



Standard Test Method for Solubility of Polymer-Modified Asphalt Materials in 1,1,1- Trichloroethane¹

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 in 1,1,1-trichloroethane of polymer-modified asphalt materials having little or no mineral matter. The test method is also acceptable for testing unmodified asphalt cements, as an alternative to Test Method D 2042.

1.2 The test method has been used successfully on polymer-modified asphalts manufactured with neoprene latex, styrene-butadiene latex (SBR), styrene-butadiene copolymer (SB), styrene-butadiene-styrene block copolymer (SBS), and ethylene-vinyl acetate copolymer (EVA). This test method may not be suitable for other polymers, such as polyethylene, which are completely insoluble in 1,1,1-trichloroethane under the conditions of this test. These polymers may float in the centrifuge tubes and be difficult to remove when decanting the solvent.

1.3 The values stated in SI units are to be regarded as the standard. The values given 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.*

2. Referenced Documents

2.1 ASTM Standards:

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

D 2042 Test Method for Solubility of Asphalt Materials in Trichloroethylene³

3. Summary of Test Method

3.1 The sample is dissolved in 1,1,1-trichloroethane 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 1,1,1-trichloroethane. The portion that is soluble in 1,1,1-trichloroethane represents the active cementing constituents. The insoluble portion is not intended to include the polymer. Additional tests to identify the insoluble residue should be conducted if it is suspected that excessive insoluble material is due to the polymer. Such tests might include infrared spectroscopy, ash content, etc.

5. Apparatus and Materials

5.1 Centrifuge:

5.1.1 A centrifuge capable of spinning two or more filled, cone-shaped, 203-mm (8-in.) centrifuge tubes at a speed that can be controlled to yield a relative centrifugal force (rcf) of 700 at the tip of the tubes shall be used.

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 shield or case strong enough to eliminate danger if any breakage occurs.

5.2 *Centrifuge Tubes*—Each centrifuge tube shall be a 203-mm (8-in.) cone-shaped tube, conforming to the dimensions given in Fig. 1, and made of thoroughly annealed glass.

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

5.4 *Oven*—An oven capable of maintaining a temperature of $110 \pm 5^\circ\text{C}$ ($230 \pm 10^\circ\text{F}$) 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.

6. Reagent

6.1 *1,1,1-trichloroethane*, reagent grade.

7. Preparation of Centrifuge Tubes

7.1 Place two centrifuge tubes in an oven at $110 \pm 5^\circ\text{C}$ ($230 \pm 10^\circ\text{F}$) for 20 min, allow to cool in a desiccator for 30 ± 5 min, and then weigh to the nearest 1 mg. Store in the desiccator until ready for use.

¹ This test method is under the jurisdiction of ASTM Committee D-4 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 on Miscellaneous Asphalt Tests.

Current edition approved Sept. 15, 1994. Published November 1994. Originally published as D 5546 – 94.

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

³ *Annual Book of ASTM Standards*, Vol 04.03.

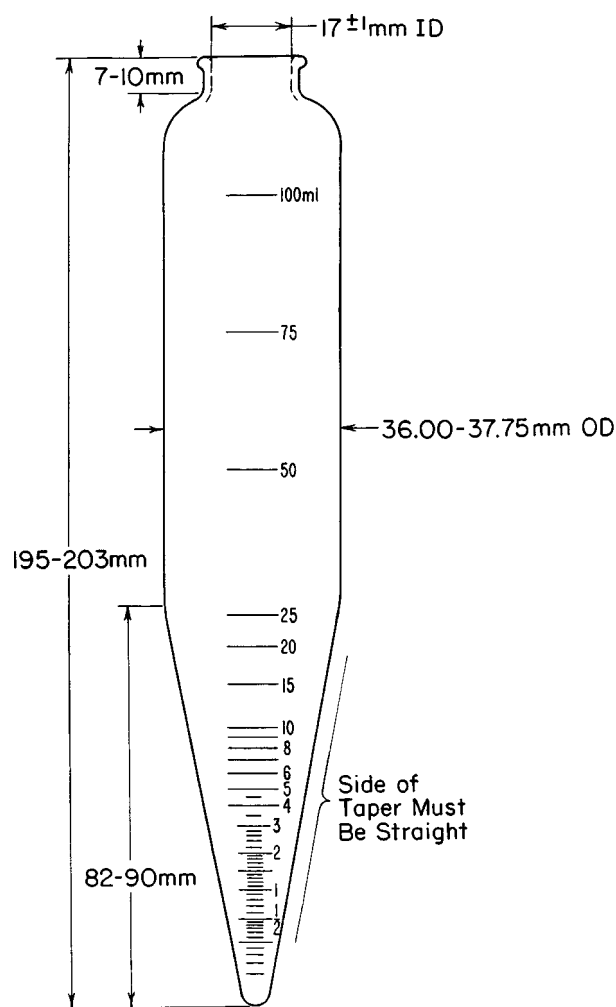


FIG. 1 203-mm (8-in.) Centrifuge Tube

8. Sample Preparation

8.1 If the sample is not fluid, heat to any convenient temperature, but in any case not more than 111°C (200°F) above the softening point (Test Method D 36). The temperature at which this test is run is not normally critical, and it 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 37.8 ± 0.25°C (100 ± 0.5°F) 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 1,1,1-trichloroethane 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 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 relative centrifugal force of 700, as calculated in 9.2.1.

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

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

where:

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

Alternatively, use the following equation:

$$r/\text{min} = 265 \sqrt{rcf/d} \quad (2)$$

where:

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

9.3 Decant the liquid from the centrifuge tubes without disturbing the insoluble material. Refill the tubes with solvent, and shake well to wash the insoluble material. Repeat the centrifuging and decanting steps after carefully relieving any built-up pressure in the tubes. Place the tubes containing the washed insoluble material in an oven at 110 ± 5°C (230 ± 10°F) for at least 20 min. Cool in a desiccator for 30 ± 5 min and weigh. Repeat the drying and weighing until constant weight is obtained.

10. Calculation and Report

10.1 Calculate either the total percentage of insoluble material or the percentage of the sample soluble in 1,1,1-trichloroethane as follows:

$$\text{percent insoluble} = (A/B) \times 100 \quad (3)$$

$$\text{percent soluble} = 100 - [(A/B) \times 100] \quad (4)$$

where:

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

10.2 Report to the nearest 0.01 % for percentages of insoluble material below 1.0. Report to the nearest 0.1 % for percentages of insoluble material above 1.0.

11. Precision and Bias

11.1 The precision of the procedure in this test method for determining the solubility of polymer-modified asphalt materials in 1,1,1-trichloroethane is being determined.

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 cement; polymer-modified asphalt; solubility; trichloroethane

 **D 5546**

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