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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Engler Specific Viscosity of Tar Products[1]

This standard is issued under the fixed designation D 1665; 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 covers the determination of specific viscosity of tars and their fluid products. It does not determine absolute viscosity, but is an empirical flow test. Only by conforming strictly to requirements of the test method are reproducible results obtained.

1.2 *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:*

D 140 Practice for Sampling Bituminous Materials[2]

E 1 Specification for ASTM Thermometers[3]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

3. Terminology

3.1 *Definition:*

3.1.1 *Engler specific viscosity*—the ratio obtained by dividing the time of flow, in s, of 50 mL of material using an Engler viscosimeter at a selected temperature by a factor representing the time of flow, in s, for an equal volume of water at 25°C. The usual temperatures for determination of specific viscosity of tar materials are 25°C, 40°C, 50°C, and 100°C, and generally the temperature is so selected that the specific viscosity is not more than 45.

4. Summary of Test Method

4.1 The time, in s, is measured for a fixed volume of liquid material to flow through an efflux tube under an accurately reproducible head and at a closely controlled temperature. The Engler specific viscosity is then calculated by dividing the efflux time by the viscometer calibration factor as determined by making the same efflux measurement for water.

5. Significance and Use

5.1 This test method is useful in characterizing the consistency of tar and tar distillates by measuring their flow properties. It is applicable to materials that are readily liquid at temperatures up to 100°C.

6. Apparatus

6.1 *Engler Viscosimeter* as shown in Fig. 1, consisting of the following:

6.1.1 *Cup*—This is a gold-plated cylindrical brass vessel of 106.0 ± 1.0 mm, *A*, inside diameter, closed at the top by a double walled lid. To the rounded bottom is attached a metal-encased tapered platinum efflux tube 20.0± 0.1 mm, *H*, long with an inside diameter of 2.90± 0.02 mm, *E*, at the top and 2.80± 0.02 mm, *F*, at the bottom. The efflux tube shall project through and extend 3.0 ± 0.2 mm, *G*, below a jacket that surrounds the cup and shall have a bottom outside diameter, including its surrounding metal, of 4.5 ± 0.2 mm, *I*. Three metal measuring points, spaced equidistantly around the circumference of the cup, are fastened to the sides and extend inwardly approximately 7 mm, then turn up at a right angle and end in sharp points which are located 52.0 ± 0.5 mm, *D*, vertically above the lower end of the efflux tube and 25.0 ± 1.0 mm, *C*, above the lowest portion of the cylindrical sidewall of the cup. They serve both for indicating when the instrument is level and for measuring the charge of material, which is approximately 250 mL.

6.1.2 *Jacket*—The cup is surrounded by a jacket which holds water or other suitable liquid serving as a constant temperature bath. In the type illustrated, the jacket is provided with a thermometer clamp and stirring device. A tripod supports the apparatus and also carries a ring burner by means of which the bath is heated. Adjustable legs on the tripod serve to level the instrument. Other arrangements of outer baths, supports, and stirring devices are acceptable, especially when it is desired to use more than one standardized cup in a single bath.

6.1.3 *Stopper*—The efflux tube in the cup is closed or opened by the insertion or withdrawal of a tapered hardwood stopper which, to leave the tube open, can be suspended by its brass pin from the hook on the cover. The stopper shall be a smooth, round wooden rod 180 mm long and 8 mm in diameter, with a brass wire pin 20 mm and 1.83 mm in diameter long inserted diametrically through the rod at a point 50 mm from the lower end, and tapered uniformly below this

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[2] *Annual Book of ASTM Standards*, Vol 04.03.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

