Designation: D7553 – 15

Standard Test Method for Solubility of Asphalt Materials in N-Propyl Bromide[1]

This standard is issued under the fixed designation D7553; 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 the degree of solubility in n-propyl bromide of asphalt materials. It is intended to be a replacement for Test Method D2042 specifying a solvent that, like trichloroethylene, is safe in that it has no flash point, and has similar solubilizing characteristics to trichloroethylene, but it is not considered to be an ozone depleter banned by the Kyoto Protocol.

NOTE 1—This method is not applicable to tars and their distillation residues or highly cracked petroleum products. For methods covering tars, pitches, and other highly cracked petroleum products, and the use of other solvents, see Test Methods D4, D2318, and D2764.

1.2 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 precaution statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4 Test Method for Bitumen Content

D2042 Test Method for Solubility of Asphalt Materials in Trichloroethylene

D2318 Test Method for Quinoline-Insoluble (QI) Content of Tar and Pitch

D2764 Test Method for Dimethylformamide-Insoluble (DMF-I) Content of Tar and Pitch

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials

D6368 Specification for Vapor-Degreasing Solvents Based on ***normal***-Propyl Bromide and Technical Grade ***normal***-Propyl Bromide

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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

3. Summary of Test Method

3.1 The sample is dissolved in n-propyl bromide and filtered through a glass fiber pad. The insoluble material is washed, dried, and weighed.

4. Significance and Use

4.1 This test method is a measure of the solubility of asphalt in n-propyl bromide. The portion that is soluble in n-propyl bromide represents the active cementing constituents.

NOTE 2—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Specification D3666 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Specification D3666 alone does not completely assure reliable results. Reliable results depend on many factors; following the suggestions of Specification D3666 or some similar acceptable guideline provides a means of evaluating and controlling some of these factors.

5. Apparatus and Materials

5.1 The assembly of a typical filtering apparatus is illustrated in Fig. 1. Details of the component parts are as follows:

5.1.1 *Bitumen Crucible or Gooch Crucible,* glazed inside and outside with the exception of outside bottom surface. The approximate dimensions shall be a diameter of 44 mm at the top tapering to 36 mm at the bottom and a depth of 20 to 30 mm.

5.1.2 *Glass Microfiber Filter Pad,* 32–34 mm diameter, fine porosity, fast flow rate, 1.5 µm particle retention.

5.1.3 *Filter Flask,* heavy-wall, with side tube, 250, 500, or 1000 mL capacity.

5.1.4 *Filter Tube,* 40 to 42 mm inside diameter.

[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 June 1, 2015. Published July 2015. Originally published 2010 as D7553 – 10. DOI: 10.1520/D7553-15.

[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.

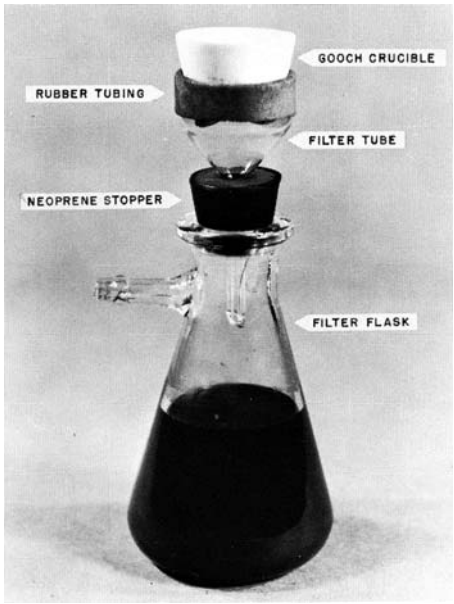

FIG. 1 Filtering Apparatus Assembly

5.1.5 *Rubber Tubing or Adapter,* for holding the crucible on the filter tube.

NOTE 3—Other suitable assemblies permitting vacuum filtration with a crucible may be used.

5.1.6 *Erlenmeyer Flask,* 125 mL.

5.1.7 *Oven,* capable of maintaining a temperature of 110 ± 5°C.

6. Reagent

6.1 *n-Propyl Bromide,* technical grade, conforming to Specification D6368. **Warning:** see Section 7.

7. Hazards

7.1 **Warning:** n-propyl bromide is toxic and should be used only under a hood or with an effective surface exhaust system in a well-ventilated area.

8. Preparation of Crucible

8.1 Place the crucible plus one thickness of the filter pad in an oven at 110 ± 5°C for 15 min, allow to cool in a desiccator for 30 ± 5 min, and then determine the mass to the nearest 0.1 mg. Designate this mass as A. Store in the desiccator until ready for use.

