



Standard Test Method for Apparent Viscosity of Asphalt Emulsion Residues and Non-Newtonian Bitumens by Vacuum Capillary Viscometer¹

This standard is issued under the fixed designation D 4957; 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 procedures primarily designed to determine the apparent viscosities of residues obtained by distilling asphalt emulsions according to Test Methods D 244. It is also recommended for use on non-Newtonian asphalts at any temperature within the capability of the apparatus. This test method is useful in characterizing rheological properties of non-Newtonian bitumens as a function of shear rate under the conditions of the test method. This test is run in straight open-end tube viscometers, normally at 140°F (60°C), but is suitable for use at other temperatures. It is applicable over the range from 50 to 500 000 P (5 to 50 000 Pa·s).

NOTE 1—The precision for this test method is based on determinations made at 140°F (60°C).

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 244 Test Methods for Emulsified Asphalts²

D 2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer²

E 1 Specification for ASTM Thermometers³

E 77 Test Method for Inspection and Verification of Thermometers³

3. Terminology

3.1 Definitions:

3.1.1 *apparent viscosity*—the determined viscosity obtained by the test method under description. Viscosity is the resistance to deformation or internal friction of a liquid expressed as the

ratio of the shear stress to shear rate, whether this ratio is constant or not. The unit of viscosity obtained by dividing the shearing stress in dynes/cm² by the rate of shear in reciprocal seconds is called the poise (P). The SI unit of viscosity is the pascal-second (Pa·s) with dimensions of N·s/m², and is equivalent to 10 P.

3.1.2 *Newtonian liquid*—a liquid in which the rate of shear is proportional to the shearing stress. The constant ratio of the shearing stress to rate of shear is the viscosity of the liquid. If the ratio is not constant, the liquid is non-Newtonian.

NOTE 2—A power law fluid is a material in which the relation between the log of the shear stress is linear with the log of the shear rate. The slope of this relation is called the shear susceptibility, *C*. If *C* is less than unity, the material is classified as pseudoplastic and the apparent viscosity decreases with increased stress. If *C* is greater than one, the material is dilatant and the apparent viscosity increases with stress. If *C* is unity the material shows Newtonian flow. Most real materials show some non-Newtonian behavior and the apparent viscosity, computed as stress divided by the shear rate, is reported.

3.1.3 *rheogram*—a rheological diagram which shows how the apparent viscosity of a material varies with the shear rate. An apparent viscosity at a specific shear rate, normally 1 s⁻¹ can be estimated from this plot. A typical rheogram with an example is shown in Fig. 1.

4. Summary of Test Method

4.1 The time is measured for a fixed volume of the liquid to be drawn up through a straight open-end capillary tube by means of vacuum, under closely controlled conditions of vacuum and temperature. The apparent viscosity in poises is calculated by multiplying the flow time in seconds by the appropriate viscometer calibration factor or calculated viscometer constant.

5. Significance and Use

5.1 This test method is useful for characterizing the flow behavior of asphalt emulsion residues and non-Newtonian bitumens. However, since non-Newtonian viscosity values depend on the level of shearing stress, its duration, and the shear history of the material, a non-Newtonian viscosity is not a unique material property. Instead, it is a parameter which is characteristic of the fluid-viscometer system under the conditions of the measurement procedure. Therefore, comparisons of

