



## Standard Test Method for Coke Residue of Creosote<sup>1</sup>

This standard is issued under the fixed designation D 168; 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 coke residue (fixed carbon) of creosote, coal tar, and creosote-coal tar solutions. As coal tars vary in their coke residue values, the method is therefore not a quantitative index of the percentage of coal tar in creosote-coal tar solutions. However, by specifying maximum amounts of coke residue as done in AWWA Standards P 2 and P 12, the method does serve to limit the amount of coal tar in such solutions; and by specifying a minimum as in Standard P 12 it ensures the presence of some coal tar. Formerly the test was also used as a check on the cleanliness of AWWA P1 creosote (absence of coal tar).

1.1.1 Test Methods 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.* See Section 6 for specific precautionary statements.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 38 Test Methods for Sampling Wood Preservatives Prior to Testing<sup>2</sup>

D 246 Test Method for Distillation of Creosote and Creosote-Coal Tar Solutions<sup>2</sup>

#### 2.2 AWWA Standards:

P 2 Creosote-Coal Tar Solution<sup>3</sup>

P 12 Creosote-Coal Tar Solution for Treating of Marine (Coastal Waters), Piles, and Timbers<sup>3</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-7 on Wood and is the direct responsibility of Subcommittee D07.06 on Treatments for Wood Products.

This test method is identical in substance with the Standard Method for the Determination of Coke Residue which is part of the American Wood-Preservers' Association Standard Methods for Analysis of Creosote and Oil-Type Preservatives (A1-78). Acknowledgment is made to the American Wood-Preservers' Association for its development of the subject matter covered in this standard.

Current edition approved July 15, 1994. Published September 1994. Originally published as D 168 – 23 T. Last previous edition D 168 – 86.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.10.

<sup>3</sup> Available from the American Wood-Preservers' Assn., P.O. Box 286, Woodstock, MD 21163-0286.

### 3. Summary of Test Method

3.1 After subjecting the creosote-coal tar solution (oil) under test to the distillation method (Standard A1, Section 2), the weighed residue at 355°C in the distillation flask is thoroughly mixed and a one-gram sample transferred to a special platinum crucible with vented cover. The crucible is then placed in a furnace maintained at  $950 \pm 20^\circ\text{C}$  for exactly 7 min. A final weighing gives the amount of coke of the distillation residue. The coke residue of the creosote-coal tar solution under test is calculated from (a) the percentage of distillation residue at 355°C, (b) the weight of the sample of distillation residue transferred to the platinum crucible, and (c) the weight of coke remaining in the platinum crucible after the coking operation.

### 4. Significance and Use

4.1 This test method is useful as an indicator of the preparation of coal tar in solution with coal tar distillate. The test method can also be used as one element in establishing uniformity of shipments and sources of supply.

### 5. Apparatus

5.1 *Crucible and Cover*—Platinum crucible and cover with dimensions as shown in Fig. 1. The cover shall be tightly fitting, shall have a depth of between 10.0 and 10.2 mm, and shall have a hole 2.0 mm in diameter at its center. The crucible without cover shall have a capacity of 25 to 30 mL. It shall have an outside diameter at the top of 34 to 35 mm, and outside diameter at the bottom of 19 to 21 mm, and a height of 40 to 45 mm. The weight of crucible with cover shall be 25 to 35 g.

5.2 *Crucible Holder*—The crucible holder shall be made from No. 20 Nichrome wire and a brass ring as shown in Fig. 2. The holder serves as a support for the platinum crucible during the coking operation.

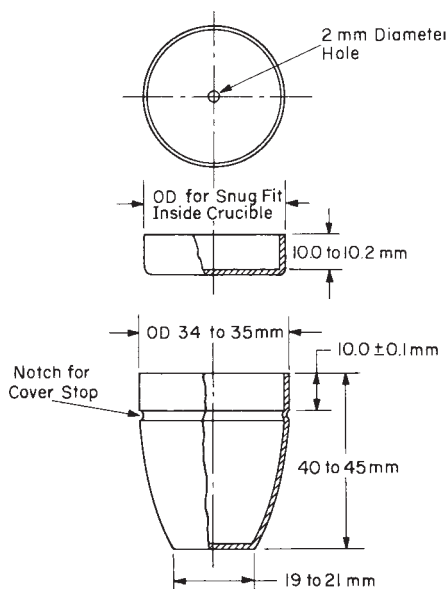


FIG. 1 Platinum Crucible

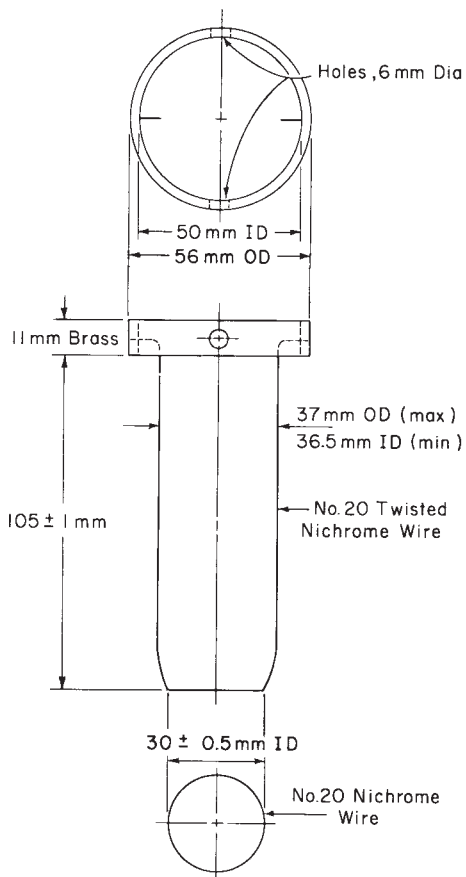


FIG. 2 Crucible Holder

5.3 *Furnace*—A vertical electric tube furnace<sup>4</sup> having a heating chamber 1½ in. (38 mm) in diameter, by 6 in. (150 mm) in depth. The power input shall be 300 to 400 W at 115 V. The furnace shall be equipped with a suitable rheostat or variable transformer and pyrometer, for control at 950 ± 20°C. A thermocouple shall be installed through a central hole in the bottom of the furnace, and sealed in place so that its junction is located 2 to 5 mm below the eventual position of the bottom of the platinum crucible.

