***Change to read:***

## ⟨911⟩ VISCOSITY■—CAPILLARY VISCOMETER METHODS

The following procedures are used to determine the viscosity of a Newtonian fluid, i.e. a fluid having a viscosity that is independent of the shearing stress rate or rate of shear. Unless otherwise directed in the individual monograph, use *Method I*.

• **METHOD I. UBBELOHDE-TYPE CAPILLARY VISCOMETER**

**Apparatus:** The determination may be carried out with an Ubbelohde-type capillary viscometer (*Figure 1*) that has the specifications described in *Table 1* or *Table 2*.

**Table 1**

| Size Number | Nominal Constant of Viscometer ($mm^2/s^2$) | Measurable Kinematic Viscosity Range ($mm^2/s$) | Internal Diameter of Tube, R (mm) (±2%) | Volume of Bulb, C (mL) (±5%) | Internal Diameter of Tube, N (mm) |
|---|---|---|---|---|---|
| 1 | 0.01 | 3.5–10 | 0.64 | 5.6 | 2.8–3.2 |
| 1A | 0.03 | 6–30 | 0.84 | 5.6 | 2.8–3.2 |
| 2 | 0.1 | 20–100 | 1.15 | 5.6 | 2.8–3.2 |
| 2A | 0.3 | 60–300 | 1.51 | 5.6 | 2.8–3.2 |
| 3 | 1.0 | 200–1,000 | 2.06 | 5.6 | 3.7–4.3 |
| 3A | 3.0 | 600–3,000 | 2.74 | 5.6 | 4.6–5.4 |
| 4 | 10 | 2,000–10,000 | 3.70 | 5.6 | 4.6–5.4 |
| 4A | 30 | 6,000–30,000 | 4.07 | 5.6 | 5.6–6.4 |
| 5 | 100 | 20,000–100,000 | 6.76 | 5.6 | 6.8–7.5 |

**Table 2**

| Size Number | Nominal Constant of Viscometer ($mm^2/s^2$) | Measurable Kinematic Viscosity Range ($mm^2/s$) | Internal Diameter of Tube, R (mm) (±2%) | Volume of Bulb, C (mL) (±5%) | Internal Diameter of Tube, N (mm) |
|---|---|---|---|---|---|
| 0 | 0.001 | 0.3–1 | 0.24 | 1.0 | 6.0 |
| 0C | 0.003 | 0.6–3 | 0.36 | 2.0 | 6.0 |
| 0B | 0.005 | 1–5 | 0.46 | 3.0 | 6.0 |
| 1 | 0.01 | 2–10 | 0.58 | 4.0 | 6.0 |
| 1C | 0.03 | 6–30 | 0.78 | 4.0 | 6.0 |
| 1B | 0.05 | 10–50 | 0.88 | 4.0 | 6.0 |
| 2 | 0.1 | 20–100 | 1.03 | 4.0 | 6.0 |
| 2C | 0.3 | 60–300 | 1.36 | 4.0 | 6.0 |
| 2B | 0.5 | 100–500 | 1.55 | 4.0 | 6.0 |
| 3 | 1.0 | 200–1,000 | 1.83 | 4.0 | 6.0 |
| 3C | 3.0 | 600–3,000 | 2.43 | 4.0 | 6.0 |
| 3B | 5.0 | 1,000–5,000 | 2.75 | 4.0 | 6.5 |
| 4 | 10 | 2,000–10,000 | 3.27 | 4.0 | 7.0 |
| 4C | 30 | 6,000–30,000 | 4.32 | 4.0 | 8.0 |
| 4B | 50 | 10,000–50,000 | 5.20 | 5.0 | 8.5 |
| 5 | 100 | 20,000–100,000 | 6.25 | 5.0 | 10.0 |

