Designation: D 2042 – ~~97~~01

ASTM
INTERNATIONAL

Standard Test Method for
Solubility of Asphalt Materials in Trichloroethylene[1]

This standard is issued under the fixed designation D 2042; 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 trichloroethylene of asphalt materials having little or no mineral matter.

NOTE 1—Use of carbon disulfide, carbon tetrachloride, and benzene has been discontinued in this method because of the safety hazards involved. 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 D 4, ~~D 2317,~~ D 2318, and D 2764.

1.2 *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:*
D 4 Test Method for Bitumen Content[2]

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

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D 2317̶8 Test Method for ̶B̶e̶ Quinz̶eoline-Insoluble (̶BQI) Content of Tar and Pitch[3]
D ̶2̶3̶1̶8̶ ̶T̶e̶s̶t̶ 2764 Test Method for ̶Q̶u̶i̶n̶o̶l̶i̶n̶e̶-̶I̶n̶s̶o̶l̶u̶b̶l̶e̶ ̶(̶Q̶I̶)̶ ̶C̶o̶n̶t̶e̶n̶t̶ ̶o̶f̶ ̶T̶a̶r̶ ̶a̶n̶d̶ ̶P̶i̶t̶c̶h̶[2]
̶D̶ ̶2̶7̶6̶4̶ ̶T̶e̶s̶t̶ ̶M̶e̶t̶h̶o̶d̶ ̶f̶o̶r̶ Dimethylformamide-Insoluble (DMF-I) Content of Tar and Pitch[4]
E 177 Practice for the Use of the Terms Precision and Bias in ASTM Test Methods[5]
2.2 AASHTO Standard:
T44 Solubility of Bituminous Materials in Organic Solvents[6]

3. Summary of Method

3.1 The sample is dissolved in trichloroethylene 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 trichloroethylene. The portion that is soluble in trichloroethylene represents the active cementing constituents.

5. Apparatus and Materials

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

5.1.1 *Gooch Crucible*, glazed inside and outside with the exception of outside bottom surface. The approximate dimensions shall be a diameter of 44 mm at the top, tapering to 36 mm at the bottom, and a depth of 24-28 mm.

5.1.2 *Glass Fiber Pad*, 3.2-cm.[7]

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

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

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

Note 2—Other suitable assemblies permitting vacuum filtration with a Gooch crucible may be used.

5.1.6 *Erlenmeyer Flask*, 125 mL.

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

[2] *Annual Book of ASTM Standards*, Vol 04.04.
[3] ̶D̶i̶s̶c̶o̶n̶t̶i̶n̶u̶e̶d̶—̶S̶e̶e̶ *1981 Annual*
[3] *Annual Book of ASTM Standards*, ̶P̶a̶r̶t̶ ̶1̶5̶.̶ Vol 05.01.
[4] *Annual Book of ASTM Standards*, Vol ̶1̶4̶.̶0̶2̶.̶ 05.02.
[5] ̶W̶h̶a̶t̶m̶a̶n̶ ̶G̶r̶a̶d̶e̶ ̶9̶3̶4̶ ̶A̶H̶ ̶g̶l̶a̶s̶s̶ ̶m̶i̶c̶r̶o̶f̶i̶b̶e̶r̶ ̶f̶i̶l̶t̶e̶r̶ ̶p̶a̶d̶,̶ ̶a̶v̶a̶i̶l̶a̶b̶l̶e̶ ̶f̶r̶o̶m̶ ̶R̶e̶e̶v̶e̶s̶ ̶A̶n̶g̶e̶l̶ ̶a̶n̶d̶ ̶C̶o̶.̶,̶ ̶C̶l̶i̶f̶t̶o̶n̶,̶ ̶N̶J̶,̶ ̶h̶a̶s̶ ̶b̶e̶e̶n̶ ̶f̶o̶u̶n̶d̶ ̶s̶u̶i̶t̶a̶b̶l̶e̶.̶
[5] *Annual Book of ASTM Standards*, Vol 14.02.
[6] Available from AASHTO, 444 N. Capitol St., NW, Suite 249, Washington, DC 20001.
[7] Whatman Grade 934 AH glass microfiber filter pad, available from Reeves Angel and Co., Clifton, NJ, has been found suitable.
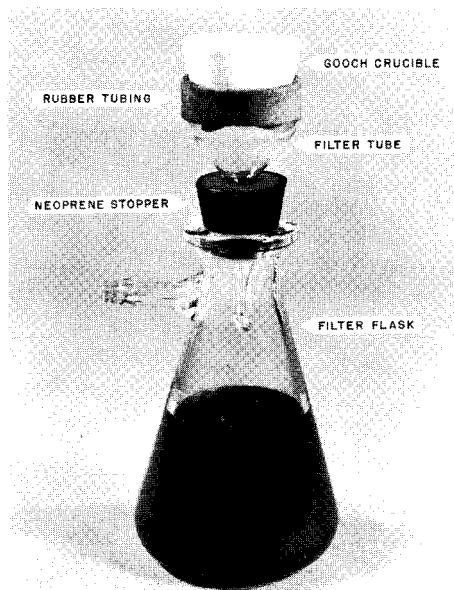
FIG. 1 Filtering Apparatus Assembly

