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American Association State
Highway Transportation Officials Standard
AASHTO No. T 62

Standard Test Method for Distillation of Creosote and Creosote-Coal Tar Solutions[1]

This standard is issued under the fixed designation D 246; 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.

1. Scope

1.1 This test method covers a procedure for the distillation of creosote and creosote-coal tar solution. 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.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 38 Test Methods for Sampling Wood Preservatives Prior to Testing

D 370 Practice for Dehydration of Oil-Type Preservatives

D 390 Specification for Coal-Tar Creosote for the Preservative Treatment of Piles, Poles, and Timbers for Marine, Land, and Freshwater Use

D 391 Specification for Creosote-Coal Tar Solution

E 1 Specification for ASTM Liquid-in-Glass Thermometers

E 1404 Specification for Laboratory Glass Conical Flasks

E 1405 Specification for Laboratory Glass Distillation Flasks

3. Summary of Test Method

3.1 A 100-g sample is distilled at a controlled rate in a 300-mL flask. The weights of distillate fractions at a series of specified temperatures and of residue at the maximum specified temperature are determined. The residue and distillates shall be tested by appropriate procedures when required by the specifier.

4. Significance and Use

4.1 Creosote quality is defined by the boiling ranges of its distillation fractions. The boiling limits of the fractions as determined by this test method must conform to the limits established by Specifications D 390 and D 391 to qualify the creosote as an acceptable preservative for its intended application.

5. Apparatus

5.1 *Flask*—Distillation flask, 300 mL, specified as Type II, Class 2 in Specification E 1405.

5.2 *Condenser Tube*—A tapered glass condenser, as shown in Figs. 1 and 2, conforming to the following dimensions:

Diameter of small end, outside	12.5 ± 1.5 mm
Diameter of large end, outside	28.5 ± 3.0 mm
Length	360 ± 4 mm
Length of tapered part	100 ± 5 mm

5.3 *Source of Heat*—A source of heat consisting of a Bunsen or Meker type gas burner or an electric heater. The electric heater[3] shall have an output variable to 600 or 750 W and removable upper and lower refractories as illustrated in Fig. 3. The temperature of the heater shall be controlled by a variable transformer or rheostat suitable for the voltage used, and shall be fitted with a clamp for mounting on a vertical support rod.

5.4 *Flask Shield for Flame Distillation*—A stainless steel shield, fitted with mica windows and lined with ⅛-in. heat-resistant ceramic board, with two-part cover made from ¼-in. "Transite" board of the forms and dimensions shown in Fig. 4.

5.5 *Flask Shield for Electric-Heater Distillation*—A stainless steel shield fitted with mica windows and cover of the same construction and dimensions as those for flame distillation (5.4), except for the height of the shield. See Fig. 5.

5.6 *Gauze*—Two sheets of 16-mesh wire gauze made with 0.51-mm (0.02-in.) diameter Nichrome wire and 125 to 152 mm in diameter or square.

5.7 *Burner Chimney for Flame Distillation*—A cylindrical metal chimney approximately 100 mm high, 95 to 105 mm in diameter, and having a peephole 25 mm in diameter centered about 32 mm below the ring support, used to protect the flame

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

[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.

[3] The "Precision" Ful-Kontrol 750-W heater with built-in variable transformer control has been found satisfactory. This heater is available for 115 V, 50/60 Hz only.