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

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

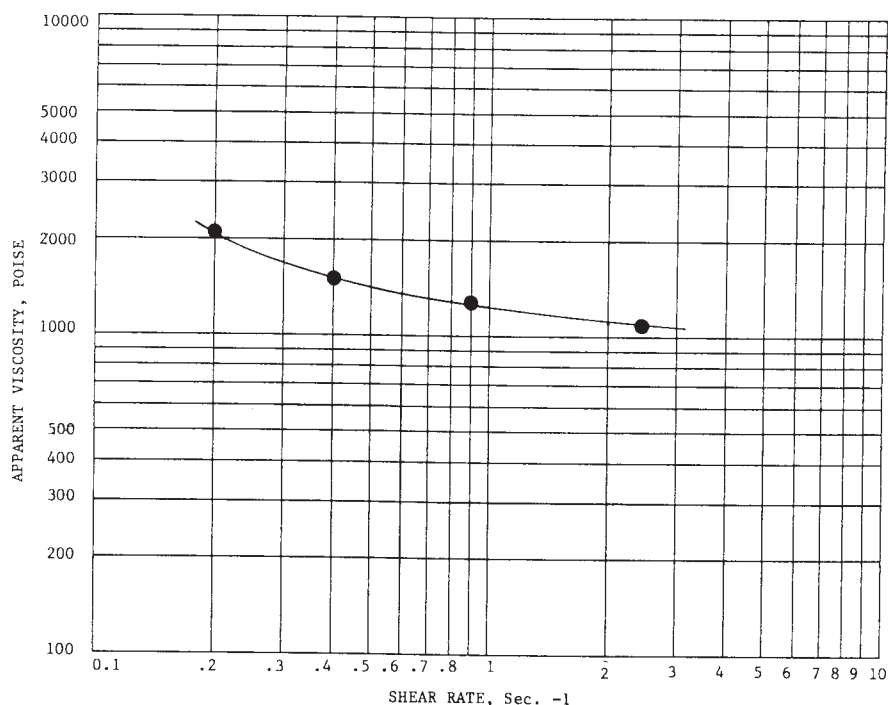


FIG. 1 Typical Log-Log Rheogram Plot with Example

non-Newtonian material behavior should only be made using apparent viscosities determined in similar viscometers under similar conditions of shearing stress and stress history. Procedures of sample preparation are especially important for repeatability or reproducibility of test results.

6. Apparatus

6.1 *Viscometers*—Capillary types, made of borosilicate glass, annealed, suitable for this test are as follows:

6.1.1 *Modified Koppers Vacuum Viscometer*, as described in Annex A1. Calibrated viscometers are available from commercial suppliers. Details regarding calibration of viscometers are given in A1.3.

6.2 *Thermometers*—Calibrated liquid in glass thermometers of an accuracy after correction of 0.04°F (0.02°C) can be used or any other thermometric device of equal accuracy. ASTM Kinematic Viscosity Thermometers 47F and 47C are suitable for the most commonly used temperature of 140°F (60°C). See Test Method D 2171, Table on Kinematic Viscosity Test Thermometers for details on specific thermometers.

6.2.1 The specified thermometers are standardized at “total immersion”, which means immersion to the top of the mercury column with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature. The practice of completely submerging the thermometer is not recommended. When thermometers are completely submerged, corrections for each individual thermometer based on calibration under conditions of complete submergence must be determined and applied. If the thermometer is completely submerged in the bath during use, the pressure of the gas in the expansion chamber will be higher or lower than during standardization, and may cause high or low readings of the thermometer.

6.2.2 It is essential that liquid-in-glass thermometers be calibrated periodically using the technique given in Test Method E 77 (see Appendix X1).

6.3 *Bath*—A bath suitable for immersion of the viscometer so that the liquid reservoir or the top of the capillary, whichever is uppermost, is at least 20 mm below the upper surface of the bath liquid, and with provisions for visibility of the viscometer and the thermometer. Firm supports for the viscometer shall be provided. The efficiency of the stirring and the balance between heat loss and heat input must be such that the temperature of the bath medium does not vary by more than ±0.05°F (±0.03°C) over the length of the viscometer or from viscometer to viscometer in the various bath positions.

6.4 *Vacuum System*—A vacuum system⁴ capable of maintaining a vacuum to within ±0.5 mm of the desired level up to and including 500 mm Hg. The essential system is shown schematically in Fig. 2. Glass tubing of 6.35 mm (¼ in.) inside diameter should be used, and all glass joints should be airtight so that when the system is closed, no loss of vacuum is indicated by the open-end mercury manometer having 1-mm graduations. A vacuum or aspirator pump is suitable for the vacuum source.

