Designation: D6703 – 14

Standard Test Method for Automated Heithaus Titrimetry[1]

1. Scope

1.1 This test method describes a procedure for quantifying three Heithaus compatibility parameters that quantify the colloidal stability of asphalts and asphalt cross blends and aged asphalts.

1.2 *Units*—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]

D8 Terminology Relating to Materials for Roads and Pavements

D3279 Test Method for*n*-Heptane Insolubles

D4124 Test Method for Separation of Asphalt into Four Fractions

D5546 Test Method for Solubility of Asphalt Binders in Toluene by Centrifuge

E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *asphaltene peptizability, n*—the tendency of asphaltenes to exist as a stable dispersion in a maltene solvent, measured by the Heithaus parameter p_a.

3.1.2 *asphalt state of peptization, n*—a measure of the ability of the combination of a maltene solvent and dispersed asphaltenes to form a stable dispersed system.

3.1.3 *colloidal suspension, n*—an intimate mixture of two substances, one of which, called the dispersed phase (or colloid), is uniformly distributed in a finely divided state through the second substance, called the dispersion medium (or dispersing medium).

3.1.4 *compatibility, n*—the state of peptization of an asphalt, which is measured quantitatively by the Heithaus parameter P.

3.1.5 *dispersed phase, n*—one phase of a dispersion consisting of particles or droplets of one substance distributed through a second phase.

3.1.6 *dispersing medium, n*—one phase of a dispersion that distributes particles or droplets of another substance, the disperse phase.

3.1.7 *flocculation, n*—the process of aggregation and coalescence into a flocculent mass.

3.1.8 *Heithaus compatibility parameters, n*—three parameters: asphaltene peptizability (p_a), maltene peptizing power (p_o), and asphalt state of peptization (P), measured using Heithaus titration methods.

3.1.9 *maltene peptizing power, n*—the ability of a maltene solvent to disperse asphaltenes, measured by the Heithaus parameter p_o.

4. Summary of Test Method

4.1 Three 40 mL reaction vials are tared (Fig. 1). Three samples of asphalt of weights 0.400 g, 0.600 g and 0.800 g are transferred to each of three reaction vials. Toluene (3.000 mL) is added to each reaction vial to dissolve the asphalt constituting three solutions which differ by concentration. Each solution is titrated with isooctane (2,2,4-trimethyl pentane) to promote onset of flocculation of the solution.

4.2 Titrations are performed by placing reaction vials separately in the apparatus illustrated in Fig. 2. Each reaction vial is separately placed into a 250 mL water-jacketed reaction vessel. A sample circulation loop is made by pumping the solution through a short path length quartz flow cell housed in an ultraviolet-visible spectrophotometer then back to the reaction vial with high flow rate metering pump. A titration loop is made by pumping titrant into the sample reaction vial at a constant flow rate using a low flow rate metering pump, thus a second reaction vessel containing titrant is placed into a second 250 mL water-jacketed reaction vessel. During a titration the

---

[1] This test method 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.

Current edition approved June 1, 2014. Published July 2014. Originally approved in 2001. Last previous edition approved in 2013 as D6703 – 13. DOI: 10.1520/D6703-14.

[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.

