Designation: D 5582 – 00

Standard Test Method for Determining Formaldehyde Levels from Wood Products Using a Desiccator[1]

This standard is issued under the fixed designation D 5582; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a small scale procedure for measuring formaldehyde emission potential from wood products. The formaldehyde level is determined by collecting air-borne formaldehyde in a small distilled water reservoir within a closed desiccator. The quantity of formaldehyde is determined by a modification of the National Institute for Occupational Safety and Health (NIOSH) 3500 chromotropic acid test procedure. Other analytical procedures may be used to determine formaldehyde emission potential provided that such methods give similar results to the chromotropic acid procedure. However, the test results and test report must be properly qualified and the analytical procedure employed must be noted. Procedures based on acetylacetone and pararosaniline have been found to give similar results to chromotropic acid in other test methods used in determining formaldehyde emission potential from wood products (see Test Method E 1333).

1.2 Wood products typically evaluated by this test method are made with urea-formaldehyde adhesives and include particleboard, hardwood, plywood, and medium-density fiberboard. This test method is used for product quality control and is a small bench test method that correlates with the large-scale acceptance test for determining formaldehyde levels from wood products, Test Method E 1333. The general desiccator testing procedure may be modified for different conditioning times to accommodate its use in manufacturing quality control. However, the test results must be properly qualified and the conditioning time employed must be noted.

NOTE 1—If modifications are made to the conditioning period for quality control purposes, it is important that the modification is consistently applied. Otherwise, the results may not be comparable.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 6 and 8.2.5.

2. Referenced Documents

2.1 *ASTM Standards:*

E 77 Test Method for the Inspection and Verification of Thermometers[2]

E 337 Test Method for Measuring Humidity With a Psychrometer (The Measurement of Wet-Bulb and Dry-Bulb Temperatures)[3]

E 1333 Test Method for Determining Formaldehyde Levels from Wood Products Under Defined Test Conditions Using a Large Chamber[4]

2.2 *HUD Document:*

24 CFR 3280, Manufactured Home Construction and Safety Standards, Federal Register, Vol 49, No. 155[5]

2.3 *NIOSH Document:*

Formaldehyde Method 3500, U.S. Department of Health, and Human Services[5]

2.4 *Other Documents:*

Minnesota Statutes Section 144.495, 325F.18, and 325F.181, Formaldehyde Gases in Building Materials[6]

3. Significance and Use

3.1 Limitations on formaldehyde levels have been established for wood panel building products made with urea-formaldehyde adhesives and permanently installed in homes or used as components in kitchen cabinets and for similar industrial products. This test method is used in conjunction with the test method referenced by HUD Rules and Regulations 24 CFR 3280 for manufactured housing and by Minnesota Statutes Section 144.495 for housing units and building materials. This test method provides a means of testing small-size samples to determine formaldehyde emission potential.

[1] This test method is under the jurisdiction of ASTM Committee D-7 on Wood and is the direct responsibility of Subcommittee D07.03 on Panel Products.

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[2] *Annual Book of ASTM Standards*, Vol 14.03.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] *Annual Book of ASTM Standards*, Vol 04.07.

[5] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

[6] Available from Print Communications, Dept. of Administration, 117 University Ave., St. Paul, MN 55155.

3.2 This test method incorporates a desiccator, with the desiccant removed, having a 250-mm (10-in.) inside diameter and a volume of approximately 10.5 L (641 in.^{3}) with the desiccator lid in place. Conditions controlled in the procedure are as follows:

3.2.1 Conditioning of panel products prior to testing,

3.2.2 Specified number, size, and edge sealing of wood specimens to be placed in the desiccator,

3.2.3 Test desiccator temperature, and

3.2.4 Samples from the 25-mL distilled water collection medium in the petri dish bottom are analyzed for formaldehyde at the end of a 2-h period in the closed desiccator.

3.3 This test method employs a single set of environmental conditions to assess formaldehyde emission potential from certain wood products. When the relationship between desiccator test values and large-chamber test values are to be determined, the values for the specific wood panel product type shall be plotted. This test method does allow a comparison of formaldehyde levels from different products for the same use.

NOTE 2—Care must be exercised in the extension of the results to actual formaldehyde emission from products under actual use conditions.

4. Interferences

4.1 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 this test method is used to accurately determine the formaldehyde emission potential from wood products made with phenol-formaldehyde adhesive systems.[7, 8]

5. Apparatus

5.1 *Desiccator*—The interior volume of the desiccator shall be 10.5 L (641 in.^{3}). Any desiccant shall have been removed, the interior of the desiccator thoroughly cleaned, and the porcelain desiccator plate replaced in the desiccator. The bearing areas of the desiccator and desiccator lid shall be greased so that the container will be air tight during the duration of the 2-h test.

