Designation: D6703 – 19

Standard Test Method for Automated Heithaus Titrimetry[1]

This standard is issued under the fixed designation D6703; 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 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 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.

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

1.5 *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]

D8 Terminology Relating to Materials for Roads and Pavements

D3279 Test Method for*n*-Heptane Insolubles

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials

D4124 Test Method for Separation of Asphalt into Four Fractions

D8055 Guide for Selecting an Appropriate Electronic Thermometer for Replacing Mercury Thermometers in D04 Road and Paving Standards

E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature

E644 Test Methods for Testing Industrial Resistance Thermometers

3. Terminology

3.1 Refer to Terminology D8 for definitions of terms relating to materials for roads and pavements.

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.2 *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.

3.2.3 *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).

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

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

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

3.2.7 *flocculation, n*—the process of aggregation and coalescence into a flocculent mass. See Test Method D3279.

3.2.8 *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.

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

---

[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 Dec. 15, 2019. Published January 2020. Originally approved in 2001. Last previous edition approved in 2014 as D6703 – 14. DOI: 10.1520/D6703-19.

[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.

4. Summary of Test Method

4.1 Three 40-mL reaction vials shall be tared (Fig. 1). Three samples of asphalt of weights 0.400 g, 0.600 g, and 0.800 g shall be transferred to each of three reaction vials. Toluene (3.000 mL) shall be 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 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 $\%T$ increase with time up to the flocculation onset point, after which values of $\%T$ 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, shall be used as the input data required to calculate compatibility parameters.

5. Significance and Use

5.1 This test method is intended as a laboratory diagnostic tool for estimating the colloidal stability of bitumen asphalt,

