Designation: D6805 – 02 (Reapproved 2016)

Standard Practice for
Infrared (IR) Procedure for Determination of Aromatic/Aliphatic Ratio of Bituminous Emulsions[1]

This standard is issued under the fixed designation D6805; 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 uses infrared analytical techniques to qualitatively determine in the laboratory a ratio of aromatic absorbance to aliphatic absorbance. This practice may be used to determine if the bitumen in the emulsion is predominantly aromatic or aliphatic in nature.

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

1.3 *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]

D2939 Test Methods for Emulsified Bitumens Used as Protective Coatings (Withdrawn 2012)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *aliphatic*—a group of organic compounds characterized by straight- or branched-chain arrangements of the constituent carbon atoms. Examples of aliphatic compounds include paraffins (alkanes), olefins (alkenes), and acetylenes (alkynes). These compounds are primarily found in petroleum sources. **Hawley's Condensed Chemical Dictionary**[4]

3.1.2 *aromatic*—a group of organic compounds made up of unsaturated cyclic hydrocarbons containing one or more rings, an example of which is benzene. Large numbers of these compounds are derived from petroleum and coal tar, and are classified as "aromatics" because of their strong and not unpleasant odor characteristics.

Hawley's Condensed Chemical Dictionary[4]

3.1.3 *infrared absorbance*—the range of wavelengths in the infrared that are absorbed by a specimen and identify its molecular components and compound structures. The infrared region of the electromagnetic spectrum includes wavelengths from 0.70 µm to approximately 300 µm, that is, longer than visible light and shorter than microwave.

Hawley's Condensed Chemical Dictionary[4]

4. Summary of Practice

4.1 A sample of bituminous emulsion from which the water has been removed using Na_2SO_4 is dissolved in carbon disulfide (CS_2), and the infrared (IR) absorbance from 2.5 to 4.2 µm is determined. The absorbance at 3.27 µm (aromatic) is divided by the absorbance at 3.40 µm (aliphatic) to obtain the IR ratio.

5. Significance and Use

5.1 The results of this practice may be used to distinguish tar-based emulsion from an asphalt-based emulsion for specification compliance purposes.

6. Apparatus

6.1 *Infrared Spectrophotometer.*

6.2 *Sealed Sodium Chloride (NaCl) IR Cell,* with 0.1 to 1 mm path length.

6.3 *IR Cell Cleaner.*

6.4 *Vacuum Pump and Dry-Ice Trap,* to protect pump.

6.5 *Syringe,* glass, 10 mL.

6.6 *Mechanical Shaker.*

6.7 *Sieves,* No. 40 (425 µm opening) and No. 100 (150 µm).

6.8 *Balance,* capable of weighing to 0.001 g.

6.9 *Pipet,* 10 mL and rubber bulb.

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[1] This practice is under the jurisdiction of ASTM Committee D08 on Roofing and Waterproofing and is the direct responsibility of Subcommittee D08.09 on Bituminous Emulsions.

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Lewis, R. J., Sr., *Hawley's Condensed Chemical Dictionary, Thirteenth Edition*, New York, Van Nostrand Reinhold, 1997.

7. Reagents and Materials

7.1 *Disposable "Medicine" Dropper.*

7.2 *Vial,* 20 mL, with cork-backed metal foil liner.

7.3 *Filter Paper,* rapid flow rate, 12.5 cm.

7.4 *Glass Funnel,* for above filter paper.

7.5 *Carbon Disulfide (CS_2),* spectroscopy grade.

7.6 *Sodium Sulfate (Na_2SO_4),* anhydrous.

8. Hazards

8.1 Carbon disulfide (CS_2) is a hazardous material and must be handled properly. Before using CS_2, read and understand the CS_2 Material Safety Data Sheet and the label on the CS_2 bottle. Use appropriate precautions, including safety equipment, when handling CS_2. Be sure to work in a properly operating hood and wear appropriate gloves.

9. Sampling, Test Specimens, and Preparation

9.1 Every effort should be taken to ensure that a representative sample is taken. Follow Section 4 from Test Methods D2939 for sampling.

