Designation: D 2994 – 98

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Test Methods for Rubberized Tar[1]

This standard is issued under the fixed designation D 2994; 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 These test methods cover the following procedures for rubberized tar:

	Section
Nonimmersed Penetration	6
Immersed Penetration	7
Volume and Weight Change	9
Flow	10
Softening Point	11
Viscosity	12
Water Content	13
Homogeneity	14

1.2 These test methods should not be used in the acceptance or rejection of materials since their precision statements have not been determined.

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 5 Test Method for Penetration of Bituminous Materials[2]

D 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation[3]

D 362 Specification for Industrial Grade Toluene[4]

D 1015 Test Method for Freezing Points of High-Purity Hydrocarbons[3]

D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[3]

D 1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids[3]

D 2398 Test Method for Softening Point of Bitumen in Ethylene Glycol (Ring-and-Ball)[5]

D 2700 Test Method for Knock Characteristics of Motor and Aviation Type Fuels by the Motor Method[6]

E 1 Specification for ASTM Thermometers[7]

3. Significance and Use

3.1 These test methods measure the properties of rubberized tar that determine its suitability as a road binder that is not sensitive to the spillage of fuel oil.

4. Apparatus

4.1 *Melting Unit*— The unit for melting laboratory samples shall be of the double-boiler type, employing a high flash-point oil as the heat transfer medium. The unit shall be equipped with mechanical agitators in the materials chamber and in the oil bath; and two metal thermometers, of the recalibrating dial type, in the range from 10 to 177°C graduated in 5°F (3°C) subdivisions.[8] A melting unit suitable for the purpose is shown in Fig. 1.[9]

4.2 *Oven*, thermostatically controlled, capable of maintaining a temperature of 100 ± 2°F (38± 1°C).

4.3 *Water Bath*, with mechanical stirrer, thermometer, heating element, and thermostatic controls capable of maintaining a water temperature of 77 ± 0.2°F (25 ± 0.1°C).

4.4 *Viscometer*, Brookfield, Model LVF or LVT.

4.5 *Thermometer*, glass, in the range from 30 to 200°C and graduated in 0.5°C subdivisions conforming to the requirements for ASTM Thermometer 16C given in Specification E 1.

4.6 *Metal Panels*— Bright tin-coated panels at least 102 mm wide and 152 mm long. The panels shall be free of any foreign material (dust, oil, etc.), shall not be warped or bent, and shall be discarded after each flow test.

4.7 *Molds*—Durable metal or plastic molds with an opening 60 mm long, 40 mm wide, and 4.2 mm deep.

4.8 *Containers*, approximately 54 mm in diameter and 35 mm deep. (Three-ounce seamless ointment boxes meet these requirements.)

4.9 *Laboratory Balances*, 2-kg and 750-g capacity, sensitive to 0.1 g and 2 mg, respectively.

4.10 *Oil Bath*, with a mechanical stirrer, thermometer, heating element, and thermostatic controls capable of maintaining bath temperatures between 90 ± 3°C and 150± 3°C.

---

[1] These test methods are under the jurisdiction of ASTM Committee D-4 on Road and Paving Materials and are the direct responsibility of Subcommittee D04.43 on Specifications and Test for Tar and Tar Products.

Current edition approved January 10, 1998. Published February 1999. Originally published as D 2994 – 71. Last previous editions D 2994 – 87 (1991).

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

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

[4] *Annual Book of ASTM Standards*, Vol 06.04.

[5] Discontinued; see 1984 *Annual Book of ASTM Standards*, Vol 04.04.

[6] *Annual Book of ASTM Standards*, Vol 05.04.

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

[8] Model BLM-100 Laboratory Melter, without the oil bath stirrer, available from the Berry Corp., Stone Rd., Lexington, KY 40503, has been found suitable for this purpose.

[9] Detailed drawings of the melting unit are available at a nominal charge from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Request Adjunct No. 12-429940-00.