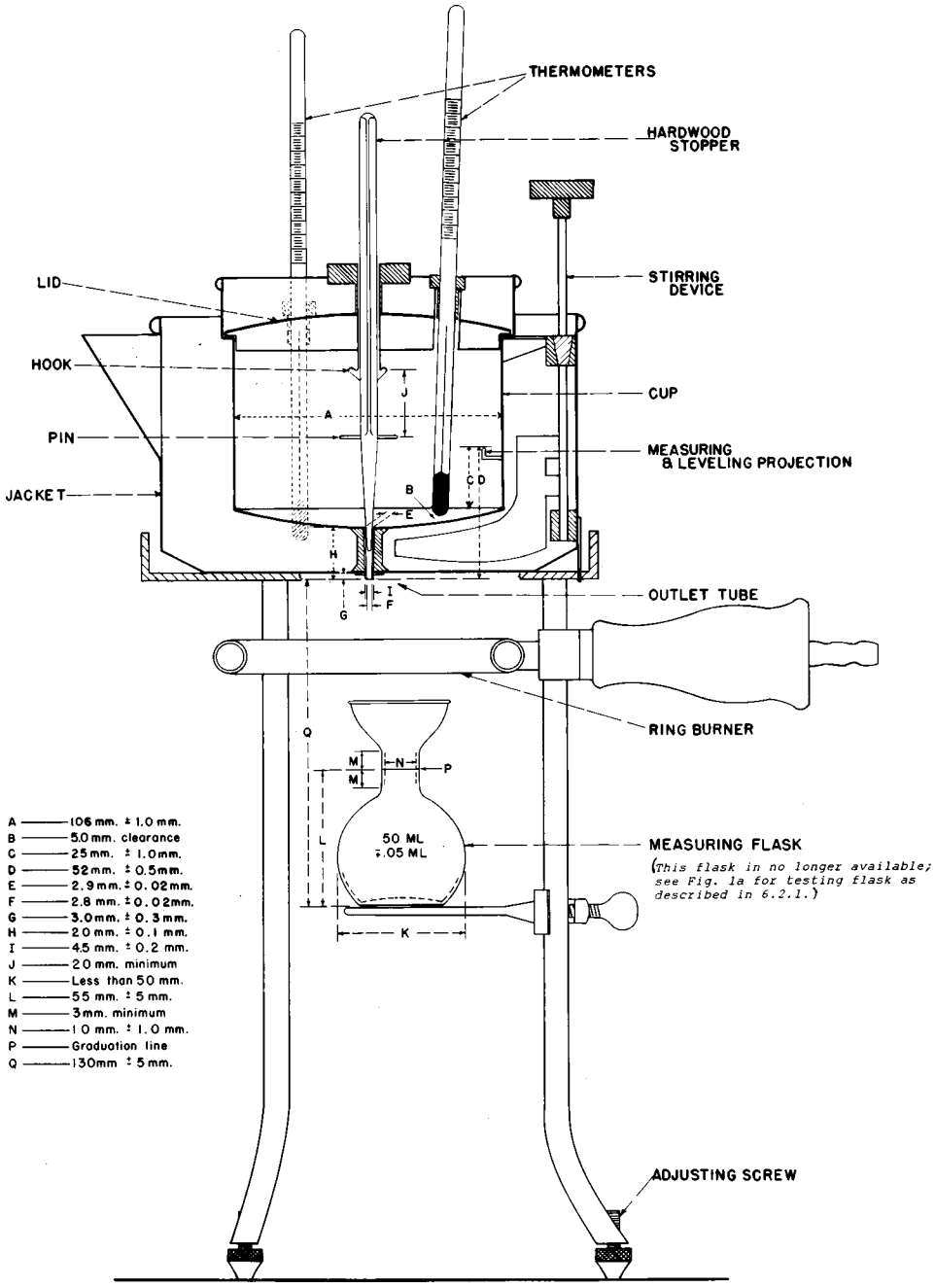

FIG. 1 Engler Viscosimeter

pin to end in a circular plane 1.6 to 2.0 mm in diameter. Above the pin the rod shall be planed or grooved on four sides to a depth of 1 mm to prevent any possible restriction of air flow.

6.2 *Receivers*—Two types are required as follows:

6.2.1 *Testing Flask*—50 mL graduate calibrated at 20°C (see Fig. 2).

6.2.2 *Calibration Flask*—For standardization purposes there shall be available a Kohlrausch flask, Fig. 3, with top enlarged above the graduation mark and calibrated to contain 200 ± 0.1 mL at 20°C.

6.3 *Thermometers*—ASTM Engler Viscosity Thermometers 23C, 24C, and 25C as required, and conforming to the requirements for these thermometers as specified in Specification E 1.

6.4 *Timer*—Stop watch or other timing device graduated in divisions of 0.2 s or less, and accurate to within 0.1 % when tested over a 60-min period.

6.5 *Strainer*—300 mm ASTM sieve conforming to Specification E 11.

7. Sampling

7.1 Samples from shipments or production vessels shall be taken in accordance with Practice D 140 and shall be free of foreign substances. Thoroughly heat and stir the sample before removing a representative portion for the determination.

8. Preparation of Sample

8.1 Stir the sample until it is homogeneous, using heat if

FIG. 2 Engler Viscosity Apparatus Using the 50 mL Graduate

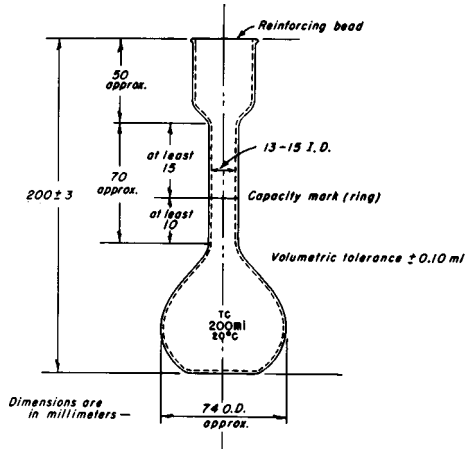

FIG. 3 Kohlrausch Sugar Flask

necessary. Avoid inclusion of air bubbles, loss of volatile or other effects, which may influence the viscosity. Strain a representative portion of the sample through the strainer to eliminate particles, and proceed in accordance with Section 10. Strain the material directly into the viscosimeter if preferred.

