

## **Chapter 2**

### **VISCOMETERS**

The measurement of viscosity is of significant importance in both industry and academia. Accurate knowledge of viscosity is necessary for various industrial processes. Various theories that are developed for prediction or estimation of viscosity must be verified using experimental data. Instruments used to measure the viscosity of liquids can be broadly classified into seven categories:

1. Capillary viscometers
2. Orifice viscometers
3. High temperature high shear rate viscometers
4. Rotational viscometers
5. Falling ball viscometers
6. Vibrational viscometers
7. Ultrasonic viscometers

A number of viscometers are also available that combine features of two or three types of viscometers noted above, such as Friction tube, Norcross, Brookfield, Viscosity sensitive rotameter, and Continuous consistency viscometers. A number of instruments are also automated for continuous measurement of viscosity and for process control. Several apparatus named after pioneers in the subject as well manufactured by popular instrument manufacturers are available for each of the classes.

#### **2.1 CAPILLARY VISCOMETERS**

Capillary viscometers are most widely used for measuring viscosity of Newtonian liquids. They are simple in operation; require a small volume of sample liquid, temperature control is simple, and inexpensive. In capillary

viscometers, the volumetric flow rate of the liquid flowing through a fine bore (capillary) is measured, usually by noting the time required for a known volume of liquid to pass through two graduation marks. The liquid may flow through the capillary tube either under the influence of gravity (Gravity Type Viscometer) or an external force. In the instruments where an external force is applied, the liquid is forced through the capillary at a predetermined rate and the pressure drop across the capillary is measured. Capillary viscometers are capable of providing direct calculation of viscosity from the rate of flow, pressure and various dimensions of the instruments. However, most of the capillary viscometers must be first calibrated with one or more liquids of known viscosity to obtain “constants” for that particular viscometer.

The essential components of a capillary viscometer are

1. a liquid reservoir,
2. a capillary of known dimensions,
3. a provision for measuring and controlling the applied pressure,
4. a means of measuring the flow rate, and
5. a thermostat to maintain the required temperature.

Several types of capillary viscometers have been designed through variation of above components, and commercially available capillary viscometers can be classified into the following three categories based on their design.

1. Modified Ostwald viscometers,
2. Suspended-level viscometers, and
3. Reverse-flow viscometers.

Glass capillary viscometers are most convenient for the determination of the viscosity of Newtonian liquids. Often the driving force is the hydrostatic head of the test liquid itself. Kinematic viscosity is generally measured using these viscometers. The same principles can also be applied to measure the viscosity of Non-Newtonian liquids, however an external pressure will be necessary to make the Non-Newtonian liquid flow through the capillary. Glass capillary viscometers are low shear-stress instruments. Usually the shear stress ranges between 10 and 150 dyn/cm<sup>2</sup> if operated by gravity only and 10 to 500 dyn/cm<sup>2</sup> if an additional pressure is applied. The rate of shear in glass capillary viscometers ranges from 1 to 20,000 s<sup>-1</sup> (based on 200 to 800 s efflux time).

### 2.1.1 THEORY

The calculation of viscosity from the data measured using glass capillary viscometers is based on Poiseuille's equation. In this section, first the derivation of Poiseuille's equation is discussed and then various corrections made to this equation are explained.

Consider a cylindrical capillary of diameter  $a$  and length  $l$ , as shown in Fig. 2.1, with a pressure difference  $\Delta P$  between the ends.  $P_1$  and  $P_2$  are pressures at two ends and the fluid is subjected to a force  $F$ .

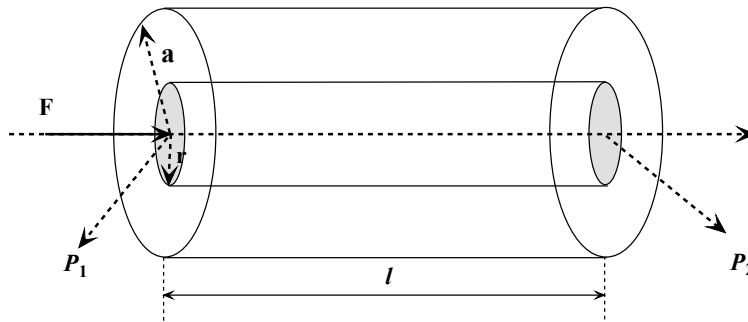


Figure 2.1. Derivation of Poiseuille's equation.

The following assumptions are made to derive Poiseuille's equation.

1. The flow is parallel to the axis of the tube everywhere, i.e., streamline flow is followed.
2. The flow is steady and there is no acceleration of the liquid at any point within the tube.
3. There is no slip at the wall, i.e., the liquid is stationary at the capillary wall.
4. The liquid is a Newtonian liquid.

Since the liquid is Newtonian, the following relationship holds.

$$\sigma = \eta \dot{\epsilon} = \eta \frac{dv}{dr} \quad (2.1)$$

In Eq. 2.1,

$\sigma$  is shear stress,

$\dot{\epsilon}$  is strain rate,  
 $\eta$  is dynamic viscosity,  
 $v$  is the velocity, and  
 $r$  is any distance from the center of the capillary.

A force balance on the cylindrical element of length  $l$  and radius  $r$  coaxial with the capillary provides the following expression.

$$\sigma \times 2\pi r l = \Delta P \times \pi r^2 \quad (2.2)$$

Substitution of Eq. (2.1) into Eq. (2.2) yields

$$\frac{dv}{dr} = \frac{\Delta P}{2\eta l} r \quad (2.3)$$

Integration of Eq. (2.3), using the boundary condition that at the wall of the capillary  $v(a) = 0$ , gives

$$v = \frac{\Delta P (a^2 - r^2)}{4\eta l} \quad (2.4)$$

It may be noted from Eq. (2.4) that the velocity distribution across the capillary is parabolic. The volumetric flow rate through the capillary can be calculated by noting that in unit time between radii  $r$  and  $r+dr$  the volume of liquid flowing is given by  $2\pi r v dr$ . The overall flow rate ( $Q$  cm<sup>3</sup>/s) can be obtained by integrating the following expression.

$$Q = \int_0^a 2\pi r v dr \quad (2.5)$$

Substitution of Eq. 2.4 into Eq. 2.5 yields

$$Q = \int_0^a \frac{2\pi \Delta P}{4\eta l} r (a^2 - r^2) dr = \frac{2\pi \Delta P}{4\eta l} \int_0^a r (a^2 - r^2) dr \quad (2.6)$$

or,

$$Q = \frac{\pi \Delta P a^4}{8 \eta l} \quad (2.7)$$

This is known as Poiseuille's equation and is used for calculation of viscosity when using a capillary viscometer. For vertical tube arrangement which is the case for most of the capillary viscometer, the hydrostatic pressure,  $\rho gh$ , depends on the height,  $h$ , of the liquid. Therefore, the pressure difference,  $\Delta p$ , in terms of hydrostatic pressure is given by

$$\Delta P = \rho gh \quad (2.8)$$

It should be noted that  $h$  is a function of time. Substitution of Eq. 2.8 into Eq. 2.7 and further rearrangement provides the expression for viscosity ( $\eta$ ).

$$\eta = \frac{\pi g h a^4}{8 l V} \rho t \quad (2.9)$$

where,

$Q = V/t$  ( $V$  is the defined volume of the liquid dispensed during the experiment and  $t$  is the time required for this volume of liquid to flow between two graduation marks in a viscometer).

