
Standard Method of Test for

Solubility of Bituminous Materials

AASHTO Designation: T 44-14¹

ASTM Designation: D 2042-01



American Association of State Highway and Transportation Officials
444 North Capitol Street N.W., Suite 249
Washington, D.C. 20001

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1. SCOPE

- 1.1. This method covers the determination of the degree of solubility in trichloroethylene or 1,1,1 trichloroethane of asphalt materials having little or no mineral matter. The portion that is soluble in trichloroethylene or 1,1,1 trichloroethane represents the active cementing constituents.
- 1.2. The values stated in SI units are to be regarded as the standard.

2. REFERENCED DOCUMENTS

- 2.1. *AASHTO Standards:*
- M 231, Weighing Devices Used in the Testing of Materials
 - R 16, Regulatory Information for Chemicals Used in AASHTO Tests
- 2.2. *ASTM Standard:*
- E 177, Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods

3. SUMMARY OF METHOD

- 3.1. The sample is dissolved in trichloroethylene or 1,1,1 trichloroethane and filtered through a filter mat. The insoluble material is washed, dried, and weighed.

4. APPARATUS AND MATERIALS

- 4.1. The assembly of the filtering apparatus is illustrated in Figure 1. Details of the component parts are as follows:

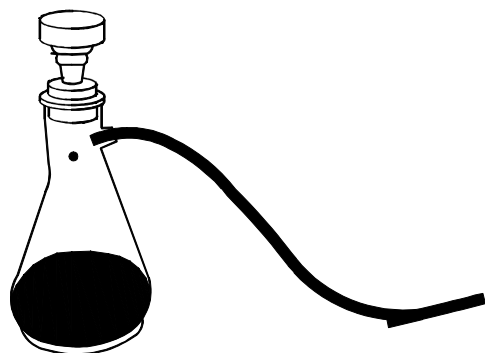


Figure 1—Filtering Apparatus Assembly

- 4.1.1. *Gooch Crucible*—glazed inside and outside with the exception of outside bottom surface. The approximate dimensions shall be a diameter of 44 mm at top, tapering to 36 mm at bottom, and a depth of 24 to 28 mm.
- 4.1.2. *Glass Fiber Pad*—32, 35, or 37 mm in diameter.²
- 4.1.3. *Filter Flask*—heavy-wall, with side tube, 250-mL capacity or larger.
- 4.1.4. *Filter Tube*—40- to 42-mm inside diameter.
- 4.1.5. *Rubber Tubing or Adapter*—for holding the Gooch crucible on the filter tube.
Note 1—Other suitable assemblies permitting vacuum filtration with a Gooch crucible may be used.
- 4.2. *Erlenmeyer Flask*—125 mL, or other suitable container.
- 4.3. *Oven*—capable of maintaining a temperature of $110 \pm 5^\circ\text{C}$ ($230 \pm 9^\circ\text{F}$).
- 4.4. *Desiccator*—of suitable size, charged with an effective desiccant.
- 4.5. *Analytical Balance*—Class A conforming to the requirements of AASHTO Specification M 231.

5. SOLVENT

- 5.1. Technical grade, Type I, trichloroethylene or technical grade 1,1,1 trichloroethane.

6. SAFETY PRECAUTIONS

- 6.1. Trichloroethylene and 1,1,1 trichloroethane are toxic materials and strict adherence to instructions in Material Safety Data Sheets are to be followed. *Caution:* Trichloroethylene and 1,1,1 trichloroethane in the presence of heat and moisture may form acids that are extremely corrosive.

7. PREPARATION OF GOOCH CRUCIBLE

- 7.1. Assemble the filtering apparatus as shown in Figure 1. Place filter pad into the Gooch crucible, moisten the pad with solvent, and seat firmly in the bottom of the crucible with light suction. Dry the crucible and contents at $110 \pm 5^\circ\text{C}$ ($230 \pm 9^\circ\text{F}$) for at least 20 min. Cool the crucible and contents in a desiccator for at least 20 min and determine the mass. Repeat this procedure until constant mass (± 0.3 mg) is obtained. Store in a desiccator until ready for use.

8. SAMPLE PREPARATION

- 8.1. If the sample is not fluid, heat the sample with care to prevent local overheating until it has become sufficiently fluid to pour, occasionally stirring the sample to aid heat transfer and to assure uniformity. Avoid the entrapment of air.

