Designation: ~~D6608 – 12~~ D6608 – 20

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~~in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 ~~It~~ This practice is limited to ~~the binder content of Trinidad Lake Modified Asphalt (Specification~~asphalt binders which are produced in accordance with Specification ~~D5710~~D5710/D5710M~~) 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~~ 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~~ mercury-containing products into your state may be prohibited by state law.

1.5 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

1.6 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

~~D140~~D140/D140M Practice for Sampling Asphalt Materials

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

~~D2172~~D2172/D2172M Test Methods for Quantitative Extraction of Asphalt Binder from Asphalt Mixtures

[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.

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials
~~D5710~~D5710/D5710M 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~~Asphalt contains sulfur compounds, most of which are removed during ~~refining,~~refining; the resulting asphalt ~~cement~~ also contains sulfur compounds which are released at the temperatures above ~~230°C.~~230 °C. Trinidad Lake Modified Asphalt contains sulfur ~~compounds,~~compounds which are released at lower temperatures between ~~170°C~~170 °C and ~~230°C.~~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.~~asphalt.

NOTE 1—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Specification D3666 are generally considered capable of competent and objective testing, sampling, inspection, etc. Users of this standard are cautioned that compliance with Specification D3666 alone does not completely ensure reliable results. Reliable results depend on many factors; following the suggestions of Specification D3666 or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.

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)~~ 1 °C and 225 ± ~~1°C (437 ± 2°F).~~ 1 °C. 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.).~~100 mm. Oil or aluminum block baths are suitable.

5.3 *Thermometric Device,* partial immersion thermometer for indicating the required test temperature, with graduations ~~1°C~~ of 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.

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~~ 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~~act as sites for the initiation of corrosion reactions.

7. Reagent

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

NOTE 2—Toluene is a suitable solvent.

8. Sample Preparation

8.1 The ~~binder~~asphalt shall be acquired either from production tanks, according to Practice ~~D140~~D140/D140M, or from asphalt mixes, according to the procedures in Test ~~Method~~Methods ~~D2172~~D2172/D2172M 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~~asphalt slowly to approximately ~~135°C~~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~~ 15 to 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~~ 44 to 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~~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).

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~~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,~~210 °C, conclude that the ~~bitumen~~ asphalt binder contains TLA and has a TLA content greater than 25 %.

10.3 If there is a color change at ~~225°C,~~225 °C, conclude that the asphalt 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

(Mandatory Information)

TABLE 1 TLA Copper Tarnishing Ranges

~~Classification~~Classification	Temperature, °C	Designation	Description	TLA Presence
~~1~~	~~210~~	~~None~~	~~Clean Strip~~	~~0 – 25 % TLA~~[A]
1	210	None	Clean strip	0–25 % TLA[A]
~~2~~	~~210~~	~~Moderate to heavy tarnish~~	~~Dark blue, black-green~~	~~> 25 % TLA~~
2	210	Moderate to heavy tarnish	Dark blue, black-green	>25 % TLA
~~3~~	~~225~~	~~Slight to moderate tarnish~~	~~Red-blue, 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~~225 °C is required for determination of the absence or presence of TLA.