For a particular viscometer, Eq. 2.9 can be rewritten as

$$\eta = K \rho t \quad (2.10)$$

where  $K$  is a constant for a viscometer and is given by

$$K = \frac{\pi g h a^4}{8 l V} \quad (2.11)$$

Equation 2.10 can be used to obtain the kinematic viscosity

$$\nu = K t \quad (2.12)$$

where

$$\nu = \frac{\eta}{\rho} \quad (2.13)$$

A number of viscometers are designed based on Eq. 2.12. The equipment is calibrated for the value of  $K$ , which is obtained by using a liquid of known viscosity and density. Once the value of  $K$  is known, the viscosity of test liquid can be obtained by measuring the time required for a known volume of sample to flow between two graduation marks.

### 2.1.1.1 Kinetic Energy Corrections

A number of factors can influence the experiment and introduce errors in the measurements. To improve the accuracy of the measurement, various corrections are made to the experimentally determined data. Among these corrections, kinetic energy and end effect corrections are most significant. In most types of viscometers, a portion of the applied force is converted to kinetic energy that sets the liquid into motion. However, as Poiseuille's equation is strictly for flow of fluid with parabolic velocity profile, a correction to Poiseuille's equation is necessary to account for the pressure used in overcoming viscous resistance.

The work done due to the kinetic energy transferred to the liquid per unit time may be expressed as

$$W_{KE} = \int_0^a \frac{1}{2} v^2 2\pi r \rho v dr \quad (2.14)$$

Substitution of Eq. 2.4 into Eq. 2.14 yields

$$W_{KE} = \int_0^a \pi \rho \frac{\Delta P^3}{4^3 \eta^3 l^3} (a^2 - r^2)^3 r dr \quad (2.15)$$

Integration of Eq. 2.15 and subsequent substitution of Eq. 2.8 provides

$$W_{KE} = \frac{\rho Q^3}{\pi^2 a^4} \quad (2.16)$$

Therefore, the work done against the viscous force is given by

$$W_{vis} = PQ - \frac{\rho Q^3}{\pi^2 a^4} \quad (2.17)$$

The effective pressure difference, then, can be written as

$$\Delta P_{eff} = P - \frac{\rho Q^2}{\pi^2 a^4} \quad (2.18)$$

Several viscometers make this correction for kinetic energy by connecting a reservoir to both ends of the capillary. However, several researchers noted that still some correction is necessary. As noted by Dinsdale and Moore<sup>1</sup>, the correction term takes the form:

$$\Delta P_{eff} = P - m \frac{\rho Q^2}{\pi^2 a^4} \quad (2.19)$$

where  $m$  is a constant which is determined experimentally.

### 2.1.1.2 End Corrections

The converging and diverging streamlines at the entrance and exit of the capillary must be taken into consideration for accurate estimation of viscosity from capillary viscometers. Couette first suggested increasing the capillary length  $l$  by  $na$  to take into account the end effects. The Poiseuille's equation after correcting for kinetic energy and end effects can be written as

$$\eta = \frac{\pi a^4 P}{8Q(l+na)} - \frac{m \rho Q}{8\pi(l+na)} \quad (2.20)$$

The values of  $n$  varied between 0 and 1.2. Equation 2.20 can be written in terms of time as follows:

$$\eta = \frac{\pi a^4 Pt}{8V(l+na)} - \frac{m \rho V}{8\pi t(l+na)} \quad (2.21)$$

Equation 2.21 for relative viscometers may be written as

$$\eta = \alpha Pt - \beta \rho t \quad (2.22)$$

The constants  $\alpha$  and  $\beta$  are determined from the experimental data using three or four liquids of known viscosity.

Equation 2.21 for gravity flow type viscometers becomes

$$\nu = At - \frac{B}{t} \quad (2.23)$$

The constants  $A$  and  $B$  are also determined from the experimental data using three or four liquids of known viscosity.

Descriptions of various capillary viscometers along with the experimental procedures are described below.

### 2.1.2 OSTWALD VISCOMETER

The most common design of gravity type viscometer is the U-tube type and best known as the Ostwald viscometer. It consists of two reservoir bulbs and a capillary tube as shown in Fig. 2.2.

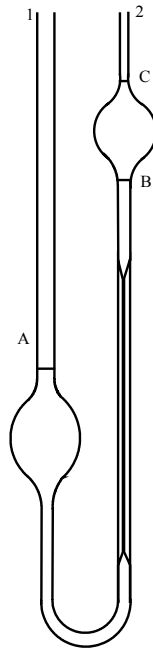


Figure 2.2. An Ostwald viscometer.



The efflux time ( $t$ ) of a fixed volume of liquid under an exactly reproducible mean hydrostatic head is measured. The viscometer is filled with the liquid until the liquid level reaches the mark  $A$ . Usually a pipette is used to accurately measure the volume of liquid added to the viscometer. The viscometer is then placed inside a constant temperature bath to equilibrate the temperature of the test liquid with the bath temperature. The liquid is drawn through the side 2 of the U-tube using a suction and then the flow is timed between marks  $C$  and  $B$ . The viscosity is calculated using Eq. 2.12. The constant  $K$  is determined from the measurement of reference liquid such as water.

Ostwald type viscometers can cause significant error in the measurement if the viscometer is not vertical in alignment. If the distance between the two sides (1 and 2) of the viscometer is  $s$ , a tilt of  $\theta$  from the vertical position can introduce a relative error in the hydrostatic head  $h$  by

$$\Delta h_{error} = 1 - \cos \theta \pm \left( \frac{s}{h} \right) \sin \theta \quad (2.24)$$

It follows from Eq. 2.24 that if  $s$  is  $0.6h$ ,  $1^\circ$  deviation from vertical axis will introduce 1% error in the head.

Another source of error in Ostwald viscometer is the requirement to use exact volume of liquid for the reference liquid and the test liquid. This requirement becomes further problematic if the measurements are made at different temperatures. The accurate knowledge of density is necessary to adjust the volume at different test temperatures.

### 2.1.3 MODIFIED OSTWALD VISCOMETERS

Several modifications to the original design of Ostwald viscometer was made to address these issues and are discussed below. Modified Ostwald viscometers can be divided into two categories.

