



Standard Test Methods for Nonvolatile Content of Resin Solutions¹

This standard is issued under the fixed designation D1259; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the determination of nonvolatile content of solutions of resins in volatile organic solvents.

1.2 Two test methods are included as follows:

1.2.1 *Test Method A*—For solutions of non-heat-reactive resins. These solutions contain resins that remain stable and release the solvent under conditions of the test. Examples are ester gums and alkyds.

1.2.2 *Test Method B*—For two types of solutions:

1.2.2.1 Solutions of heat-reactive resins. These solutions contain resins that undergo condensation or other reactions under the influence of heat. Examples include the formaldehyde reaction products of urea, melamine, and phenols.

1.2.2.2 Solutions that release solvent slowly. Examples include epoxy resin solutions.

1.3 Test Methods A and B differ primarily in the drying times and types of oven used.

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

1.5 *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. Summary of Test Methods

2.1 In both test methods, a weighed specimen of resin solution is spread under pressure between two weighed sheets of aluminum or tin foil. The coated foil sheets are separated and then dried. The weight of residue is determined and the nonvolatile content is calculated. The test method is unique in that it provides for drying of a very thin film of resin, thus

minimizing chances for volatiles to be trapped and held during the heating operation.

2.2 Either a gravity-convection or a forced-ventilation oven and a 30-min heating period at 105°C are used in Test Method A.

2.3 A forced-ventilation oven and a 2-h heating period at 105°C are used in Test Method B.

3. Significance and Use

3.1 The nonvolatile content of resin solutions is useful to coatings producers and users for the determination of the total solids available for film formation and for the estimation of the volatile organic content.

TEST METHOD A—NON-HEAT-REACTIVE RESIN SOLUTIONS

4. Apparatus

4.1 *Ovens*:

4.1.1 Gravity-convection type, maintained at $105 \pm 2^\circ\text{C}$, with vents open.

4.1.2 Forced-ventilation type, maintained at $105 \pm 2^\circ\text{C}$. For ovens with adjustable air flow rate, set the control damper at 50 %.

4.2 *Aluminum or Tin Foil*, from 38 to 50 μm (0.0015 to 0.0020 in.) in thickness. Either one piece 150 by 300 mm (6 by 12 in.), or two 150 by 150-mm (6 by 6-in.) pieces may be used. The foil must be perfectly smooth; if it becomes wrinkled during the initial handling, roll smooth as directed in 5.2.

4.3 *Plate Glass*—Two pieces about 5 mm ($3/16$ in.) thick; one piece 140 by 140 mm ($5\frac{1}{2}$ by $5\frac{1}{2}$ in.) and one piece 180 by 180 mm (7 by 7 in.).

4.4 *Device for Weighing Specimens*²—Apparatus that will prevent loss of volatile matter during the weighing operation such as any of the following, or equivalent:

4.4.1 *Syringe*, Luer, 2 or 5-mL capacity,

4.4.2 *Weighing Buret*, Smith, 10-mL capacity, or

4.4.3 *Bulb Pipet*, dropping, with 50-mL Erlenmeyer flask.

¹ These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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² The bulb pipet is available from most scientific supply houses.

4.5 *Roller, for Smoothing Foil*—Use a ground and polished cylinder, preferably stainless steel, approximately 7 in. (180 mm) long and 2 in. (50 mm) in diameter.

4.6 *Foil Trays*, two types as follows:

4.6.1 Trays measuring 165 by 300 mm (6½ by 12 in.) , for use with (6 by 12-in.) foil, constructed from No. 22-gauge aluminum sheet in accordance with dimensions shown in Fig. 1. Several trays may be stacked in the oven to permit running several specimens simultaneously.

4.6.2 Trays measuring 165 by 165 mm (6½ by 6½ in.), for use with (6 by 6-in.) foil, with holder, shall be constructed from No. 22-gauge aluminum sheet, as shown in Fig. 2.

5. Procedure

5.1 Use the following procedure with the 150 by 300-mm (6 by 12-in.) foil sheets and the 165 by 300-mm (6½ by 12-in.) trays. Alternatively two 150 by 150-mm (6 by 6-in.) foil sheets may be used in a similar manner with the 165 by 165-mm (6½ by 6½-in.) trays. In handling the foil, avoid wrinkling or creasing the sheets until after the specimen has been dried. Sheets may be rolled for convenience of handling and making the initial weighing, but must be kept smooth throughout the pressing and drying operations.