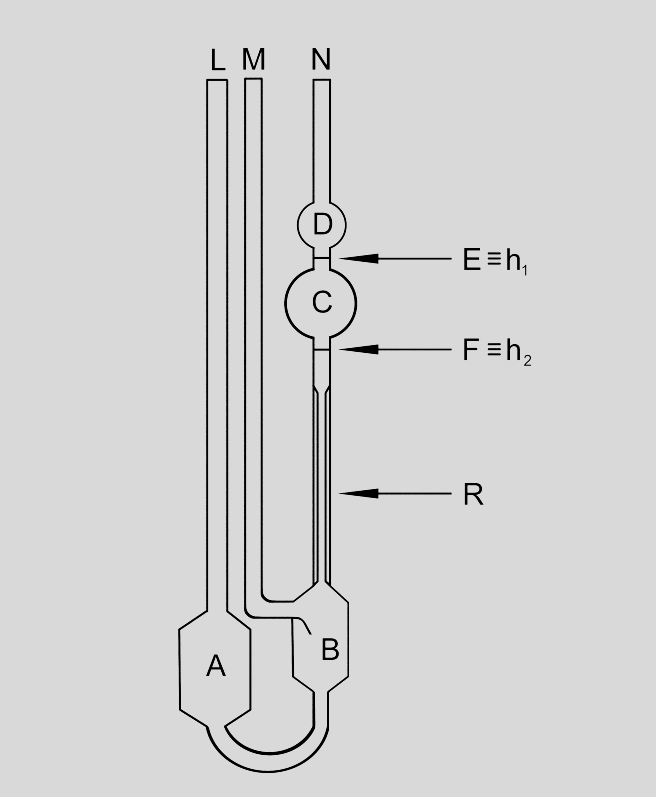


Figure 1. Ubbelohde-Type Capillary Viscometer

**Procedure:** Fill the viscometer through tube (L) with a sufficient quantity of the sample liquid that is appropriate for the viscometer being used or by following the manufacturer's instructions. Carry out the experiment with the tube in a vertical position. Fill bulb (A) with the liquid, and also ensure that the level of liquid in bulb (B) is below the exit to the ventilation tube (M). Immerse the viscometer in a water or oil bath stabilized at the temperature specified in the individual monograph, and control the temperature to ±0.1°, unless otherwise specified in the individual monograph. Maintain the viscometer in a vertical position for a time period of NLT 30 min to allow the sample temperature to reach equilibrium. Close tube (M), and raise the level of the liquid in tube (N) to a level about 8 mm above mark ($E \equiv h_1$). Keep the liquid at this level by closing tube (N) and opening tube (M). Open tube (N), and measure the time required for the level of the liquid to drop from mark ($E \equiv h_1$) to ($F \equiv h_2$), using an appropriate accurate timing device. [NOTE—In *Table 1*, the minimum flow time should be 350 s for size no. 1, and 200 s for all other sizes. In *Table 2*, the minimum flow time should be 300 s for size no. 0, and 200 s for all other sizes.]

**Calibration:** Calibrate each viscometer at the test temperature by using fluids of known viscosities of appropriate viscosity standards to determine the viscometer constant, *k*. The viscosity values of the calibration standards should bracket the expected viscosity value of the sample liquid. Determine the viscometer constant at the same temperature as the sample liquid under test.

Calculate the viscometer constant, *k*, in $mm^2/s^2$, from the equation:

$$k = \eta/(\rho \times t)$$

$\eta$ = known viscosity of the liquid (mPa · s)
$\rho$ = density of the liquid (g/mL)
$t$ = flow time for the liquid to pass from the upper mark to the lower mark (s)

**Calculation of kinematic and Newtonian viscosities of sample fluid:** A capillary viscometer is chosen so that the flow time, *t*, ranges between 200 and 1000 s, and the kinematic energy correction is typically less than 1%. If the viscosity constant, *k*, is known, use the following equation to calculate the kinematic viscosity, *v*, in $mm^2/s$, from the flow time, *t*, in s.

$$v = k \times t$$

If the density of the fluid is known at the temperature of the viscosity measurement, then the Newtonian viscosity, $\eta$, in mPa · s, is calculated by the following equation:

$$\eta = v \times \rho$$

$\rho$ = density of the fluid (g/mL)

The flow time of the fluid under examination is the mean of NLT three consecutive determinations. The result is valid if the percentage of the relative standard deviation (%RSD) for the three readings is NMT 2.0%.