6.5 *Timer*—A stopwatch or other timing device graduated in divisions of 0.1 s or less and accurate to within 0.05 % when tested over intervals of not less than 15 min shall be used. Electrical timing devices may be used only on electrical circuits the frequencies of which are controlled to an accuracy of 0.05 % or better.

6.5.1 Alternating-current frequencies that are intermittently and not continuously controlled, as provided by some public

⁴ The vacuum control system marketed by Cannon Instrument Co., P.O. Box 16, State College, PA, 16801, has been found satisfactory for this purpose.

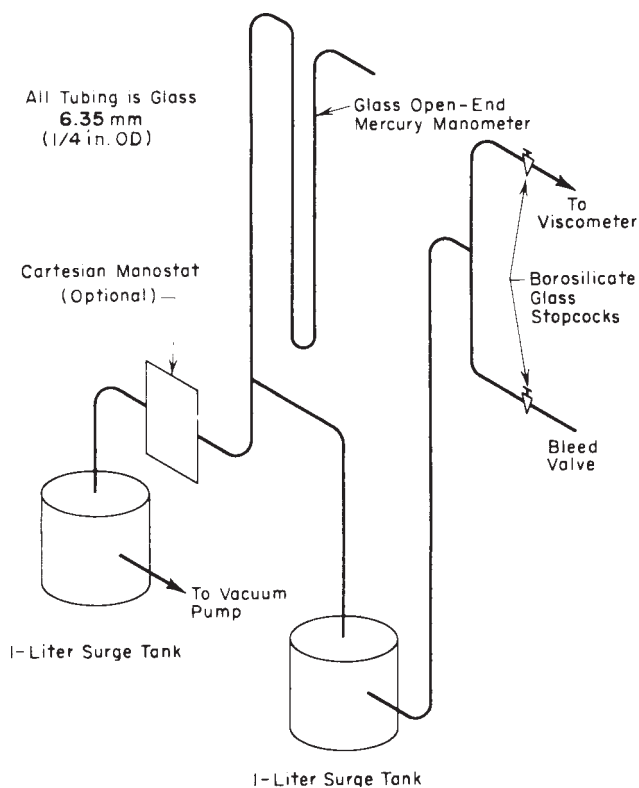


FIG. 2 Suggested Vacuum System for Vacuum Capillary Viscometers

power systems, can cause large errors, particularly over short timing intervals, when used to actuate electrical timing devices.

6.6 *Sample Preparation Oven*—A suitable oven for semi-continuous operations with control of temperature up to $396 \pm 4^\circ\text{F}$ ($202 \pm 2^\circ\text{C}$) is required. It should have a fast heating rate capability in order not to delay testing when needed on short notice.

7. Sample Preparation

7.1 *Asphalt Emulsion Residue*—If the sample is the residual product from the emulsion distillation test at 500°F (260°C), pour a suitable portion of the total residue into a 50-mL beaker and allow to cool to $356 \pm 9^\circ\text{F}$ ($180 \pm 5^\circ\text{C}$). Then stir this portion of the sample at 1 r/s for 10 s. Following this, pour the proper amount into the viscometer as in Section 8. Alternatively, allow this portion of the residue to cool completely and set aside for future testing. This material should then be handled as in 7.2.

7.2 *Ambient Sample*—Heat the sample in an oven maintained at $383 \pm 4^\circ\text{F}$ ($195 \pm 2^\circ\text{C}$). Stir the sample occasionally until homogeneous and pour into a 50-mL preheated beaker. Stir the sample at approximately 1 r/s for 10 s.

NOTE 3—Because of the nature of some asphalts and asphalt emulsion residues, their shear and thermal history prior to testing may cause variations in test results. Careful sample preparation is most important for consistent test results.