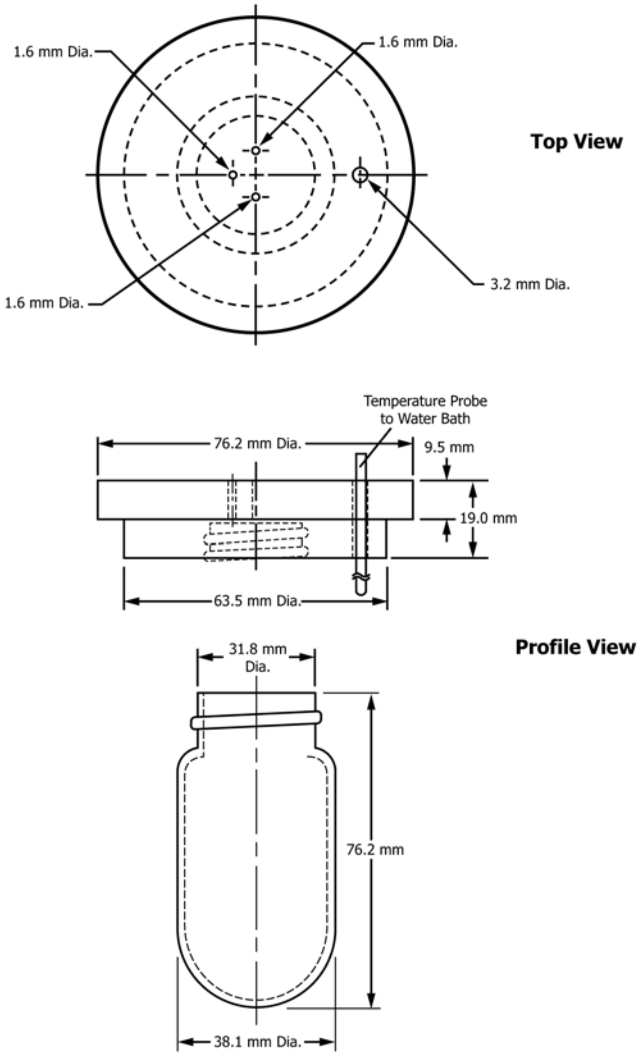

FIG. 1 Reaction Vial (40 mL) with TFE-fluorocarbon Cover and Temperature Probe

output signal from a spectrophotometer is recorded using a data acquisition system (computer) to record the change in percent transmittance $\%T$ of detected radiation at 740 nm plotted as a function of time t (Fig. 3), as the titrated solution passes through a quartz flow cell.

4.3 The spectrophotometer output signal measures turbidity of the sample solution as a titration experiment proceeds to a flocculation onset point, corresponding to the onset of flocculating asphaltene phase separating from the solution. Fig. 3 illustrates a plot of $\%T$ versus t for three test solutions. Values of $\%T$ are observed to increase with time up to the flocculation onset point, after which values of $\%T$ are observed to decrease with time. The time required to reach flocculation onset t_f multiplied by the titrant flow rate gives the titrant flocculation volume V_T.

4.4 The measured weight of each asphalt sample, W_a, the volume of toluene initially used to dissolve each sample V_S, and the volume of titrant at onset of flocculation V_T represent the input data required to calculate compatibility parameters.

5. Significance and Use

5.1 This test method is intended primarily as a laboratory diagnostic tool for estimating the colloidal stability of bitumen asphalt, asphalt cross blends, aged asphalt, and heavy oil residuum. Historically, bituminous asphalt and heavy oil residua have been modeled as colloidal suspensions in which a polar associated asphaltene moiety (the dispersed phase) is suspended in a maltene solvent moiety (the dispersing medium) (refer to Test Methods D3279, D4124, and D5546 for further definition of asphalt fraction materials). The extent to which these two moieties remain in state of peptization is a measure of the compatibility (colloidal stability) of the suspension. Compatibility influences the physical properties of these materials, including rheological properties, for example, phase angle and viscosity. This test method and other similar test methods, along with the classical Heithaus test, measures the overall compatibility of a colloidal system by determining a parameter referred to as the state of peptization, P. The value of P commonly varies between 2.5 to 10 for unmodified or neat