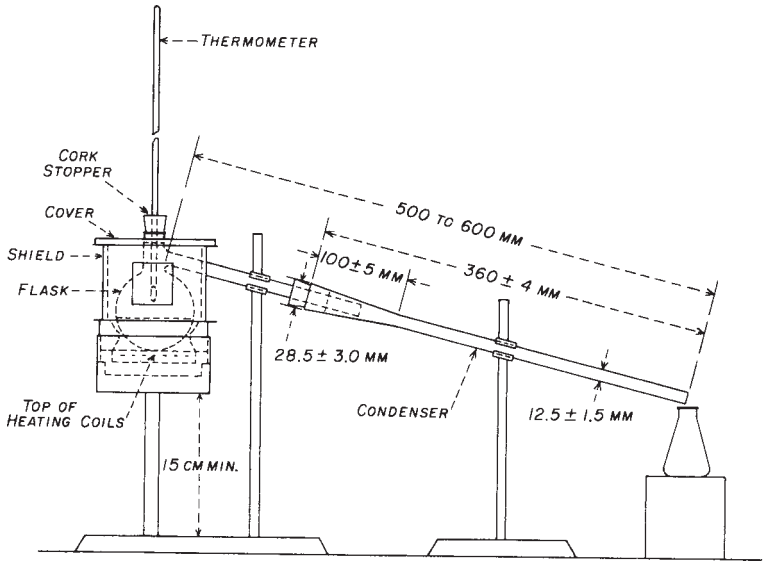


FIG. 1 Apparatus Assembly for Flame Distillation

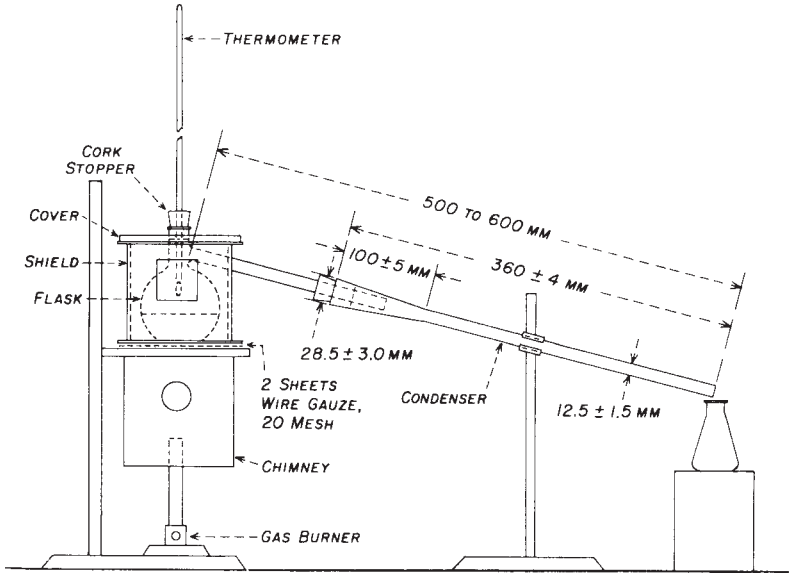


FIG. 2 Apparatus Assembly for Electric-Heater Distillation

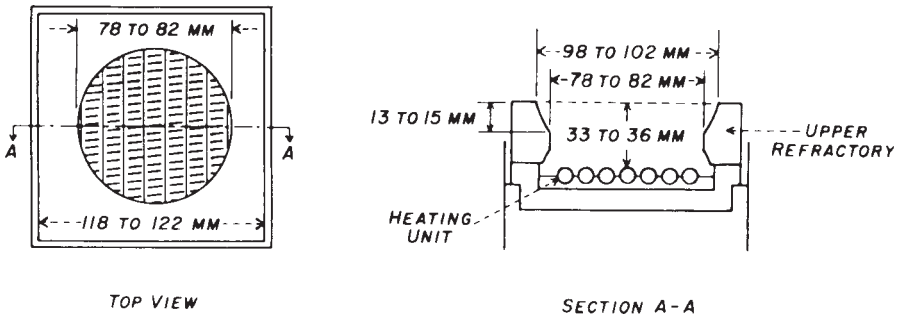
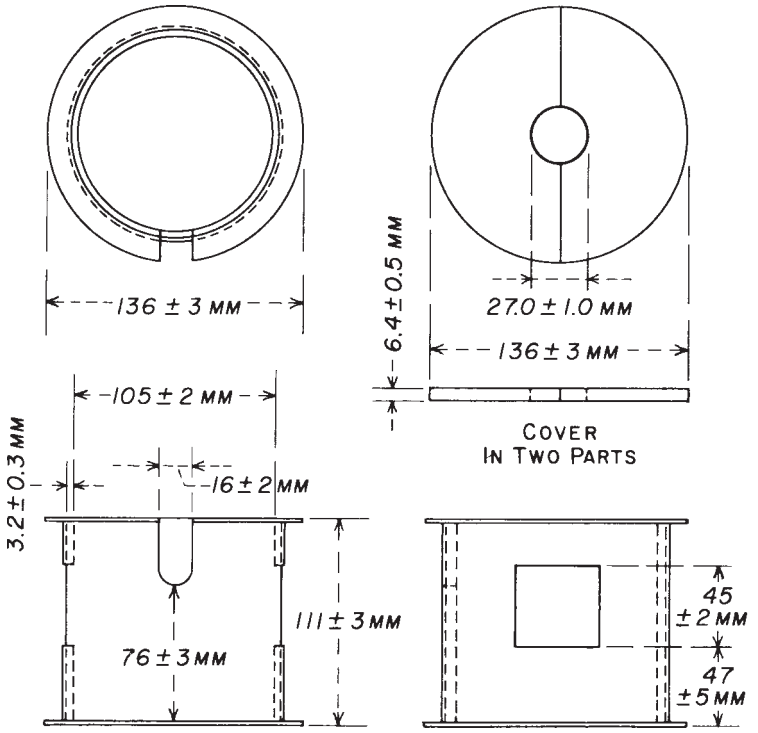


