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IS 13745 (1993): Method for determination of formaldehyde content in particle board by extraction method called perforator method [CED 20: Wood and other Lignocellulosic products]



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Indian Standard

**METHOD FOR DETERMINATION OF
FORMALDEHYDE CONTENT IN PARTICLE
BOARD BY EXTRACTION METHOD CALLED
PERFORATOR METHOD**

UDC 674·816-41 : 543 [547·281·1]

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Wood Products Sectional Committee had been approved by the Civil Engineering Division Council.

The emission of formaldehyde from a particle board 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.

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 result must be considered in relation to the specific board conditions at the time of testing as outlined above.

There is not necessarily a correlation between the perforator value and the formaldehyde emission of the board.

The temperature of conditioning of boards has been given as $27 \pm 2^{\circ}\text{C}$ in 3.3 and temperature required for calibrating the test apparatus has been given as 27°C under 6. However, for the purpose of export of boards to European countries, values of $20 \pm 2^{\circ}\text{C}$ and 20°C respectively may have to be adopted.

In the formulation of this standard considerable assistance has been taken from EN 120 : 1984 'Particle Boards. Determination of Formaldehyde Content by Extraction Method Called Perforator Method', European Committee for Standardization (CEN).

The composition of the committee responsible for the preparation of this standard is given at Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

AMENDMENT NO. 1 NOVEMBER 2011
TO
IS 13745 : 1993 METHOD FOR DETERMINATION OF
FORMALDEHYDE CONTENT IN PARTICLE
BOARD BY EXTRACTION METHOD CALLED
PERFORATOR METHOD

(Page 1, clause 1, Note, third line) — Substitute ‘5 mg/100 g’ *for* ‘10 mg/100 g’.

(CED 20)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHOD FOR DETERMINATION OF FORMALDEHYDE CONTENT IN PARTICLE BOARD BY EXTRACTION METHOD CALLED PERFORATOR METHOD

1 SCOPE

This standard covers method for determination of formaldehyde content of particle boards by an extraction method known as Perforator Method.

NOTE — The method given in this standard is used for the determination of formaldehyde content of particle boards of 10 mg/100 g and above. In the range below this value the results are difficult to interpret.

2 PRINCIPLE

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

3 SAMPLING AND PREPARATION OF TEST PIECES

3.1 Sampling

3.1.1 The test pieces are to be taken evenly distributed over the width of the (cooled) board excluding a strip of 500 mm width at a frontside of the board.

3.1.2 Take 18 test pieces of 25 mm × 25 mm × 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.

3.2 For Production Control

If this method is used for production control, the board selected for sampling is immediately cut up. The test pieces taken from the board shall be stored in a hermetically sealed container at ambient temperature.

The formaldehyde determination should be carried out not more than 24 hours after sampling.

3.3 For Other Purposes

If this method is used for other purposes, for example for boards already installed, the methods chosen for sampling, preparation of test pieces, and conditioning which influence the final result shall be as agreed between the parties and laid down in the test report.

Unless otherwise agreed, the boards shall be conditioned for 43 days and the test pieces for 6 days at $27 \pm 2^\circ\text{C}$ and $(65 \pm 5)\%$ relative humidity.

4 REAGENTS

For the analysis, 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 Iodine, standard solution with concentration (I_2) = 0.01 mol/l. Standardization of the solution shall be regularly checked. 1 ml of this solution corresponds to 0.3 mg formaldehyde.

4.3 Sulphuric acid solution, obtained by dilution of concentrated sulphuric acid with water at a ratio of 1:1 (volume).

4.4 Sodium thiosulphate, standard solution with concentration ($\text{Na}_2\text{S}_2\text{O}_3$) = 0.01 mol/l.

4.5 Sodium hydroxide, standard solution with concentration (NaOH) = 1 mol/l.

4.6 Starch solution of 1 percent (m/m).

5 APPARATUS

Approved laboratory equipment, like those given in 5.1 to 5.9 shall be used.

5.1 Precision balance accurate to 0.001 g.

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

5.3 Extraction Apparatus

The apparatus (see Fig. 1) shall consist of:

- a) spiral condenser, total length approximately 400 mm; cone 45/40, socket 29/32 (Part 1)
- b) conical adaptor, socket 45/40, cone 71/51 (Part 2)
- c) filter insert, porosity P 160 (100 to 160 μm), bowl and filter diameter 60 mm (Part 3)
- d) perforator attachment 1 000 ml with cock (4 mm bore, socket 71/51, male 29/32) (Part 4)
- e) conical adaptor, socket 29/32, cone 45/40 (Part 5)