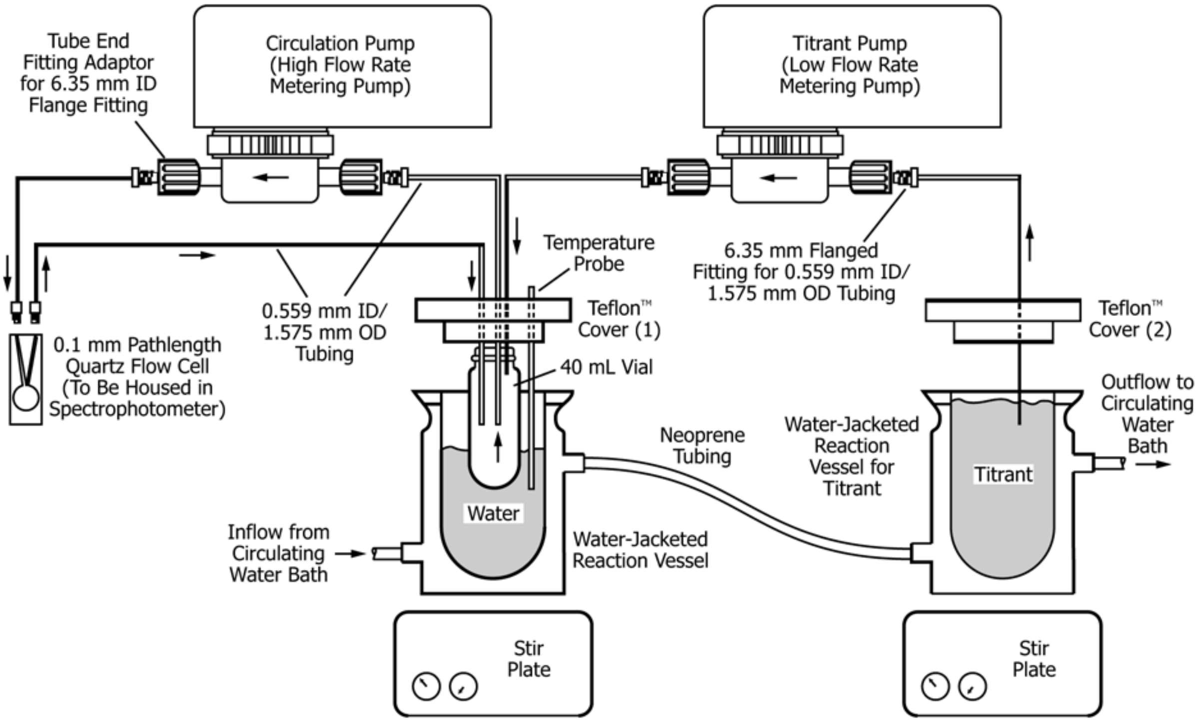

FIG. 2 Automated Titration Apparatus

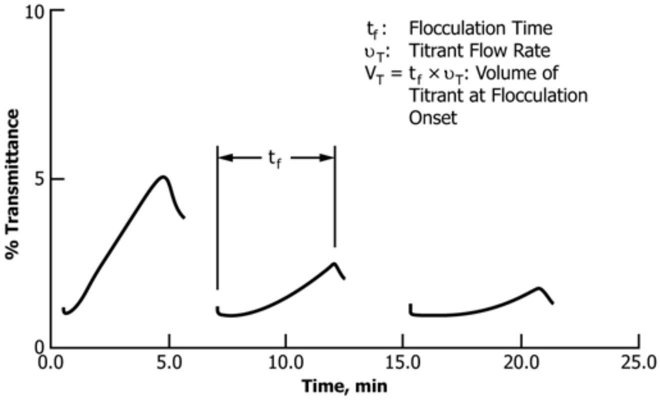

FIG. 3 Onset of Flocculation Peaks Measured at Three Successively Increasing Concentrations (Solvent: Toluene, Titrant: Isooctane)

asphalts. Materials calculated to have low values of P are designated incompatible. Materials calculated to have high P values are designated compatible. Values in P are calculated as a function of two parameters that relate to the peptizability of the asphaltene moiety (the asphaltene peptizability parameter, p_a) and the solvent power of the maltene moiety (the maltene peptizing power parameter, p_o). Values of p_a and p_o are calculated as functions of the quantities C_{min} and FR_{max}. Values of C_{min} and FR_{max} are determined from experimental variables, the weight of asphalt (W_a), the volume of solvent (V_S) to dissolve the weight of asphalt, and the volume of titrant (V_T) added to initiate flocculation.

6. Apparatus

6.1 *UV-visible Spectrophotometer,* wavelength scanning range from 200 to 1000 nm, with adjustable aperture or attenuator.

6.2 *Digital Acquisition System (computer).*

6.3 *Water-Jacketed Reaction Vessel,* 250-mL, two.

6.4 *TFE-fluorocarbon Covers,* two.

6.4.1 *TFE-fluorocarbon Cover No. 1,* (see Fig. 1), threaded to hold a 40 mL reaction vial. Three holes, 1.5 mm diameter, concentric to the cover's center are tapped to set within the inside diameter of the vial when attached to the TFE-fluorocarbon cover. One additional hole, 3.0 mm, is tapped off center, positioned just to the outside of where the reaction vial is positioned in the TFE-fluorocarbon cover. This hole allows the temperature probe to be inserted into the water-filled reaction vessel.

6.4.2 *TFE-fluorocarbon Cover No. 2,* as a lid for the second 200-mL, water-jacketed reaction vessel, containing titrant. Dimensions: thickness, 2.0 mm; diameter, 70 mm. One hole 1.5 mm in diameter tapped through the cover's center. This cover is identical to the cover described in 6.4.1 except for the number of holes, and is not threaded.

6.5 *High Flow Rate Metering Pump*—Flow rate range from 0.5 to 10.0 mL/min; flow rate consistency, ± 0.1 mL/min; and piston chamber resistant to damage from solvent contact.

6.6 *Low Flow Rate Metering Pump*—Flow rate range from 0.100 to 1.000 mL/min; flow rate consistency, ±0.002 mL/min; and piston chamber resistant to damage from solvent contact.

6.7 *Magnetic Stirring Plates,* two.

6.8 *Refrigerated Water Bath Circulator*—Temperature variation, ±0.1°C; temperature range from 0 to 100°C.