• **METHOD II. OSTWALD-TYPE CAPILLARY VISCOMETER**

**Apparatus:** The determination may be carried out with an Ostwald-type capillary viscometer (*Figure 2*).

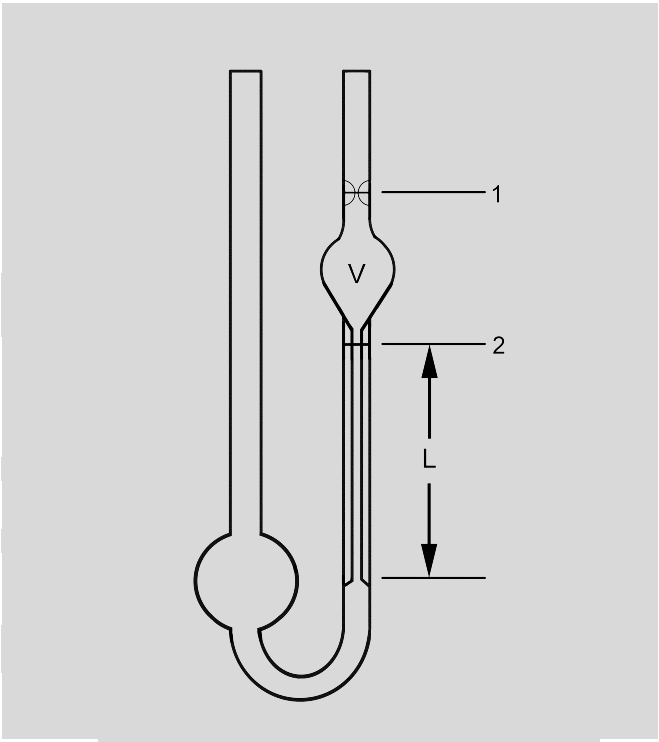


Figure 2. Ostwald-Type Capillary Viscometer

**Procedure:** Fill the tube with an amount of the sample that is appropriate for the viscometer being used or by following the manufacturer's instructions. The volume of fluid used should be such that the lower bulb is not entirely emptied when the fluid is drawn up through the capillary tube to the uppermost graduation mark. Carry out the experiment with the tube in a vertical position. Immerse the viscometer in a water or oil bath stabilized at the temperature specified in the individual monograph, and control the temperature to ±0.1°, unless otherwise specified in the individual monograph. Maintain the viscometer in a vertical position for a time period of NLT 30 min to allow the sample temperature to reach equilibrium. Using suction, draw the fluid up through the capillary tube until the meniscus is at the level of the uppermost graduation. With both the filling and capillary tubes open to atmospheric pressure, record the time, in s, required for the liquid to flow from the upper mark to the lower mark in the capillary tube. [NOTE—The minimum flow time should be 200 s.]

**Calibration** and **Calculation of kinematic and Newtonian viscosities of sample fluid:** Proceed as directed in *Method I*.■2S (*USP35*)

***Add the following:***

## ■⟨912⟩ ROTATIONAL RHEOMETER METHODS

The principle of the method is to measure the force (torque) acting on a rotor when it rotates at a constant angular velocity (rotational speed) in a liquid. Rotational rheometers/viscometers are used for measuring the viscosity of Newtonian fluids, i.e., a fluid having a viscosity that is independent of the shearing stress or rate of shear, or the apparent viscosity of non-Newtonian fluids, which may exhibit different rheological behavior, depending on shear rate, shear stress, and temperature. The following procedures are used to determine the viscosity of Newtonian fluids or the apparent viscosity of non-Newtonian fluids. The calculated viscosity of Newtonian fluids should be the same (within experimental error), regardless of the rate of shear (or rotational speed). Given the dependence of viscosity on temperature, the temperature of the substance being measured should be controlled to within ±0.1°, unless otherwise specified in the individual monograph. Unless otherwise directed in the individual monograph, use *Method I*.

• **METHOD I. SPINDLE RHEOMETERS (RELATIVE RHEOMETERS—SPINDLE VISCOMETERS)**

**Apparatus:** In the spindle rheometer, the apparent viscosity is determined by rotating a cylinder- or disc-shaped spindle, as shown in *Figures 1* and *2*, respectively, immersed in a large volume of liquid.