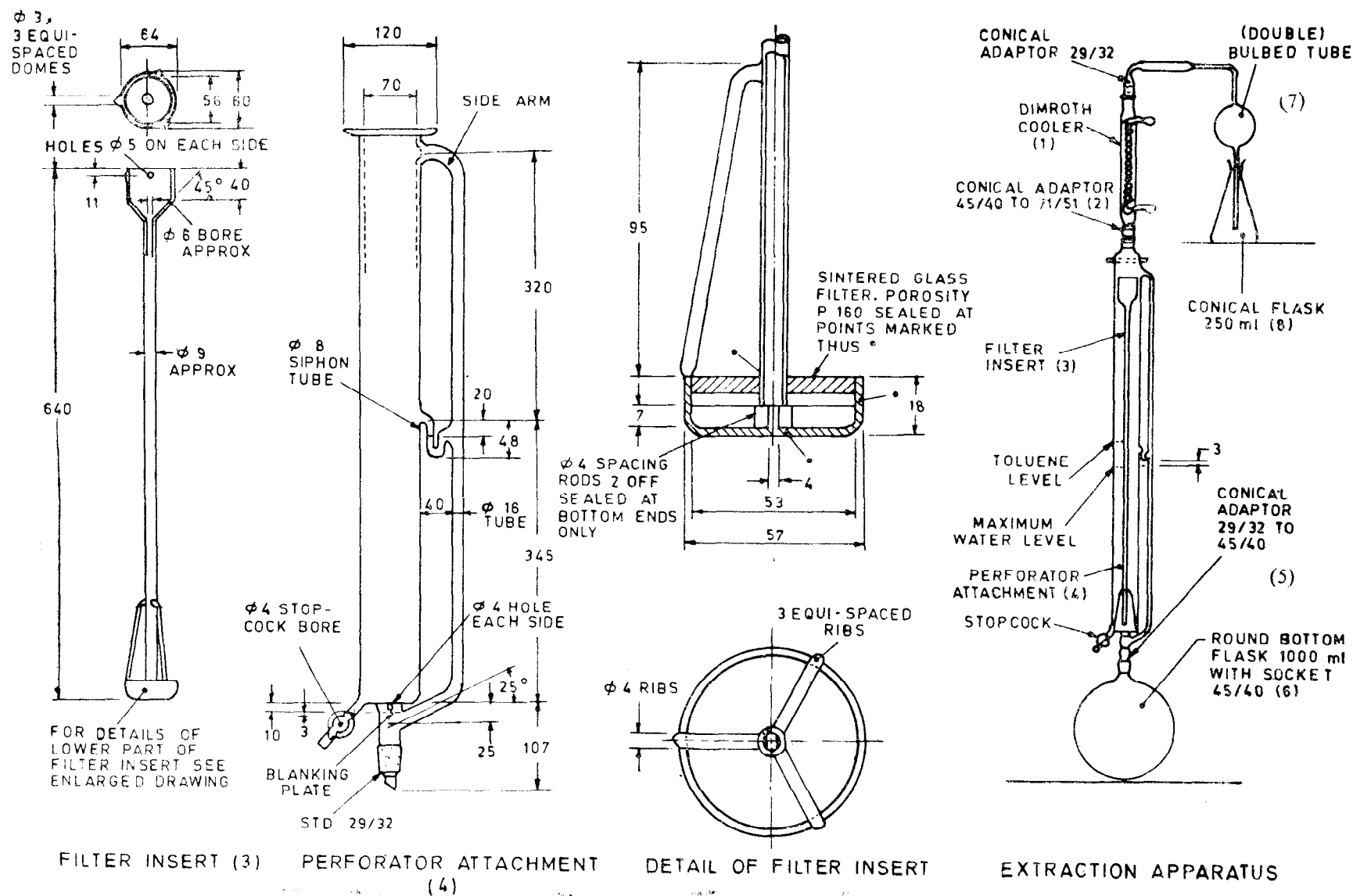


FIG. 1 EXTRACTION APPARATUS FOR DETERMINATION OF FORMALDEHYDE CONTENT IN PARTICLE BOARDS

- f) 1 000 ml round-bottomed flask, socket 40/50 (Part 6)
- g) (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) (Part 7)
- h) Absorption bulb (for example, Conical flask 250 ml) (Part 8).

5.4 Volumetric flask of 2 000 ml calibrated at 27°C.

5.5 Conical flask, 250 ml.

5.6 Precision burette 50 ml calibrated at 27°C.

5.7 Pipette 100 ml calibrated at 27°C.

5.8 Pipette 25 ml calibrated at 27°C.

5.9 Two measuring cylinders, 25 ml.

6 EXECUTION OF THE TESTS

6.1 Number of Determination

The determinations are to be carried out in duplicate.

The individual values of a duplicate determination may deviate by the maximum of 5 mg/100 g, but by not more than 10 percent related to the greater of the two single values; otherwise a third determination is to be carried out.

