Designation: D6608 – 12

Standard Practice for the Identification of Trinidad Lake Asphalt in Asphalt Mixes[1]

This standard is issued under the fixed designation D6608; 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 practice shall be used for the qualitative determination of Trinidad Lake Asphalt in Trinidad Lake Modified Asphalt Cements obtained from production blends or recovered binder from asphalt paving mixtures.

1.2 The values stated is SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 It is limited to the binder content of Trinidad Lake Modified Asphalt (Specification D5710) used in the construction of asphalt pavements.

1.4 **Warning—**Mercury has been designated by the United States Environmental Protection Agency and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/index.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state may be prohibited by state law.

1.5 *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]

D140 Practice for Sampling Bituminous Materials

D1856 Test Method for Recovery of Asphalt From Solution by Abson Method

D2172 Test Methods for Quantitative Extraction of Bitumen From Bituminous Paving Mixtures

D5710 Specification for Trinidad Lake Modified Asphalt

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Summary of Test Method

3.1 A polished copper strip is immersed in a given quantity of sample and heated at a temperature and for a given time characteristic of the material being tested. At the end of this period the copper strip is removed, washed, and compared with the classifications of the TLA Copper Tarnishing Ranges for copper strips.

4. Significance and Use

4.1 Bitumen contains sulfur compounds most of which are removed during refining, the resulting asphalt cement also contains sulfur compounds which are released at the temperatures above 230°C . Trinidad Lake Modified Asphalt contains sulfur compounds, which are released at lower temperatures between 170°C and 230°C . The release of sulfur from Trinidad Lake Modified Asphalt tarnishes the copper strip and appears to be related directly to the total TLA content. The copper strip test is designed to assess the qualitative presence of TLA in asphalt cements.

5. Apparatus

5.1 *Test Tubes,* 25 by 150 mm.

5.2 Provide a bath, which can maintain a constant temperature of 210 ± 1°C (410 ± 2°F) and 225 ± 1°C (437 ± 2°F). The bath should have suitable supports to hold the test tubes in a vertical position and immersed to a depth of about 100-mm (4-in.). Oil or aluminum block baths are suitable.

5.3 *Thermometric Device,* partial immersion thermometer for indicating the required test temperature, with graduations 1°C or less. The ASTM 2C thermometer is suitable. Any other thermometric device of equal accuracy may be used.

5.4 *Polishing Vise,* to hold the copper strip firmly without marring the edge while polishing. Any suitable holder may be used provided that the surface is held tightly and the surface being polished is supported above the surface of the holder.

5.5 *Viewing Test Tubes,* flat glass test tubes, are convenient for protecting corroded strip and may be used for close inspection and storage.

[1] This practice is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 on Miscellaneous Asphalt Tests.

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[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.

6. Materials

6.1 *Polishing Materials:*

6.1.1 Silicone carbide grip paper of varying degrees of fineness including 65 µm (240 grit) paper or cloth.

6.1.2 Silicone carbide grains 105 µm (150–mesh).

6.1.3 Absorbent cotton (cotton wool), pharmaceutical grade.

6.2 *Copper Strips:*

6.2.1 *Specifications*—Use strips 12.5 mm wide, 1.5 to 3.0 mm thick, cut 75 mm long from smooth-surface, hard temper, cold-finished copper of 99.9+ % purity; electrical bus bar stock is generally suitable (see Annex A1). The strips may be used repeatedly but should be discarded when the surfaces become deformed on handling.

6.2.2 *Surface Preparation*—Remove all surface blemishes from all six sides of the strip with silicone carbide paper of such varying degrees of fineness as are needed to accomplish the desired results efficiently. Finish with 65–µm (240 grit) silicone carbide paper or cloth, removing all marks that may have been made by other grades of paper used previously. Immerse the strip in wash solvent from which it can be withdrawn immediately for final preparation (polishing) or in which it can be stored for future use.

6.2.2.1 As a practical manual procedure for the surface preparation, place a sheet of paper on a flat surface, moisten it with kerosene or wash solvent, and rub the strip against the paper with a rotary motion, protecting the strip from contact with the fingers using an ashless filter paper. Alternatively, the surface of the strip can be prepared by use of motor-driven machines using appropriate grades of dry paper or cloth.

6.2.3 *Final Preparation*—Remove a strip from the wash solvent. Holding it in the fingers protected with ashless filter paper, polish first the ends and then the sides with the 105–µm (150–mesh) silicone carbide grains picked up from a clean glass plate with a pad of cotton (cotton wool) moistened with a drop of wash solvent. Wipe vigorously with fresh pads of cotton (cotton wool) and subsequently handle only with stainless steel forceps. *Do not touch with fingers*. Clamp in a vise and polish the main surfaces with silicone carbide grains on absorbent cotton. Do not polish in a circular motion. Rub in the direction of the long axis of the strip, carrying the stroke beyond the end of the strip before reversing direction. Clean all metal dust from the strip by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled. When the strip is clean, immediately immerse it in the prepared sample.