10. Dehydration of Sample

10.1 Dehydration is not necessary since a drying agent (Na_2SO_4) will be added in a later step.

11. Preparation of Working Sample

11.1 Stir thoroughly to obtain representative sample.

11.2 *Dissolution in Solvent (CS_2):*

11.2.1 Weigh 0.2 ± 0.01 g of bituminous emulsion into a vial using a disposable "medicine" dropper and add about 1 g of anhydrous Na_2SO_4. Using a 10 mL pipet and rubber bulb, add 10 mL of CS_2 to the vial, bituminous emulsion, and Na_2SO_4. Cap vial and mix on mechanical shaker for 15 min. Then filter quickly (to minimize CS_2 evaporation) through fast filter paper into another clean vial; save filtrate.

NOTE 1—CS_2 is preferred, as any hydrogen-containing solvent will cause interference during the IR analysis.

12. Preparation of Apparatus

12.1 Remove any previous solution of CS_2 and bituminous emulsion from the IR cell using a cell cleaner (blowing the solution out) or by using a vacuum source and an appropriate dry-ice trap. Clean the cell with fresh CS_2; the cell should be filled with fresh CS_2 at least 3 times.

12.2 Because the moisture in the air easily etches the NaCl cell, store sodium chloride (NaCl) cell in a desiccator.

13. Procedure

13.1 *Determination of IR Spectrum:*

13.1.1 Determine spectrum within 1 h of extraction with CS_2.

13.1.2 Fill the IR reference cell, using a dropper or syringe, with the CS_2 solvent.

13.1.3 Fill the IR sample cell, using a dropper or syringe, with the CS_2/bituminous emulsion filtered solution.

NOTE 2—Follow the filling directions supplied by the cell(s) manufacturer.

13.1.4 Referring to the instructions supplied with the particular IR instrument being used, determine the IR spectrum from 2.5 to 4.2 µm in the absorbance mode. Linearly ruled chart paper is used in the absorbance mode; however, if the instrument can only be operated in the Transmittance mode, use logarithmically ruled chart paper (if log paper is not available, follow Transmittance mode for linear paper calculation procedure).

13.1.5 Record the following information on the chart paper:

13.1.5.1 Sample number,

13.1.5.2 Sample identification,

13.1.5.3 Date of analysis,

13.1.5.4 Analyst's name,

13.1.5.5 Cell used (NaCl), and

13.1.5.6 Solvent used (CS_2).

13.1.6 Draw a baseline from 2.7 to 4.0 µm.

14. Calculation of Results

14.1 The determination of absorbance (A) is dependent on the operational mode of the IR instrument and the chart paper used. Record aromatic as $A_{(aromatic)}$. Record aliphatic absorbance as $A_{(aliphatic)}$. The various methods are explained below:

14.1.1 *Absorbance Mode (Linear Paper)*—Determine absorbance at 3.27 µm (aromatic) and at 3.40 µm (aliphatic). Absorbance can be determined by recording the value of the peak on the chart and subtracting the value of the baseline under the peak. Estimate values to the nearest tenth (0.1). See Absorbance Graph in Appendix X2 (Fig. X2.2).

14.1.2 *Transmittance Mode (Log Paper)*—The procedure is essentially the same as in 14.1.1 except the peak direction is reversed.

14.1.3 *Transmittance Mode (Linear Paper)*—Read values as in 14.1.2, except divide the peak value by the baseline value to obtain the Transmittance Percentage (T). See Transmittance Graph in Appendix X2 (Fig. X2.1). Calculate Absorbance (A) according to the equations below:

$$A_{(aromatic)} = log\frac{1}{T_{(aromatic)}} = -logT_{(aromatic)} \tag{1}$$

$$A_{(aliphatic)} = log\frac{1}{T_{(aliphatic)}} = -logT_{(aliphatic)} \tag{2}$$

14.2 The IR ratio is calculated as follows:

$$\text{IR ratio} = \frac{A_{(aromatic)}}{A_{(aliphatic)}} \tag{3}$$

15. Report

15.1 Report aromatic/aliphatic IR ratio to nearest hundredth (0.01).

16. Precision and Bias

16.1 *Precision:*

16.1.1 The repeatability standard deviation of the IR ratio has been determined to be 0.10 absolute.

16.1.2 The reproducibility of this practice is being determined and will be available in 2005.

17. Keywords

17.1 absorbance; aliphatic; aromatic; asphalt; bituminous; coal tar; emulsion; infrared; sealer

APPENDIXES

(Nonmandatory Information)

X1. TYPICAL AROMATICITY INDICES (*Ia*) OF VARIOUS MATERIALS

Sample	*Ia*
Asphalt (aliphatic) Emulsion	0.03
Coal Tar (aromatic) Emulsion	2.23
50/50 Asphalt/Coal Tar Emulsion	0.09
Typical Aromatic Emulsion Blend[A]	1.94

[A] Contains aromatic coal tar and aromatic petroleum compounds.