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

6.2 Determination of Moisture Content

The moisture content is determined on a sample of 5 to 6 test pieces (25 mm × 25 mm).

The sample is weighed (*see* 5.1), to an accuracy of 0.1 percent on a watchglass (about 120 mm diameter) and it is dried in a well ventilated drying oven (5.2) at a temperature of $103 \pm 2^\circ\text{C}$ to constant mass (about 12 h).

Constant mass is regarded as having been reached when the mass of the test piece does not differ by more than 0.1 percent over a period of 4 hours.

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

6.3 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 toluene.

About 100 g of test pieces are weighed to an accuracy of 0.1 g and put into the round bottom flask (5.3). 600 ml of toluene (4.1) is added. Subsequently, the round-bottom

flask is connected to the perforator. About 1 000 ml of distilled water is poured into the perforator attachment, leaving a space of 10 to 20 mm between the surface of the water and the siphon outlet. Then the condenser and the gas absorption equipment are connected. The absorption bulb of the gas equipment (5.3) is filled with about 100 ml distilled water.

When the apparatus is complete, the cooling and heating are turned on.

Toluene must flow back regularly throughout the whole period of perforation, with a reflux rate of 50 to 70 drops per minute.

Care must be taken that no water flows back from the absorption bulb (Part 8) into other parts of the equipment during the extraction process.

The extraction is carried on for two hours, counting from the moment that the first bubbles pass through the filter insert. The heating must be such that the onset of bubbling occurs between 20 and 30 minutes after turning on the heating device.

After these two hours the heating is switched off and the gas absorption bulb is removed.

The water contained in the perforator is transferred, after cooling to ambient temperature, into the volumetric flask by means of the stop-cock. 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 on 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.

6.4 Blank Test

The test must be repeated without test pieces with new toluene from the same bottle as used for the perforation.

6.5 Titration

6.5.1 Principle

The formaldehyde is oxidized by an excess of iodine in sulphuric acid solution, unused iodine is back titrated with thiosulphate.

6.5.2 Procedure

Transfer 100 ml of the formaldehyde solution in the volumetric flask (5.4) into the conical flask (5.5). Add 50 ml of iodine solution (4.2) and 20 ml of sodium hydroxide solution (4.5). Close the flask and keep it in the dark for 15 minutes. Carefully add 10 ml sulphuric acid (4.3). The liberation of excess iodine causes a brown colour to appear. Back titrate this excess iodine with the thiosulphate in the burette (5.6) in the presence of starch (4.6) as indicator.

The presence of other products capable of being oxidized by iodine (ethyl alcohol, acetone, etc) shall be avoided.

7 RESULTS

7.1 Moisture Content

The moisture content of the particle board, M in percent is calculated by the equation :

$$M = \frac{M_1 - M_0}{M_0} \cdot 100$$

where

M_1 is the mass in g of the test piece before drying, and

M_0 is the mass in g of the test piece after drying.

7.2 Perforator Value

The formaldehyde content is expressed in mg formaldehyde per 100 g of dry board.

This value is called the perforator value and is calculated according to the following equation:

$$\text{Perforator value} = \frac{3.0 (V_0 - V_1) (100 + M)}{m} \text{ mg/100 g}$$

where

V_0 is the consumption in ml of 0.010 mol/l thiosulphate solution for the blank test,

V_1 is the consumption in ml of 0.010 mol/l thiosulphate solution for the test,

M is the moisture content of the particle board in percent, and

m is the mass in g of test pieces before the extraction.

NOTE — 1 ml 0.01 mol/l thiosulphate solution corresponds to 0.5 ml 0.01 mol/l iodine solution and 0.15 mg formaldehyde.

Results shall be rounded off to the next integer.

8 TEST REPORT

In the test report the following minimum

information shall be given with reference to the present standard:

- a) Origin of the boards,
- b) Place, location* and state† of the board at the time of sampling (in particular the moisture content),
- c) Type of the board,
- d) Thickness of the board (mm),
- e) Bulk density of the board (kg/m³),
- f) Date of manufacture of the board,
- g) Date of sampling,
- h) Date of the titration,
- j) Moisture content (%), at the time of testing according to 6.2,
- k) Perforator value (mg formaldehyde/100 g dry board),
- m) Description of further details ‡, and
- n) At a conspicuous place of the test report, the following text must be included:

The emission of formaldehyde from a particle board 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.

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 result obtained must be considered in relation to the specific board conditions at the time of testing as outlined above.

There is not necessarily a correlation between the perforator value and the formaldehyde emission of the board.

*For example, ceiling, floor, wall, etc.

†For example, moisture content, surface coating, finishing.

‡ Report of all the information about the operations which are not in accordance with standard (sampling of test pieces, conditioning, etc).

ANNEX A

(Foreword)

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