Designation: D4 – 86 (Reapproved 2010)

Standard Test Method for Bitumen Content[1]

This standard is issued under the fixed designation D4; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.
Although this test method was withdrawn by the Society in June 1966 at the request of Committee D04, the Committee on Standards has approved its reinstatement under the jurisdiction of Committee D08 on the basis that Procedure No. 2 still applies to their interest. Procedure No. 1 has been replaced by Method D 2042, Test for Solubility of Asphalt Materials in Trichloroethylene.

1. Scope

1.1 This test method covers the determination of bitumen content in materials containing at least 25 % bitumen. Bitumen content may usually be expeditiously and accurately determined by Procedure No. 1, Section 7. However, some bituminous materials containing finely divided mineral matter may clog the filter or the mineral residue may not be easily retained, in which cases Procedure No. 2, Section 8, shall be followed.

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

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 and regulatory limitations prior to use.* For specific precautionary information, see Section 4.

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D370 Practice for Dehydration of Oil-Type Preservatives
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

3. Apparatus, Reagents, and Materials

3.1 *Filtering Crucible,* porcelain, high-form, 25- to 40-mL capacity, with fine-porosity bottom, 7-µm maximum porosity.

NOTE 1—Selas grade 01, size FC 30 or FC 40, or equivalent. Available from various laboratory supply houses.

3.2 *Celite Analytical Filter Aid (CAFA)*, dried to constant mass at 105°C and stored in a tightly stoppered container.

NOTE 2—Use Celite 211 or Celite 505.

3.3 *Beakers*—One 30-mL beaker, Griffin low-form, and one 150-mL beaker, Griffin low-form.

3.4 *Carbon Disulfide,* cp.

NOTE 3—For filled asphaltic materials, trichloroethylene may be directly substituted for carbon disulfide if desired. However, carbon disulfide shall be used in referee testing.

3.5 *Filtering Flask,* with crucible adapter.

3.6 *Drying Oven.*

3.7 *Bunsen Burner or Muffle Furnace.*

3.8 *Suction Pump.*

3.9 *Analytical Balance.*

3.10 *Desiccator.*

3.11 *Evaporating Dish.*

3.12 *Watch Glasses.*

4. Safety Precautions

4.1 Carbon disulfide is extremely flammable. The vapor will often ignite spontaneously on contact with a hot surface such as a hot plate, oven, or radiator. When using this solvent, be sure that the filtration is conducted under a hood and well away from flame or other sources of heat. Before placing crucibles or other containers in the oven, all traces of carbon disulfide should be removed (as indicated by the disappearance of all odor). Otherwise, the vapors will ignite. Carbon disulfide is also very irritating to the skin and direct contact should be avoided.

5. Preparation of Sample

5.1 The sample shall be representative, and if it contains more than 2 % of water it shall be dehydrated in accordance with Test Method D370. If the material is hard and brittle, it may be ground and dried at a temperature below the temperature of volatilization of the material.

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[1] This test method is under the jurisdiction of ASTM Committee D08 on Roofing and Waterproofing and is the direct responsibility of Subcommittee D08.03 on Surfacing and Bituminous Materials for Membrane Waterproofing and Built-up Roofing.

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[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.

6. Crucible Preparation

6.1 If the crucible, after thorough cleaning (see 6.2), has been used for less than six determinations, clean it as follows: Remove the CAFA mat completely, wash the crucible with distilled water, dry, and ignite in a muffle furnace for 1 h at about 800°C. Cool the crucible slowly by placing it in a drying oven for 1 h after removal from the furnace to prevent cracking and place it in a desiccator while still warm.

6.2 After the crucible has been used for six determinations, remove any residual ash from pores in the filtering area by boiling in 1 + 1 hydrochloric acid. Then boil the crucible in distilled water, thoroughly back wash with distilled water, dry, and ignite as in 6.1.

7. Procedure No. 1

7.1 Weigh approximately 2 g of the sample (size sample to obtain 0.1 to 0.3 g of insoluble matter) into a tared 150-mL beaker and add 100 mL of carbon disulfide to the beaker in small portions, with continuous agitation, until all lumps disappear and nothing adheres to the beaker. Cover the beaker with a watch glass and set it aside for 15 min.

7.2 Transfer 0.45 to 0.55 g of dry CAFA to a clean, dry, filtering crucible. Distribute the Celite evenly over the bottom. Immediately weigh and record the weight of crucible plus Celite.

7.3 Decant the carbon disulfide solution carefully through the CAFA mat (previously wetted with carbon disulfide) in the prepared crucible, with or without light suction as may be necessary, retaining as much sediment as possible in the beaker until the solution has drained through the mat. Wash the beaker with a small amount of carbon disulfide and transfer all sediment from the beaker to the mat. Do not allow the mat to dry out at any time until filtration is complete. Wash with carbon disulfide until the filtrate is substantially colorless, then apply strong suction to remove the remaining carbon disulfide. Remove the crucible from the adapter, wash the bottom free of any bitumen, and place the crucible on top of the oven until practically all of the carbon disulfide has been driven off. (**Warning—**see 4.1). Place in the oven at 110 ± 10°C for at least 20 min. Cool in a desiccator and weigh.

7.4 If insoluble matter adheres to the beaker, dry the beaker in the oven at 110°C and weigh. Add the mass of this adherent material as a correction to the mass of the insoluble matter in the crucible.