6.9 *Quartz Flow Cell,* 0.20 mm path length[3] with 6.35 mm flanged fittings.

6.10 *TFE-fluorocarbon Tubing,* 0.559 mm inside diameter/1.575 mm outside diameter.

6.11 *Reaction Vials,* 40 mL volume capacity.

6.12 *"4-hole" TFE-fluorocarbon cover and "1-hole" TFE-fluorocarbon cover.*

6.13 *TFE-fluorocarbon-Coated Magnetic Stir Bars.*

6.14 *Stopwatch.*

6.15 *Syringe,* 5.000 cc, glass, gas-sealed, and resistant to solvents that it will be used to sample.

6.16 *TFE-fluorocarbon Tube Fittings (4),* including standard 6.35 mm flanged fittings for 0.559 mm inside diameter/1.575 mm outside diameter TFE-fluorocarbon tubing.

6.17 *Neoprene Tubing,* 13 mm inside diameter.

6.18 *Tubing Clamps,* sized to fit 13 mm inside diameter tubing.

6.19 *Digital Probe Thermometer,* °C (calibrated to ±0.2°C). Probe length, >80-mm, probe diameter, 3.0 mm.

6.20 *Graduated Cylinders,* two. Volumes: 1.000 ± 0.001 mL and 10.0 ± 0.1 mL.

6.21 *Argon Gas Supply.*

6.22 *Laboratory Jacks*—Laboratory jacks may be used as stands for metering pumps.

6.23 *Beakers,* two. Volume: 500 mL.

6.24 *Polypropylene Rinse Bottles,* two. Volume: 200 mL.

6.25 *TFE-fluorocarbon Lined Caps,* for 40 mL reaction vials.

7. Reagents

7.1 *Purity of Reagents*—HPLC grade chemicals shall be used in all sample preparations and tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Isooctane* (2,2,4-trimethylpentane), HPLC grade.

7.3 *Toluene,* HPLC grade.

7.4 *Toluene,* reagent grade.

8. Assembly

8.1 *Installation Requirements:*

8.1.1 It is recommended that the following assembly be conducted in a fume hood. The fume hood should be of sufficient size to accommodate all pieces of the apparatus and supplies needed to perform the test method.

8.1.2 The fume hood should be equipped with a pump or house vacuum line for the assembly of a vacuum trap, used during the procedural cleanup step (see 10.2.8).

8.2 *Assembly* (Fig. 2):

8.2.1 *Circulation Loop Assembly*—A sample (circulation loop) is assembled using a high flow rate metering pump plumbed between a short path length flow cell and a TFE-fluorocarbon cover (fitted to a 40 mL reaction vial/200 mL water-jacketed reaction vessel assembly) using 0.559 mm inside diameter/1.575-mm outside diameter TFE-fluorocarbon tubing fitted with standard 6.2 mm flange fittings adaptable to 0.559 mm inside diameter/1.575 mm outside diameter tubing.

8.2.1.1 Position one of the 200-mL, water-jacketed reaction vessels on one of the stir plates, next to the cuvette cell housing of the UV-visible spectrophotometer.

8.2.1.2 Position a 0.1-mm path length flow cell in the cell housing of the spectrophotometer and secure it into place.

8.2.1.3 Position the high flow rate metering pump on a laboratory jack next to the stir plate. Attach a 6.35 mm flanged fitting to one end of a 100 mm long piece of 0.559 mm inside diameter/1.575 mm outside diameter TFE-fluorocarbon tubing and attach the flanged fitting provided with the flow cell to the opposite end of this piece of tubing. Fasten the tubing between the inflow end of the flow cell and the outflow end of the high flow rate metering pump.

8.2.1.4 Attach a second flanged fitting provided with the flow cell to one end of a second 300 mm long piece of 0.559 mm inside diameter/1.575 mm outside diameter TFE-fluorocarbon tubing, leaving the other tubing end free. Fasten the flanged fitting end of this tubing to the outflow end of the flow cell.

8.2.1.5 Attach a 6.35 mm flanged fitting to a third 200 mm long piece of 0.559 mm inside diameter/1.575 mm outside diameter TFE-fluorocarbon tubing, leaving the other tubing end free. Fasten this fitting to the inflow end of the high flow rate metering pump. The two free ends of tubing (8.2.1.4 and

---

[3] The sole source of supply of the apparatus known to the committee at this time is Starna Cells, Inc. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.2.1.5) will lead to the 40 mL reaction vial, positioned through the holes provided in the top of the "4-hole" TFE-fluorocarbon cover.