FIG. 3 Upper Part of Electric Heater

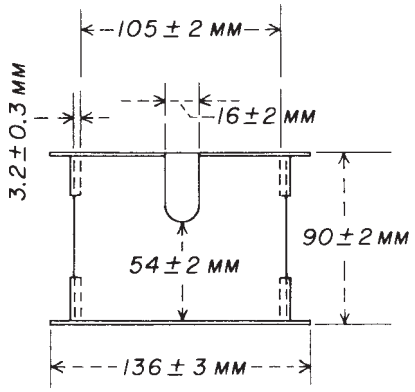
from air currents. The top of the shield should be flanged to permit its being suspended from the ring support.

5.8 *Receivers*—Standard 125-mL conical flasks, specified as Type I, Class 1 in Specification E 1404.



NOTE 1—Flanged open-end cylinder made of 22-gage stainless steel with ⅛-in. heat-resistant ceramic board lining.
NOTE 2—Two mica windows are provided at right angles to the end slot.

FIG. 4 Shield and Cover for Flame Distillation



NOTE 1—Flanged open-end cylinder made of 22-gage stainless steel with ⅛-in. asbestos lining.

FIG. 5 Shield for Use with Electric Heater

5.9 *Balance and Weights*, accurate to 0.05 g.

5.10 *Thermometer*—An ASTM high-distillation thermometer having a range from −2 to +400°C and conforming to the requirements for Thermometer 8C as prescribed in Specification E 1.

6. Preparation of Sample

6.1 Thoroughly stir or otherwise mix the sample immediately before removing the portion for testing, to ensure that such portion will be representative of the sample. If warming is necessary, take care to avoid loss of volatile material.

7. Dehydration of Sample

7.1 If not more than 3.0 % of water is present, the sample may be tested without previous dehydration. If the water content exceeds 3.0 %, dehydrate a representative portion of the sample before distillation, in accordance with Practice D 370. In case of dispute, the determination shall be carried out on a dehydrated sample.

8. Apparatus Assembly

8.1 Assemble the apparatus as described in 8.1.1-8.1.3 and as shown in Figs. 2 and 3.

8.1.1 Suspend the burner chimney by its flange from the support ring, place the specified two sheets of wire gauze on the burner chimney, and place the flask shield on the upper sheet of gauze. In case the electric furnace is used, attach the electric heater to a vertical support so that at the end of the distillation, it can be lowered for a distance of at least 6 in. (15 cm). Place the upper refractory on the heater with its larger opening facing upwards. Set the flask shield on the upper refractory.

8.1.2 Insert the thermometer through a regular-length No. 13 cork, rolled before use, in the neck of the flask so that the bottom of the cork is from 23 to 28 mm above the lowest point of the juncture between the tubulature and the neck of the flask, and the bottom of the bulb is 12 to 13 mm from the surface of the liquid in the flask. The exact location of the thermometer bulb may be determined by calculating the number of divisions on the thermometer which are equal to 12 to 13 mm, lowering the thermometer through the cork until the tip of its bulb

touches the surface of the 100-g sample in the flask, and then raising the thermometer the calculated number of degrees to give the correct distance from the surface of the liquid. Align the stem on the axis of the bulb through the neck of the flask.

8.1.3 In the case of the flame distillation apparatus, place the flask in the flask shield with its bulb resting on the gauze. In the case of the electric-heater distillation apparatus, place the flask in the flask shield and support the flask so that its bottom is between 4 and 7 mm above the heating coils. Connect the condenser tube to the tubulature of the flask with a tight cork joint, having the tubulature project 30 to 50 mm through the cork. The distance from the neck of the flask to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. Support the condenser tube in a position so that it is in alignment with the tubulature of the flask, and the thermometer is vertical. Place the shield cover on the flask shield around the neck of the flask.

9. Procedure

9.1 Weigh the flask to the nearest 0.05 g and then weigh 100.0 ± 0.1 g of the sample into it. Assemble the apparatus as described in Section 8.

9.2 Apply heat to the flask so that, 45 s after the first drop of distillate falls from the end of the condenser, the distillation rate is 80 to 100 drops per minute (Note 1). Maintain this rate throughout the distillation. If the sample contains over 1 % water, heat the flask and contents carefully until the vapor temperature reaches 170°C to distill off the water before continuing the distillation as previously described. Warm the condenser tube whenever necessary to prevent accumulation of solid distillates in the tube.

NOTE 1—If an electric metronome is not available to measure the distillation rate, not less than 20 drops nor more than 25 drops of distillate shall fall from the end of the condenser in the time interval 45 to 60 s after the first drop or in any given 15-s time interval thereafter.