5.2 Weigh the foil to 0.1 mg. Open and place half the foil, with the shiny side up, on the 180 by 180-mm (7 by 7-in.) glass plate. If necessary, roll smooth with the metal roller. By means of the weighing device, weigh by difference a 0.9 to 1.1-g specimen of the resin solution, to 0.1 mg. Place the specimen on the center of that area of the foil covering the glass plate. Place the other half of the foil on top. Cover the foil with the second glass plate, centering the glass on the foil, and press down sufficiently to cause the specimen to spread uniformly into a thin film, about 75 mm (3 in.) in diameter. The pressure that must be exerted depends on the viscosity of the sample. In case a specimen of low viscosity should extend beyond the

edge of the foil, repeat the determination, allowing a few minutes for a portion of the solvent to evaporate from the weighed specimen before covering and pressing it.

5.3 After pressing, open the foil to its full length and place it in the foil tray. Place the tray in either a gravity-convection or a forced-ventilation oven at 105 ± 2°C for 30 min.

5.4 Remove the tray from the oven and then carefully remove the foil sheet from the tray. Return the dried film surfaces to the face-to-face position. While the foil is still warm, fold the edges together to enclose completely the dried film. Without undue delay, weigh to 0.1 mg.

6. Calculation

6.1 Calculate the percent nonvolatile content, *C*, as follows:

$$C = [(A - B) \times 100] / S$$

where:

- A* = weight of foil plus dried solids, g,
- B* = weight of foil, g, and
- S* = weight of sample taken, g.

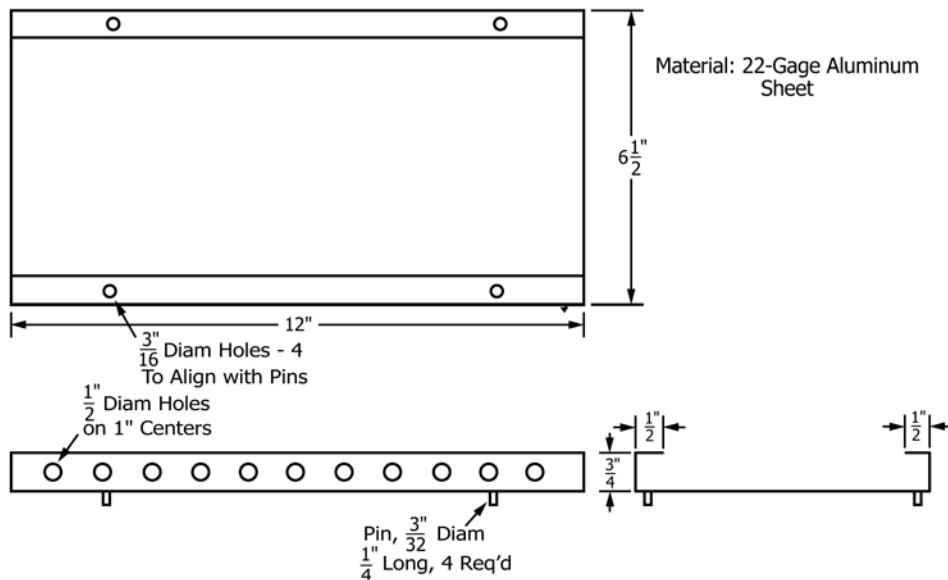
7. Precision and Bias

7.1 The following criteria should be used for judging the acceptability of results at the 95 % confidence level:

7.1.1 *Repeatability*—The difference between two results, each the mean of duplicate determinations obtained by the same analyst, is normally about 0.2 %, absolute. Two such results should be considered suspect if they differ by more than 0.5 %, absolute.

7.1.2 *Reproducibility*—The difference between two results, each the mean of duplicate determinations, obtained by analysts in different laboratories is normally about 0.4 %, absolute. Two such results should be considered suspect if they differ by more than 1.0 %, absolute.

7.1.3 No bias has been determined for this test method.



NOTE 1—Millimetre dimensions appear in section on Apparatus.