NOTE 4—In cases where the asphalt is not sufficiently fluid to pour at 356°F or too fluid to pour without splattering during transfer into the viscometer; other pouring temperatures may be used providing there is agreement between interested parties.

8. Procedure

8.1 Follow the general procedure described as follows, however, the specific details of the Modified Koppers viscometer are described in Annex A1.

8.1.1 Maintain the bath at the test temperatures to within $\pm 0.05^\circ\text{F}$ ($\pm 0.03^\circ\text{C}$). Apply the necessary corrections, if any, to all thermometer readings.

8.1.2 Select a clean, dry viscometer that will give a flow time between 50 and 200 s for the C zone of a Modified Koppers viscometer. Preheat the viscometer filling tube and capillary tube separately in the oven at $383 \pm 4^\circ\text{F}$ ($195 \pm 2^\circ\text{C}$) for 5 min to assist in eliminating air bubbles when the sample is poured.

8.1.3 Charge the viscometer filling tube by pouring the prepared sample to within ± 2 mm of the fill line.

NOTE 5—In cases where highly viscous materials are being tested, the possibility of air entrapment in the sample is greater. To alleviate this problem, a slight vacuum on the fill tube before the capillary tube is placed in position is suggested. This can be accomplished using a light vacuum source attached to the fill tube for 30 s. A simple cork and tube should suffice to achieve the desired effects.

8.1.4 Place the charged viscometer filling tube in an oven maintained at $383 \pm 4^\circ\text{F}$ ($195 \pm 2^\circ\text{C}$) for a period of 10 ± 2 min to allow large air bubbles to escape.

8.1.5 Remove the viscometer filling tube and capillary tube from the oven and then properly position the capillary tube into the filling tube. Insert the viscometer in a holder and position the viscometer in the bath so that the uppermost timing mark is at least 20 mm below the surface of the bath liquid.

NOTE 6—Capillary action on certain small-diameter viscometer tubes may cause a rise of the sample above the first timing mark. If this is a problem, the capillary tube should be withheld from contact with the sample for a few minutes prior to running the test, then positioned quickly and allowed to equilibrate a minute or two.

8.1.6 Establish the vacuum to within ± 0.5 mm of the desired level and connect the vacuum system to the viscometer with the toggle valve or stopcock closed in the line leading to the viscometer.

8.1.7 After the viscometer has been in the bath for 30 ± 5 min, start the flow of asphalt in the viscometer by opening the toggle valve or stopcock in the line leading to the vacuum system.

8.1.8 Measure to within 0.1 s the time required for the leading edge of the meniscus to pass between successive pairs of timing marks.

8.1.9 Upon completion of the test, clean the viscometer. There are numerous methods that can be used. Two suggested methods are given in A1.4 and A1.5.

9. Calculation

9.1 Select the appropriate viscosity constant for any set of timing marks from the calibration constants given by the manufacturer.

9.1.1 Using these appropriate viscometer constants, calculate and report the apparent viscosity to three significant figures according to the following equation:

$$\text{Apparent Viscosity, } \eta_{\text{zone}} = K_{\text{zone}} \cdot t \quad (1)$$

where:

- η_0 = apparent viscosity in poise for particular timing zone,
- t = time it takes the asphalt meniscus to traverse the particular zone or interest in seconds, and
- K_{zone} = viscometer constant for the particular timing zone in P/s.

9.1.2 The shear rate associated with this apparent viscosity is calculated according to the following equation:

$$\text{Shear rate, } \dot{\gamma}_{zone} = 4 L s^{-1} / R t = \text{Shear Constant} / t \quad (2)$$

where:

- $\dot{\gamma}$ = shear rate in reciprocal seconds, s^{-1}
- R = tube radius in mm,
- L = length in mm of the zone for which the time was recorded,
- t = time in the zone in seconds, s, and values for shear constant are found in Table A1.1.

