



## Standard Test Method for Coking Value of Tar and Pitch (Modified Conradson)<sup>1</sup>

This standard is issued under the fixed designation D2416; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of the coking value of tar and pitch having an ash content not over 0.5 % as determined by Test Method D2415.

1.2 Coking values by this test method are practically the same as those obtained by Test Method D189, but results are more reproducible. The apparatus used is identical, except that an electric furnace is substituted for the gas flame.

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 of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D140 Practice for Sampling Bituminous Materials

D189 Test Method for Conradson Carbon Residue of Petroleum Products

D370 Practice for Dehydration of Oil-Type Preservatives

D2415 Test Method for Ash in Coal Tar and Pitch

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

### 3. Summary of Test Method

3.1 A sample of the tar or pitch is vaporized and pyrolyzed for a specified time at a specified temperature in special standardized equipment that limits the available oxygen supply. The percentage of residue is reported as the coking value.

### 4. Significance and Use

4.1 This test method is useful for indicating the relative coke-forming propensities and for evaluating and characteriz-

ing tars and pitches. This test method can also be used as one element in establishing the uniformity of shipments or sources of supply.

### 5. Apparatus (see Fig. 1)

5.1 *Crucible*—Wide-form, *a*, either porcelain, glazed throughout, or silica, *a*; 29 to 31-mL capacity, 46 to 49 mm in rim diameter.

5.2 *Skidmore Crucible*—Iron crucible, *b*, flanged and ringed, 65 to 82-mL capacity, 53 to 57 mm inside and 60 to 67 mm outside diameter of flange, 37 to 39 mm in height, supplied with a cover without delivery tubes and having the vertical opening closed. The horizontal opening of about 6.5 mm shall be kept clean. The outside diameter of the flat bottom shall be 30 to 32 mm.

5.3 *Metal Crucible*—Spun sheet-iron or nickel crucible, *c*, with cover; 78 to 82 mm in outside diameter at the top, 58 to 60 mm in height, and approximately 0.8 mm in thickness. At the bottom of this crucible, and level before each test, shall be a layer of about 25 mL of sand, or enough to bring the Skidmore crucible, with cover on, nearly to the top of the sheet-iron crucible. The sand shall be dry and screened to pass through a 65-mesh screen and to be retained on a 200-mesh screen.

5.4 *Wire Support*—Triangle (65 mm) of bare Nichrome wire 1.5 to 2.0 mm in cross section, having an opening small enough to support the bottom of the metal crucible (see 5.3). The triangle, *d*, shall have its arms bent to form a cradle that will support the metal crucible with its bottom level with the bottom of the insulator (see 5.6).

5.5 *Hood*—Circular, sheet-iron, *e*, from 120 to 130 mm in diameter, the height of the lower perpendicular side to be from 50 to 53 mm; provided at the top with a chimney 50 to 60 mm in height and 50 to 56 mm in inside diameter, which is attached to the lower part having the perpendicular sides by a cone-shaped member, bringing the total height of the complete hood to 125 to 130 mm. The hood may be made from a single piece of metal, providing it conforms to foregoing dimensions. A bridge, *g*, made of approximately 3 mm iron or nichrome wire, and having a height of 50 mm above the top of the chimney, shall be attached to handle the chimney when positioning.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

Current edition approved Oct. 1, 2009. Published November 2009. Originally approved in 1965. Last previous edition approved in 2004 as D2416-84(2004). DOI: 10.1520/D2416-84R09.

<sup>2</sup> 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.

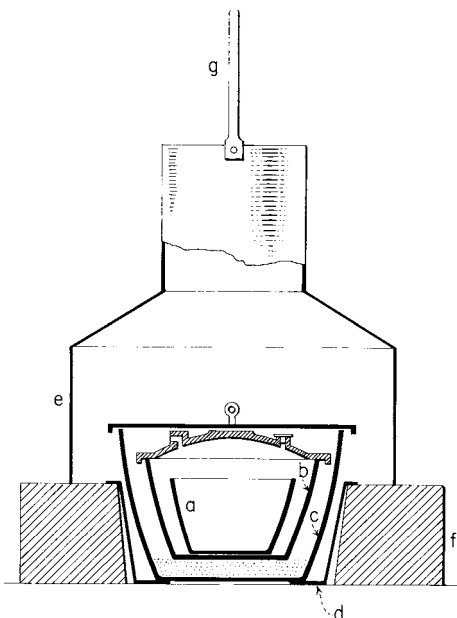


FIG. 1 Apparatus for Determining Carbon Residue

5.6 *Insulator*—Ceramic block or refractory ring, *f*, 150 to 175 mm in diameter if round, or on a side if square, 32 to 38 mm in thickness, provided with an inverted cone-shaped opening through the center; 83 mm in diameter at the bottom and 89 mm in diameter at the top.

5.7 *Vertical Electric Furnace*<sup>3</sup>—Bore a hole in the bottom and insert the thermocouple of the pyrometer controller so that its junction is centered exactly 38 mm below the furnace opening.

5.8 *Pyrometer Controller*<sup>4</sup>—The pyrometer controller should be checked periodically against a calibrated reference thermocouple, placed alongside but not touching, to ensure that the furnace is operating at the desired temperature. Appropriate changes in the controller circuit should be made, when necessary, to maintain the required temperature.