- a. Constant volume viscometer at filling temperature
  - a. Cannon-Fenske routine viscometer
  - b. Cannon Manning semi-micro viscometer
  - c. Pinkevitch viscometer
- b. Constant volume at the test temperature
  - a. Zeitfuchs viscometer
  - b. SIL viscometer
  - c. BS/U-tube viscometer
  - d. BS/U-tube miniature viscometer

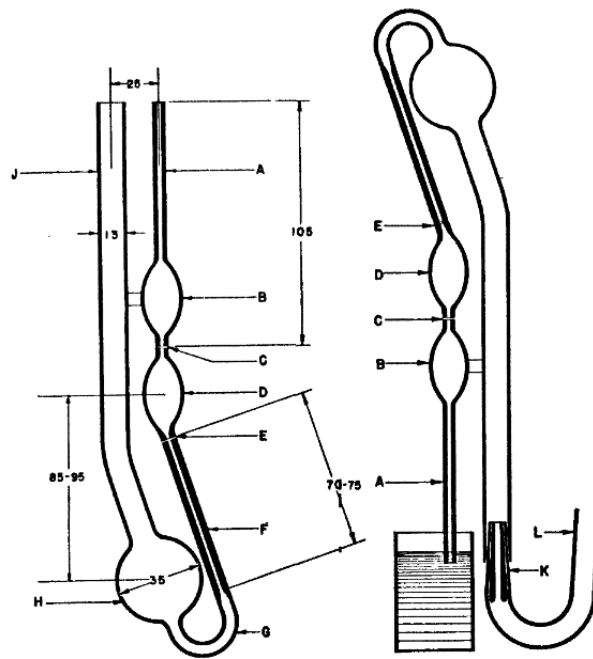
### 2.1.3.1 Cannon-Fenske Routine Viscometer

Ostwald viscometer is modified by Cannon and Fenske<sup>2,3</sup> in such a manner that the upper bulb (Bulb *B*) and lower bulb (Bulb *D*) lie in the same vertical axis in order to reduce the error in the mean head caused by the deviation of the viscometer from the vertical position. A schematic drawing of a Cannon-Fenske Routine viscometer of Size 25 is shown in Fig. 2.3. The Cannon-Fenske routine viscometers are designed for measuring the kinematic viscosity of transparent Newtonian liquids in the range of 0.5 to 20,000 cSt (mm<sup>2</sup>/s). The Cannon-Fenske viscometers can also be used to measure shear stress versus shear rate relations that is useful to the study of non-Newtonian liquids, wax crystallization, and oil flow characteristics at low temperatures.

Cannon-Fenske Routine viscometer that is used to measure viscosity of transparent liquids is based on standard ASTM D-445 and D-446 and ISO 3104 and 3105 methods. The general procedure for using a Cannon-Fenske Viscometer is discussed below. Before any measurement, the viscometer must be cleaned using a suitable solvent or solvents. Although it is desirable to dry the viscometer by passing clean, dry, filtered air through the instrument to remove the final traces of solvents, filtered air may not be readily available in laboratories. In that case, the viscometer may be given a final rinse with acetone and then dried in an oven. The viscometer should be periodically cleaned with acid to remove trace deposits that might occur due to repeated use. One of the best acids for cleaning glasses is chromic acid. It is also advisable to filter the liquid sample to remove solid suspensions before filling the viscometer.

The viscometer shown in Figs. 2.3a and 2.3b depicts the arrangement convenient for filling the apparatus. The sample is drawn into the apparatus by inserting the tube *A* in the inverted position into the liquid (free from air bubbles) kept in a beaker and liquid is drawn applying suction to the arm as shown in Fig. 2.3a, through bulbs *B* and *D* up to the etched mark *E*. The viscometer is turned to its normal position, wiped carefully, inserted into a holder and placed in a thermostat. The viscometer is aligned vertically in the bath by means of a small plumb bob in tube *G*, if a self-aligning holder is not used. After reaching the equilibrium conditions at the required temperature, suction is applied to tube *A*, to bring the sample in to bulb *D* and allowed to reach a point slightly above mark *C*. The time required for the liquid meniscus to pass from the mark at *C* to mark *E* is recorded. The measurement should be repeated several times and the average time of all the runs should be used in the calculation of kinematic viscosity, which is obtained by multiplying the efflux time in seconds by the viscometer

constant. If the efflux time observed is less than 200s, the observation should be repeated with another viscometer with a smaller capillary.



(a) Dimensional sketch of size 25 (b) Method of filling

*Figure 2.3.* Cannon-Fenske Routine viscometer of Size 25 shown with the dimensions in millimeters and its filling procedure.

A single viscometer is not capable of measuring the viscosity over the entire range. The main limitation is the size of the capillary *F*. Table 2.1 lists the range for measuring viscosity using various size Cannon-Fenske Routine viscometers.

### 2.1.3.2 Cannon-Manning Semi-micro Viscometer

The Cannon-Manning Semi-Micro viscometer is a modified Ostwald type model requiring approximately 1 mL of the sample and is capable of measuring the kinematic viscosity of transparent Newtonian liquids in the same range of 0.4 to 20,000 cSt as that of Cannon-Fenske Routine viscometer<sup>4</sup>. The apparatus is shown in Fig. 2.4.

Table 2.1. Kinematic viscosity range of various size Cannon-Fenske Routine viscometers.

Viscometer Size	Kinematic Viscosity Range, $\text{mm}^2/\text{s}$	Diameter of Capillary Tube, $F$ , mm ( $\pm 2\%$ )
25	0.5 – 2 *	0.30
50	0.8 – 4	0.44
75	1.6 – 8	0.54
100	3 – 15	0.63
150	7 – 35	0.78
200	20 – 100	1.01
300	50 – 250	1.27
350	100 – 500	1.52
400	240 – 1,200	1.92
450	500 – 2,500	2.35
500	1,600 – 8,000	3.20
600	4,000 – 20,000	4.20

\*: Minimum flow time is 250 s. Minimum Flow time for other size viscometers is 200 s.

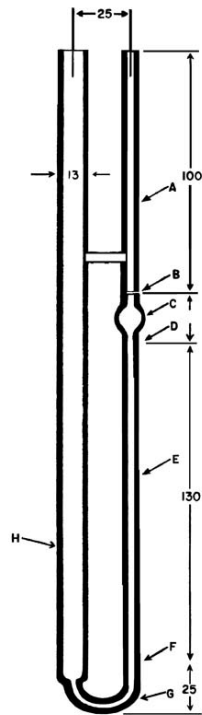


Figure 2.4. A Cannon-Manning Semi-Micro viscometer of size 25 for measuring viscosity of transparent liquid (Modified from ASTM D446-04, American Society for Testing and Materials International, West Conshohocken, PA, USA).