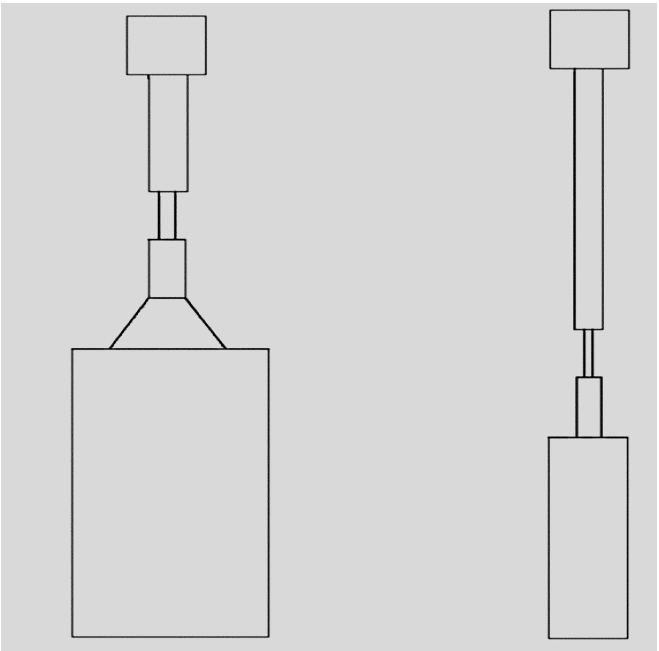

Figure 1. Cylinder-shaped spindles

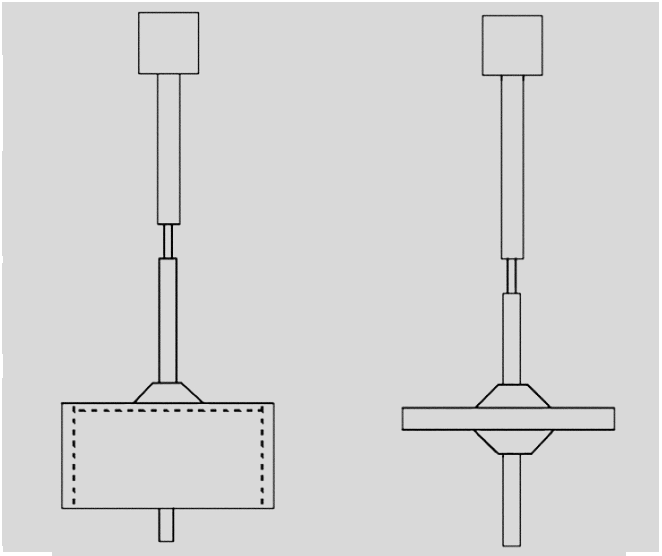

Figure 2. Disc-shaped spindles

**Procedure:** Under these test conditions the shear rate varies between the outer surface of the spindle and the inner surface of the beaker or cup containing the test substance. As a result, the following additional information must be described along with the measured viscosity:

1. Size and geometry of spindle
2. Angular velocity of the spindle
3. Inner dimensions of the test substance container
4. Temperature of the test substance
5. Use of instrument accessories, such as a spindle guard

The preparation of the test specimen, including its temperature equilibration, is specified in each individual monograph. Follow the instrument manufacturer's recommendations regarding sample loading, spindle selection, and rheometer operation.

**Calibration:** Select at least two calibration standards whose viscosities differ by an appropriate value within the viscosity range of the test substance under measurement for a particular rheometer configuration. Measure the apparent viscosities of each standard, as described above, at multiple rotational speeds.

A rheometer is deemed to be calibrated if the measured apparent viscosities are within ±5% of the stated values.

Generally, calibration, operation, and cleaning of rheometers should be performed according to the recommendations of the instrument manufacturer.

• **METHOD II. CONCENTRIC CYLINDER RHEOMETERS**

**Apparatus:** In the concentric cylinder rheometer, the apparent viscosity is determined by placing the liquid in the gap between the inner cylinder and the outer cylinder. Both controlled-stress and controlled-rate rotational rheometers are available commercially in configurations with absolute geometries (e.g., very small annular gaps between concentric cylinders) that can provide consistent meaningful rheological data for non-Newtonian fluids. Controlled-stress rheometers provide controlled-stress input and measurement of the resulting shear rate. Controlled-rate rheometers provide controlled-shear rate input and determination of the resultant shear stress, measured as torque, on the rotor axis. Concen-