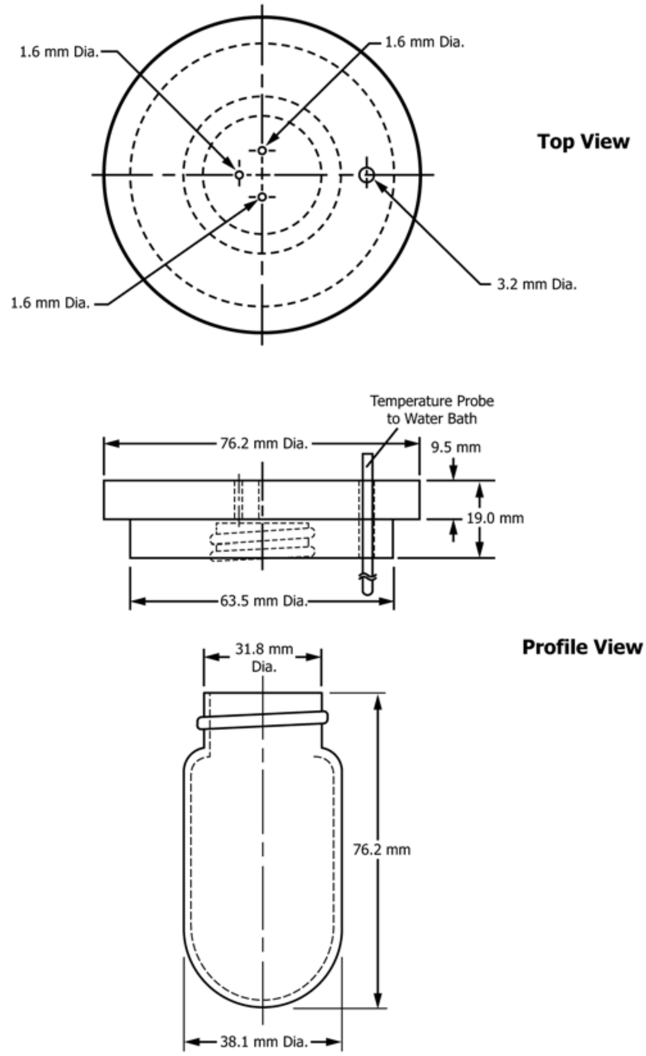

FIG. 1 Reaction Vial (40 mL) with TFE-fluorocarbon Cover and Temperature Probe

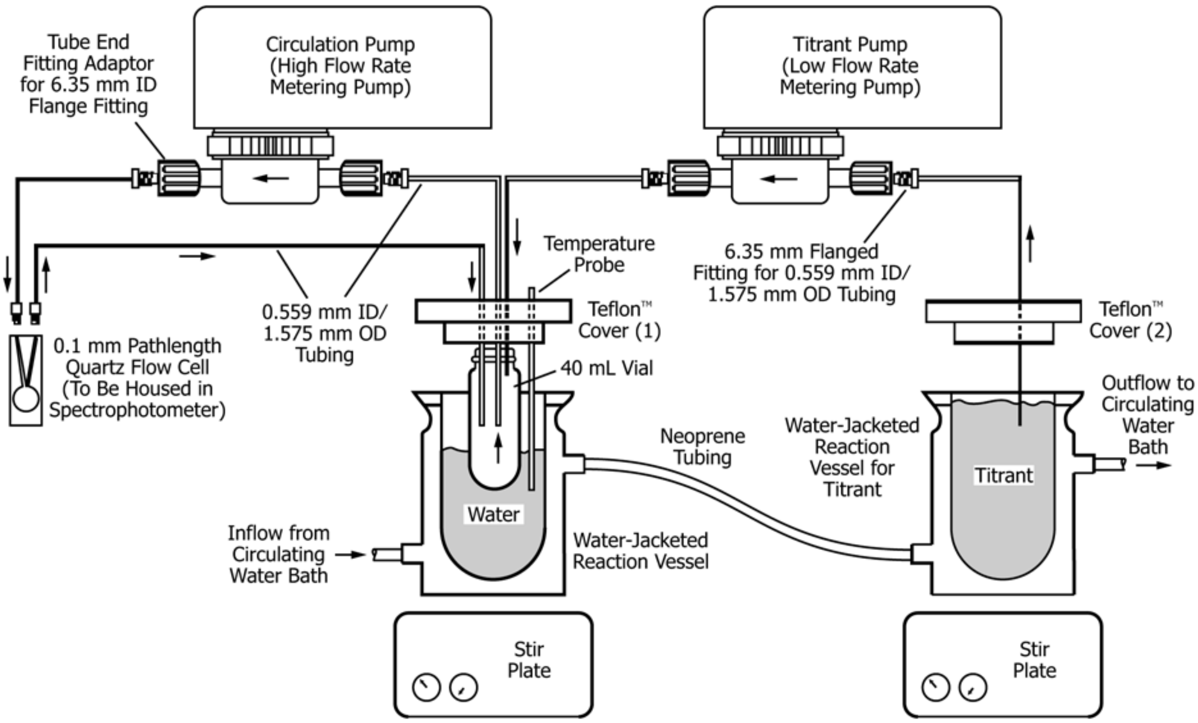

FIG. 2 Automated Titration Apparatus

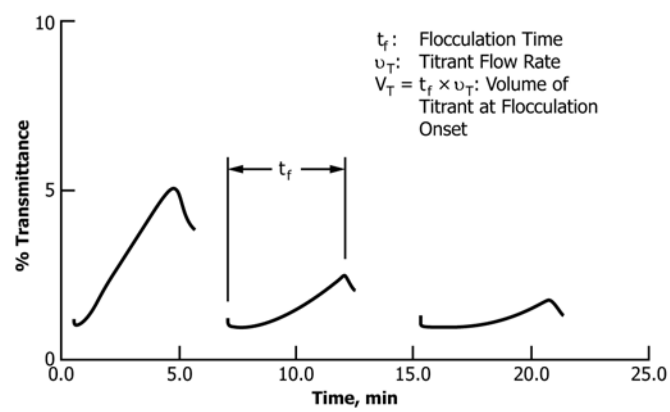

FIG. 3 Onset of Flocculation Peaks Measured at Three Successively Increasing Concentrations (Solvent: Toluene, Titrant: Isooctane)

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 and D4124 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 may influence 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, may be recommended as a measure of 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 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,* shall have a wavelength scanning range from 200 to 1000 nm, with adjustable aperture or attenuator.