8.2.2 *Titrant Loop Assembly*—A titrant dispenser (titrant loop) is assembled using a low flow rate metering pump plumbed between the reaction vial and titrant vial using 0.559 mm inside diameter/1.575 mm outside diameter flanged fitting.

8.2.2.1 Position a 200 mL water-jacketed reaction vessel on a second stir plate, next to the high flow rate metering pump/laboratory jack assembly.

8.2.2.2 Position the low flow rate metering pump on a second laboratory jack next to the 200 mL water-jacketed reaction vessel/stir plate assembly.

8.2.2.3 Attach a 300 mm piece of 0.559 mm inside diameter/1.575 mm outside diameter TFE-fluorocarbon tubing fitted with one 6.35 mm flanged fitting to the inflow end of a low flow rate metering pump.

8.2.2.4 The free end of the tubing is placed through the hole provided in the second TFE-fluorocarbon cover into the 200 mL water-jacketed reaction vessel.

8.2.2.5 Attach a 200 mm piece of 0.559 mm inside diameter/1.575 mm outside diameter TFE-fluorocarbon tubing fitted with a standard 6.35 mm flange fitting to the outflow end of the low flow rate metering pump. The free end of tubing runs to the 30 mL reaction vial.

8.2.3 *Refrigerated Water Bath Circulator Assembly:*

8.2.3.1 Using 13 mm inside diameter neoprene tubing and tubing clamps, plumb between the water outflow nozzle of the first 200 mL water-jacketed reaction vessel and the inflow nozzle of the second 200 mL water-jacketed reaction vessel.

8.2.3.2 Plumb two additional pieces of 13 mm inside diameter neoprene tubing between the inflow and outflow couplers of the refrigerated water bath circulator and the two 200 mL water-jacketed reaction vessel's nozzles.

9. Preparation and Calibration

9.1 *UV-Visible Spectrophotometer:*

9.1.1 See the manufacturer's instructions and specifications for operation of the UV-visible spectrophotometer.

9.1.2 Set the UV-visible spectrophotometer to the percent transmittance detection mode.

9.1.3 Set the wavelength of the spectrophotometer to 740 nm (see Note 1).

NOTE 1—A wavelength of 740 nm has been selected as the detection wavelength for the present test method. At this wavelength the light source scatters light when transmitted through a turbid solution of flocculating particles, but will otherwise not promote absorption of light by molecular species (asphaltenes) present in a test sample.

9.1.4 Calibrate the spectrophotometer in accordance with the manufacturer's instruction and specifications. Calibration is to be performed using toluene as the 100 % transmittance spectral background.

9.1.4.1 Guidelines for properly obtaining a reference background spectrum for a reference solvent are referenced in Practices E169.

9.2 *Refrigerated Water Bath Circulator and Water-Jacketed Reaction Vessel Assembly:*

9.2.1 Set the refrigerated circulating water bath temperature to 25.0 ± 0.1°C in accordance with the manufacturer's instruction and specifications.

9.2.2 Fill both 200 mL water-jacketed reaction vessel chambers one-half full with water. Place a small TFE-fluorocarbon stir bar in the bottom of each reaction vessel chamber.

9.2.3 Fill a 40 mL reaction vial with isooctane (2,2,4-trimethylpentane). Place a small clean stir bar into the reaction vial chamber.

9.3 *Pumps and Tubing Assemblies:*

9.3.1 Cut the lengths of the tubing from the high flow rate metering pump and low cell assembly to achieve a minimum total solution-circulation loop assembly volume, < 0.25 mL.

9.3.2 Adjust the high flow rate metering pump to flow at 10 mL/min. Time the flow rate with a stopwatch and 10.0 mL graduated cylinder. Report the average and standard deviation flow rate for three measurements.

9.3.3 Adjust the low flow rate metering pump to flow at 0.350 mL/min. Time the flow rate with a stopwatch and 1.000 mL graduated cylinder. Report the average and standard deviation flow rate for three measurements.

9.4 *Data Acquisition System*—Setup and operation of data acquisition system is performed based on the manufacturer's instructions and specifications.

10. Procedure

10.1 *Preparation of Samples:*

10.1.1 For a single material analysis, label and tare three 40-mL reaction vials fitted with TFE-fluorocarbon lined caps. Weigh into each of the three vials, 0.400 g, 0.600 g, and 0.800 g, respectively, of asphalt or heavy residua to an accuracy of ±0.001 g. Record these sample weights.

10.1.2 Flood each sample vial with argon gas. Seal the reaction vials with TFE-fluorocarbon lined caps (Note 2).

NOTE 2—Dry samples in TFE-fluorocarbon lined capped vials sealed under a blanket of argon gas may be stored for several weeks before samples are tested, if stored in a cool dark place.

