Designation: D 453 – 94 (Reapproved 2000)$^{\epsilon 1}$

Standard Test Method for Tar Acids in Creosote-Coal Tar Solutions[1]

This standard is issued under the fixed designation D 453; 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 Department of Defense.

ϵ^1 NOTE—A keyword was added editorially in March 2000.

1. Scope

1.1 This test method covers the determination of tar acids in creosote and creosote-coal tar solution.

1.1.1 Test Method D 38 covers the sampling of wood preservatives prior to testing.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 38 Test Methods for Sampling Wood Preservatives Prior to Testing[2]
D 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation[3]
D 246 Test Method for Distillation of Creosote and Creosote-Coal Tar Solutions[2]
D 836 Specification for Industrial Grade Benzene[4]
D 843 Specification for Nitration Grade Xylene[4]
E 1 Specification for ASTM Thermometers[5]

2.2 *British Wood Preserving Association Standard:*
British Standard 144 on Wood Preservation[6]

3. Summary of Test Method

3.1 Acoustic solution is separated and acidified with H_2SO_4 to liberate tar acids that are measured in a graduated funnel.

4. Significance and Use

4.1 This test method covers the determination of tar acid content of creosote coal tar solutions so that comparison can be made with existing international standards, for example, British Standard 144.

5. Apparatus

5.1 *Creosote Distillation Equipment,* as described in Test Method D 246.

5.2 *Separatory Funnels*—Two Squibb-type separatory funnels with stoppers, 250-mL capacity.

5.3 *Type II Separatory Funnel*—The Type II glass separatory funnel shall have a total capacity of not less than 260 mL and not more than 650 mL, and shall conform to the requirements shown in Fig. 1 except that the bulb at the top may be replaced by a 500-mL Squibb-type bulb. The capacity of the lower bulb from the stopcock to the first graduation mark shall be 65.0 mL, and above this mark the stem shall be graduated accurately to a total capacity of 100.0 mL in 0.2-mL increments. The graduation marks shall be numbered for each 2 mL.

5.4 *Thermometer*—An ASTM Low Softening Point Thermometer having a range from − 2 to 80°C conforming to the requirements for Thermometer 15C as described in Specification E 1 or other thermometer of suitable range and precision.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Sodium Hydroxide Solution* (20 %)—Mix 200 g of 50 % sodium hydroxide (NaOH) solution with 300 g of water.

NOTE 1—Carbonate-free (clear) 50 % NaOH solution may be purchased and is convenient and safe to use.

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[1] This test method is under the jurisdiction of ASTM Committee D07 on Wood and is the direct responsibility of Subcommittee D07.06 on Treatments for Wood Products.

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[2] *Annual Book of ASTM Standards*, Vol 04.10.

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

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

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

[6] Available from Premier House, 150 South Hampton Row, London, England, WUC 1BSAL.

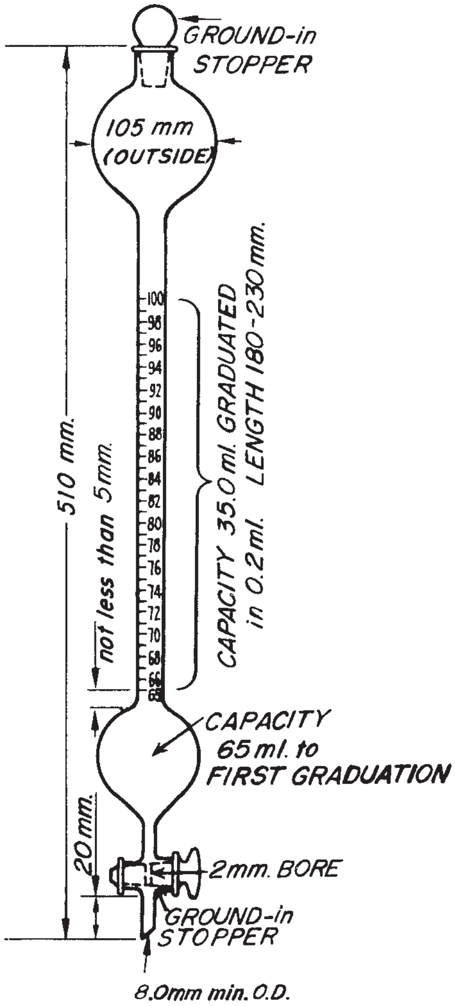

FIG. 1 Type II Glass Separatory Funnel, 260-mL Minimum Capacity

6.3 *Sulfuric Acid Solution* (40 %)—Mix 100 g of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) carefully with stirring into 150 g of water.

6.4 *Benzene* (C_6H_6)—Industrial grade, meeting the requirements of Specification D 836.

6.5 *Xylene*—($C_6H_4(CH_3)_2$)—Nitration grade, meeting the requirements of Specification D 843.

7. Preparation of Sample

7.1 Warm the sample, if necessary, to dissolve crystalline material before taking portion for analysis. Determine water content of the sample by Test Method D 95.

8. Procedure

8.1 Distill 100 g of the sample, weighed to the nearest 0.1 g, to 355°C by the procedure described in Test Method D 246, and collect the distillate in a Squibb-type separatory funnel. Add 50 mL of C_6H_6 to the freshly distilled fraction and extract the resulting solution with three successive 30-mL portions of 20 % NaOH solution. After each portion of caustic solution is placed in the funnel, shake vigorously, allow to settle completely, then draw off the lower layer into the second Squibb separatory funnel before placing the next 30-mL portion in the funnel containing the sample.

8.2 Wash the combined caustic layers in the second separatory funnel with 50 mL of $C_6 \cdot H_6$. Draw off the washed caustic layer into a 250-mL beaker and heat on a hot plate under an efficient hood until the odor of benzene is no longer detectable. Cool the NaOH solution in a water and ice bath. Then acidify with 40 % H_2SO_4 solution until the sodium sulfate solution is definitely acid to litmus, taking care to keep the mixture cool at all times.

8.3 Place 65 to 66 mL of $C_6H_4(CH_3)_2$ in the *Type II* separatory funnel. Submerge the lower portion of the funnel in a water bath at room temperature. After 30 min record the temperature of the bath to the nearest 0.1°C and read the volume of solvent to the nearest 0.1 mL. Pour the sulfate solution with the liberated tar acids through the solvent layer. Draw off the sulfate solution at the bottom of the separatory funnel into the original beaker, washing down the sides of the beaker, and pour it back into the funnel. Repeat this operation until all visible oil is transferred to the separatory funnel. Finally, with all sulfate solution in the funnel, shake well and allow to stand until the layers have separated and no drops of solvent are dispersed in the sulfate solution. Then draw off the sulfate layer slowly and completely, immerse the lower part of the funnel in the water bath, and adjust the temperature to within 0.5°C of the recorded temperature. After 30 min read the volume of the solvent layer. The increase in volume of the solvent is the tar acid content of the sample taken for distillation.

9. Calculation and Report

9.1 Calculate the tar acids content of the original dry sample as follows:

$$\frac{\text{volume of tar acids, mL}}{\text{weight of dry sample, g}} \times 100 \tag{1}$$

9.2 Report tar acids as mL/100 g of dry sample.

10. Keywords

10.1 coal tar; creosote; solution; tar acids

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