5.2 *Petri Dish and Beaker*—A clean 400-mL beaker to be inverted as a reservoir support and the bottom of a 100 by 20-mm petri as a distilled water reservoir dish shall be available for each desiccator test.

5.3 *Test Room or Area*—A room or test area capable of being maintained at 24 ± 1°C (75 ± 2°F) shall be available for conducting desiccator tests.

NOTE 3—If liquid-in-glass thermometers are used for determining or checking the temperature of the test area, see Test Method E 77.

5.4 Examples of acceptable reagents, materials, and equipment are provided in Appendix X1.

6. Hazards

6.1 *Chromotropic Acid Reagent Treatment (see* 8.2.4 *and* A3.5*)*—During this hazardous operation, the operator shall wear rubber gloves, apron, and a full face mask or be protected from splashing by a transparent shield such as a hood window. The solution becomes extremely hot during the addition of sulfuric acid. Add slowly to avoid loss of sample due to splattering.

6.2 *Cleaning Chemicals for Glassware*—Appropriate precautions shall be taken if cleaning chemicals are considered to be hazardous.

7. Test Specimens

7.1 Use eight 70 ± 2 by 127 ± 2-mm (2 ¾ by 5-in.) by panel thickness specimens for each desiccator test. Cut specimens from the sample panel or panel segment to obtain adequate representation of areas within the panel or panel segment. The fresh cut edges and ends of each specimen shall be at least 25 mm (1 in.) from the edges and ends of the sample panel or panel segment. When a product has significantly different emission characteristics for each surface and has only one surface exposed to the building space, also use sixteen 70 by 127-mm (2 ¾ by 5-in.) test pieces to prepare eight 70 by 127-mm double-piece back-to-back specimens.

7.2 *Specimen Edge Sealing*—Remove sawdust and loose splinters from each test specimen. Coat the edges and ends of each single or double-piece specimen by immersion in melted paraffin wax. Apply at least two coats. The wax shall cover no more than 5 mm (3⁄16 in.) of either face around the coated perimeter.

7.3 *Specimen Conditioning*—Then condition the specimens on edge, spaced apart, so air can freely circulate across all surfaces for seven days ±4 h at 24 ± 1.7°C (75 ± 3°F) and 50 ± 10 % relative humidity. The formaldehyde concentration in the air within 30 cm (12 in.) of where the specimens are conditioned shall be not more than 0.1 ppm during the conditioning period.

NOTE 4—Conditioning time less than seven days and specimens with edges and ends not coated with paraffin wax may be used for quality control or informational testing; however these and other test method modifications shall be clearly indicated in the test report. Modifications to conditioning time or edge treatment, or both, will affect the test results; therefore, correlation to other test methods may need to be re-established.

NOTE 5— If liquid-in-glass thermometers or psychrometers, or both, are used for determining or checking the temperature or the relative humidity, or both, of the conditioning area, see Test Methods E 77 and E 337.

8. Procedure

NOTE 6—A list of test apparatus and chemical reagents are provided in Appendix X1.

8.1 *Test Procedure for Materials:*

8.1.1 Conduct tests in a room maintained at 24 ± 0.6°C (75 ± 1°F). Equilibrate the desiccator, petri dish bottom, and distilled water to 24 ± 0.6°C.

8.1.2 Before each test, wipe the desiccator with a clean rag or paper towel moistened with distilled water, and then dry with a clean dry rag or paper towel.

NOTE 7—Formaldehyde can be used as a constituent of wet-strength resins for paper and of permanent-press resins for fabrics. The type of rag or paper towel selected for cleaning must be formaldehyde-free.

[7] Hakes, D., Johnson, G., and Marhevka, J., *Procedure for Elimination of Phenol Interference in the Chromotropic Acid Method for Formaldehyde*, American Industrial Hygiene Association, April 1984.

[8] *Technical Bulletin No. 415*, National Council of the Paper Industry for Air and Stream Improvement Inc. (NCASI), 1983.

8.1.3 Apply a light coating of vacuum grease to the desiccator lid and desiccator. Avoid excessive use of vacuum grease.

8.1.4 Arrange specimens as prepared in 7.1 and 7.2 and condition as in 7.3 on top of the porcelain desiccator plate around an inverted 400-mL beaker as a 100 ± 7-mm (4 ± ¼-in.) high support inside the desiccator for the petri dish bottom distilled water reservoir. Specimens should be arranged so that air has access to all surfaces and edges.

8.1.5 Pipet 25 mL of distilled water into the bottom portion of petri dish.

8.1.6 Carefully lower the petri dish bottom containing distilled water into the desiccator until it rests upon the inverted 400-mL beaker.

8.1.7 Slide the desiccator lid into place making sure a good seal is obtained.

8.1.8 Observe and record the time.

8.1.9 Maintain the desiccator test room at 24 ± 0.6°C (75 ± 1°F). Record the temperature at 30-min intervals. Alternatively, use a continuous temperature recorder. Report any temperature range deviations.