10.1.3 A minimum of 4 h prior to testing, add 3.000 ± 0.002 mL of HPLC-grade toluene to each of three samples in a set using a 5.000-cc syringe. Allow the samples to dissolve completely prior to testing (Note 3).

NOTE 3—The minimum time requirement for complete dissolution of most concentrated samples to dissolve at room temperature will be in excess of 4 h. A 24-h period of dissolution is recommended for non-time-restricted applications. Solutions may be gently heated over a water bath to promote more rapid dissolution of sample solution.

10.2 *Sample Analysis:*

10.2.1 Place a small TFE-fluorocarbon coated magnetic stir bar into a 40 mL reaction vial containing the sample solution. Screw the 40 mL reaction vial into the "4-hole" TFE-fluorocarbon cover. Place the 40 mL reaction vial with sample/TFEfluorocarbon cover assembly into the circulation loop 200-mL water-jacketed reaction vessel. Adjust the stir plate stirring rate to stir the sample solution at a relatively high stirring rate to cause a smooth vortex in the stirred solution but slow enough to avoid splashing the solution.

10.2.2 Clear the high flow rate metering pump of solvent that may remain during calibration or prior cleaning (see 10.2.8). Run the two free ends of the TFE-fluorocarbon tubing (extending from the high flow metering pump and flow cell), through two tapped holes in the TFE-fluorocarbon cover. Extend tubing ends down toward the bottom of the 40-mL reaction vial into the solution but avoid contact with the stir bar. Engage the high flow rate metering pump to begin circulating the sample. Adjust the two tube end heights in the solution to eliminate air bubbles in the tubing line.

10.2.3 Place the free end of the TFE-fluorocarbon tubing (extending from the low flow rate metering pump, titrant loop), through the third hole in the TFE-fluorocarbon cover, down into the 40-mL reaction vial. The tubing should be positioned well above the surface of the solution.

10.2.4 Place a thermo-probe through the fourth larger hole in the TFE-fluorocarbon cover. Monitor the temperature of the water bath so that it is maintained at 25.0 ± 0.1°C.

10.2.5 Engage the low flow rate metering pump while simultaneously engaging the data acquisition system to start the analysis.

10.2.6 Allow the titration to proceed until the maximum inflection point in %*T* is detected.

10.2.7 Record the temperature of the solution and the flocculation time (t_f) at the flocculation onset.

10.2.8 At the completion of a test, disengage the pump and withdraw the two ends of tubing from the solution. Flush the remaining solution into a large solvent waste beaker by reengaging the circulation loop pump. Use a squirt bottle filled with toluene to rinse the ends of the tubing. Flush the circulation loop with several milliliters of fresh toluene. Clear the circulation loop after flushing the remaining solvent out of the line. Use vacuum to draw any remaining solvent from the circulation loop.

10.2.9 Repeat the steps given in 10.2 for additional solutions.

11. Calculation

11.1 *Measured Variables:*

11.1.1 Sample weight, W_a (g).

11.1.2 Volume of solvent (toluene), V_S (mL).

11.1.3 Detection wavelength, λ_D (nm).

11.1.4 Titrant flow rate, υ_T (mL/min).

11.1.5 Flocculation time at peak apex (flocculation onset), t_f (min).

11.1.6 Solution temperature at flocculation onset, T_{sln} (°C).

11.2 Calculate the volume of titrant (V_T (mL)) required to initiate flocculation by multiplying the time required to deliver titrant (reported as the peak flocculation time t_f, (min) and the titrant flow rate, υ_T (mL/min) as shown in Eq 1.

$$V_T = t_f \upsilon_T \tag{1}$$

11.3 Calculate the flocculation ratio (*FR*) and the dilution ratio concentration (*C*) for each of the three samples using the values of V_T, V_S, and W_a and Eq 2 and 3.

$$FR = \frac{V_S}{V_S + V_T} \tag{2}$$

$$C = \frac{W_a}{V_S + V_T} \tag{3}$$

11.4 Plot the values of *FR* versus values of *C* for each of the three samples (Fig. 4). Draw a line through the three points. Extrapolate the line to the *x*- and *y*-axes to determine the dilution ratio concentration minimum (C_{min}) and the flocculation ratio maximum (FR_{max}). The value of C_{min} is the point at which the line intercepts the *x*-axis. The *y* intercept is $FR_{\max}$.

