

EXPERIMENT NO: 13
SOLUBILITY OF BITUMINOUS MATERIAL
(AASHTO DESIGNATION : T 44-93
ASTM DESIGNATION : D 2042-81)



1. SCOPE

1.1 This method covers the determination of the degree of solubility in trichloroethylene or 1, 1, 1 trichloroethylene of asphalt minerals having little or no mineral matter.

2. SUMMARY OF METHOD

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

3. SIGNIFICANCE AND USE

3.1 This method is a measure of the solubility of asphalt in trichloroethylene or 1, 1, 1 trichloroethylene. The portion that is soluble in trichloroethylene or 1, 1, 1 trichloroethylene represents the active cementing constituents.

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:

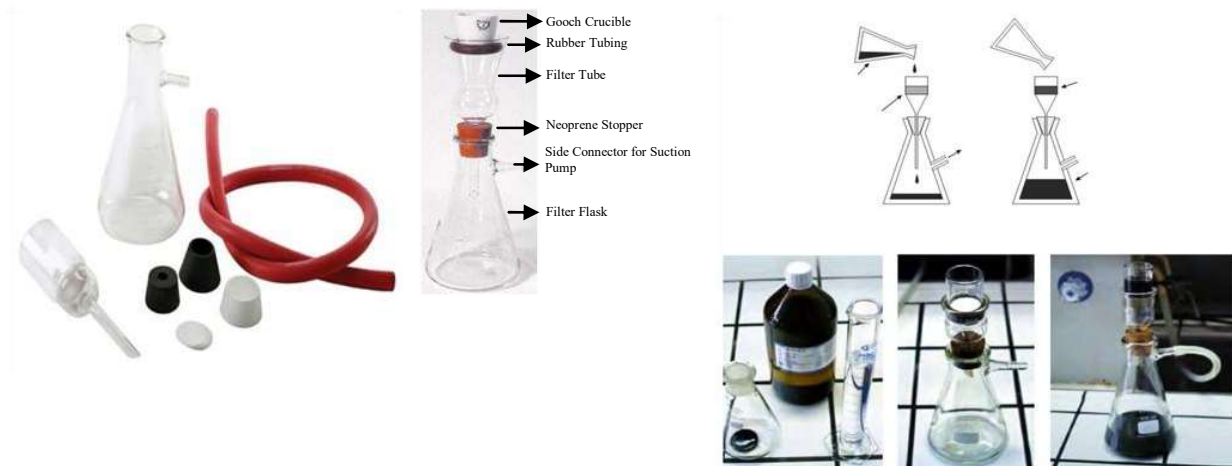


Figure 13.1 Component parts of the filtering apparatus

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 28 mm.

4.1.2 Glass Fiber Pad of 3.7 cm in diameter.

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 an AASHTO Specification M 231.

5. SOLVENT

5.1 Technical grade, Type 1, Trichloroethylene or technical grade 1,1,1 trichloroethylene.

6. SAFETY PRECAUTIONS

6.1 "Trichloroethylene and 1,1,1 trichloroethylene are toxic material and strict adherence to instructions in Material Safety Data Sheets are to be followed". Caution: Trichloroethylene and 1,1,1 trichloroethylene 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. Place filter pad into the gooch crucible, moisten the pad with solvent and seat firmly in the bottom of the crucible with light suction. Place in an oven at $110 \pm 5^{\circ}\text{C}$ ($230 \pm 9^{\circ}\text{F}$) for at least 20 minutes, cool in desiccator and weigh to the nearest 0.1 mg. Repeat the drying and weighing 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 to any convenient temperature, but in any case not more than 100°C or 180°F above the softening point.

9. PROCEDURE

9.1 Note safety precautions in Section 6. Transfer approximately 2 g of the sample into a tared 25 ml Erlenmeyer flask or other suitable container. Allow the container and its contents to cool to ambient temperatures and weigh to the nearest 1 mg. Add 100 ml of the trichloroethylene or 1,1,1 trichloroethylene to the container in small portions with continuous agitation until all lumps disappear and no undissolved sample adhere to the container. Stopper the flask or otherwise cover the container and set aside for at least 15 minutes.

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 hour before filtering.

9.2 Place the previously prepared and weighed 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. 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, and then apply strong suction to remove the remaining solvent. Remove the crucible from the tube and wash the bottom free of any dissolved matter. Place in an oven at $110 \pm 5^\circ\text{C}$ ($230 \pm 9^\circ\text{F}$), for at least 20 min. Cool in a desiccator and weigh to the nearest 0.1 mg. Repeat the drying and weighing until constant weight (± 0.3 mg) is obtained. Fiber glass 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:

$$\text{Insoluble, percent} = A/B \times 100$$

$$\text{Soluble, percent} = 100 - (A/B \times 100)$$

Where,

A = total weight insoluble, and

B = total weight of sample.

10.1.1 For percentage of insoluble less than 1.0, report to the nearest 0.01 percent; for percentage of insoluble 1.0 or more report to the nearest 0.1 percent.

11. PRECISION

11.1 estimates of standard deviation for this procedure and the criteria for judging the acceptability of results (95 percent confidence level) are indicated in the table entitled "Standard Deviations".

The estimates of standard deviation are based on the following:

	Asphalts
Materials	4
Replications	3
Solvents	4
Laboratories	26
Degree of freedom:	
within-laboratory	159
between Laboratory variability	81
Standard deviation (S) of data:	
within-laboratory variability	0.035
between laboratory variability	0.090