6. Reagent

6.1 *Trichloroethylene*, technical grade.

7. Safety Precautions

7.1 Trichloroethylene is toxic, and good ventilation should be provided. It is more flammable than carbon tetrachloride.

8. Preparation of Gooch Crucible

8.1 Place the Gooch crucible plus one thickness of the glass fiber pad in an oven at 110 ± 5°C for 15 min, allow to cool in a desiccator, 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 111°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 37.8 ± 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. 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 trichloroethylene 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).~~ 7).

10.2 Place the previously prepared and weighed Gooch crucible in the filtering tube. Wet the glass fiber pad with a small portion of trichloroethylene and decant the solution through the glass fiber 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, 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 trichloroethylene is removed (see safety precautions in Section 6). 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 3—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 dessicator, 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:

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

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

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 %.

12. Precision

12.1 The following precision statement was formulated when it was permissible to use carbon disulfide, carbon tetrachloride, and benzene, as well as trichloroethylene and before the substitution of the glass fiber pad for asbestos. It is included in the test method as a guide ~~only. A precision statement based on the use of trichloroethylene and the glass fiber pad will be prepared.~~ only.

12.2 Estimates of standard deviations ($\sigma\rho'$) for this procedure and the criteria for judging the acceptability of results (95 % confidence level) are as follows: ~~The estimates of standard deviation are based on the following:~~

	Within-Laboratory Variability[A]		Between-Laboratory Variability[A]	
	Standard Deviation[B]	Repeat-ability[C]	Standard Deviation[B]	Reproduc-ibility[D]
Asphalts, solubility more than 99 % (applicable when either carbon disulfide, carbon tetrachloride, trichloroethylene, or benzene are used)	0.035	0.10	0.090	0.26

[A] For definitions of terms and recommended use of precision indexes, see Practice E 177.

[B] The standard deviations shown (σ_p') 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 of the same sample should be considered suspect if they differ by more than the stated amount. As defined in Practice E 177, this is the "difference two-sigma" limits 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 Practice E 177, this is the" difference two-sigma" limits for multilaboratory-operator-machine-day precision.

12.3 The estimates of standard deviation are based on the following:

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

12.4 AASHTO Materials Reference Laboratory (AMRL) data for Test Method D 2042 (and its AASHTO equivalent standard, T44) were analyzed in 2001. These data represent approximately 13 200 repetitions of the test, conducted on 132 samples having solubility values between 99.5 % and 100.0 %. For samples within this range, the analysis suggests the following precision values are appropriate:

	Within-Laboratory Variability[A] Standard Deviation[B] = 0.01 + (0.75× % Insoluble)	Repeatability[C]	Between-Laboratory Variability[A] Standard Deviation[B]	Reproducibility[D]
Multilab Standard Deviation (1S)	= 0.01 + (0.75× % Insoluble)			
Single-Operator Standard Deviation (1S)	Asphalts, solubility more than 99 % (applicable when either carbon disulfide, carbon tetrachloride, trichloroethylene, or benzene are used)	0.035	0.10	0.090	0.26
Single-Operator Standard Deviation (1S)	Asphalts, solubility more than 99 % (applicable when either carbon disulfide, carbon tetrachloride, trichloroethylene, or benzene are used)	0.035	0.10	0.090	0.26 = 0.01 + (0.25 × % Insoluble)

A For definitions of terms and recommended use of precision indexes, see Practice E 177.

[B] The standard deviations shown (σ_{P}') 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 of the same sample should be considered suspect if they differ by more than the stated amount. As defined in Practice E 177, this is the " difference two-sigma" limits 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 Practice E 177, this is the " difference two-sigma" limits for multilaboratory-operator-machine-day precision.

13. Keywords

13.1 asphalt; Gooch crucible; solubility; trichloroethylene