Cannon-Manning Semi-Micro viscometer may be modified by including another timing bulb on the top for measuring viscosity for opaque liquids. The apparatus is charged with the liquid by inverting the instrument and applying suction to tube *H*, immersing tube *A* in the liquid. The liquid is drawn to the filling mark *F*. The apparatus is wiped and placed in the thermostat, in the vertical position. After the apparatus reaches equilibrium, suction is applied to tube *A* (pressure to tube *H*) and the liquid is drawn to a position slightly above the timing mark *B*. The suction is released and the efflux time is recorded. Cannon-Manning Semi-Micro viscometer is also available in different sizes based on the viscosity range. The size-number and the viscosity range are same as that given in Table 2.1 for Cannon-Fenske Routine viscometer.

### 2.1.3.3 Pinkevich Viscometer

The Pinkevich viscometer<sup>5</sup> is a modified form of Ostwald type viscometer used for the determination of the kinematic viscosity of transparent Newtonian liquids in the range of 0.6 to 17,000 cSt. A sketch of Pinkevich viscometer is presented in Fig. 2.5. The viscometer is made of transparent colorless heat resistant glass. The capillary should be straight without any bends, and unevenness and should have a constant internal diameter over its entire length. The joint between the capillary and the tubing should be such as to give a smooth transition without abrupt changes in the direction. The internal diameter of the neck between bulbs *B* and *D* should not be smaller than the capillary diameter. The ring marks should be clearly visible and in a plane perpendicular to the capillary. Ring mark *E* should be 3 mm below the conical end of the bulb *D*. Each viscometer must be etched with the diameter of the capillary and a serial number. Breakage of the lower U-shaped tube between the cylindrical and capillary tubes can be eliminated by the use of an elastic oil resistant hose connection between the tubes at the bottom and a clamping tube at the top. The capillary diameters are in the ranges from 0.4 mm (for 1 to 1.5 cSt) to 4 mm (for 2000 to 15,000 cSt). Minimum efflux time is to be 120 s.

A rubber tube is attached to the side outlet *H*. The top arm *G* is covered with a finger, the viscometer is inverted and arm *A* is inserted into the sample. The sample is sucked up to the ring mark *E*, using a suction bulb, water pump or any other means of creating vacuum. Care is taken to avoid air bubbles, separations, or films in arm *A* or bulbs *B* and *D*. When the sample reaches the ring mark *E*, the viscometer is removed from the sample and quickly inverted to the normal position. The rubber tube is removed and the viscometer is wiped carefully. The viscometer is placed in the thermostat

and allowed to reach the required temperature. The rubber tube is connected to the top of the arm *A* and the sample is sucked up to a level slightly above the ring mark *C*. The suction is released and the time required by the liquid to pass between the marks *C* and *E* is noted. Pinkevich viscometers are also designated as their size number referring to various viscosity ranges. The size-number and the corresponding viscosity ranges are given in Table 2.2.

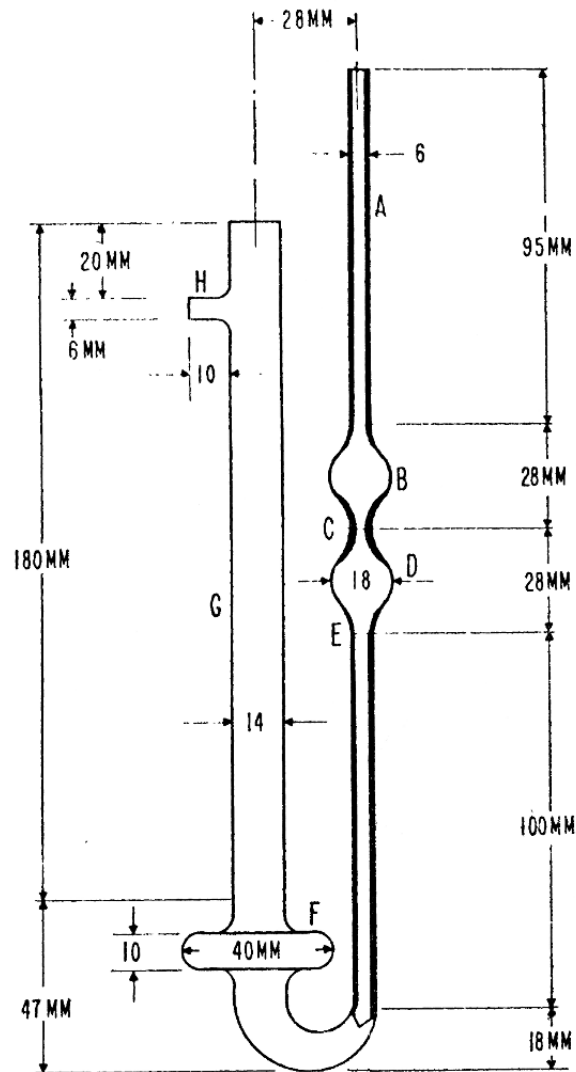


Figure 2.5. Pinkevich viscometer for transparent liquids. (Modified from ASTM D446-04, American Society for Testing and Materials International, West Conshohocken, PA, USA).

Table 2.2. The size-number and corresponding viscosity ranges of Pinkevich viscometers.

<i>Size No</i>	<i>Kinematic Viscosity Range, mm<sup>2</sup>/s (cSt)</i>	<i>Diameter of the Capillary, mm</i>
0	0.6 – 1.7	0.40
1	1.7 – 8.5	0.60
2	5.4 – 27	0.80
3	13 – 65	1.00
4	28 – 140	1.20
5	70 – 350	1.50
6	200 – 1,000	2.00
7	520 – 2,600	2.50
8	1,060 – 5,300	3.00
9	1,980 – 9,900	3.50
10	3,400 – 17,000	4.00

#### 2.1.3.4 Zeitfuchs Viscometer

Zeitfuchs viscometer<sup>6-8</sup> is normally used to measure the kinematic viscosity of Newtonian oils in the range of 0.6 to 3,000 cSt at temperatures between 50 and 250 °C. The constructional features shown in Fig. 2.6, enable cleaning, filling and measurement of the efflux time without removing the viscometer from the thermostat.

The apparatus is charged by pouring the test liquid into the filling tube 1, approximately to the filling line *D*. A screened funnel can be used to filter the sample before pouring into the viscometer. After the apparatus attains the desired temperature, the sample is drawn into the measuring bulb by partially opening the stop cock located over flow tube 2 that is under vacuum, and partially closing the vent to the atmosphere at the top of the capillary tube 3 with a finger. The excess liquid is allowed to flow into the over flow trap and thus to the trap provided in the vacuum line. When the liquid in the filling tube reaches a point 2 to 5 mm above the reference mark *C*, it is held at that level by alternately closing and opening the vent to the atmosphere. Finally, the working volume is adjusted by drawing the meniscus in the filling tube exactly to the mark *C*, making sure that the liquid completely fills the viscometer between the mark and the tip of the over flow. After the final adjustment of the working volume, the connection to the vacuum source is removed and the vent tube 3 is opened to the atmosphere to allow the flow of the liquid to the measuring bulb. The efflux time is carefully measured.

