Designation: D 369 – 84 (Reapproved 2002)

American Association State
Highway Transportation Officials Standard
AASHTO No. T74

Standard Test Method for Specific Gravity of Creosote Fractions and Residue[1]

This standard is issued under the fixed designation D 369; 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.

1. Scope

1.1 This test method covers a convenient and accurate means of determining the specific gravity of (*1*) creosote fractions entirely liquid at 38°C, (*2*) creosote fractions containing solids at 38°C, and (*3*) distillation residues. It is also suitable for determining the specific gravity of oil-type preservatives when the quantity available is too small for the hydrometer method as given in Test Method D 368. Test Methods D 38 cover 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 246 Test Method for Distillation of Creosote and Creosote-Coal Tar Solutions[2]

D 368 Test Method for Specific Gravity of Creosote and Oil-Type Preservatives[2]

D 390 Specification for Coal-Tar Creosote for the Preservative Treatment of Piles, Poles, and Timbers for Marine, Land, and Fresh Water Use[2]

D 391 Specification for Creosote-Coal Tar Solution[2]

E 1 Specification for ASTM Thermometers[3]

3. Summary of Test Method

3.1 This is a test method for the determination of specific gravity of fractions from the distillation of creosote and creosote-coal tar solutions as measured by means of pycnometer.

4. Significance and Use

4.1 This is a test method to determine the conformance of specified fractions of creosote and creosote-coal tar solutions to Specifications D 390 and D 391.

5. Apparatus

5.1 *Pycnometers*, of the Gay-Lussac type, 10-mL or 25-mL capacity, and of the Hubbard-Carmick type, 25-mL capacity. The Hubbard-Carmick type pycnometer shall be used for determining the specific gravity of distillation residue.

5.2 *Water Bath*, consisting of a vessel filled with sufficient water to permit maintaining a temperature of 38.0 ± 0.1°C.

5.3 *Thermometer*—An ASTM Low Softening Point Thermometer having a range from −2 to +80°C, and conforming to the requirements for Thermometer 15C as prescribed in Specification E 1.

5.4 *Balance and Weights*—An analytical balance and weights accurate to 0.5 mg.

6. Calibration of Pycnometers

6.1 Before calibration, grind the stopper into the neck of the pycnometer by partially filling the pycnometer with water, inserting the stopper into the neck, and rotating the stopper by hand with very light pressure. During this operation, keep the ground surfaces wet and occasionally flush with water. When the wet stopper can be rotated freely with no tendency for the ground surfaces to stick together, using no more pressure than the weight of the stopper, the pycnometer is in condition for calibration.

6.2 Thoroughly clean the pycnometer with hot chromic acid cleaning solution. Empty the acid from the pycnometer, flush thoroughly with distilled water, dry in an oven at about 110°C, cool in a desiccator for 15 to 30 min, and weigh to the nearest 0.001 g. Designate this weight *P*.

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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.

Current edition approved Dec. 28, 1984. Published February 1985. Originally published as D 369 – 30 T. Last previous edition D 369 – 67 (1979).

This method is identical in substance with the Standard Method for the Determination of the Specific Gravity of Distillation Fractions which is part of the American Wood-Preservers' Association Standard Methods for Analysis of Creosote and Oil-Type Preservatives (A1). Acknowledgement is made to the American Wood-Preserver's Association for its development of the subject matter covered in this standard.

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

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

6.3 Fill the pycnometer with freshly boiled distilled water at room temperature. (A pipet or drawn-out medicine dropper facilitates this operation.) Insert the stopper with a rotary motion to secure a firm seat, making sure that no air is entrapped, then completely immerse the pycnometer in the water bath at 38.0°C for 30 min.

6.4 While still in the water bath, raise the pycnometer so that the top of the stopper is slightly over the water level in the bath and wipe off the water on the flat top surface of the stopper with soft absorbent paper, taking precautions not to remove water from the capillary tube. Remove the pycnometer from the water bath, dry the surface and weigh to the nearest 0.001 g. Then remove the stopper, add distilled water and reinsert the stopper as described above. Return the pycnometer to the water bath for 30 min, remove, and weigh as before. (It is advisable to have the previously determined weights on the balance pan to expedite the reweighing.) Repeat this operation until three successive weighings check within 0.010 g; if this proves impossible, regrind the stopper and repeat the calibration until three weighings agree within 0.010 g. Designate this weight W_1. Remove the water and thoroughly dry the pycnometer in an oven at about 110°C.

NOTE 1—Calibration of the pycnometer is not necessary before making each determination. Dependent on usage, recalibration should be done at sufficient time intervals to ensure that the calibration is accurate. In case of dispute, calibration is mandatory.