9.1.3 If desired, a mean shear stress may be calculated from the following equation:

$$\text{Shear Stress, } \tau = \eta \cdot \dot{\gamma} \quad (3)$$

where:

- τ = shear stress in dynes/cm²,
- η = apparent viscosity in poise, P, and
- $\dot{\gamma}$ = shear rate in reciprocal seconds, s^{-1} .

In this way, values of apparent viscosity, shear rate and shear stress for several zones can be computed.

9.2 If a broader range of shear rates is desired, the vacuum may be increased or decreased on a subsequent run and new viscosity constants calculated according to the relationship as follows:

$$[K_{zone}]_2 = \frac{(P_2 - h)}{(P_1 - h)} [K_{zone}]_1 \quad (4)$$

where:

- h = head of liquid in the capillary at the midpoint of the zone (mm of Hg) found in Annex A1, Table A1.1
- $[K_{zone}]_1$ = viscometer constant in P/s,
- $[K_{zone}]_2$ = viscometer constant for the vacuum applied in P/s,
- P_1 = nominal vacuum applied, generally, 300 mm of Hg, and
- P_2 = vacuum applied (mm of Hg),

9.2.1 Repeat the computation procedure of 9.1 for the times obtained with the new vacuum. Decrease the vacuum to increase the time of flow for materials that are too fluid. Alternatively, vacuum may be increased to reduce time of flow for materials that are too viscous. Report the test conditions with the results.

9.3 The apparent viscosity, η , from 9.1.1 may be plotted against the shear rate on log-log coordinates to obtain a rheogram or plot illustrating the variation of viscosity with shear rate. The B zone value does not necessarily reflect the apparent viscosity of the material. An example rheogram is shown in Fig. 1.

10. Report

10.1 Report the test temperature and vacuum with the viscosity test result, for example, viscosity at 140°F (60°C) and 150 mm Hg vacuum, in poises.

10.2 Unless otherwise stipulated, always report the apparent viscosity test result obtained from the rheogram at a specific shear rate, preferably 1 s^{-1} .

10.3 It is suggested that a form sheet as shown in Fig. 3 be used to record the measured and calculated data, and an applicable rheogram of apparent viscosity to shear rate (or their logarithms) should be plotted for the data in different zones. A log-log rheogram plot with example is shown in Fig. 1.

Sample No. _____ Viscometer No. _____ Vacuum Hg. _____ mm Date _____

Zone	Zone Time	Viscosity Constant K	Viscosity η	Shear Constant	Shear Rate $\dot{\gamma}$	Shear Stress τ

Temp. _____ Type Viscometer _____ Size _____

Remarks: _____

Pouring Temp. _____ by: _____

FIG. 3 Test Data Report Form

10.4 Report the pouring temperature of the sample.

11. Precision and Bias

11.1 The following criteria should be used for judging the acceptability of results for tests performed at 140°F (60°C) and 300 mm vacuum (95 % probability):

11.1.1 *Repeatability*—Duplicate results by the same operator should not be considered suspect unless they differ: *a*) for Newtonian materials, by more than 7 % of their mean; and *b*)

for non-Newtonian materials, by more than 10 % of their mean (where flow time in the *C* zone is 50 to 200 s) on the same size tube.

11.1.2 *Reproducibility*—The results submitted by each of two laboratories should not be considered suspect unless the two results differ: *a*) for Newtonian materials, by more than 10 % of their mean; and *b*) for non-Newtonian materials, by more than 37 % of their mean (where flow time in the *C* zone is 50 to 200 s) on the same size tube.

