Designation: D 5546 – 01

Standard Test Method for Solubility of Asphalt Binders in Toluene by Centrifuge[1]

This standard is issued under the fixed designation D 5546; 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 determination of the degree of solubility of asphalt binders in toluene using centrifugal separation. The method is an alternative to Test Method D 2042, and may be preferable to Test Method D 2042 when testing modified asphalt binders.

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 36 Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus)[2]

D 96 Test Methods for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure)[3]

D 2042 Test Method for Solubility of Asphalt Materials in Trichloroethylene[4]

3. Summary of Test Method

3.1 The sample is dissolved in toluene and centrifuged to separate the insoluble material. The insoluble material is dried and weighed.

4. Significance and Use

4.1 This test method is a measure of the solubility of polymer-modified asphalt in toluene. The portion that is soluble in toluene represents the active cementing constituents. Additional tests to characterize the insoluble residue may be conducted. Such tests might include infrared spectroscopy, microscopy, ash content, and so forth.

5. Apparatus and Materials

5.1 *Centrifuge*:

5.1.1 A centrifuge with a swinging bucket rotor capable of spinning two or more filled centrifuge tubes shall be used. The centrifuge shall be capable of delivering a minimum relative centrifugal force (RCF) of 700 at the tip of the tubes, and maintaining this RCF for a minimum of 10 min.

5.1.2 The revolving head, trunnion rings, and trunnion cups, including the cushions, shall be constructed soundly to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall support the tubes firmly when the centrifuge is in motion. The centrifuge shall be enclosed by a metal chamber strong enough to eliminate danger if any breakage occurs. The centrifuge chamber shall be isolated from potential ignition sources.

5.2 *Centrifuge Tubes*—For referee testing, each centrifuge tube shall be a 203-mm cone-shaped tube conforming to the dimensions given in Fig. 1. For routine quality assurance testing, alternate centrifuge tubes may be used. Alternate centrifuge tubes shall have a cone shaped bottom, a length between 150-210 mm, and a nominal capacity of 100 ml. All centrifuge tubes shall be made of thoroughly annealed glass and shall be marked at the 100-ml level.

NOTE 1—Centrifuge tubes meeting the requirements of Test Methods D 96 are widely available, and are suitable for use in this test. The 203-mm centrifuge tubes described in D 96 are suitable for referee testing. The 167-mm centrifuge tubes described in D 96 are suitable for routine quality-assurance testing.

5.3 *Erlenmeyer Flask*—A 125-mL Erlenmeyer flask shall be used.

5.4 *Oven*—An explosion-proof, ventilated oven capable of maintaining a temperature of 130 ± 5°C shall be used to dry the insoluble material.

5.5 *Balance*—A balance or scale with a capacity of 100 g or higher, sensitive to 0.1 mg and accurate within 0.1 % of the load.

NOTE 2—Static electricity may cause unstable mass measurements, due in part to the characteristics of the glass centrifuge tubes. This problem can be minimized by mounting a passive ion source inside the balance draft shield. Passive ion sources are available from electronic balance manufacturers, distributors and service companies.

6. Reagent

6.1 *Toluene*, reagent grade.

[1] This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 on Miscellaneous Asphalt Tests.

Current edition approved Dec. 10, 2001. Published February 2002. Originally published as D 5546 – 94. Last previous edition D 5546 – 94a.

[2] *Annual Book of ASTM Standards*, Vol 04.04.

[3] Discontinued. See *2000 Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 04.03.