11.5 Using values of FR_{max} and C_{min}, calculate Heithaus parameters p_a, the peptizability of asphaltenes, p_o, solvent power of maltenes, and *P*, state of peptization for the sample set using Eq 4-6 respectively.

$$p_a = 1 - FR_{max} \tag{4}$$

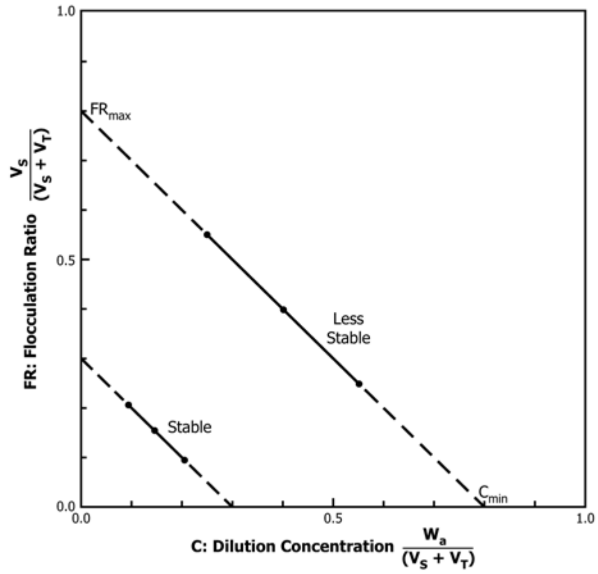

FIG. 4 Flocculation Ratio Versus Dilution Concentration for One Stable Asphalt and One Less Stable Asphalt

$$p_o = FR_{max}\left[\left(\frac{1}{C_{min}}\right)+1\right] \quad (5)$$

$$P = \frac{p_o}{1 - p_a} \quad (6)$$

12. Report

12.1 Report the calculated values of p_a, p_o, and P for each material tested. For duplicate samples tested, report the average values of p_a, p_o, and P.

12.2 Report the average temperature of the solution of flocculation onset calculated from temperatures measured for all titrations in a set vessel to calculate Heithaus parameters.

13. Precision and Bias

13.1 *Precision*[5]—A precision statement for this standard has not been developed. This test method is intended for research or informational purposes only. This standard should not be used for acceptance or rejection of a material for purchasing purposes. A standard deviation range for this test method was determined by testing eight asphalts. The repeatability standard deviation ranges from 0.002 to 0.866. Appendix X1, SAMPLE DATA SETS, reports the precision for multiple measurements obtained for one test asphalt.

13.2 *Bias*—Bias has not been determined since there is no accepted reference material suitable for determining the bias for the procedure in this test method.[6]

14. Keywords

14.1 asphalt; bitumen; coke; colloidal stability; compatibility; heavy oil residua

---

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D04-1019.

[6] The development of an automated Heithaus procedure was undertaken by the Western Research Institute, under FHWA contract, to bring precision to an acceptable level.

APPENDIX

(Nonmandatory Information)

X1. SAMPLE DATA SETS

X1.1 Data from Sample Analysis

X1.1.1 Seven sample sets of a Lloydminster heavy crude oil (asphalt) were prepared by weighing eight samples at an average mass of 0.403 ± 0.002 g, seven samples at an average mass of 0.604 ± 0.004 g, and seven samples at an average mass of 0.803 ± 0.003 g into 30-mL round bottom reaction vials.

X1.1.2 A 3.000 ± 0.005 mL aliquot of HPLC grade toluene was added to each of the 21 samples prior to testing.

X1.1.3 Each sample vial was blanketed under dry Argon gas, caped with a Teflon lined cap, and stored in a dark environment.

X1.1.4 Samples were allowed to stand undisturbed for no less than a 24 h period prior to analysis as described by the procedure given in 10.2.

X1.1.5 As per subsection 9.3.3, the low flow rate metering pump was calibrated to flow at 0.301 ± 0.001 mL/min for sample tests conducted.

X1.1.6 All tests were conducted in order of sample set, refer to Table X1.1.

X1.1.7 Heithaus compatibility parameters were determined as described in Section 11. The data that were determined are presented in Tables Table X1.1 and Table X1.2.

X1.1.8 The data presented in Table Table X1.2, represent typical values for the three Heithaus compatibility parameters for the material tested.