Zeitfuchs viscometer is available in seven sizes. The viscosity range is mainly determined by the size of the capillary of the tube. The viscosity range for different size viscometers is given in Table 2.3.

Table 2.3. Size number and viscosity range of Zeitfuchs viscometers.

Size No.	Kinematic Viscosity Range, $\text{mm}^2/\text{s}$ (cSt)	Diameter of the Capillary, $\text{mm}$ ( $\pm 2\%$ )
1	0.6 – 3	0.42
2	2 – 10	0.59
3	6 – 30	0.78
4	20 – 100	1.16
5	60 – 300	1.54
6	200 – 1000	2.08
7	600 – 3000	2.76

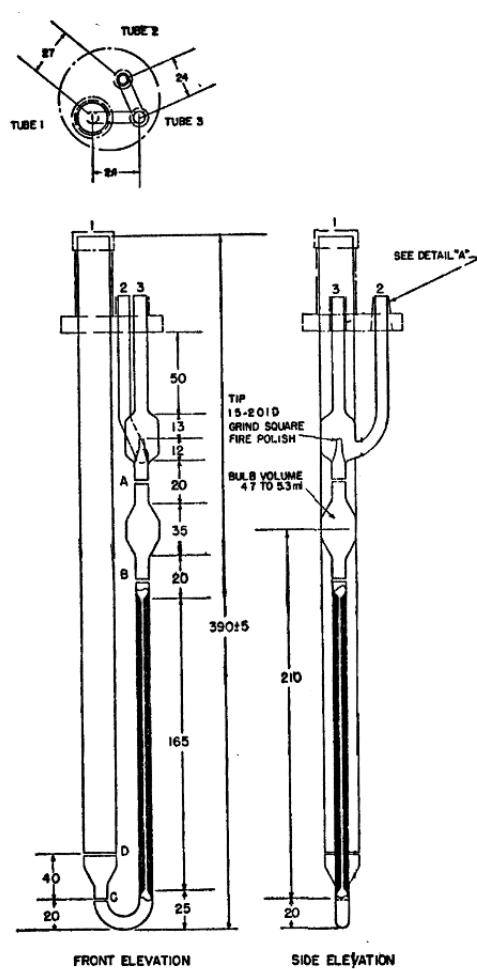


Figure 2.6. Zeitfuchs viscometer for measuring viscosity of transparent liquids.



### 2.1.3.5 SIL Viscometer

SIL viscometer<sup>9</sup> is useful for the determination of the kinematic viscosity of transparent Newtonian liquids in the range of 0.6 to 10,000 cSt. The special features include capability to minimize the effects of surface tension and better precision in terms of volume determination. Fig. 2.7 gives a schematic representation of the apparatus. The volume of the bulb changes with the range of viscosity to be studied and is: 3 mL for the range of 0.6 to 3 cSt, 4 mL for the range of 2 to 30 cSt, and 5 mL for the range of 20 to 20,000 cSt. Generally, SIL viscometers are available in 8 sizes and their number and the corresponding viscosity range are given in Table 2.4.

The viscometer is charged by tilting it by about 30° from the vertical, with bulb *A* below the capillary. Enough sample is introduced into tube 1 to fill bulb *A* completely and for slight overflow into the gallery. The quantity of sample introduced should provide a level of liquid between 6 and 12 mm above the opening *D*, after the viscometer reaches the required temperature. The excess liquid from the gallery is removed by applying suction through tube 3. Time is allowed to ensure complete drainage of the liquid from the lower end of the capillary. A slight suction is applied to tube 2, until its upper meniscus reaches the middle of the bulb *C*. The suction is removed and the efflux time for the flow of liquid is noted.

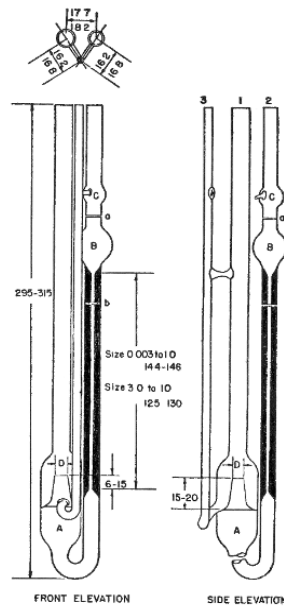


Figure 2.7. A SIL viscometer.

Table 2.4. Size number and viscosity range of SIL viscometers.

<i>Size No.</i>	<i>Kinematic Viscosity Range, mm<sup>2</sup>/s (cSt)</i>	<i>Diameter of the Capillary, mm</i>
0C	0.6 – 3	0.41
1	2 – 10	0.61
1C	6 – 30	0.73
2	20 – 100	1.14
2C	60 – 300	1.50
3	200 – 1,000	2.03
3C	600 – 3,000	2.68
4	2,000 – 10,000	3.61

### 2.1.3.6 BSU-tube Viscometer

The BSU-tube viscometer is generally used when IP Method 71 is required<sup>10</sup>. The viscometer is useful for the determination of the kinematic viscosity in the range of 0.9 to 10,000 cSt. Fig. 2.8 presents the essential features of the apparatus.

The cleaned viscometer is placed in the thermostat, taking care to ensure that the capillary arm is vertical, as a deviation of 1° from the vertical will introduce an error of approximately 0.2% in the measurement. The viscometer is charged with a slight excess of the filtered sample, by means of a long pipette to minimize the wetting of the tube above the filling mark *G*. The apparatus is allowed to attain the required temperature and the volume is adjusted to bring the liquid level to within 0.2 mL of the filling mark by applying suction. Once the desired temperature is attained, suction or pressure is applied to bring the liquid level up to a point about 1 cm above the timing mark *E*. The suction or pressure is removed and time required for the liquid to pass from the top edge of the mark *E* to the top edge of the mark *F* is noted. BSU viscometer is available in 8 sizes and their sizes are designated using letters and are given in Table 2.5.

Table 2-5. Size number and viscosity range of BSU-Tube viscometers.