FIG. 1 Tray for 150 by 300-mm (6 by 12-in.) Foil

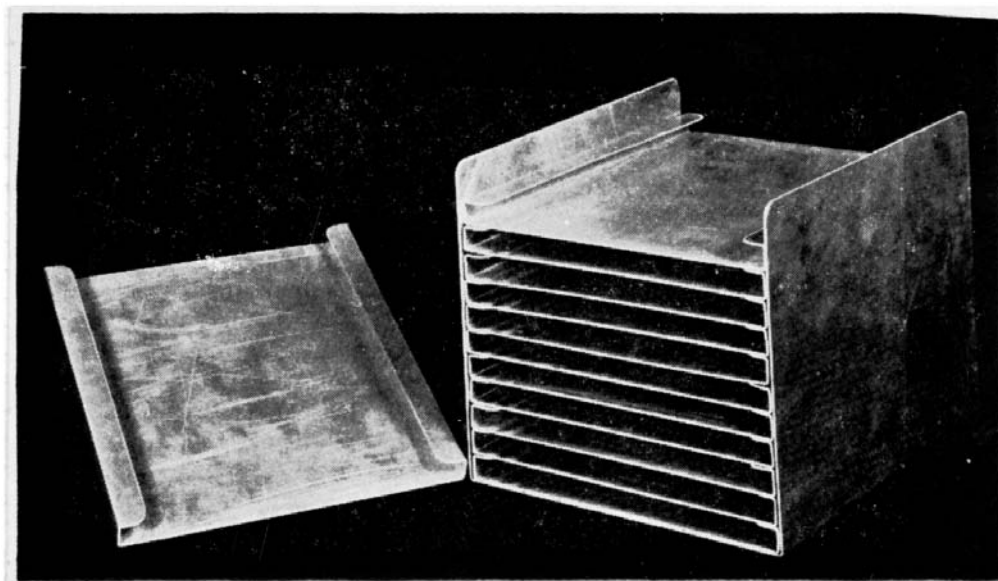


FIG. 2 Trays and Holder for 150 by 150-mm (6 by 6-in.) Foil

TEST METHOD B—HEAT-REACTIVE RESIN SOLUTIONS AND SOLUTIONS THAT RELEASE SOLVENT SLOWLY

8. Apparatus

8.1 *Oven*—Forced-ventilation type, maintained at $105 \pm 2^\circ\text{C}$. For ovens with adjustable air flow rate, set the control damper at 50 %.

8.2 The remainder of the apparatus is identical with that given in 4.2 – 4.6.

9. Procedure

9.1 Weigh the specimen of resin solution and press it between two sheets of foil as described in 5.1 and 5.2.

9.2 After pressing, open the foil to its full length and place it in the foil tray. Then place the tray in a forced-ventilation oven at $105 \pm 2^\circ\text{C}$ for 2 h. When using 150 by 150-mm (6½ by 6½-in.) trays and holder, place the assembly in the oven with the open ends perpendicular to the direction of air flow.

9.3 Complete the determination as described in 5.4.

10. Calculation

10.1 Calculate the nonvolatile content as described in Section 6.

11. Precision and Bias

11.1 The following criteria should be used for judging the acceptability of results at the 95 % confidence level.

11.1.1 For Heat-Reactive Resin Solutions:

11.1.1.1 *Repeatability*—The difference between two results, each the mean of duplicate determinations obtained by the same analyst, is normally about 0.3 %, absolute. Two such results should be considered suspect if they differ by more than 0.7 %, absolute.

11.1.1.2 *Reproducibility*—The difference between two results, each the mean of duplicate determinations obtained by analysts in different laboratories, is normally about 0.7 %, absolute. Two such results should be considered suspect if they differ by more than 1.7 %, absolute.

11.1.1.3 No bias has been determined for this test method.

11.1.2 For Solutions that Release Solvent Slowly:


11.1.2.1 *Repeatability*—The average difference between two results each the average of duplicate determinations, obtained by the same analyst is normally about 0.1 %, absolute. Two such results should be considered suspect if they differ by more than 0.3 %, absolute.

11.1.2.2 *Reproducibility*—The average difference between two results obtained by analysts in different laboratories will approximate 0.2 %. Two such results should be considered suspect if they differ by more than 0.5 %, absolute.

11.1.2.3 Bias cannot be determined for this method because there are no acceptable reference standards.

12. Keywords

12.1 foil method; heat-reactive; non-heat-reactive; nonvolatile; resin solution

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