SLOVENSKI STANDARD
SIST EN 717-2:1996

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Lesne plošče - Ugotavljanje sproščanja formaldehida - 2. del: Ugotavljanje sproščanja formaldehida po metodi plinske analize

Wood-based panels - Determination of formaldehyde release - Part 2: Formaldehyde release by the gas analysis method

Holzwerkstoff - Bestimmung der Formaldehydabgabe - Teil 2: Formaldehydabgabe nach der Gasanalyse-Methode

Panneaux a base de bois - Détermination du dégagement de formaldéhyde - Partie 2: Emission en formaldéhyde par la méthode d'analyse de gaz

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English version

Wood-based panels - Determination of formaldehyde release - Part 2: Formaldehyde release by the gas analysis method

Panneaux à base de bois - Détermination du dégagement de formaldéhyde - Partie 2: Emission en formaldéhyde par la méthode d'analyse de gas

Holzwerkstoff - Bestimmung der Formaldehydabgabe - Teil 2: Formaldehydabgabe nach der Gasanalyse-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

Contents

Foreword

This European Standard has been prepared by the 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of an identical text or by endorsement, at the latest by May 1995, and conflicting national standards shall be withdrawn at the latest by May 1995.

The gas analysis method was developed during 1965 to 1973 by the European Federation of Associations of Particleboard Manufacturers FESYP. A summary appeared in 1969 under the title "FESYP Perforator Method, FESYP Gas Analysis Method" as Special Booklet 1/1969 of FESYP.

During the subsequent years this procedure was modified and improved in certain respects by the Wilhelm-Klauditz-Institut, Fraunhofer Working Group for Wood Research (WKI), Braunschweig. In September 1984 the gas analysis method became a German standard (DIN 52 368).

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 of 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: Walk-in-chamber reference method [1])

EN 717-3 Wood-based panels - Determination of formaldehyde release - Part 3: Formaldehyde release by the flask method [1])

In accordance with the Common CEN/CENELEC Rules 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.

[1]) At present at the draft stage

1 Scope

This European Standard describes a procedure for determination of accelerated formaldehyde release from 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 326-1 Wood-based panels - Sampling, cutting and inspection - Part 1: Sampling and cutting of test pieces and expression of test results

3 Principle

A test piece of known surface area is placed in a closed chamber in which the temperature, humidity, airflow and pressure are controlled to defined values. Formaldehyde released from the test piece mixes with the air in the chamber. This air is continually drawn from the chamber and passes through gas wash bottles, containing water, which absorbs the released formaldehyde. At the end of the test, the formaldehyde concentration is determined photometrically. The formaldehyde release is calculated from this concentration, the sampling time and the exposed area of the test piece and is expressed in milligrammes per square meter per hour (mg/m^2h).

4 Reagents

4.1 General

Reagents of recognized analytical purity and distilled or demineralised water (referred throughout the following text as distilled water) shall be used for the analysis.

4.2 Acetylacetone solution

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

4.3 Ammonium acetate solution

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

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

5 Apparatus

5.1 The test apparatus (see figure 1) comprises the following main components:

5.1.1 Air filter (1)

5.1.2 Wash bottle, 500 ml, containing ca. 400 ml distilled water (2)

5.1.3 Desiccator, 500 ml, containing silica gel (3)

5.1.4 Air pump (4)

5.1.5 Needle valve (5)

5.1.6 Equipment for measuring rate of air flow through apparatus (6)

5.1.7 Test chamber (length: 555 mm, diameter: 96 mm, internal volume: 4 017 ml) with double casing of stainless steel or glass (7)

5.1.8 Heating equipment for air (e.g. copper coil inside the double casing) (8)

5.1.9 Thermostat (9)

5.1.10 Magnetic valves (10)

5.1.11 4 pairs of wash bottles, 100 ml (21)

5.1.12 Pressure monitor (22)

5.1.13 Temperature monitor (23)

5.2 Laboratory equipment

5.2.1 Ventilated oven, as described in EN 322

5.2.2 Spectrophotometer with cells of 50 mm optical path length and capable of measuring absorbance at 412 mm

5.2.3 Water bath, capable of maintaining a temperature of (40 ± 1) °C

5.2.4 6 volumetric flasks, 100 ml (calibrated at 20 °C)

5.2.5 4 volumetric flasks, 250 ml (calibrated at 20 °C)

5.2.6 2 volumetric flasks, 1 000 ml (calibrated at 20 °C)

5.2.7 8 wash bottles, 100 ml

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

5.2.9 5 flasks, 50 ml, (with stoppers)

5.2.10 Microburette

5.2.11 Burette, 50 ml, graduated in 0,05 ml (calibrated at 20 °C)

5.2.12 Balance, capable of measuring to 0,001 g

6 Test pieces

6.1 Selection of test pieces for production control

Sampling and cutting of the test pieces shall be in accordance with EN 326-1.

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

- For the determination of formaldehyde release, 3 test pieces of 400 mm x 50 mm x board thickness.
- For the determination of moisture content, 5 or 6 test pieces of 25 mm x 25 mm x board thickness.

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

6.2 Selection of test pieces for other purposes

The procedure of taking test pieces (e.g. from boards already installed) shall be noted and described in the test report. The number and dimensions of the test pieces shall be as given in 6.1.

6.3 Preparation of test pieces

The test pieces shall be edge sealed.

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

7 Procedure

7.1 Number of determinations

Determinations shall always be made in duplicate using two different test pieces. If the individual values of a duplicate determination differ from each other by more than 0,5 mg/m^{2}h then a third determination shall be made.