9. PROCEDURE

- 9.1. Note safety precautions in Section 6. Transfer approximately 2 g of the sample into a tared 125-mL Erlenmeyer flask or other suitable container. Allow the container and its contents to cool to ambient temperatures and determine the mass to the nearest 1 mg. Add 100 mL of the trichloroethylene or 1,1,1 trichloroethane to the container, stopper flask and agitate as necessary until the sample is dissolved and no undissolved material adheres to the container.

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 $37.8 \pm 0.25^\circ\text{C}$ ($100 \pm 0.5^\circ\text{F}$) for 1 h before filtering.

- 9.2. Place the previously prepared and tared Gooch crucible in the filtering tube.

Wet the filter pad with a small portion of clean solvent and decant the solution through the filter pad of the crucible with light suction.

When the insoluble matter is appreciable, retain as much of it as possible in the container until the solution has drained through the filter pad. If the filter paper becomes clogged with insoluble material and the solution cannot drain through, the use of additional crucibles is permitted, provided they are conditioned as described in Section 7.1. 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 the container, thoroughly wash the insoluble matter in the crucible with solvent until the filtrate is substantially colorless, then apply strong suction to remove the remaining solvent. Remove the crucible from the tube and wash the bottom free of any dissolved matter. Dry the crucible and contents at $110 \pm 5^\circ\text{C}$ ($230 \pm 9^\circ\text{F}$) for at least 20 min. Cool the crucible and contents in a desiccator for at least 20 min and determine the mass. Repeat this procedure until constant mass (± 0.3 mg) is obtained.

Fiberglass filter pads should be used only one time.

10. CALCULATIONS AND REPORT

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

$$\text{Insoluble, percent} = \frac{C - A}{B} \times 100 \quad (1)$$

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

where:

A = mass of crucible and filter;

B = total mass of sample; and

C = mass of crucible, filter, and insoluble material.

- 10.1.1. Report the percentage of insoluble material to the nearest 0.1 percent.

11. PRECISION

- 11.1. Estimates of standard deviations for this procedure and the criteria for judging the acceptability of results (95 percent confidence level) are indicated in Table 1.

Table 1—Standard Deviations

	Standard Deviations			
	Within-Laboratory Variability ^a		Between-Laboratory Variability ^a	
	Standard Deviation ^b	Repeatability ^c	Standard Deviation ^b	Reproducibility ^d
Asphalts solubility more than 99 percent (trichloroethylene or 1,1,1 trichloroethane)	0.035	0.10	0.090	0.26

^a For definition of terms and recommended use of precision indexes, see the Recommended Practice for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material (ASTM E 177).

^b The standard deviations shown represent the estimated standard deviation of the measurement process for the stated conditions. They are calculated by multiplying the standard deviations of the applicable data by the factor:

$$1 + \frac{1}{4(N-1)}$$

where *N* is the number of tests in the set of data.

^c Two results obtained by an operator on the same sample should be considered suspect if they differ by more than the stated amount. As defined in ASTM E 177, this is the “difference two-sigma” limit for single-laboratory-operator-machine-multiday precision.

^d Two results obtained by operators in different laboratories should be considered suspect if they differ by more than the stated amount. As defined in ASTM E 177, this is the “difference two-sigma” limit for multilaboratory-operator-machine-day precision.

The estimates of standard deviation are based on the following:

	Asphalts
Materials	4
Replications	3
Solvents	4
Laboratories	26
Degrees of freedom:	
Within-laboratory variability	159
Between-laboratory variability	81
Standard deviation(s) of data:	
Within-laboratory variation	0.035
Between-laboratory variation	0.090

11.2. AASHTO Materials Reference Laboratory (AMRL) data for T 44 and ASTM D 2042 were analyzed in 2001. These data represent approximately 13,200 repetitions of the test conducted on 132 samples having solubility values between 99.5 percent and 100 percent. For samples within this range, the analysis suggests the following precision values are appropriate:

$$\text{Multilaboratory standard deviation (1s)} = 0.01 + (0.75 \times \% \text{ insoluble})$$

$$\text{Single-operator standard deviation (1s)} = 0.01 + (0.25 \times \% \text{ insoluble})$$

¹ Similar, but not technically identical to ASTM D 2042-01.

² Whatman Grade 934 AH glass microfiber filter pad, available from Reeve Angel & Co., Clifton, NJ, has been found suitable.