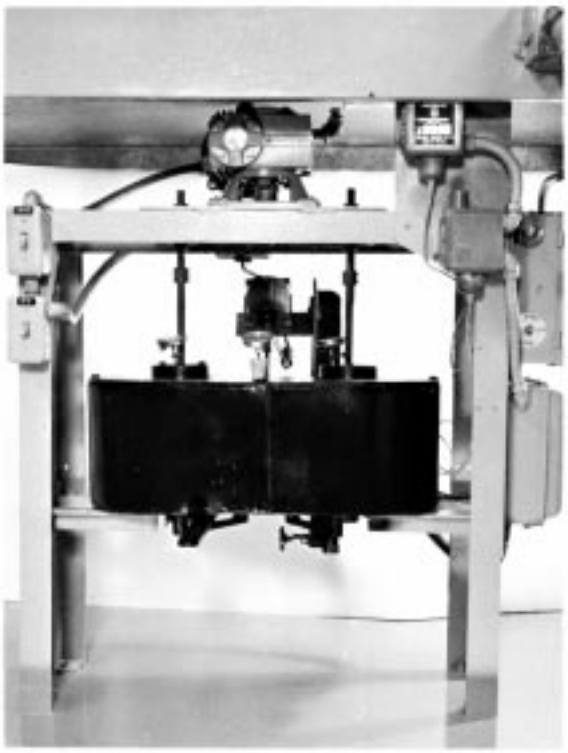

FIG. 1 Rubberized Tar Melting Unit

4.11 *Paint Cans*, 1-pt capacity from which the lips have been removed.

5. Preparation of Samples

5.1 The portion of the sample selected for all of the tests except the water content and homogeneity tests shall weigh approximately 1000 g. Melt the sample in the unit specified in 4.1. Stirring shall be continuous during the melting operation. Melting time shall not exceed 60 min. The temperature of the sample during melting shall not exceed 245°F (118°C) and during pouring of test specimens the temperatures shall be 235 ± 10°F (113 ± 6°C). The temperature of the heat-transfer oil shall not exceed 275°F (135°C).

NONIMMERSED PENETRATION

6. Procedure

6.1 Pour a portion of the sample prepared, as described in 5.1, into a 3-oz container filling it to a depth of about 1 in. (25 mm). Determine the nonimmersed penetration in accordance with Test Method D 5; the test should be conducted at the same time the immersed penetration test is conducted (8.1).

IMMERSED PENETRATION

7. Test Fuel

7.1 The test fuel shall be composed of 70 volume % of *iso*octane and 30 volume % of industrial grade toluene.[10] The *iso*octane shall meet the following requirements:

[10] ASTM Reference Fuel B, which is available from Phillips Petroleum Co., Special Products Div., Bartlesville, OK.

		ASTM Test Method
ASTM motor octane number	100.0 ± 0.1	D 2700
Density at 20°C, g/mL	0.69193 ± 0.00015	D 1217
Refractive index, N_D at 20°C	1.39145 ± 0.00015	D 1218
Freezing point, min, °C	−107.442	D 1015
Distillation: 50 % recovery,° C	99.238 ± 0.025	[A]
Increase from 20 to 80 % recovery, max,° C	0.20	

[A] For the equipment and method used, see *Journal of Research*, Nat. Bureau Standards, JRNBA, Vol 44, No. 3, 1950, pp. 309 and 310 (RP2079).

The toluene shall meet the requirements given in Specification D 362.

8. Procedure

8.1 Immerse a test specimen identical with that used for the nonimmersed test for 18 h in 500 mL of clean test fuel maintained at 100 ± 2°F (38 ± 1°C). The containers for the test fuel and specimen shall be semi-closed, that is, a moderately tight lid with a ⅛-in. (3-mm) round hole cut in the lid to eliminate pressure buildup. More than one specimen of the same material may be immersed in the same container providing the volume of test fuel per specimen is maintained at 500 mL. The overall dimensions of the container shall be such that a minimum of ½in. (13 mm) of test fuel covers the surface of the test specimens. Use a covered constant-temperature water bath conforming to 4.3 to maintain the container, test fuel, and specimens at the required temperature. Immediately after the 18-h immersion period, dry the specimens under a 12-in. (305-mm) diameter electric fan at 77 ± 2°F (25 ± 1°C) for 1 h and conduct the penetration test in accordance with Test Method D 5. Placement of the specimens in relation to the electric fan shall be such that an air velocity from 150 to 500 ft/min over the surface of the specimens is maintained.