ANNEX

(Mandatory Information)

A1. MODIFIED KOPPERS VACUUM CAPILLARY VISCOMETER

A1.1 Scope

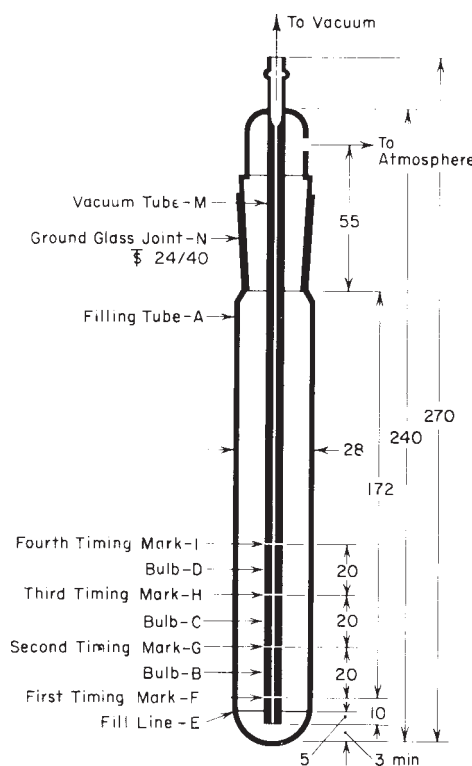
A1.1.1 The Modified Koppers vacuum capillary viscometer is available in four sizes (Table A1.1), covering a range from 42 to 200 000 P. Sizes 50 through 200 are best suited to viscosity measurements of asphalt emulsion residues at 140°F (60°C).

A1.2 Apparatus

A1.2.1 Details of design and construction of the Modified Koppers vacuum capillary viscometer are shown in Fig. A1.1. The size numbers, approximate radii, approximate bulb factors, *K*, and viscosity ranges for the series of Modified Koppers vacuum capillary viscometers are given in Table A1.1.

A1.2.2 This viscometer consists of a separate filling tube, A, and precision-bore glass capillary vacuum tube, M. These two parts are joined by borosilicate ground glass joint, N, having a 24/40 standard taper. The measuring bulbs, B, C, D, E, and F on the capillary are 20-mm long capillary segments, separated by timing marks.

A1.2.3 A viscometer holder can be made by drilling a 28 mm hole through the center of a No. 11 rubber stopper and slitting the stopper between the hole and the edge. When placed in a 2-in. (51-mm) diameter hole in the bath cover, it holds the viscometer in place.



All dimensions are in millimetres.

FIG. A1.1 Modified Koppers Vacuum Capillary Viscometer

TABLE A1.1 Modified Koppers Vacuum Viscometer

Tube Size	50	100	200	400	
Shear Constant	320	160	80	40	
Radius	0.25	0.50	1.0	2.0	
Zone	<i>h</i>	<i>K</i>	<i>K</i>	<i>K</i>	<i>K</i>
<i>B</i>	1.1	8.0	32	128	500
<i>C</i>	2.6	4.0	16	64	250
<i>D</i>	4.1	2.6	10	42	160
<i>E</i>	5.5	2.0	7.8	31	125
<i>F</i>	7.0	1.6	6.2	25	100

where:

- K* = the viscometer constant in P/s
- h* = the head of liquid in mm Hg and is the same for all tube sizes
- radius = mm

A1.3 Calibration of Viscometers

A1.3.1 See procedure X4. in D2171 and apply to the method described in A1.1 and A1.2.

A1.4 Cleaning the Viscometer

A1.4.1 Method 1:

A1.4.1.1 Upon completion of the test, remove the viscometer from the bath and place it in an inverted position into a waste can. Place in an oven maintained at 347°F (175°C) until the asphalt is drained thoroughly from the viscometer. Alternately remove the vacuum tube from the filling tube, and draw heated solvent through the vacuum tube to remove the asphalt.

Clean the vacuum tube and the filling tube thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. This cleaning with solvent should be done with a separate vacuum system discharging into an isolated receiver.

NOTE A1.1—Do not allow viscometers containing asphalt or solvents to remain in high temperature ovens for extended periods of time, as this may cause organic deposits to accumulate on the viscometer.

A1.4.1.2 Dry the tube by passing a slow stream of filtered dry air through the capillary for 2 min, or until the last trace of solvent is removed. Periodically clean the instrument with chromic acid cleaning solution to remove organic deposits, rinse thoroughly with distilled water followed by residue-free acetone, and dry with clean dry air.