TABLE X1.1 Sample Masses (g) Prepared for Seven Sample Sets of a Lloydminster Heavy Crude Oil (Asphalt) Material

Set	Sample 1	Sample 2	Sample 3
1	0.40201	0.60092	0.80000
2	0.40440	0.60052	0.80572
3	0.40508	0.60476	0.80254
4	0.40143	0.60170	0.80046
5	0.40427	0.60838	0.80037
6	0.40053	0.60095	0.80528
7	0.40242	0.60866	0.80725
Average	0.403	0.604	0.803
Standard Deviation	0.002	0.004	0.003

TABLE X1.2 Heithaus Compatibility Parameters Measured for Seven Sample Sets of a Lloydminster Heavy Crude Oil (Asphalt) Material

Set	P_a	P_o	P
1	0.6639	1.10	3.27
2	0.6678	1.03	3.09
3	0.6778	0.85	2.63
4	0.6762	0.83	2.57
5	0.6621	1.00	2.97
6	0.6773	0.77	2.39
7	0.6627	0.84	2.49
Average	0.670	0.9	2.8
Standard Deviation	0.007	0.1	0.3

REFERENCES

(1) Heithaus, J. J., "Measurements and Significance of Asphaltene Peptization," *American Chemical Society, Div Petrol Chem Preprints*, Vol 5, 1960, pp. A23-A37.

(2) Heithaus, J. J., "Measurements and Significance of Asphaltene Peptization," *Journal of Inst. Petrol*, Vol 48, 1962, pp. 45-53.

(3) Redelius, P. G., "Ageing of Bitumen Studied by Colloidal Stability," IV International Conference: "Durable and Safe Pavements," Kielce, May 5-6, 1998.

(4) Schabron, J. F., and Pauli, A. T., "Coking Indexes Using the Automated Heithaus Titration and Asphaltene Solubility," *American Chemical Society, Div. Petrol. Chem. Preprints*, Vol 44, No. 2, 1999, pp. 187-189.

(5) Barth, Edwin J., *Asphalt Science and Technology*, Gordon and Breach Science Publishers, Inc., New York, NY, 1962.

(6) Redelius, P. G., "Solubility Parameters and Bitumen," *Fuel*, Vol 79, 2000, pp. 27-35.

(7) Andersen, S. I., "Flocculation Onset Titration of Petroleum Asphaltenes," *Energy and Fuels*, Vol 13, 1999, pp. 315-322.

(8) Nellensteyn, F. J., "Relation of the Micelle to the Medium in Asphalt," *Ins Petrol Technology*, Vol 14, 1928, pp. 134-138.

(9) Pfeiffer, J. P., and Saal, R. N. J., "Asphalt Bitumen as Colloidal System," *Phys. Chem.*, Vol 44, 1940, pp. 139-149.

(10) Pauli, A. T., and Branthaver, J. F., "Relationship Between Asphaltenes, Heithaus Compatibility Parameters, and Asphalt Viscosity," *Petroleum Science and Technology*, Vol 16, Nos. 9 and 10, 1998, pp. 1125-1147.

(11) Pauli, A. T., and Branthaver, J. F., "Rheological and Compositional Definitions of Compatibility as They Relate to the Colloidal Model of Asphalt and Residua," *American Chemical Society Div Petrol Chem Preprints*, Vol 44, No. 2, 1999, pp. 190-193.

(12) Hotier, G., and Robin, M., "Action De Divers Diluants Sur Les Produits Pétrliers Lourds: Mesure, Interprétation et Prévision de la Flocculation des Asphalténes," *Revue de L'Institut Francais du Pétrole*, Vol 38, No. 1, 1983, pp. 101-120.

(13) Reichert, C., Fuhr, B., and Klien, L., "Measurement of Asphaltene Flocculation in Bitumen Solutions," Presented at the 36th Annual Technical Meeting of the Petroleum Society of CIM, Edmonton, 1985, *Paper No. 85-36-18*, pp. 757-765.

(14) Pauli, A. T., "Asphalt Compatibility Testing Using the Automated Heithaus Titration Test," *American Chemical Society, Div of Fuel Chem Preprints*, Vol 41, No. 4, 1996, pp. 1276-1281.

(15) Horst, L., Rahimian, I., and Schorling, P., "The Colloidal Stability of Crude Oils and Crude Oil Residues," *Petroleum Science and Technology*, Vol 17, Nos. 3 and 4, 1999, pp. 349-368.

(16) Robertson, R. E., Branthaver, J. F., Harnsberger, P. M., Petersen, J. C., Dorrence, S. M., McKay, J. F., Turner, T. F., Pauli, A. T., Huang, S.-C., Huh, J.-D., Tauer, J. E., Thomas, K. P., Netzel, D. A., Miknis, F. P., Williams, T., Duvall, J. J., Barbour, F. A., Wright, C., Salmans, S. L., and Hansert, A. F., 2001d, "Fundamental Properties of Asphalts and Modified Asphalts," Volume II: Final Report, New Methods, FHWA-RD-99-213. U. S. Department of Transportation, Federal Highway Administration, McLean, VA.