VOLUME AND WEIGHT CHANGE

9. Procedure

9.1 Prepare a specimen as described in 5.1. Condition the sample at 77 ± 2°F (25 ± 1°C) for not less than 1½ nor more than 2 h, then weigh the specimen in air to the nearest 0.01 g. Place the specimen in a water bath conforming to 4.3 maintained at a temperature of 77 ± 0.2°F (25 ± 0.1°C) for not less than 1½ nor more than 2 h, then weigh the sample while submerged in water at the same temperature as the water bath. Determine the difference between the weight in air and weight in water and record it as the volume in cubic centimetres of the specimen plus container before immersion in fuel.

9.2 After the weight in water is obtained, dry the specimen with a clean, dry cloth and immerse it as described in 8.1, and then dry the specimen under a 12-in. (305-mm) electric fan at 77 ± 2°F (25 ± 1°C) for 1 h. Reweigh the specimen in air and in water as described in 9.1. Determine the difference between the weight in air and weight in water, and record it as the volume of the sample in cubic centimetres after immersion in fuel.

FLOW

10. Procedure

10.1 Pour a portion of the sample, prepared in accordance

with 4.1, into a mold conforming to 4.7 resting on a bright tin panel conforming to 4.6. Fill the mold with an excess of material. Allow the test specimen to cool at 77 ± 2°F (25 ± 1°C) for at least ½h; then trim the specimens flush with the face of the mold with a heated metal knife or spatula. Remove the mold and place the panel containing the sample in a forced-draft oven conforming to 4.2 maintained at 100 ± 2°F (38 ± 1°C) for 1 h. During the test mount the panel so that the longitudinal axis of the specimen is at an angle of 75± 1° with the horizontal and the transverse axis is horizontal. Measure the change in length in centrimetres of the specimen during the 1-h test period, and report as the flow.

SOFTENING POINT

11. Procedure

11.1 Determine the softening point of the rubberized-tar in accordance with Test Method D 2398.

VISCOSITY

12. Procedure

12.1 Fill the 1-pt paint can specified in 4.11 to within about ½ in. (13 mm) of the top with material prepared in accordance with 5.1. Place the container in the oil bath specified in 4.10 and condition the sample to the approximate temperatures shown in the tabulation. Determine the viscosity of the sample by use of a viscometer conforming to 4.4 at each of the following temperatures, with the spindles and revolutions per minute specified. Take readings 60 s after the spindle is actuated.

Temperature, °C	Spindle No.	Revolutions per minute
90	4	6
105	4	6
120	2	6

12.2 Plot the viscosity in centipoises at each actual test temperature on semi-log graph paper and draw the best possible straight line through the three points. Read the viscosity where the line intercepts the test temperatures indicated above.

WATER CONTENT

13. Procedure

13.1 Determine the water content of the sample in accordance with Test Method D 95.

HOMOGENEITY

14. Procedure

14.1 Melt approximately 1000 g of material as described in 5.1 at a temperature of 295 ± 5°F (146 ± 3°C) and pour through a No. 20 (850-µm) sieve. The temperature of the oil bath in the melter shall not exceed 320°F (160°C) during preparation of the sample for testing. After the sample has passed through the sieve, examine the sieve for material retained on the sieve.

NOTE 1—Preheating the sieve prior to pouring the sample will facilitate passage of the sample through the sieve and identification of material retained on the sieve.

15. Precision and Bias

15.1 Work is underway to develop a precision statement for D 2994. This test method should not be used to accept or reject materials until the precision statement is available.