9. Standardization and Calibration of Viscosimeter

9.1 The efflux time for 200 mL of distilled water at 20.0°C with an acceptable Engler viscosimeter shall be between 50.0 and 52.0 s. Determine this time and the factor representing the efflux time for 50.0 mL of water at 25.0°C, as described in 9.1.1-9.1.6:

9.1.1 Clean the inner vessel and efflux tube of the viscosimeter with appropriate solvents, and finish by washing several times with pure methyl or 95 % ethyl alcohol and rinsing several times with distilled water.

NOTE 1—In cleaning the viscosimeter take particular precautions to avoid injury to the efflux tube and measuring points. Use only a soft cloth in the cup, and soft tissue in the efflux tube. Avoid wires or similar substances and corrosive liquids. To prevent an air seal, keep the lid and lip of the cup clean at all times. After a viscosimeter has been used with bituminous materials, pay particular attention to cleaning the metal surrounding the bottom end of the efflux tube. Failure to do this may cause erratic and erroneous results.

9.1.2 Immediately after cleaning the viscosimeter, close the efflux tube with a stopper which has never been in contact with tar, oil, or similar materials. Fill the outer bath with water at slightly below or above 20°C as found necessary to maintain the inner temperature at 20°C. Fill the inner vessel approximately to the top of the fixed gage points with freshly boiled distilled water at 20.0°C. Level the instrument so the tips of the gage points lie in a plane parallel to the surface of the water, and add or remove water with a pipet until its surface is even with the extreme tips of all gage points. Place the lid and thermometer in position and maintain the inner temperature at 20.0°C for at least 3 min with frequent stirring; agitate the contents of the inner cup by holding the stopper firmly and rotating the cover back and forth and around, occasionally stirring the outer bath. Dry the bottom of the efflux tube and the area surrounding it by wiping. Carefully lift the stopper until water runs into and completely fills the efflux tube, and adjust until a hemispherical drop about 4.5 mm in diameter hangs from and covers the lower end of the tube. Then allow to stand without agitation for 1 min.

9.1.3 Place a dry calibration flask 240 ± 10 mm below the discharge end, and adjust it so the flow will strike the narrow portion of the neck of the flask near or slightly below the calibration line. Start the timer and simultaneously withdraw the stopper, suspending it by the lid hook. Determine the time, in seconds, for flow of 200 mL. Repeat this determination, starting the flow under conditions described above until at least three successive determinations, varying not more than 0.2 s, are obtained. If the results obtained from three or more tests do not check within 0.2 s, clean the viscosimeter again and make additional trials, until three or more results agree within 0.2 s.

9.1.4 Make another series of determinations as above, starting with the instrument freshly washed with alcohol, then with distilled water and refilled as before. The average results from the second series shall agree with the average from the first series within 0.2 s. Take the efflux time for 200 mL at 20.0°C as the mean of the averages of at least two series of determinations agreeing within 0.2 s. This time for an acceptable viscosimeter shall be between 50.0 and 52.0 s.

9.1.5 Make additional runs as necessary, beginning with a newly cleaned viscosimeter until two successive series are in substantial agreement.

9.1.6 The factor representing efflux time for 50 mL of water at 25.0°C has been found to be equivalent to the efflux time for 200 mL of distilled water at 20.0°C multiplied by 0.224.

10. Procedure

10.1 Thoroughly clean and dry the cup and outlet tube of the viscosimeter as described in 9.1.1 and insert the stopper. Fill the outer bath and bring it to the required temperature of test. Maintain the bath not more than 1°C high for tests at 25°C,

40°C, or 50°C, and not more than 2 or 3°C high for tests at 100°C.

10.2 Pour the material into the cup until it exactly reaches the tops of the three measuring points when the instrument is level. Position the 50 mL testing flask so that the bottom of the flask is 130 ± 5 mm below the discharge end of the efflux tube, and adjust it so that the effluent will strike the narrow portion of the neck of the flask near or slightly below the calibration line.

10.3 Place the lid and inner thermometer into position and maintain the bath, with frequent agitation, at such a temperature that the material in the viscosimeter cup remains at the test temperature. Maintain these conditions for 3 min. Check the accuracy of the temperature reading by holding the stopper firmly in position and rotating the cover at short intervals during the first 2 min, but do not disturb the material during the last minute. When these conditions have been met, withdraw the stopper from the efflux tube, simultaneously start the timer, and suspend the stopper by the hook on the cover. Determine the time in seconds for 50 mL of material to flow from the viscosimeter.

NOTE 2—Once the material has started to flow through the efflux tube, do not use the ring burner, but maintain the required temperature by the addition or removal of water at suitable temperatures, or by an auxiliary burner momentarily directed at the outside cylindrical portion of the water jacket.

11. Calculation

11.1 Calculate the Engler specific viscosity by dividing the time of flow for 50 mL of material at the selected temperature by the factor, as previously determined, according to the following formula:

$$\text{Engler specific viscosity at } t = \frac{\text{s for flow of 50 mL at } t}{\text{factor}} \quad (1)$$

where:

t = selected temperature of test, °C.

12. Precision and Bias

12.1 Results should not differ from the mean by more than the following amounts:

Repeatability (one operator and apparatus)	4 %
Reproducibility (different operators and apparatus)	6 %

13. Keywords

13.1 tar; engler specific gravity