A1.4.1.3 Chromic acid cleaning solution may be prepared, by adding, with the usual precautions, 800 mL of concentrated sulfuric acid to a solution of 92 g of sodium dichromate in 458 mL water. The use of similar commercially available sulfuric

acid cleaning solutions is acceptable. Non chromium-containing, strongly oxidizing acid cleaning solutions may be substituted so as to avoid the disposal problems of chromium-containing solutions.

A1.4.1.4 The use of alkaline glass cleaning solutions may result in a change in viscometer calibration and is not recommended.

A1.5 Method 2

A1.5.1 Alternatively, the viscometer fill tube and capillary tubes can be warmed over a Bunsen burner and the major part of the bitumen blown from the tube using compressed air. Place it in a sonic cleaner bath for a few minutes with an appropriate solvent to clean it quickly. Similarly, the 50-cm³ glass beakers used to prepare the samples are heated over a burner to melt the excess bitumen. Allow it to run into a suitable receiver. Then place it into a sonic cleaning bath to be cleaned quickly of any residual asphalt.

APPENDIX

(Nonmandatory Information)

X1. ICE POINT DETERMINATION AND RECALIBRATION OF KINEMATIC VISCOSITY THERMOMETERS

X1.1 To achieve an accuracy of $\pm 0.02^{\circ}\text{C}$ for calibrated kinematic viscosity thermometers, it is required that a check at the ice point be made and the corrections altered for the change seen in the ice point. It is recommended that the interval of checking be every six months; for a new thermometer, check monthly for the first six months.

X1.2 A detailed procedure for the measurement of the ice point and recalibration of thermometers is described in 6.5 of Test Method E 77. The suggestions in the following sections of this appendix are given specifically for mercury-in-glass kinematic viscosity thermometers and may not apply to other thermometers.

X1.2.1 The ice point reading of kinematic viscosity thermometers shall be taken within 60 min after being at the test temperature for not less than 3 min. The ice point reading shall be expressed to the nearest 0.01°C or 0.02°F .

X1.2.2 Select clear pieces of ice, preferably made from distilled or pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the crushed ice and add sufficient distilled and preferably pre-cooled water to form a slush, but not enough to float the ice. As the ice melts, drain off some of the water and add more crushed ice. Insert the thermometer packing the ice gently about the stem, to a depth approximately one scale division below the 0°C (32°F) graduation. It may be necessary to repack the ice around the thermometer because of melting.

X1.2.3 After at least 3 min have elapsed, tap the stem gently, and observe the reading. Successive readings taken at least 1 min apart should agree within one tenth of a division.

X1.2.4 Record the ice point reading and compare it with the previous reading. If the reading is found to be higher or lower than the reading corresponding to a previous calibration, readings at all other temperatures will be correspondingly increased or decreased.

X1.2.5 The ice point procedure given in X1.1-X1.2.4 is used for the recalibration of kinematic viscosity thermometers, and a complete new calibration of the thermometer is not necessary in order to meet the accuracy ascribed to this design thermometer.

X1.3 It is recommended that these kinematic viscosity thermometers be stored vertically when not in use so as to avoid the separation of the mercury column.

X1.4 It is recommended that these kinematic viscosity thermometers be read to the nearest $\frac{1}{5}$ of a division using appropriate magnification. Since these thermometers are typically in a kinematic viscosity bath (which has vision through the front), the thermometer is read by lowering the thermometer such that the top of the mercury column is 5 to 15 mm below the surface of the bath liquid. Be careful to ensure that the expansion chamber at the top of the thermometer is above the lid of the constant temperature bath. If the expansion chamber is at elevated or lowered temperatures from ambient temperatures, a significant error can occur. This can be as much as one or two thermometer divisions. A reading glass such as used for reading books may be useful to ensure reading the scale to $\frac{1}{5}$ of a division.

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