Designation: D1994 – 07 (Reapproved 2015)

Standard Test Method for Determination of Acid Numbers of Hot-Melt Adhesives[1]

This standard is issued under the fixed designation D1994; 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 determination of acid numbers of hot-melt adhesives.

1.2 This test method is applicable for hot-melt and hot-melt/acid systems that are soluble under the conditions described. The hot melt must also give light- or medium-colored solutions when dissolved. If this is not the case another method must be used.

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

1.4 *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.* Specific caution statements are given in 7.1 and 7.2.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D907 Terminology of Adhesives

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

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

4. Significance and Use

4.1 Acidic hot-melt adhesives are useful in many applications, as the acid functionality can contribute to better substrate wetting and better adhesion to polar, nonporous surfaces.

4.2 Acidic hot-melt adhesives are also quite corrosive to conventional iron and steel adhesive application equipment. The acid number determination will tell an equipment manufacturer if corrosion-resistant equipment for an application should be recommended. The need for corrosion-resistant equipment will vary depending on the acid number of the hot-melt adhesive and type of metal that will be bonded.

5. Reagents

5.1 *Potassium Hydrogen Phthalate*—pH standard, dried and purified (suitable as an acidimetric standard).

5.2 *Potassium Hydroxide/Methanol Titrant* (0.1 *N*)—Dissolve 6 to 7 g of potassium hydroxide (KOH) in methanol, industrial grade, and dilute to 1 L with methanol. This solution should be standardized before use (see Section 6).

5.3 *Phenolphthalein Indicating Solution* (2.5 g/L)—Dissolve 0.5 g of phenolphthalein in 200 mL of methanol.

5.4 *Toluene*—Reagent grade.

5.5 *Deionized Water.*

6. Calibration and Standardization

6.1 *Preparation of Standard Acid Solution*—Dissolve 0.25 to 0.35 g of potassium hydrogen phthalate in 100 mL of deionized water.

6.2 *Titration of Standard Acid Solution*—Add 2 mL of the phenolphthalein indicating solution and titrate the entire standard acid solution using the methanolic KOH solution prepared in 5.2.

6.3 To calculate the normality of the methanolic KOH, use the following formula:

$$N\ KOH = \frac{(\text{g of phthalate}) \times 1000}{204.23\ (\text{mL of KOH solution})}$$

$$= \frac{\text{g of phthalate}}{\text{mL of KOH solution}} \times 4.90$$

7. Procedure

7.1 Dissolve 1.80 to 2.10 g of adhesive in 200 mL of toluene. (Xylene, reagent grade, may provide a safer alternate solvent.) Use a thermocouple or thermometer to monitor the temperature of the solution, and heat the solution (with stirring) to 80 to 90°C (176 to 194°F) to accelerate dissolution.

[1] This test method is under the jurisdiction of ASTM Committee D10 on Packaging and is the direct responsibility of Subcommittee D10.14 on Tape and Labels.

Current edition approved Oct. 15, 2015. Published October 2015. Originally approved in 1991. Last previous edition approved in 2007 as D1994 – 07. DOI: 10.1520/D1994-07R15.

[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.

(**Warning**—Toluene boils at 111°C (232°F), so monitor the solution temperature closely and do not exceed 90°C (194°F).) Continue heating and stirring the solution until the sample dissolves in the toluene (15 to 30 min normally).

7.2 When the sample is dissolved, turn off the heat and continue to stir the solution until it has cooled to 55 to 60°C (131 to 140°F). (**Warning**—Methanol boils at 65°C (149°F) so do not titrate until solution cools to at least 60°C (140°F).) When the solution has cooled to 55 to 60°C titrate immediately as in 6.2 until a light pink color remains after 10 s. A white background under the beaker will make the endpoint easier to see.

7.3 If precipitation of the adhesive occurs during the titration, reheat the flask.

8. Calculation

8.1 Calculate the acid number of the sample with the following equation:

$$\text{acid number (milligrams KOH/g of sample)} = \frac{(\text{millilitres of KOH solution}) \times (\text{KOH normality}) \times 56.1}{\text{grams of adhesive}}$$

8.2 Duplicate analyses should agree within ±5 % of each other. Duplicates found to be outside this range suggest at least a partial insolubility of the adhesive in the solvent or a partial precipitation of the adhesive during the titration.

9. Report

9.1 Report the following information:

9.1.1 Complete identification of the adhesive used.

9.1.2 Conditioning and test conditions: temperature and relative humidity.

9.1.3 Normality of methanolic KOH.

9.1.4 Acid number.

10. Precision and Bias

10.1 No data currently exists on repeatability or reproducibility for the test method. An interlaboratory study using Practice E691 will be conducted to generate data to revise Section 10 to bring it into conformance with the Precision and Bias section in the ASTM Form and Style Manual.

11. Keywords

11.1 acid number; hot-melt adhesive