9. Sample Preparation

9.1 If the sample is not fluid, heat to any convenient temperature, but in any case not more than 100°C above the softening point. Normally the temperature at which this test is run is not 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 38.0 ± 0.3°C for 1 h before filtering.

10. Procedure

10.1 Note safety precautions in Section 7. Transfer approximately 2 g of the sample into a tared 125-mL Erlenmeyer flask or other suitable container. Smaller sample sizes may be necessary if more than 0.5 % insoluble material is expected. Allow the sample to cool to ambient temperature and then determine the mass to the nearest 1 mg. Designate this mass as B. Add 100 mL of the n-propyl bromide 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 9).

10.2 Place the previously prepared and weighed crucible in the filtering tube. Wet the filter pad with a small portion of n-propyl bromide and decant the solution through the filter pad of the crucible with or without light suction as may be necessary. When the insoluble matter is appreciable, retain as much of it as possible in the container until the solution has drained through the mat. Wash the container with a small amount of solvent and, using a stream of solvent from a wash bottle, transfer all insoluble matter to the crucible. Use a "policeman" if necessary to remove any insoluble matter adhering to the container. Rinse the policeman and container thoroughly. Wash the insoluble matter in the crucible with solvent until the filtrate is substantially colorless, and then apply strong suction to remove the remaining solvent. Remove the crucible from the tube, wash the bottom free of any dissolved matter, and place the crucible on top of an oven or on a steam bath until all odor of the n-propyl bromide is removed (see safety precautions in Section 7). Place the crucible in an oven at 110 ± 5°C for at least 20 min. Cool the crucible in a desiccator for 30 ± 5 min and determine its mass to the nearest 0.1 mg. Repeat the drying and weighing until constant mass (±0.3 mg) is obtained. Designate this mass as C.

NOTE 4—To obtain precise results, the cooling time in the desiccator must be approximately the same (within ±5 min) after all heatings. For example, if the mass of the empty crucible is determined after a 30-min cooling period in the desiccator, the mass of the crucible containing the insoluble matter should be determined after a 30 ± 5-min cooling period in the desiccator. Either empty crucibles or crucibles containing insoluble matter that have remained in a desiccator overnight should be reheated in an oven for at least 30 min, then allowed to cool for the prescribed period before the mass is determined.

11. Calculation and Report

11.1 Calculate either the total percentage of insoluble matter or the percentage of the sample soluble in the solvent used as follows:

$$\%\ Insoluble = \left(\frac{C - A}{B}\right) \times 100 \quad (1)$$

$$\%\ Soluble = \left(\frac{B - (C - A)}{B}\right) \times 100 \quad (2)$$

where:

A = mass of crucible and filter,
B = mass of sample, and
C = mass of crucible, filter and insoluble material.

11.2 For percentages of insoluble less than 1.0, report to the nearest 0.01 %. For percentages of insoluble 1.0 or more, report to the nearest 0.1 %.

TABLE 1 Solubility (Mass %)

Sample	Average[A] $\bar{X}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
Modified and Unmodified Paving Asphalts, Roofing Asphalts, and Emulsified Asphalt Residues[B]	99.7677	0.0576	0.0921	0.1612	0.2580
Natural Asphalts or High Mineral Filler Asphalts	66.9931	10.1178	16.9740	28.3300	47.5272

[A] The average of the laboratories' calculated averages.
[B]The results from nine different specimen types were averaged to produce these statistics

12. Precision and Bias[3]

12.1 The precision of this test method is based on an interlaboratory study of Test Method D7553 conducted in 2010. Fifteen laboratories participated in this study. Each of the labs was asked to report three replicate test results for ten different sample materials, nine which were an assortment of different sources of modified and unmodified paving asphalts, roofing asphalts, and emulsified asphalt residues, and a single specimen type representing natural asphalts or high mineral filler asphalts. Every "test result" reported represents a single determination or measurement. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D04-1034.

12.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

12.1.1.1 Repeatability limits are listed in Table 1.

12.1.2 *Reproducibility limit "R"*—Two test results shall be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

12.1.2.1 Reproducibility limits are listed in Table 1.

12.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

12.1.4 Any judgment in accordance with statements 12.1.1 and 12.1.2 would have an approximate 95 % probability of being correct.

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method. Therefore, no statement on bias is being made.

12.3 The precision statement was determined through statistical examination of 435 results, from fifteen laboratories, on a total of ten different sample materials from various sources throughout the Western Hemisphere.

13. Keywords

13.1 asphalt; n-propyl bromide; solubility

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D04-1034.