X2. ABSORBANCE AND TRANSMITTANCE GRAPHS USED IN CALCULATING AN AROMATIC INDEX (Ia)

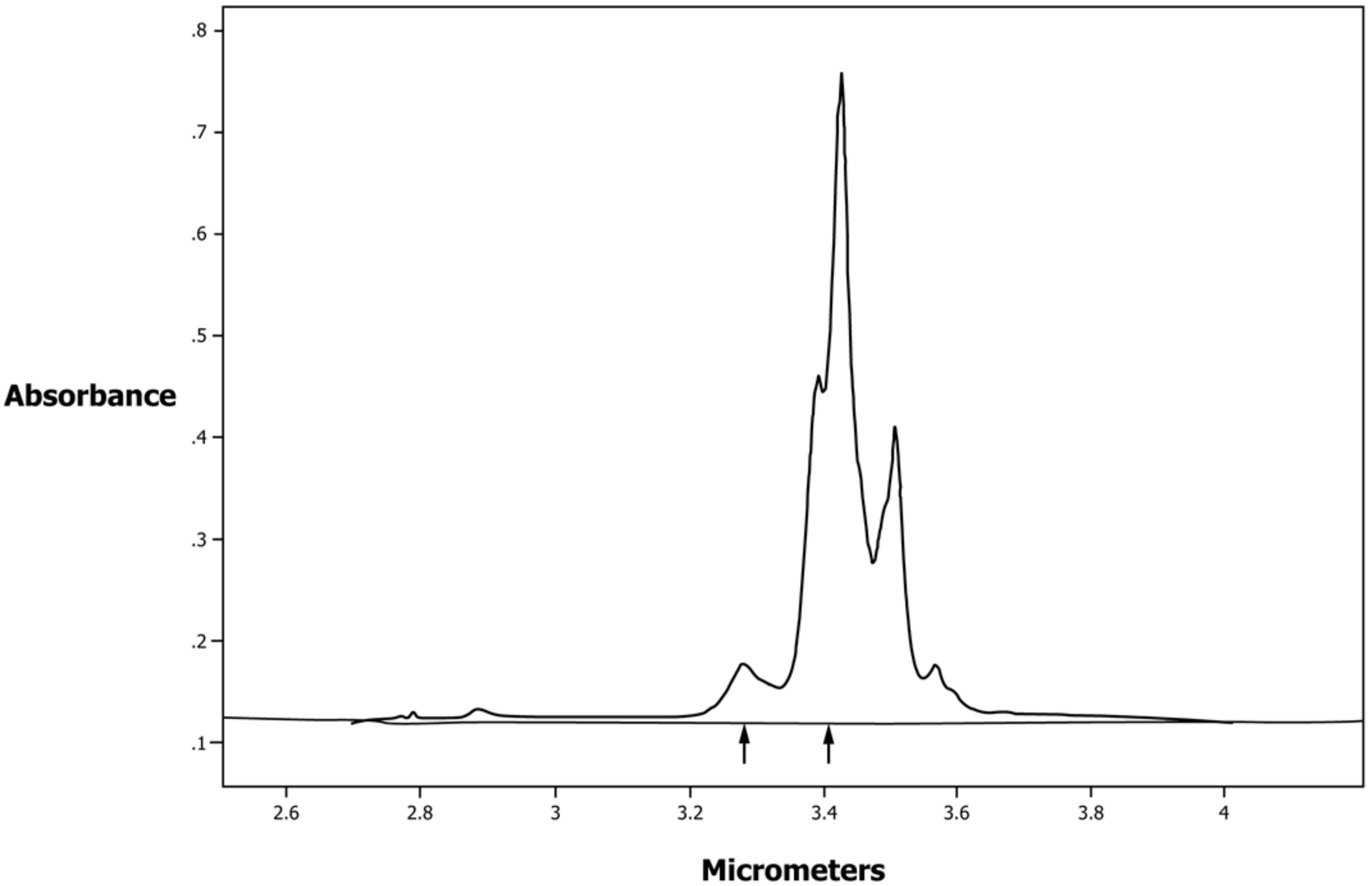

