Designation: D 1579 – 01

Standard Test Method for Filler Content of Phenol, Resorcinol, and Melamine Adhesives[1]

This standard is issued under the fixed designation D 1579; 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 Department of Defense. , as part of Federal Test Method Standard No. 175a.

1. Scope *

1.1 This test method is suitable for measuring the filler content of phenol, resorcinol, and melamine resin-base adhesives mixed with hardener or catalyst, that set at room, intermediate, and high temperatures. Results are expressed on the basis of the nonvolatile content of the mixed, liquid adhesive.

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:*

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 terms used in this test method are defined in Terminology D 907.

4. Significance and Use

4.1 It is possible to modify the properties of phenol, resorcinol, and melamine-based adhesives by adding fillers to the adhesive and in doing so affect the way the adhesive performs. This test method makes possible accurate determination of these filler levels for purposes of quality control and characterization of the adhesive.

5. Apparatus

5.1 *Erlenmeyer Flask*, or beaker, of 100-mL capacity.

5.2 *Analytical Balance*, accurate to 0.001 g.

5.3 *Circulating-Air Oven*, capable of maintaining a temperature of 105 ± 1°C (221 ± 1.8°F).

5.4 *Desiccator*, with drying agent and tray.

5.5 *Sintered Glass Crucible*, of coarse or medium porosity.

6. Sampling

6.1 Except in special cases, take a sample from each component of the adhesive. When possible, use a composite sample of each component chosen from three or more separate containers, chosen at random. Also take samples from containers which appear to be nonrepresentative and test such samples separately. Place the samples immediately in clean, dry, airtight containers, filled to prevent excessive air space above the adhesive and transported to the testing laboratory. Take precautions to reduce evaporation or drying of the adhesive to a minimum. Mix the adhesive in the can thoroughly if there is a tendency for the materials to separate before a sample is taken.

6.2 Test three specimens of each sample, unless otherwise required.

7. Procedure

7.1 Mix the adhesive in accordance with the instructions of the manufacturer.

7.2 *Phenol and Resorcinol Base*—Accurately weigh to the nearest 1 mg approximately 2 g of the freshly mixed adhesive (cut film adhesives into small pieces approximately 13-mm (0.5-in) square before weighing) in a 100-mL beaker or Erlenmeyer flask, add 50-mL of absolute methyl alcohol, and stir the mixture thoroughly for 3 min. Filter the mixture promptly through a previously weighed, sintered glass crucible of coarse or medium porosity and wash the contents of the beaker onto the filter with another 50-mL portion of methyl alcohol. Wash the residue in the crucible with at least 50-mL of boiling distilled water, dry to approximately constant weight at 105 ± 1°C (221 ± 1.8°F), cool in a desiccator, and weigh accurately.

7.3 *Melamine Base*—Accurately weigh to the nearest 1 mg approximately 2 g of freshly mixed adhesive in a 100-mL beaker or Erlenmeyer flask, add 50 mL of a cold solution made of 20 parts (by volume) of glacial acetic acid, 20 parts of 95 % ethyl alcohol, and 60 parts of distilled water. Thoroughly stir this mixture for 1 min and add 0.5 g of accurately weighed (to the nearest 1 mg) acid-washed diatomaceous earth which has

[1] This test method is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D 14.30 on Wood Adhesives.

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

***A Summary of Changes section appears at the end of this standard.**

been previously oven-dried overnight at 105 ± 1°C (221± 1.8°F) and cooled in a desiccator. Continue to stir for 2 min and filter the suspension immediately through a previously weighed sintered glass crucible of medium porosity. Wash carefully all the solid material remaining in the beaker onto the filter with the above mentioned solvent. Wash the residue on the filter with a total of at least 30 mL of this solvent, remove the acid with cold distilled water, and finally with 20 mL of 95 % ethyl alcohol. After the preliminary drying by suction, dry the residue at 105 ± 1°C (221 ± 1.8°F) for 16 h, cool in a desiccator, and weigh accurately to the nearest 1 mg.

8. Calculation

8.1 *Phenol and Resorcinol Base Adhesives*—Consider the insoluble residue determined in 7.2 as the filler content of the adhesive. Calculate the residue as a percentage of the nonvolatile content (see 8.2.1) of the adhesive as follows:

$$\text{Filler, \%} = (r/an) \times 100 \quad (1)$$

where:
r = weight of insoluble residue,
a = weight of adhesive sample, and
n = % nonvolatile content/100.

8.2 *Melamine Base Adhesives*—Consider the insoluble residue determined in 7.3 as the filler content of the adhesive. Calculate the residue as the percentage of the nonvolatile content (see 8.2.1) of the adhesive as follows:

$$\text{Filler, \%} = [(r - d)/an] \times 100 \quad (2)$$

where:
d = weight of diatomaceous earth, and other symbols are the same as defined in 8.1.

8.2.1 Determine the nonvolatile content of the adhesive in accordance with Test Method D 1582.

9. Report

9.1 Report the following:

9.1.1 Complete identification of the adhesive tested, including type, catalyst, hardener, mixing proportions of components, source, lot number, condition, etc.

9.1.2 Number of specimens tested, and

9.1.3 Percent of filler in individual specimens, and the average for the sample.

10. Precision and Bias

10.1 The precision and bias statement for this test method has not been determined yet. Results are being expected by April 2003.

11. Keywords

11.1 filler; melamine; non-volatile content; phenol; resorcinol

SUMMARY OF CHANGES

Subcommittee D14.30 has identified the location of selected changes to this standard since the last issue, D 1579 - 97, that may impact the use of this standard.

(1) No significant changes were made during this review.