5.4 *Gas Burner*—A gas burner of the bunsen or Meker type.

NOTE 1—In case of dispute, the tube furnace method shall be considered the standard method.

6. Precautions

6.1 Conduct the test in a part of the laboratory free of drafts.

6.2 It is important that the tip of the inner cone of a bunsen burner flame shall be below the bottom of the crucible. The burner should, therefore, be adjusted so that the inner cone is 60 to 70 mm in height. The bottom of the crucible shall be 5 to 10 mm above the tip of the inner cone.

7. Procedure

7.1 Heat and adjust the temperature of the uncovered, empty furnace to 950 ± 20°C. Clean the platinum crucible and cover, place them in the crucible holder, and suspend them in the temperature-adjusted furnace for at least 5 min. Remove the holder with contents from the furnace and cool the crucible and cover to about 150°C, being careful not to contaminate the bottom of the crucible. Then place the crucible with cover in a desiccator until they have cooled to room temperature and weigh (*P*). Record all weighings to the nearest 1 mg.

7.2 If the coke residue determination is made shortly after completion of the distillation test (AWPA Standard A1, Section 2) transfer a 1 ± 0.1-g portion of the well-stirred distillation residue at 355°C directly from the distillation flask to the tared platinum crucible. Since the tare weight (*P*) includes the weight of the platinum cover, the cover must also be on the balance pan during this operation.

7.3 If the residue is in a storage container, carefully heat the container and contents in a water bath or on a steam bath until sufficiently liquid for pouring. Then transfer a 1 ± 0.1-g portion to the platinum crucible as directed in the preceding paragraph. After the desired amount of distillation residue has been transferred to the platinum crucible, insert its cover firmly and weigh (*Q*). Weight of sample of distillation residue taken for coking:  $Q - P = W$ .

7.4 Place the covered crucible with sample in the crucible holder and suspend in the furnace at 950 ± 20°C for exactly 7 min. At the end of this period immediately remove the crucible holder from the furnace, cool the crucible and contents to about

<sup>4</sup> Two furnaces have proved satisfactory: (1) The Fieldner, Hoskins Electric Furnace, Type FA-120, with suitable rheostat or variable transformer and pyrometer. When using this furnace the platinum crucible shall be supported in the crucible holder described under 2.2. (2) "Hevi-Duty" Furnace, Type MU-86 725 W, with suitable rheostat or variable transformer and pyrometer. When using this furnace the platinum crucible shall be located in the center of the furnace, seated in a Nichrome basket suspended from the cover furnished with the furnace. This furnace should be adjusted to temperature with the cover in place.

150°C as before, and transfer to a desiccator. When at room temperature, weigh (*R*). Weight of coke in sample of distillation residue:  $R - P = A$ .

7.5 If a gas burner of the bunsen or Meker type is used (Note 1), it shall have a free flame 200 mm in height. The crucible shall sit in a Nichrome triangle with approximately two thirds of its height below the triangle and with the bottom of the crucible 60 to 80 mm above the top of the burner. Assurance of the desired temperature of test may be indicated by the fusion of crystals of potassium chromate in the crucible when exposed in the specified position for the test. Expose the crucible and contents to the full flame of the burner for exactly 7 min (see 6). Then transfer to a desiccator, cool, and weigh the crucible to the nearest 1 mg.

## 8. Calculation

8.1 Calculate the percentage of coke residue on the basis of original oil (creosote-coal tar solution) as follows:

$$\text{Coke residue} = \frac{A \times B}{W} \quad (1)$$

where:

*W* = weight of sample of distillation residue taken for coking,

*A* = weight of coke in sample of distillation residue, and

*B* = percentage of distillation residue at 355°C of original oil (creosote-coal tar solution).

*Example:*  $W = 1.100$  g;  $A = 0.308$  g;  $B = 29.0$  %

$$\text{Coke residue} = \frac{29.0 \times 0.308}{1.100} = 8.1 \quad (2)$$

*ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.*

*This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).*

## 9. Report

9.1 Report the calculated amount of coke residue to the nearest 0.1 %.

## 10. Precision

10.1 *Coke Residue, 0 to 2.5 % Range:*

10.1.1 *Repeatability*—Duplicate values by the same operator should not be considered suspect unless they differ by more than 0.3 % absolute.

10.1.2 *Reproducibility*—The values reported by each of two laboratories should not be considered suspect unless they differ by more than 0.5 % absolute.

NOTE 2—The estimated standard deviation of repeatability is 0.12 % absolute at 16 degrees of freedom (df). The estimated standard deviation of reproducibility is 0.18 % absolute at 14 df.

10.2 *Coke Residue, 2.5 to 12 % Range:*

10.2.1 *Repeatability*—Duplicate values by the same operator should not be considered suspect unless they differ by more than 12 % relative.

10.2.2 *Reproducibility*—The values reported by each of two laboratories should not be considered suspect unless they differ by more than 14 % relative.

NOTE 3—The estimated standard deviation of repeatability is 4.1 % relative at 24 df. The estimated standard deviation of reproducibility is 4.9 % relative at 21 df.

## 11. Keywords

11.1 coke; creosote; residue