<i>Size No.</i>	<i>Kinematic Viscosity Range, mm<sup>2</sup>/s (cSt)</i>	<i>Capillary Diameter, mm (± 2%)</i>
A	0.9 – 3	0.50
B	2 – 10	0.71
C	6 – 30	0.88
D	20 – 100	1.40
E	60 – 300	2.00
F	200 – 1,000	2.50
G	600 – 3,000	4.00
H	2,000 – 10,000	6.10

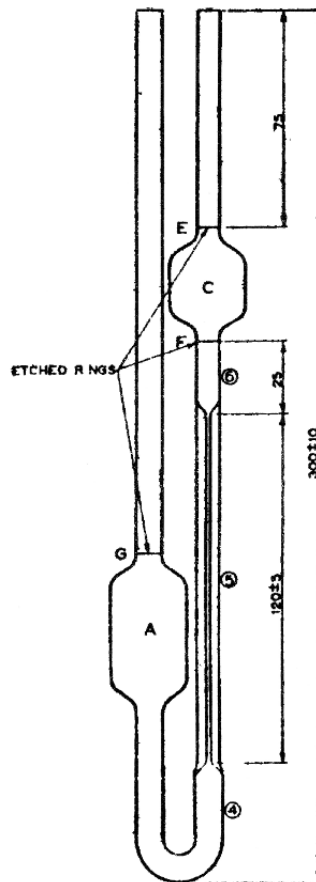


Figure 2.8. BSU-Tube viscometer for measuring viscosity of transparent liquid (Modified from ASTM D446-04, American Society for Testing and Materials International, West Conshohocken, PA, USA).

### 2.1.3.7 BSU-Miniature Viscometer

BSU-Miniature viscometer is designed to measure the kinematic viscosity in the range of 0.2 to 100 cSt. The basic design of the viscometer is same as that of the BSU viscometer shown in Fig 2.8. The diameter of the capillary tube ranges from 0.2 to 0.65 mm and the volume of the sample reservoir is fixed at 0.50 ( $\pm 5\%$ ) mL. BSU-Miniature viscometer is available in five sizes. Table 2.6 provides the kinematic viscosity range corresponding to the size number.

Table 2.6. Size number and viscosity range of BSU-Miniature Tube viscometers.

Size Number	Kinematic Viscosity Range, $\text{mm}^2/\text{s}$ (cSt)	Diameter of Capillary Tube, $\text{mm}$ ( $\pm 2\%$ )
M1	0.2 – 1	0.20
M2	1 – 5	0.30
M3	3 – 15	0.40
M4	8 – 40	0.50
M5	20 – 100	0.65

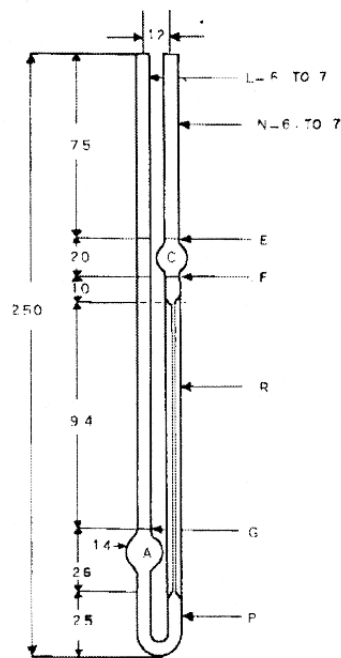


Figure 2.9. A BSU-Miniature Tube viscometer (Reproduced from ASTM D446-04 with permission of American Society for Testing and Materials International, West Conshohocken, PA, USA).

### 2.1.4 SUSPENDED LEVEL VISCOMETERS FOR TRANSPARENT LIQUID

In suspended level viscometers the test liquid is suspended in the capillary so that it becomes completely filled. The advantage of suspended level viscometers is that they ensure a uniform driving head of liquid independent of the quantity of sample charged into the viscometer. As a result, viscometer constant becomes independent of temperature. The effect

of surface tension is greatly reduced by making the diameter of the lower meniscus approximately equal to the average diameter of the upper meniscus. The suspended level viscometers include the following types.

- a. Ubbelohde
- b. FitzSimons
- c. Atlantic
- d. Cannon-Ubbelohde
- e. Cannon-Ubbelohde semi-micro
- f. BS/IP/SL
- g. BS/IP/SL(S)
- h. BS/IP/MSL

#### 2.1.4.1 Ubbelohde Viscometer

Ubbelohde suspended level viscometer<sup>11,12</sup>, shown schematically in Fig. 2.10, is useful for the determination of the kinematic viscosity of transparent Newtonian liquids in the range of 0.3 to 100,000 cSt. A Ubbelohde viscometer possesses the same viscometer constant at all temperatures. This property is advantageous when measurements are to be made at a number of different temperatures. The liquid is induced to flow only down the walls of the bulb below the capillary, thus forming a suspended level, ensuring that the lower liquid level is automatically fixed and coincides with the lower end of the capillary, avoiding the need to fill the viscometer with a definite volume of the liquid and application of corrections for the expansion of glass due to changes in temperature.

The viscometer is charged by tilting the instrument by about 30°C from the vertical, with the bulb *A* below the capillary, by introducing the liquid into tube 1 up to the lower filling line. Care should be taken to see that the liquid does not go above the upper filling line when the viscometer is brought to the vertical position. The U-tube must be filled completely at the bottom and should be free from air bubbles and particulate matter. The viscometer is positioned in a thermostat maintained at the required temperature. After desired temperature is attained, a plug is placed over tube 3 and suction is applied to tube 2, until the liquid reaches the center of the bulb *C*. The suction is disconnected from tube 2, the plug is removed from tube 3 and is immediately placed over tube 2 until sample drops away from the lower end of the capillary. The plug is removed and the efflux time is noted.

The advantages of Ubbelohde type viscometers are speed, accuracy (within  $\pm 0.1\%$ ), small sample size (about 11 mL is sufficient), low susceptibility to errors (due to drainage, and alignment), and cost

effectiveness (the equipment is cheaper than the other models providing the same type of accuracy). The main concern with this viscometer is the prospect of clogging (specially, in small capillaries).

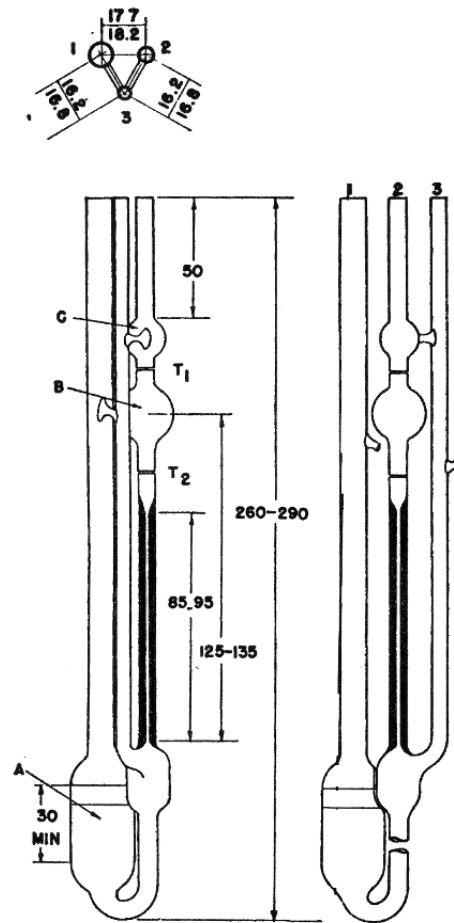


Figure 2.10. Ubbelohde suspended level viscometer for measuring viscosity of transparent liquids (Modified from ASTM D446-04, American Society for Testing and Materials International, West Conshohocken, PA, USA).