8.1.10 After 120 ± 1 min, remove the desiccator lid and carefully remove the petri dish. Proceed immediately to 8.2.1. When running multiple desiccator tests, initiate 8.2.1 within 10 min, otherwise cover the petri dish or dishes with parafilm while awaiting analysis.

8.2 *Analysis of Water Samples*:

8.2.1 Gently swirl the petri dish and pipet 4 mL of the solution into each of two 16 by 150-mm screw cap test tubes for duplicate analysis. Label to avoid subsequent error. Alternatively, use three tubes for triplicate analysis.

8.2.2 Pipet 4 mL of distilled water into a 16 by 150-mm screw capped test tube to act as a "blank."

8.2.3 Add 0.1 mL of 1 % chromotropic acid reagent to each test tube and shake to mix.

8.2.4 Slowly and carefully pipet 6.0 mL concentrated sulfuric acid into each test tube (**Precaution**—See 6.1.) and allow to flow down the side of test tube. Allow the volumetric pipet to drain. **Do not blow out.** Before placing caps on test tubes, check the condition of the polytetrafluoroethylene (PTFE) cap liners to make sure they are clean and not deteriorated.

8.2.5 Ensure adequate mixing by use of a vibrating laboratory mixer or other means. Mixing is complete when there is no sign of stratification. If absorbance readings routinely exceed 1.0 or if spectrophotometric analysis is performed within 2 h, heat capped test tubes to 95°C or place in a boiling water bath for 15 ± 2 min to ensure that the chemical reaction is complete. After removal, allow the test tubes to cool to room temperature. Carefully vent test tubes to release pressure. (**Warning**—Avoid rapid mixing as heating and pressure will increase and potentially break the test tube.)

8.2.6 Allow the tubes to cool to room temperature. Do not accelerate the cooling. Avoid cooling tubes in direct sunlight as this may alter color chromogen development. Transfer the solution to cuvettes (if necessary). At this point, small bubbles may be rising through the solution. Do not make absorbance readings until the solution is clear.

8.3 *Absorbance Readings*:

8.3.1 Prior to performing this test method for the first time, a calibration curve shall be developed. See Annex A3.

8.3.2 Standardize the spectrophotometer to 100 % transmittance (zero absorbance) using distilled water at 580 nm in accordance with the instrument's operating instructions. This gives an indication that the instrument is in proper working order. Read the "blank" against distilled water because an absorbance above 0.040 (using a 12-mm cell path length) or above 0.030 (using a 10-mm cell path length) for the reagent blank indicates contamination of the reagent blank or improper solution preparation.

8.3.3 Zero the instrument on the reagent blank if absorbance is not greater than 0.040 (12-mm path length) or 0.030 (10-mm path length) compared to distilled water as zero. Alternately, leave the instrument zeroed on distilled water, and subtract the absorbance of the reagent blank from the absorbance of the sample solutions.

8.3.4 Read and record absorbance at 580 nm of each sample prepared (see 9.1 for calculation).

8.3.5 When a precise desiccator value is required and the sample solution is found to fall outside the stated absorbance range (greater than 1.0 or as determined in A3.12), repeat 8.2.1-8.3.4. Otherwise, report the desiccator value associated with a greater than 1.0 absorbance value. When 8.2.1-8.3.4 are repeated, appropriately dilute the sample solution to fall within the preferred absorbance range of the spectrophotometer. Make dilution by pipetting x mL of text solution to (4-x mL) of distilled water for a total of 4 mL (that is, 1 mL of test solution + 3 mL distilled water = 4 mL total). Rerunning the distilled water" blank" is not required. Use average sample determinations as the sample absorbance. Read micrograms (µg) of formaldehyde from the calibration curve. (See Annex A3.)

9. Calculation

9.1 Calculate formaldehyde concentration in weight per unit volume in the solution from the petri dish aliquot in the desiccator:

$$c_t = \frac{c_s}{D \times 4} \quad (1)$$

where:

c_t = µg of formaldehyde per mL of sampled solution,
c_s = µg of formaldehyde in 4-mL aliquot of sample read from calibration curve, and
D = dilution factor, for example:

$$\frac{1 \text{ mL } (\textit{original volume})}{4 \text{ mL } (\textit{final volume})} D = 0.25 \quad (2)$$

If no dilution is made, $D = 1$.

9.2 When the temperature at which the test is conducted differs from 24 by ¼ °C (75 by ½ °F) or more, adjust the desiccator value obtained to a standard temperature of 24°C (75°F) using a formula developed by Berge et al.[9] Annex A1 contains a table of conversion factors for use at different

[9] Berge, A., Mellagaard B., Hanetho, P., and Ormstad, E., *Formaldehyde Release from Particleboard—Evaluation of a Mathematical Model*, Holz Als Roh-und Werkstoff 38, 1980, pp. 252–255.