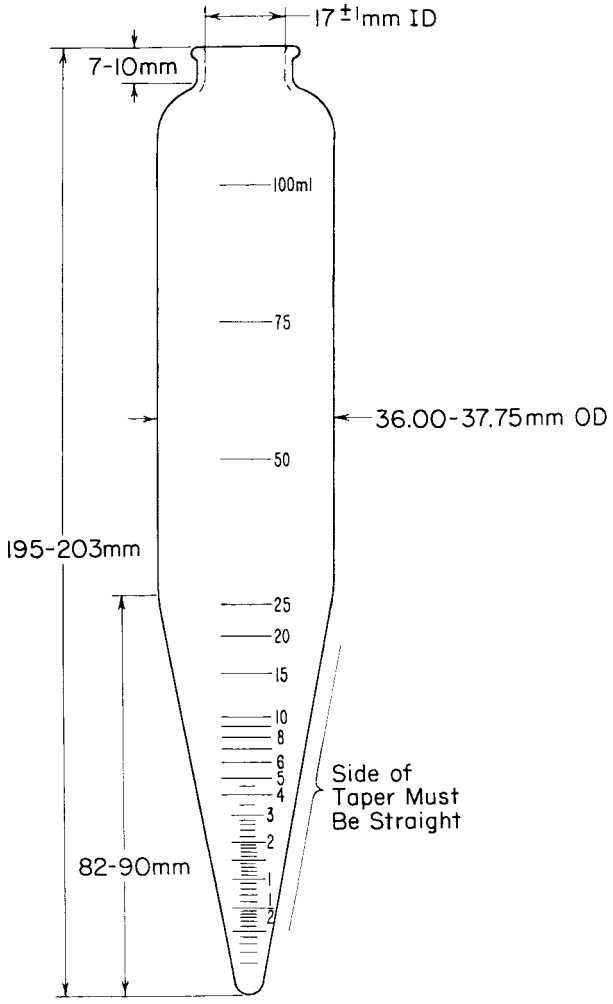

FIG. 1 203-mm (8-in.) Centrifuge Tube

7. Preparation of Centrifuge Tubes

7.1 Place two clean centrifuge tubes in an oven at 130 ± 5°C for 20 min, allow to cool in a desiccator for a minimum of 30 min, and then weigh to the nearest 1 mg. Store in the desiccator until ready for use.

8. Sample Preparation

8.1 If the sample is not fluid, heat to any convenient temperature, but in any case not more than 100°C above the softening point (Test Method D 36). The test may be performed at the laboratory air temperature. For referee tests, however, the flask and sample in solution shall be placed in a water bath maintained at 25 ± 1°C for about 1 h before centrifuging.

9. Procedure

9.1 Transfer approximately 2 g of the sample into a tared 125-mL Erlenmeyer flask. Weigh accurately to the nearest 1 mg. Add 100 mL of the toluene to the container in small portions, with continuous agitation, until all lumps disappear and no undissolved sample adheres to the container. Stopper the flask or otherwise cover the container, and set aside for at least 15 min (see Section 8).

9.2 Transfer the solution to two clean pre-weighed centrifuge tubes. Use additional solvent to ensure that all insoluble matter is transferred to the centrifuge tubes, and fill both tubes to the 100-mL mark. Stopper the tubes tightly and shake well. Carefully remove the stoppers to relieve any pressure in the tubes, and replace the stoppers. Place the tubes in the centrifuge on opposite sides to provide proper balance, and spin for 10 min at a rate sufficient to produce a minimum relative centrifugal force of 700, as calculated in 9.2.1.

9.2.1 Calculate the minimum required speed of the rotating head in revolutions per minute (r/min) as follows:

$$\text{r/min} = 1335\sqrt{rcf/d} \quad (1)$$

where:
rcf = relative centrifugal force (700), and
d = diameter of swing (mm) measured between tips of opposite tubes when in rotating position.

9.3 After centrifuging the samples, inspect each tube and note the presence of any film or particulate matter floating on the surface.

NOTE 3—Any floating material represents insoluble matter with a density less than toluene (0.87 g/cc at 25°C). The only known insoluble materials that will float under these conditions are particles which contain an entrained gas. Any such floating material cannot be reliably quantified by this method, due to potential loss during decanting .

9.4 Decant the liquid from the centrifuge tubes without disturbing the insoluble material. Refill the tubes with toluene, and shake well to wash the insoluble material. Repeat the centrifuging and decanting steps after carefully relieving any built-up pressure in the tubes.

9.5 Place the tubes containing the washed insoluble material in the oven at 130 ± 5°C for at least 20 min. Cool in a desiccator for a minimum of 30 min and weigh. Repeat the drying and weighing until constant weight is obtained.

9.6 Alternate methods of drying the tubes, such as vacuum desiccation, are also acceptable. However, the method described in 9.5 shall be the referee method.

10. Calculation and Report

10.1 Calculate the percentage of the sample soluble in toluene as follows:

$$\text{percent soluble} = 100 - [(A/B) \times 100] \quad (2)$$

where:
A = total weight of insoluble material, and
B = weight of sample.

10.2 Report the results to the nearest 0.01 %.

10.3 If both tubes contained floating insoluble material, as described in 9.3, append the words “or less” to the numeric result, and report that “This sample contained an undetermined quantity of low-density insoluble matter.”

11. Precision and Bias

11.1 Available precision information for this test is summarized in the following table:

Number of Labs	Binder Type	Repetitions	Mean	Std Dev
1	PMA	4	99.71 %	0.20 %
1	Neat + TLA	4	99.19 %	0.23 %
4	Neat	8	99.97 %	0.03 %

Additional precision information is being developed and will

be included in future revisions of this standard.

11.2 Bias has not been determined since there is no accepted reference material suitable for determining the bias for the procedure in this test method.

12. Keywords

12.1 asphalt binder; centrifuge; solubility; toluene