5.9 *Sieve*—U. S. Standard No. 30 (600- $\mu$ m), conforming to Specification E11.

## 6. Bulk Sampling

6.1 Samples from shipments shall be taken in accordance with Practice D140, and shall be free of foreign substances. Thoroughly mix the sample immediately before removing a representative portion for the determination or for dehydration.

<sup>3</sup> The sole source of supply of a Crucible furnace, Hoskins No. FD104, or equivalent, known to the committee at this time is National Element, Inc., 422 Oliver Street, Troy, MI 48084. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>4</sup> Omega Model D921 Digital Controller with 15 amp relay, available from Omega Engineering, Inc., Stamford, CT; or Thermo Electric Model 700, available from Thermo Electric, Saddle Brook, NJ; or equivalent. Minimum line voltage required for 220 V furnace is 205 V. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

## 7. Dehydration of Sample

7.1 *Hard Pitch*—If the solid bulk sample contains free water, air-dry a representative portion.

7.2 *Soft Pitch*—If the presence of water is indicated by surface foam on heating, maintain a representative portion of the bulk sample at a temperature between 125 and 150°C in an open container until the surface is free from foam. Take care not to overheat, and remove heat source immediately when foam subsides.

7.3 *Tar*—Dehydrate a representative portion of the bulk sample in accordance with Test Method D370, but stop the distillation when the temperature reaches 170°C. Separate any oil from the water that has distilled over (if crystals are present, warm sufficiently to ensure their solution), and thoroughly mix the oil with the residual tar in the still after the latter has cooled to a moderate temperature.

## 8. Preparation of Working Sample

8.1 *Hard Pitch*—If the pitch can be crushed at room temperature, prepare a 20-g working sample by suitable crushing, mixing, and quartering of a representative portion of the dry sample. The crushing can be done with a small jaw crusher and a mortar and pestle. No particle in the representative sample should be larger than 5 mm in any dimension. Crush this sample so that all of it will pass the No. 30 (600- $\mu$ m) sieve but have a minimum of fine particles.

8.2 *Soft Pitch*—If the pitch is too soft to grind and too sticky to mix, heat a representative portion of the dry sample to the lowest temperature that will permit passage through the No. 30 (600- $\mu$ m) sieve, taking care to avoid excessive loss of volatile matter. Do not exceed 10 min for this melting period. Pass the heated sample through the No. 30 (600- $\mu$ m) sieve to remove foreign matter.

8.3 *Tar*—Heat a representative portion of the dry tar rapidly to the lowest temperature that will permit passage through the No. 30 (600- $\mu$ m) sieve, then filter through this sieve to remove foreign matter.

8.4 *Preservation of Samples*—Store samples as large lumps or as solidified melts in closed containers. Do not save crushed samples for future analyses since changes in composition sometimes occur in pulverized pitch.

## 9. Procedure

9.1 Ignite a clean porcelain or silica crucible in a muffle furnace at 900°C or over a gas flame for 1 h. Cool in a desiccator and weigh to the nearest 1 mg.

9.2 Transfer a 3-g representative portion of the dry tar or pitch to the tared crucible and weigh to the nearest 5 mg. Place this crucible in the center of the Skidmore crucible. Level the sand in the metal crucible and place the Skidmore crucible in the exact center. Apply covers to both the Skidmore and metal crucibles, the one on the latter fitting loosely to allow free exit to the vapors as formed.

9.3 Place the triangle on the insulator, center the metal crucible in the insulator with its bottom resting on top of the triangle, and cover the whole with the sheet-iron hood and chimney in order to distribute the heat uniformly during the coking process. The assembly, including the insulator ring will be at approximately room temperature.

9.4 Cover the opening of the furnace with the furnace lid and set the controller to maintain a temperature of  $900 \pm 10^\circ\text{C}$  (see 5.8). With the furnace at this temperature, remove the lid, immediately center the assembly (see 9.3) over the furnace opening and start timing the test period. The positioning of the assembly must be done quickly to avoid excessive loss of heat. Apply heat to the sample for exactly 30 min without changing the setting of the controller. The temperature should recover to  $900^\circ\text{C}$  in 15 min. At the end of 30 min, remove the assembly from the heat source and allow to cool for about 15 min. Remove the porcelain crucible with heated tongs, place in a desiccator, cool, and weigh.

## 10. Calculation

10.1 Calculate the coking value of the sample as follows:

$$\text{Coking value, wt. \%} = 100A/B \quad (1)$$

where:

$A$  = weight of residue, and  
 $B$  = weight of sample.

## 11. Report

11.1 Report the coking value to the nearest 0.1 wt %.

## 12. Precision

12.1 The following criteria shall be used for judging the acceptability of results (95 % probability):

12.1.1 *Repeatability*— Duplicate values by the same operator shall not be considered suspect unless the determined percentages differ by more than 1.0.

12.1.2 *Reproducibility*— The values reported by each of two laboratories, representing the arithmetic average of duplicate determinations, shall not be considered suspect unless the reported percentages differ by more than 2.0.

## 13. Keywords

13.1 coking value; Conradson

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