FIG. X2.1 Absorbance Graph

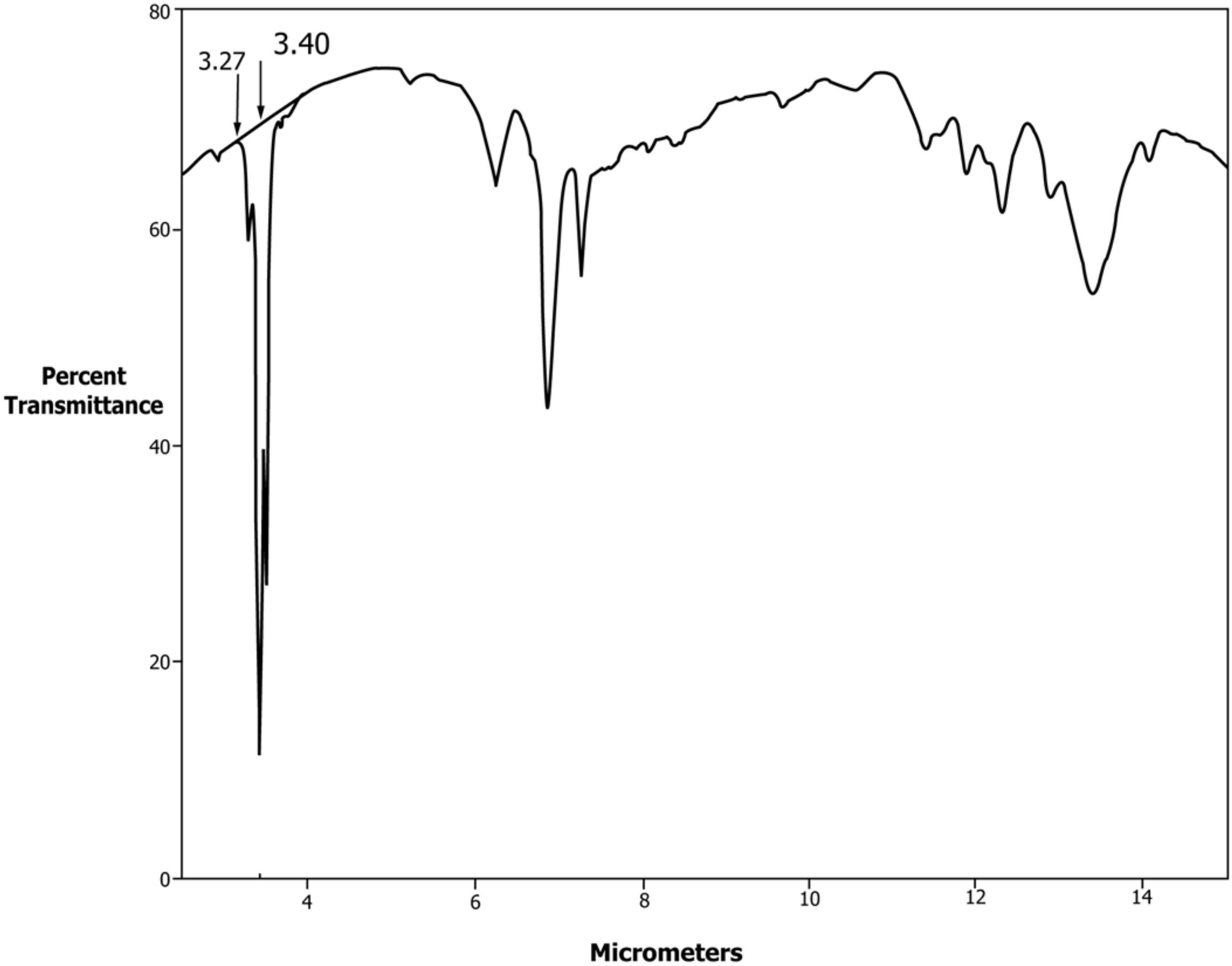

FIG. X2.2 Transmittance Graph

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