Designation: D 5062 – 96 (Reapproved 2001)

Standard Test Method for Resin Solution Dilutability by Volumetric/Gravimetric Determination[1]

This standard is issued under the fixed designation D 5062; 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 both volumetric and gravimetric determination of resin solution dilutability which gives a numerical value for the overall solubility of the resin expressed as percent dilutability.

1.2 This test method is applicable only if the test solution is of sufficient clarity to allow accurate visual judgement of the end point and of low enough viscosity for efficient mixing to take place.

1.3 This test method is primarily for, but not limited to, resins used in the printing ink industry.

1.4 The percent solvent tolerance of a resin can be determined using this test method if the solvent in the resin solution and the dilution solvent are the same.

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. Referenced Documents

2.1 *ASTM Standards:*

D 1725 Test Method for Viscosity of Resin Solutions[2]

E 1 Specification for ASTM Thermometers[3]

3. Terminology

3.1 *Definition of Term Specific to This Standard:*

3.1.1 *resin solution dilutability*—the maximum amount of diluent tolerated to reach a defined degree of turbidity; beyond this point, resin precipitation will occur.

4. Summary of Test Method

4.1 A sample of resin solution is weighed into a glass beaker that is placed over a piece of 10 point print (standard newspaper print).

4.2 The dilution solvent is added slowly from a buret until the newsprint can no longer be read (cloud point) when viewed from the top of the beaker.

4.3 The solvent dilutability is calculated by weight, or volume.

5. Significance and Use

5.1 This test method provides a means for resin producers and users as well as solvent and varnish manufacturers to rate various types of resins for solubility by assigning a numerical dilutability value. This percent dilutability value can be used to differentiate resin types for end users and can be utilized as a quality control tool by resin manufacturers.

5.2 When running a series of these tests the same lot or batch of dilution solvent must be used throughout to ensure reproducible results.

6. Apparatus

6.1 *Glass Beaker*, 150-mL (51-mm diameter, 79-mm height).

6.2 *Graduated Buret*, 50 mL.

6.3 *Constant Temperature Water Bath* at 25°C.

6.4 *Thermometer*, 0 to 40°C range with subdivisions of 0.5°C conforming to Specification E 1.

6.5 *Magnetic Stirring Bar and Stirring Plate or Stirring Rod*.

6.6 *Sheet of Newsprint*, with 10-point (Note), No. 31 old-style type, lower case letters 1.5-mm high with normal spacing, upper and lower case with no italicized or bold letters.

NOTE 1—The term point is derived from the American Point System. 72 points = 1 in.

7. Reagents and Materials

7.1 *Resin Solution*—A resin solution prepared in such a way as to provide an accurate percent solids and to have sufficient clarity to allow accurate visual judgment of the cloud point. The solution must also have low enough viscosity at 25°C to allow efficient mixing during the addition of the dilution solvent.

7.2 *Dilution Solvent*—The dilution solvent for this test is typically, but not limited to, a hydrocarbon. The dilution solvent should have minimal loss by evaporation at room temperature, and be agreed upon between the purchaser and the supplier.

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.37 on Ink Vehicles.

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[2] *Annual Book of ASTM Standards*, Vol 06.03.

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

7.3 *Standard Ink Oils*.

8. Procedure

8.1 *Preparation of Resin Solution*:

8.1.1 The resin solution may be prepared by either heating the resin/solvent combination as described in Test Method D 1725, or dispersing using a high-speed mixer such as a blender. Please note results may vary dependent on method of preparation.

8.1.2 The ratio of resin to solvent in the preparation should correspond as much as possible to the intended end use of the material, but should be chosen to avoid difficulty in effecting solution at a low enough viscosity for efficient mixing during dilution.

8.1.3 The precision of weighing the resin and solvent should ensure a maximum deviation of ±1 % in the desired concentration. This percent solids must not be altered during resin solution preparation by solvent loss.

8.2 Weigh a 150-mL glass beaker with the magnetic stirrer bar or stirring rod. Record this tare weight as W_1.

8.3 Accurately weigh 10 ± 0.1 g of the resin solution into the beaker. Record this sample weight as W_2.

8.4 Place the beaker in a 25°C constant temperature bath until the resin solution reaches 25 ± 1°C as measured with the thermometer specified in 6.4.

8.5 Prepare the dilution solvent by bringing it to a temperature of 25 ± 1°C before adding it to the buret.

8.6 Place the beaker over the piece of 10-point print and begin adding solvent slowly (5 to 6 mL/min) while stirring. Ensure that the sample temperature remains at 25 ± 1°C, which may require periodic return of the beaker to the water bath. If the sample has started to form a haze it should not be returned to the water bath.

8.7 Observe the 10-point print from the top of the beaker looking through the solution.

8.8 If the resin solution begins to cloud, add solvent slowly one drop at a time while continuing to mix. Do not return the sample to the water bath at this point. If more than 100 g is required, the resin solution can be considered infinitely soluble unless otherwise specified in an agreement between the purchaser and supplier.

8.9 The end point is reached when the 10-point print cannot be read looking through the solution from the top of the beaker.

8.10 Record the final weight of the beaker and its contents (W_3).

8.11 Record the sample temperature at the end point.

9. Calculation

9.1 Calculate the percent resin solution dilutability, *D*, as follows:

$$D = \frac{W_3 - (W_1 + W_2)}{W_2} \times 100 \quad (1)$$

9.2 Calculate the percent solvent tolerance of the resin, *S*, (percent resin solids at cloud point) as follows:

$$T = \frac{[W_2 \times (S/100)]}{W_2 + [W_3 - (W_1 + W_2)]} \times 100 \quad (2)$$

where S = resin in solution, %.

10. Report

10.1 Report the following information:

10.1.1 The identification of the resins and the solvent in the solution as well as the dilution solvent,

10.1.2 The method used to prepare the resin solution,

10.1.3 The percent solids of the resin solution, and

10.1.4 The percent resin dilutability value or volume of solvent added per weight solution, and operating conditions including air temperature, relative humidity, initial resin solution temperature, final resin solution temperature, initial dilution solvent temperature, and final dilution solvent temperature.

11. Precision

11.1 An interlaboratory study was conducted in which one operator in each of eight laboratories determined the percent dilutability of two common resin solutions and two dilution solvents completed in duplicate on two different days. The pooled within-laboratory standard deviation was found to be 3.25 % of the value measured with 12 df. The between-laboratory standard deviation was 7.63 % of the value measured with 28 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

11.2 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator should be considered suspect if they differ by more than 9.19 % of the value measured.

11.3 *Reproducibility*—Two results, each the mean of duplicate determinations obtained by operators in different laboratories, should be considered suspect if they differ by more than 23.38 % of the value measured.

12. Keywords

12.1 dilutability value; percent dilutability; printing ink resins; resins; resin solution cloud point; resin solution dilutability; solvent tolerance