7.5 If a determination of mineral matter is required, ignite the crucible from 7.3 until it glows with a dull red color. Hold at this temperature until all carbon has been consumed; then raise the temperature to produce a bright red. After cooling, add to the residue about five times its mass of saturated ammonium carbonate solution and let it digest for 1 h at room temperature in a covered beaker; then dry in an oven at 105 to 110°C to constant mass (Note 4). Add the corrections described in 7.4 and, if needed, 7.6 to the mass of mineral matter in the crucible.

NOTE 4—In the event that water-soluble salts insoluble in carbon disulfide are present, the amount of these salts may be determined in accordance with the procedure described in the 1937 Report of Committee D04 on Road and Paving Materials.[3]

7.6 If there is any question involving the amount of mineral matter that may have passed through the filter, a correction may be determined as described in 8.5.

8. Procedure No. 2

8.1 Weigh approximately 2 g of the sample into a tared 50-mL beaker. Add to the beaker about 0.5 g, weighed to the nearest 0.001 g, of freshly dried CAFA. Cover with about 25 mL of carbon disulfide and stir the filter aid into the liquid. Let stand, covered with a watch glass, at least 1 h, stirring occasionally in order to dissolve the sample completely.

8.2 Transfer 0.45 to 0.55 g of dry CAFA to a clean, dry, filtering crucible. Distribute the Celite evenly over the bottom. Immediately weigh and record the mass of crucible plus Celite.

8.3 Immediately before starting the filtering process, stir the filter aid into the liquid. Wet the CAFA mat in the crucible with carbon disulfide. Pour the solution from the beaker onto the pad, filling the crucible to the top. Apply light suction and, as the liquid filters through, pour the remaining contents of the 50-mL beaker into the crucible. Wash the beaker with a small amount of carbon disulfide and transfer all sediment from the beaker to the mat. Do not allow the CAFA mat to dry out until filtration is complete. Wash with carbon disulfide until the filtrate is substantially colorless, then apply strong suction to remove the remaining carbon disulfide. Remove the crucible from the adapter, wash the bottom free of any bitumen, and place on top of the oven until practically all of the carbon disulfide has been driven off. Place in the oven at 110 ± 10°C for at least 20 min. Cool in a desiccator and weigh.

8.4 If insoluble matter adheres to the beaker, dry the beaker in the oven at 110°C and weigh. Add the mass of this adherent material as a correction to the mass of the insoluble matter in the crucible.

8.5 Ignite the evaporating dish to a dull red heat, cool in a desiccator, and weigh. Pour the filtrate from the filtering flask into the dish and wash the flask thoroughly with carbon disulfide, putting these washings into the dish also. Burn off the carbon disulfide in a hood and ignite the residue until no black or glowing spots remain. Extreme care must be exercised during the ignition to prevent the light mineral matter from being blown out of the dish. Cool in a desiccator and weigh immediately. This mass shall be added as a correction to the weight of the insoluble matter in the crucible.

8.6 If a determination of mineral matter is required, heat the crucible and its contents from 8.3 until they glow with a dull red color. Hold at this temperature until all carbon has been consumed; then raise the temperature to produce a bright red. After cooling, add to the residue about five times its mass of saturated ammonium carbonate solution and let it digest for 1 h at room temperature in a covered beaker; then dry in an oven at 105 to 110°C to constant mass (Note 4). Add the corrections described in 8.4 and 8.5 to the mass of mineral matter in the crucible.

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[3] *Proceedings*, ASTM, Vol 37, Part I, 1937, p. 395.

8.7 The mass of additional filter aid used must be subtracted from the total mass of insoluble residue and also from the total mass of mineral matter in the crucible in order to obtain net mass.

9. Calculation

9.1 Calculate the bitumen content, as follows:

$$\%\ \text{Bitumen}\ (\%\ \text{Soluble in Carbon Disulfide}) = 100 - \left(\frac{A}{B} \times 100\right) \quad (1)$$

where:

A = total mass of insoluble material present, g, and
B = total mass of water free sample used, g.

9.1.1 Report to the nearest 0.1 %.

10. Precision

10.1 Estimates of standard deviations (σ_ρ') for this procedure and the criteria for judging the acceptability of results (95 % confidence level) are listed in Table 1.

11. Keywords

11.1 bitumen; coal tar; solubility

TABLE 1 Estimates of Standard Deviations

	Within-laboratory variability[A]		Between-laboratory variability[A]	
	Standard deviation, σ_ρ'[B]	Repeatability[C]	Standard deviation σ_ρ'[B]	Reproducibility[D]
Tars, liquid grades (applicable when carbon disulfide is used)	0.11	0.31	0.22	0.61
Tars, semisolid (applicable when carbon disulfide is used)	0.17	0.48	0.83	2.34

	Liquid Tars	Semisolid Tars
Materials	1	1
Replications	3	3
Solvents	1	1
Laboratories	8	8
Degrees of freedom:		
Within-laboratory variability	14	15
Between-laboratory variability	6	7
Standard deviation (*S*) of data		
Within-laboratory variation	0.103	0.167
Between-laboratory variation	0.208	0.798

[A] For definitions of terms and recommended use of precision indexes, see Practice E177. The estimates of standard deviation are based on the following:

[B] The standard deviations shown ($\sigma\rho'$) 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 Practice E177, 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 E177 this is the "difference two-sigma" limits for multilaboratory-operator-machine-day precision.

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