**Designation: D 4426 – ~~96~~ 01**

# 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 D 4426; 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 and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

[1] This test method is under the jurisdiction of ASTM Committee D-14 on Adhesives and is the direct responsibility of Subcommittee D14.30 on Wood Adhesives.
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D 907 Terminology of Adhesives[2]
D 1582 Test Method for Nonvolatile Content of Liquid Phenol, Resorcinol, and Melamine Adhesives[2]

## 3. Terminology

3.1 *Definitions:*—Many of the terms in this test method are defined in Terminology D 907.

~~3.1.1 *nonvolatile content, n*—the portion of a material that remains after volatile matter has been evaporated under specified ambient or accelerated conditions. (Sometimes called *solids content*.)~~

~~3.1.1.1 *Discussion*—The percentage by weight of nonvolatile matter in an adhesive will vary depending on the analytical procedure used. A standard test method must be used to obtain consistent results. Some liquids may remain, depending on the conditions of the test.~~

~~3.1.2 *solids content, n*—See *nonvolatile content*.~~

## 4. Summary of Test Method

4.1 A weighed ~~sample~~ 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 D 1582 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 gage, 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-gage 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.
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 ~~material,~~ 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.)

[2] *Annual Book of ASTM Standards*, Vol 15.06.

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 ~~sample~~ resin to the nearest 0.1 mg (0.000002 lb). Weigh rapidly to minimize loss by evaporation.

10.5 Repeat 10.2-10.4 ~~for two~~ twice more for a total of three specimens per resin sample~~s~~.

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 specimen~~sets~~ per shelf.

10.7 Dry the ~~samples~~ 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 specimen~~sets~~ and report the results to ~~three significant figures only (nearest one tenth of 1 %). Do not omit a single result that does not agree closely with~~ the ~~remaining two.~~ one-hundredth percent (0.01). Report all results. If an individual specimen result~~s~~ var~~y~~ies 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, etc. However, expect a maximum 95 % confidence~~ level. Four resins of varying type, solids level and caustic level were evaluated in a series of ~~±1.0 % between laboratories when analyzing~~ 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 stan~~u~~dard 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 a~~s~~n 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 ~~one sample. Within a laboratory,~~ the bias of the proca~~e~~dure in Test Method D 4226 for measur~~ w~~ing phenolic resin nonvolatile content because no mate~~w~~rial having an accepted reference value is~~m~~ availabl~~e~~r.

## 14. Keywords

14.1 laminating; nonvolatile content; phenolic; resins; wood