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American Association State
Highway and Transportation Officials Standard
AASHTO No. T81

Standard Test Method for Xylene-Insoluble Matter in Creosote[1]

This standard is issued under the fixed designation D 367; 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.

ε[1] NOTE—Keywords were added editorially in March 2000.

1. Scope

1.1 This test method covers the determination of the xylene-insoluble matter in creosote and creosote-coal tar solution. Since this method is empirical, strict adherence to all details of the procedure is necessary for close agreement of results among laboratories.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 38 Test Methods for Sampling Wood Preservatives Prior to Testing[2]

D 370 Test Method for Dehydration of Oil-Type Preservatives[2]

D 845 Specification for Five-Degree Xylene[3]

3. Summary of Test Method

3.1 The sample is digested in hot xylene and filtered. The insoluble matter is washed, dried, and weighed.

4. Significance and Use

4.1 The test is useful in evaluating and characterizing creosote and as one element in establishing the uniformity of shipments and sources of supply.

5. Apparatus and Materials

5.1 *Filtering Crucibles*, porcelain with fine-porosity bottom 40-mL capacity, high form, maximum pore diameter, 7μ m[4].

5.2 *Filter Apparatus, Filter Flask and Tube*, with crucible adapter, and means for producing a vacuum.

5.3 *Diatomaceous Silica Analytical Filter*[5], shall be dried to a constant weight at 105°C and stored in tightly stoppered container. (Any other grade of filtering medium should not be used because porosities differ.)

5.4 *Balance and Weights*, accurate to 0.5 mg.

5.5 *Xylene*, five-degree distillation range, conforming to Specification D 845.

5.6 *Acetone*, boiling at a pressure of 760 mm Hg within a range of 1.0°C, which shall include the temperature of 56.1°C.

6. Preparation of Filter Crucible

6.1 Make and record all weighings to the nearest 0.5 mg.

6.2 Clean a crucible, if used for less than six determinations as follows. Remove the mat, wash the crucible with distilled water, dry and ignite in a muffle furnace for 1 h at about 800°C. Cool the crucible slowly to prevent cracking and place it in a desiccator while still warm.

6.3 After a crucible has been used for six determinations, remove any residual ash from pores in the filtering area by boiling in 1 + 1 hydrochloric acid. Then boil the crucible in distilled water, thoroughly back wash with distilled water, dry, and ignite as above.

6.4 Transfer 0.45 to 0.55 g of diatomaceous silica to a clean, filtering crucible, distributing it evenly over the bottom. Dry in an oven at 105 to 110°C for 30 min. Cool in a desiccator and weigh. Record the weight of crucible plus diatomaceous silica.

7. Procedure

7.1 Take the original, undehydrated sample and, if necessary, heat and stir until any crystalline material is in solution and the sample is homogeneous. Determine the percentage of water in accordance with Test Method D 370.

7.2 Weigh the following size sample, into a 100-mL beaker:

[1] 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.

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This test method is identical in substance with the Standard Method for the Determination of the Amount of Material Insoluble in Xylene which is part of the American Wood-Preservers' Association Standard Methods of Analysis of Creosote and Oil Type Preservatives (A1-62). Acknowledgment is made to the American Wood Preservers' Association for its development of the subject matter covered in this standard.

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

[3] Discontinued; see *1981 Annual Book of ASTM Standards*, Part 29.

[4] Selas Grade 01. size FC40, or equivalent has been found satisfactory.

[5] Celite, available from the Arthur H. Thomas Co., 3rd and Vine Sts., Philadelphia, PA 19105, has been found satisfactory.

New creosote	10 ± 1 g
Used creosote	5.0 ± 0.5 g
Creosote solutions	2.0 ± 0.1 g

7.3 Calculate and record the weight of the sample.

7.4 Warm 50 mL of xylene to a temperature of 50 to 60°C, and immediately add it to the sample while stirring thoroughly. Continue stirring until the sample is dispersed and the bottom of the beaker is clean. Bring the beaker or flask containing the solution to boiling on a hot plate.

7.5 Insert the filter tube with the adapter in the filter flask and place the previously prepared and tared crucible in the adapter. Fill the crucible before the xylene has been drawn entirely through the diatomaceous silica. Take care that the diatomaceous silica is never free from liquid, either during the addition of the solution containing the sample, or during the subsequent washing with xylene.

7.6 Wash the beaker, thermometer, or stirring rod, and crucible with hot xylene. Pass all washes through the filter. Use a suitable policeman to sweep the insoluble particles into the crucible. Wash the crucible and contents with the hot xylene, allowing each wash to pass almost through the filter before the next is added, until the washings are colorless.

7.7 Reduce the suction and wash the contents with acetone until the washings are colorless. Four additions of 5 mL each are usually sufficient. Remove the crucible, and wipe the outside clean with a tissue moistened with xylene.

8. Calculation

8.1 Calculate the xylene-insoluble matter as a percentage of the water-free preservative, as follows:

$$\text{Xylene-insoluble matter, \%} = [100(A - B)/C] \times [100/(100 - D)] \quad (1)$$

where:

A = weight of crucible after filtration,
B = weight of prepared crucible before filtration,
C = weight of sample used, and
D = percentage of water in the sample.

9. Report

9.1 Report the amount of xylene-insoluble matter to the nearest 0.1 %.

10. Precision

10.1 The following criteria should be used for judging the acceptability of results:

10.1.1 *Repeatability*—The average difference between two results obtained by the same analyst on different days will approximate 0.1 % absolute. Two such values should be considered suspect if they differ by more than 0.2 % absolute.

10.1.2 *Reproducibility*—The average difference between two results obtained by analysts in different laboratories will approximate 0.2 % absolute. Two such values should be considered suspect if they differ by more than 0.5 % absolute.

11. Keywords

11.1 creosote; insoluble; xylene; xylene-insoluble