There are 16 types of Ubbelohde viscometers covering the kinematic viscosity in the range of 0.3 to 100,000 cSt. In Table 2.7 is lists the size number of Ubbelohde viscometers and corresponding kinematic viscosity range.

Table 2.7. Size number and corresponding viscosity range of Ubbelohde viscometers.

<i>Size No.</i>	<i>Kinematic Viscosity Range, mm<sup>2</sup>/s (cSt)</i>	<i>Capillary Diameter, mm (± 2%)</i>
0	0.3 – 1	0.24
0C	0.6 – 3	0.36
0B	1 – 5	0.46
1	2 – 10	0.58
1C	6 – 30	0.78
1B	10 – 50	0.88
2	20 – 100	1.03
2C	60 – 300	1.36
2B	100 – 500	1.55
3	200 – 1,000	1.83
3C	600 – 3,000	2.43
3B	1,000 – 5,000	2.75
4	2,000 – 10,000	3.27
4C	6,000 – 30,000	4.32
4B	10,000 – 50,000	5.20
5	20,000 – 100,000	6.25

#### 2.1.4.2 FitzSimons Viscometer

FitzSimons viscometer<sup>13</sup>, useful for the determination of the kinematic viscosity of transparent Newtonian liquids, has a range of 0.6 to 1200 cSt. A schematic diagram of the apparatus is shown in Fig. 2.11. This model permits sample filling, observation of efflux time and cleaning of the apparatus without removing the viscometer from the thermostat. FitzSimons viscometer, which is available both with one capillary and two capillaries, is shown in Fig. 2.11a and 2.11b, respectively. Being a viscometer with a spherical shouldered outlet tube, liquids with different surface tensions do not produce different mean effective heads and no correction for surface tension is required.

About 10 mL of the sample is filtered into the viscometer through tube 1 and sufficient time is allowed to enable the sample attain the required temperature. A finger is placed over tube 3, and the liquid is drawn in by applying suction to tube 2 to a level above the upper mark but below the center of the smaller bulb *C*. This operation is to be carried out slowly and cautiously to avoid formation of air bubbles in the sample. When a double capillary model is being used, tube 2 when not in use should be kept closed by means of a flexible tube when not in use. The suction on tube 2 is released and the finger is removed from tube 3. The efflux time for the free flow of sample through the capillary is measured. Prior to the actual experiment, the viscometer should be thoroughly cleaned by means of a

solvent, such as petroleum ether or benzol. FitzSimons viscometer is available in six sizes that cover from 0.6 to 1,200 cSt (Table 2.8).

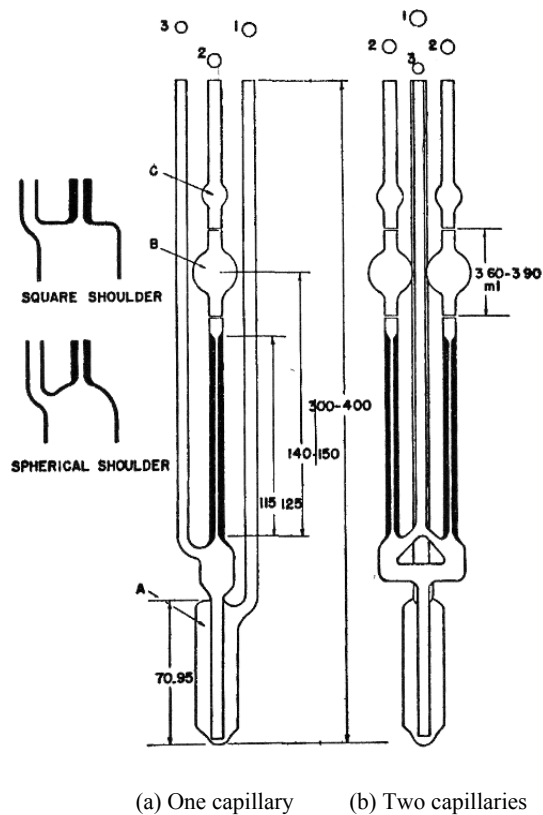


Figure 2.11. FitzSimons suspended level viscometer for measuring viscosity of transparent liquid (Reproduced from ASTM D446-04 with permission of American Society for Testing and Materials International, West Conshohocken, PA, USA).

Table 2.8. Size number and corresponding viscosity range of FitzSimons viscometers.

Size No.	Kinematic Viscosity Range, $\text{mm}^2/\text{s}$ (cSt)	Capillary Diameter, mm ( $\pm 2\%$ )
1	0.6 – 3.0	0.43
2	2 – 10	0.60
3	7 – 35	0.81
4	20 – 100	1.05
5	50 – 250	1.32
6	240 – 1,200	1.96



### 2.1.4.3 Atlantic Viscometer

Atlantic viscometers<sup>14</sup> are available in eleven sizes that cover the kinematic viscosity of transparent Newtonian liquids in the range of 0.7 to 5,000 cSt and are given in Table 2.9. The temperature range is from the dew point to 100 °C.

This apparatus does not have a U-tube type arrangement in contrast to the other models, but it is well suited for routine use with applied air pressure. Provision for filling, measuring efflux time and cleaning without removing from the thermostat are some attractive features of this type. Fig. 2.12 presents the apparatus schematically. The volume of the efflux bulb is  $3.2 \pm 5\%$  mL. The capillary diameter is between 0.41 and 2.69 mm and the length is 100 mm. The three-way stopcock has a curved outlet for connection to a vacuum manifold, making the charging of the viscometer a simple operation. The capillary is connected to a large diameter tube so that the liquid can flow along the wall to form a suspended level without the need for an air vent, simplifying the constructional features considerably.

The viscometer is mounted vertically in the constant temperature bath with the enlargement *B*, resting on the top split collar and the lower end of the capillary *E*, 20 mm below the bottom of the bath. A number of viscometers can be accommodated in a thermostat. The filtered sample is poured into a carefully cleaned 50 mL beaker. The viscometer is charged by positioning the beaker containing the sample under the lower opening of the viscometer and keeping the opening immersed in the liquid. A small vacuum is applied by means of the provision through the three-way stopcock *A*. The sample is drawn into the apparatus until the upper bulb *C* is approximately half filled. The stopcock *A* is closed. After the required temperature is achieved, the stopcock is turned to the vent position to allow the flow of the liquid. The efflux time is measured.

Table 2.9. Size number of various Atlantic viscometers.

