This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D4426 – 01 (Reapproved 2021)

Standard Test Method for Determination of Percent Nonvolatile Content of Liquid Phenolic Resins Used for Wood Laminating[1]

This standard is issued under the fixed designation D4426; 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 the recommended procedure for the determination of the nonvolatile or total solids content of liquid phenolic resins used for wood laminating.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D907 Terminology of Adhesives

D1582 Test Method for Nonvolatile Content of Liquid Phenol, Resorcinol, and Melamine Adhesives

3. Terminology

3.1 *Definitions*—Many of the terms in this test method are defined in Terminology D907.

4. Summary of Test Method

4.1 A weighed specimen is oven-dried, cooled in a desiccator, and weighed. The percent nonvolatile content is calculated, based on the amount remaining.

5. Significance and Use

5.1 This test method is intended as a fast and economical determination of the nonvolatile content of liquid phenolic resins used for wood laminating and is useful for general comparative purposes. For greater precision and accuracy, Test Method D1582 is recommended.

6. Apparatus

6.1 *Disposable Aluminum Foil Dishes,* 60-mm (2.4-in.) diameter by 16-mm (0.60-in.) depth.

NOTE 1—A glass dish may be used if the pH of the resin is greater than ten.

6.2 *Desiccator,* charged with Drierite or equivalent desiccant.

6.3 *Mechanical Convection Oven.*

6.4 *Analytical Balance,* accurate to ±0.1 mg (0.000002 lb).

6.5 *Eye Droppers, or Dropping Pipettes,* 2-mL capacity.

6.6 *Anemometer.*

6.7 *Stainless Steel Trays,* fabricated 30 gauge, 83 by 250 mm (3.25 by 9.8 in.), with 19-mm (0.75-in.) sides and open ends.

6.8 *Modified Oven Shelves,* fitted with fabricated 18-gauge 19 by 19-mm (0.75 by 0.75-in.) stainless steel angle stop positioned 108 mm (4.25 in.) from back edge. Shelf-stop ensures consistent solids dish placement and prevents placement at the rear of the oven where airflow is erratic.

6.9 *Laboratory Hotplate,* with heating capacity to 270°C (518°F).

7. Oven Setup and Periodic Check Specifications

7.1 *Specifications:*

7.1.1 *Intake Port*—6.4 mm (0.25 in.) open.

7.1.2 *Exhaust Port*—6.4 mm (0.25 in.) open.

7.1.3 *Internal Airflow*—91.4 ± 1 m/min (300 ± 3 ft/min) at 40°C (104°F).

7.1.4 *Temperature*—125 ± 1°C (257 ± 2°F).

7.2 A 6.4-mm (0.25-in.) metal spacer is a convenient tool for setting port openings.

[1] This test method is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.30 on Wood Adhesives.

Current edition approved April 1, 2021. Published April 2021. Originally approved in 1984. Last previous edition approved in 2013 as D4426 – 01 (2013). DOI: 10.1520/D4426-01R21.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States

7.3 Measure with Davis-type anemometer placed as close as possible to the geometric center of the cavity with the door shut.

7.4 Set the oven temperature at 125°C (257°F) and record the actual temperature for a period of 2 h. The control range shall be ±1°C (2°F) during the check period.

8. Sampling

8.1 Take a representative sample of the lot of resin being evaluated of a sufficient quantity for the tests. While the tests consume less than 5 g (0.01 lb) of resin, a sample of at least 250 g (0.55 lb) is recommended to ensure that it is representative and that rechecks can be made if desired.

8.2 To ensure that surface evaporation of solvent does not affect the accuracy of the results, mix the resin sample thoroughly just prior to the test.

9. Sample Dish Preparation

9.1 Inscribe an identifying number on each dish to be used in the test. (Prepare three specimens from each sample.)

9.2 Place each dish on a hotplate set at 270°C (518°F) for about 15 s to flash off a thin coating of oil. (A whitish smoke will cease to be given off when all the oil has dissipated.)

9.3 Place the heat-treated dishes in a desiccator for at least 5 min.

10. Procedure

10.1 Adjust oven to 125 ± 1°C (257 ± 2°F).

10.2 Weigh a clean, previously numbered and heat-treated, aluminum dish to the nearest 0.1 mg (0.000002 lb) on an analytical balance.

10.3 Using an eyedropper or dropping pipette, add approximately 1 g (0.002 lb) of resin to the center of the dish. (Allow resin to remain as a "button.")

10.4 Reweigh the dish and resin to the nearest 0.1 mg (0.000002 lb). Weigh rapidly to minimize loss by evaporation.

10.5 Repeat 10.2 – 10.4 twice more for a total of three specimens per resin sample.

10.6 Place dishes on a stainless steel tray and set the tray in the oven. Trays are positioned with tray side against shelf-step and parallel with airflow. Use only the top three shelf positions, with no more than two trays of specimens per shelf.

10.7 Dry the specimens at 125 ± 1°C (257 ± 2°F) for 105 ± 3 min.

10.8 Remove the dishes with the dried specimens and place in a desiccator to cool for at least 5, but not more than 15 min.

10.9 When cool, weigh each specimen as rapidly as possible to the nearest 0.1 mg (0.000002 lb). Inspect each specimen visually at the time of weighing. If any of the three specimens obviously deviates from the other two, or from usual normal appearance, discard all three specimens and rerun the test.

11. Calculation

11.1 Calculate the percent of nonvolatile matter as follows:

$$\text{Nonvolatile matter, } \% = (A/B) \times 100$$

where:

A = net weight of dried residue, and
B = net weight of liquid resin specimen.

11.2 Calculate the average of the three specimens and report the results to the one-hundredth percent (0.01). Report all results. If an individual specimen result varies more than ±0.5 % from the average, rerun the test set.

12. Report

12.1 Report the results as percent of nonvolatile content.

13. Precision and Bias

13.1 *Precision*—The precision of this test method varies with resin type, solids content and caustic level. Four resins of varying type, solids level and caustic level were evaluated in a series of round-robin tests to determine repeatability and reproducibility of this test method. The data generated by these round-robin tests will be available at a later date.

13.1.1 *Repeatability*—The average repeatability standard deviation has been determined to be 0.12 % (0.08–0.15), with an average repeatability at 95 % of 0.34 % (0.21–.43) for four different resins.

13.1.2 *Reproducibility*—The average reproducibility standard deviation has been determined to be 0.20 % (0.10–0.30), with an average reproducibility at 95 % of 0.56 % (0.28–0.83) for four different resin samples.

13.2 *Bias*—No information can be presented on the bias of the procedure in Test Method D4426 for measuring phenolic resin nonvolatile content because no material having an accepted reference value is available.

14. Keywords

14.1 laminating; nonvolatile content; phenolic; resins; wood

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/