensured that enough analytical solution is available for the spectrophotometric determination. Furthermore it should be taken into consideration that measurement errors could have increasing influence on the result.

7.5.5 Calibration curve

The calibration curve (see Figure 4) 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.

7.5.5.1 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