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

7.2 Determination of moisture content

Moisture content shall be determined according to EN 322 using a separate sample (see 6.1).

7.3 Determination of formaldehyde release

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

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

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

Place a test piece in the pre-heated test chamber. After closing the test chamber and starting the test, the test piece is uniformly exposed to practically formaldehyde free, heated air (60 ± 0,5) °C with a relative humidity of (2 ± 1) %. Immediately set the airflow into the chamber to (60 ± 3) l/h, using the needle valve (5.1.5), and the air volume meter (5.1.6). This air is led into one of a series of pairs of wash bottles via a magnetic valve (5.1.10).

As the formaldehyde released from the test piece shall be determined at hourly intervals (up to 4 h from starting the test), a new series of wash bottles has to be connected up every hour. This exchange should be automatic.

Over the whole test period, the overpressure in the test chamber is monitored (5.1.12). An overpressure of 1 000 to 1 200 Pa shall be maintained throughout the entire test period.

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

NOTE: Care should be taken that the combined contents of the bottles and rinsings does not exceed 250 ml.

Make up the flask to the mark with distilled water and determine the formaldehyde content as specified in 7.4.

7.4 Determination of formaldehyde content of the aqueous solutions

7.4.1 General

The formaldehyde content of the aqueous solution from each one hour sampling period shall be determined photometrically.

7.4.2 Principle

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

NOTE: Other suitable photometric procedures may also be used.

7.4.3 Procedure

10 ml are taken from the aqueous solution (7.3) with a pipette (5.2.8) and added to 10 ml acetylacetone solution (4.2) and 10 ml ammonium acetate solution (4.3) in a 50 ml flask (5.2.9). The flask is stoppered, shaken and warmed for 15 min in a water bath (5.2.3) at 40 °C. The 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 (5.2.2). A blank value is determined in parallel with distilled water and taken into consideration in the determination of the gas analysis value.

7.4.4 Calibration curve

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

7.4.4.1 Formaldehyde standard solution

Reagents:

- Standard iodine solution $c(I_2)$ = 0,05 mol/l
- Standard sodium thiosulphate solution $c(Na_2S_2O_3)$ = 0,1 mol/l
- Standard sodium hydroxide solution c(NaOH) = 1 mol/l
- Standard sulphuric acid solution $c(H_2SO_4)$ = 1 mol/l
- starch solution 1 % m/m

The solutions shall be standardised before use.

About 1 g formaldehyde solution (concentration 35 % to 40 %) is diluted in a 1 000 ml volumetric flask (5.2.6) with distilled water and made up to the mark. The exact formaldehyde concentration of this solution is determined as follows:

20 ml of the formaldehyde standard solution are mixed with 25 ml iodine solution and 10 ml sodium hydroxide solution. After 15 minutes standing, protected from light, 15 ml of sulphuric acid solution is added. The surplus iodine is back-titrated with the thiosulphate solution. At the end of the titration some drops of starch solution are added as an indicator. A blank test with 20 ml distilled water is carried out in parallel.

The formaldehyde content is calculated by the following equation:

$$c(HCHO) = (V_o - V) \times 15 \times c(Na_2S_2O_3) \times 1\,000/20 \quad (1)$$

where:

$c(HCHO)$ is the formaldehyde concentration, in milligramms per litre

$c(Na_2S_2O_3)$ is the thiosulfate concentration in mols per litre

V is the volume of thiosulphate titration solution, in millilitres

V_o is the volume of thiosulphate titration solution for the blank, in millilitres

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

7.4.4.2 Formaldehyde calibration solution

Using the concentration determined in 7.4.4.1, calculate the volume which will contain 3 mg formaldehyde. Transfer this volume, using a microburette, to a 1 000 ml volumetric flask and make up to the mark with distilled water. 1 ml of this calibration solution contains 3 µg formaldehyde.

7.4.4.3 Determination of the calibration curve

Pipette either zero, 5, 10, 20, 50 or 100 ml of formaldehyde calibration solution (7.4.4.2) in a 100 ml volumetric flask (5.2.4) and make up to the mark with distilled water. 10 ml of each dilution are analysed photometrically by the same procedure as described above (7.4.3). The absorbance values are plotted against the formaldehyde concentrations (c) (between 0 and 0,003 mg/ml) on millimetre graph paper (see example in figure 2). The slope (f) is either determined graphically, or calculated.

8 Expression of results

8.1 Gas analysis value

From each 1 hour sampling period the gas analysis value G_i is determined and calculated by the following equation:

$$G_i = \frac{(A_S - A_B) \times f \times V}{F} \quad [mg/m^2h] \qquad (2)$$

Where:

G_i is the formaldehyde content of the solution from each hourly sample in milligramms divided by the area of the exposed, unsealed surface

i is the first, second, third or fourth hour

A_S is the absorbance of the solution from the wash bottles

A_B is the absorbance of distilled water

f is the slope of the calibration curve for standard formaldehyde solution, in milligramms per millilitre

F is the combined area of the emitting (unsealed) surfaces, in square metres

V is the volume of the volumetric flask, in millilitres

8.2 Calculation of results

As a rule the formaldehyde content of the liquid absorbent, taken during the first hour is lower than the content of the second hour, as the temperature of the test piece over the first hour does not reach 60 °C immediately. In this case the gas analysis value is calculated from the sum of the contents for hours 2 to 4 and is related to the surface area (F) of the test piece. If the maximum of formaldehyde content is reached during the first hour, the sum of all 4 hourly samples is used for the calculation.