9.3 Collect the distillate fractions in tared receivers, changing receivers as the thermometer indicates the maximum temperature, corrected as described in 9.4, for each specified fraction. The following fractions are specified:

Up to 210°C
210 to 235°C
235 to 270°C
270 to 315°C
315 to 355°C

9.4 Do not change the position of the thermometer during the distillation. Make no correction for emergent stem of the thermometer, but if the barometric pressure is outside the range from 756 to 765 mm, adjust (but do not report) the temperature in accordance with Table 1.

9.5 When the maximum temperature specified for the test is indicated by the thermometer, immediately remove the flame and the flask-shield cover; or when the electric heater is used as a source of heat, immediately remove the flask shield cover and drop the electric heater a distance of at least 6 in. Allow the apparatus to cool for at least 5 min, or until no vapors are visible. Drain any oil remaining in the condenser tube into the receiver containing the last fraction.

TABLE 1 Adjustment of Distillation Test Temperatures for Barometric Pressure

Barometric Pressure, mm Hg	Fractionation Temperatures for Various Barometric Pressure Ranges, °C				
786 to 795	212	237	272	317	357
776 to 785	211	236	271	316	356
766 to 775	211	236	271	316	356
756 to 765	210	235	270	315	355
746 to 755	209	234	269	314	354
736 to 745	209	234	269	314	354
726 to 735	208	233	268	313	353
716 to 725	208	233	267	312	352
706 to 715	207	232	267	312	351
696 to 705	207	231	266	311	351
686 to 695	206	231	265	310	350
676 to 685	205	230	265	309	349
666 to 675	205	230	264	309	348
656 to 665	204	229	264	308	348
646 to 655	204	228	263	307	347
636 to 645	203	228	262	307	346
626 to 635	202	227	262	306	345
616 to 625	202	226	261	305	345
606 to 615	201	226	260	305	344
596 to 605	201	225	260	304	343

9.6 Weigh the receivers containing the distillate fractions to the nearest 0.05 g. Remove the cork and thermometer and weigh the flask and residue to the nearest 0.05 g.

9.7 Should the fraction of 210°C contain water, determine the water volume and calculate the net weight of oil distillate, assuming that 1 mL of water weighs 1 g. The amount of water contained in this fraction may be determined by either of the following methods:

9.7.1 Transfer the fraction, after weighing, to a tube or cylinder graduated in 0.1 mL. Rinse the receiver several times with xylene, adding the rinsings to the tube or cylinder containing the fraction or,

9.7.2 The fraction to 210°C may be collected in a tared 5-mL graduated cylinder having a flared top. After weighing, add xylene; this will result in a clear separation of the water and oil distillate.

9.8 If tests of the residue are required, replace the cork and thermometer in the flask and lower the thermometer until its bulb is in the liquid residue. If the residue is not completely fluid, heat it carefully to a temperature not exceeding 150°C by holding the bulb of the flask over a wire gauze heated by a gas burner or by immersion in a suitable bath, the temperature of which does not exceed 150°C. Incline the flask and rotate it so that the fluid residue will flow around the side, and collect any oils that have condensed on the upper surfaces of the flask. Mix the contents of the flask until they are homogeneous. Allow the residue to cool to a temperature at which it can be readily poured from the flask without loss of volatile material and then pour it into the desired testing equipment or into a suitable receptacle. Cover the receptacle.

10. Calculation

10.1 Convert the distillation results to the water-free basis by means of the following equations:

Fraction to 210°C:

$$\text{Percentage (water-free basis)} = (F - W) \times 100/(100 - W) \quad (1)$$

Second and Subsequent Fractions Including Residue:

$$\text{Percentage (water-free basis)} = F \times 100/(100 - W) \quad (2)$$

where:
F = fraction or residue, g, and
W = millilitres of water, g, in fraction distilling to 210°C.

10.2 Add the percentages (water-free basis) of the fractions to obtain cumulative percentages to the specified temperatures.

11. Report

11.1 Report the cumulative percentages to the specified temperatures and distillation residue, on a water-free basis, to the nearest 0.1 %.

12. Precision and Bias

12.1 The following criteria should be used for judging the acceptability of results at the 95 % probability level:

12.1.1 *Repeatability*—Duplicate determinations by the same operator should not be considered suspect unless the reported percentages differ by more than 1.1.

12.1.2 *Reproducibility*—The results submitted by two different laboratories should not be considered suspect unless the reported percentages differ by more than 2.9.

13. Keywords

13.1 coal-tar; creosote; distillation; wood-preservative