7. Procedure for Fractions Entirely Liquid Below 38°C

7.1 Carefully heat the fraction to a temperature below 38°C in a water bath or by direct heat with an asbestos board under the container until the distillation fraction is entirely liquid. Fill the dry Gay-Lussac pycnometer by means of a warm pipet or warm drawn-out medicine dropper until the neck is about one half full, avoiding the inclusion of air bubbles. Insert the stopper with a rotary motion to secure a firm seat, making sure no air is entrapped. Place the filled pycnometer in the water bath at 38.0°C so that the top of the stopper is slightly above the water level, and allow to remain for at least 30 min. All this time the capillary tube should be completely filled with oil. Carefully wipe off with soft absorbent paper any oil from the top flat surface of the stopper while the pycnometer is still in the water bath. Then remove the pycnometer, dry and weigh to the nearest 0.001 g. Remove the stopper, refill the pycnometer with liquid fraction, and repeat the determination as described above until two successive weighings agree within 0.010 g. Designate this weight W_2.

8. Procedure for Fractions Containing Solids at 38°C

8.1 Carefully heat the fraction in a water bath or by direct heat with an asbestos board under the container until the distillation fraction is entirely liquid. By means of a warm pipet or warm drawn-out medicine dropper, transfer a sufficient amount of the fraction to the dry Gay-Lussac or Hubbard-Carmick pycnometer until it is approximately one half full, avoiding the inclusion of air bubbles and contact of the oil with the ground glass surface of the neck of the pycnometer. (Permitting the stream of liquid to impinge on the side of the pycnometer below the ultimate liquid level aids in preventing inclusion of air bubbles.) Cool the pycnometer to room temperature and weigh with the stopper. Designate this weight W_3.

8.2 Add freshly boiled distilled water to the partially filled pycnometer until it is about three quarters full. Partially immerse the pycnometer without the stopper in a small water bath, maintained at 90 to 95°C, and allow to remain until the fraction is liquid and free of air bubbles. Any entrapped air bubbles can be removed with a heated fine wire loop.

8.3 Cool the pycnometer and contents to a temperature of about 25°C and then add cool freshly boiled distilled water until the neck is about one half full. Insert the stopper with a rotary motion to secure a firm seat, making sure that all air is excluded. Completely immerse the pycnometer in the water bath at 38.0°C and allow sufficient time for an equilibrium crystal state to be established. One hour is usually sufficient. While still in the water bath, raise the pycnometer until the top of the stopper is above the water level and wipe off the water on the flat surface of the stopper with soft absorbent paper, taking precautions not to remove water from the capillary tube. Remove the pycnometer and partially immerse in an auxiliary water bath at room temperature until the water in the capillary tube of the stopper has contracted approximately ½ in. (13 mm). Then wipe the pycnometer dry with soft absorbent paper and weigh. Remove the stopper, add freshly boiled distilled water, reinsert the stopper, and return the pycnometer to the water bath at 38.0°C. After 30 min, remove and weigh the pycnometer as described above. Repeat these operations until two successive weighings agree within 0.010 g. Designate this weight W_4.

9. Procedure for Distillation Residue

9.1 Melt the distillation residue in accordance with Test Method D 246. Carry out the determination as in 8.1 and 8.3, using the Hubbard-Carmick pycnometer.

10. Calculation

10.1 *Fractions Entirely Liquid at 38°C*—Calculate specific gravity as follows:

$$\text{sp gr at } 38.0/15.5°\text{C} = [(W_2 - P)/(W_1 - P)] \times 0.99393 \quad (1)$$

10.2 *Fractions Containing Solids at 38°C and Distillation Residue*—Calculate specific gravity as follows:

$$\text{sp gr at } 38.0/15.5°\text{C} = \frac{W_3 - P}{(W_1 - P) - (W_4 - W_3)} \times 0.99393 \quad (2)$$

where:

P_1	=	weight of empty pycnometer,
W_1	=	weight of pycnometer full of water,
W_2	=	weight of pycnometer full of liquid fraction,
W_3	=	weight of pycnometer partially filled with solid fraction or distillation residue,
W_4	=	weight of pycnometer full of solid fraction or residue and water, and
0.99393	=	the ratio of the density of water at 38.0°C to the density of water at 15.5°C, that is, 0.99299/0.99905.

11. Report

11.1 Report the specific gravity at 38.0/15.5°C to the nearest one thousandth unit of specific gravity.

12. Precision

12.1 *Repeatability*—Duplicate determinations by the same operator should not be considered suspect unless the reported results differ by more than the following:

Fractions entirely liquid below 38°C	0.003
Fractions containing solids at 38°C	0.006
Distillation residue	0.008

12.2 *Reproducibility*—The results submitted by two different laboratories should not be considered suspect unless the reported results differ by more than the following:

Fractions entirely liquid below 38°C	0.008
Fractions containing solids at 38°C	0.010
Distillation residue	0.010

13. Keywords

13.1 creosote; fractions; preservative; residue; specific gravity