6.2.3.1 It is important to polish the whole surface of the strip uniformly to obtain a uniformly stained strip. If the edges show wear (surface elliptical) they will likely show more corrosion than the center. The use of a vise will facilitate uniform polishing.

6.2.3.2 It is important to follow the order of preparation with the correctly sized silicone carbide material as described in 6.2.2 and 6.2.3. The final preparation is with 105–µm silicone carbide grains. This is a larger grain size than the 65–µm paper used in the surface preparation stage. The reason for this use of larger silicone carbide grains in the final preparation is to produce asperities (controlled roughness) on the surface of the copper which acts as sites for the initiation of corrosion reactions.

7. Reagent

7.1 *Wash Solvent*—Any volatile, sulfur free hydrocarbon solvent may be used that would show no tarnish when tested at 210°C or 225°C.

NOTE 1—Toluene is a suitable solvent.

8. Sample Preparation

8.1 The binder shall be acquired either from production tanks, according to Practice D140, or from asphalt mixes, according to the procedures in Test Method D2172 and Test Method D1856. Store the sample material in a clean, dark glass or metal container or other suitable containers such that the properties of the sample are not affected. Fill the containers as completely as possible and seal immediately.

8.2 To produce a test sample, heat the recovered binder slowly to approximately 135°C with constant stirring, by hand, until molten. Stir the sample thoroughly again before decanting into the sample test tubes.

9. Procedure

9.1 Place 15–20 mL of the molten recovered binder sample, which is free of any entrained water or suspended water, into each of two clean and dry test tubes. Insert a polished copper strip into each test tube within one (1) minute of completing the final strip preparation. Ensure that the strip is vertically held in the sample with approximately 44–57 mm of the strip projecting out of the sample mixture.

9.2 Place the test tubes in the bath, which has been maintained at 210°C and leave for 25 min.

9.3 *Strip Examination:*

9.3.1 Empty the contents of each test tube into a 150–mL tall-form beaker, allowing the strip to slide out gently to avoid breakage. Remove the strip immediately with clean stainless steel forceps and immerse in the wash solvent for not longer than 30 s. Remove and blot dry with quantitative filter paper. Inspect the strip for evidence of tarnishing or color change while holding at an angle of approximately 45°. Compare the strip to the classification given in the TLA Copper Tarnishing Ranges (see Table 1).

TABLE 1 TLA Copper Tarnishing Ranges

Classification	Temperature, °C	Designation	Description	TLA Presence
1	210	None	Clean Strip	0 – 25 % TLA[A]
2	210	Moderate to heavy tarnish	Dark blue, black-green	> 25 % TLA
3	225	Slight to moderate tarnish	Red-blue, green	< 25 % TLA
4	225	None	Clean strip	0 % TLA

[A] Further testing at 225°C is required for determination of the absence or presence of TLA.

9.3.2 In handling the test strip during inspection, care must be taken to avoid damage by marking or staining. A flat glass tube, which can be stoppered with absorbent cotton, may be used as a viewing aid.

9.4 If the surface of the strip is unchanged, raise the bath temperature to 225°C and allow to stand for 25 min. Replace the strip in the test tube and place in the bath for 25 min. Repeat paragraph 9.3 for strip examination.

10. Interpretation

10.1 Interpret the Trinidad Lake Asphalt presence and content of the sample accordingly as the appearance of the test strip agrees with one of the strips in the TLA Copper Tarnishing Standard in Table 1.

10.2 If there is a color change at 210°C, conclude that the bitumen contains TLA and has a TLA content greater than 25 %.

10.3 If there is a color change at 225°C, conclude that the binder contains TLA and has a TLA content of less than 25 %.

11. Report

11.1 Report the results as the presence of greater than 25 % of TLA, the presence of less than 25 % of TLA or the absence of TLA in the asphalt mix.

12. Keywords

12.1 asphalt mix; copper strip finish; copper tarnishing; sulfur compound; Trinidad Lake Asphalt

ANNEX

A1. COPPER QUALITY

A1.1 Copper Quality

A1.1.1 Hard-tempered, cold-finished type (ETP) electrolytic tough pitch copper. [3]

[3] Conforming to Copper Development Assn. (CDA), United States of America No. 110, or to British Standard (BS) 1036:1952, which have proper quality.