Size No.	Kinematic Viscosity Range, mm <sup>2</sup> /s (cSt)	Capillary Diameter, mm ( $\pm 2\%$ )
0C	0.7 – 3	0.42
0B	1 – 5	0.46
1	2 – 10	0.56
1C	6 – 30	0.74
1B	10 – 50	0.83
2	20 – 100	1.00
2C	60 – 300	1.31
2B	100 – 500	1.48
3	200 – 1,000	1.77
3C	600 – 3,000	2.33
3B	1,000 – 5,000	2.64

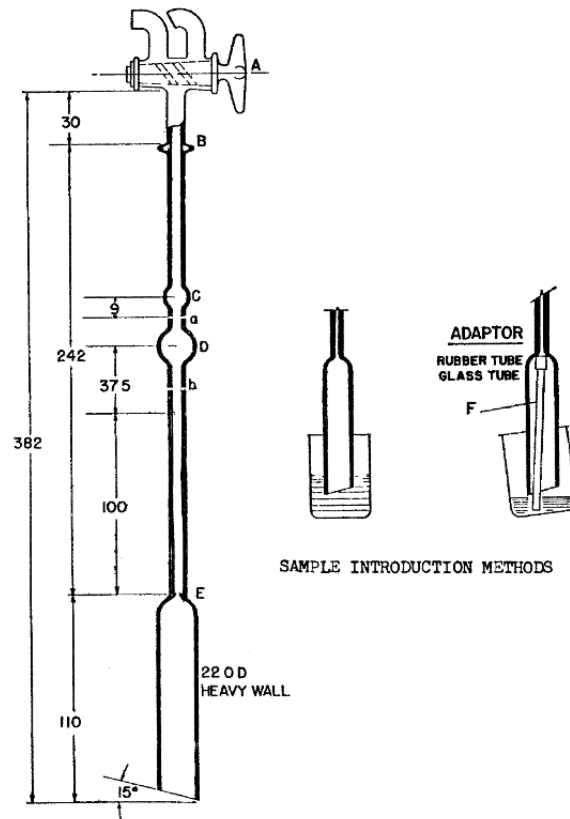


Figure 2.12. Atlantic viscometer for measuring viscosity of transparent liquid (Reproduced from ASTM D446-04 with permission of American Society for Testing and Materials International, West Conshohocken, PA, USA).

#### 2.1.4.4 Cannon - Ubbelohde Dilution Viscometer

The Cannon-Ubbelohde, Cannon-Ubbelohde Dilution, Cannon-Ubbelohde Semi-Micro, and Cannon-Ubbelohde Semi-Micro Dilution suspended level viscometers are normally used for the determination of the kinematic viscosity of transparent Newtonian liquids in the range of 0.4 to 100,000 cSt. The size number for various viscosity ranges is shown in Table 2.10. Fig. 2.13 shows a Cannon-Ubbelohde and Cannon-Ubbelohde Dilution Viscometer for transparent liquids. The volume of the bulb *B* of the Cannon-Ubbelohde viscometer for Size No. 25 which has a kinematic viscosity range of 0.5 to 2 cSt is 1.5 mL and it is 3 mL for the rest of the size numbers. The capillary diameters are in the range of  $0.31 \pm 0.02$  mm to  $5.6 \pm 0.1$  mm. For Cannon-Ubbelohde Semi-Micro-Dilution viscometer, the

volume of the bulb *B* is 0.3 mL, bulb *A* is 30 mm long and the capillary diameters are in the range of  $0.22 \pm 0.1$  mm to  $3.70 \pm 0.05$  mm. Eight to twelve milliliters of the sample is charged into tube 1 (approximately 1 mL for the Semi-Micro size). The viscometer is placed vertically in the thermostat. After thermal equilibrium is established, a finger is placed over tube 3, and suction is applied to tube 2 until the liquid reaches the center of tube 2 until the sample drops away from the lower end of the capillary. The finger over tube 2 is removed and efflux time is measured. A Cannon-Ubbelohde semi micro dilution viscometer is shown in Fig. 2.14.

The Cannon-Ubbelohde Dilution viscometer is useful for measuring viscosity of polymers. Molecular size and shape of large polymer molecules can be related to their kinematic viscosity measured using dilute solutions of the polymers. The Cannon-Ubbelohde Dilution viscometer has an extra-large reservoir which allows polymer solutions to be diluted several times. Dilute polymer solutions frequently appear to exhibit changes in kinematic viscosity when the shear rate is changed. Cannon-Ubbelohde Dilution viscometer allows up to five fold dilution of the sample. The Semi-Micro Dilution Viscometer requires only 1 mL sample and provides up to twenty fold dilution.

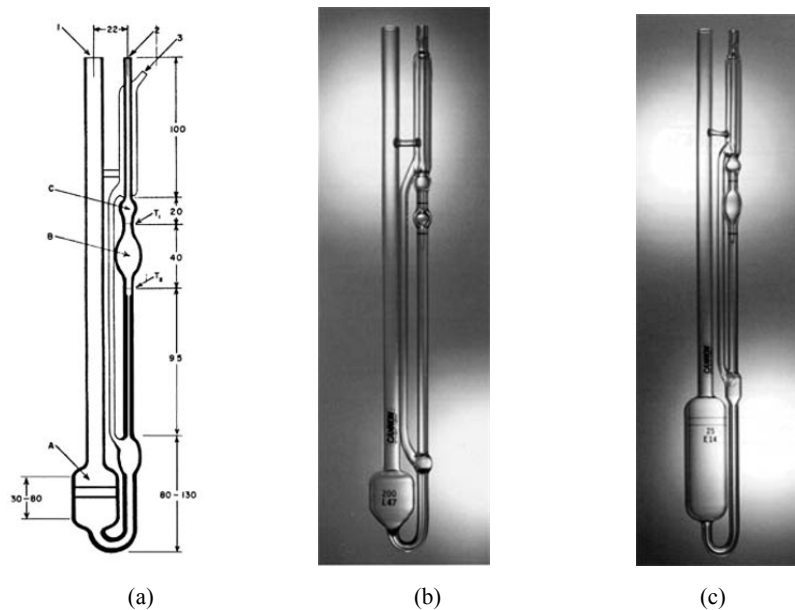


Figure 2.13. Cannon-Ubbelohde and Cannon-Ubbelohde Dilution viscometers. (a) Line diagram of a Cannon-Ubbelohde viscometer (b) Picture of Cannon-Ubbelohde viscometer (c) Picture of Cannon-Ubbelohde dilution viscometer (Courtesy of Cannon Instrument Co., State College, PA, USA).

Table 2.10. Size number and corresponding viscosity range of Cannon-Ubbelohde, Cannon-Ubbelohde Dilution, Cannon-Ubbelohde Semi-Micro, and Cannon-Ubbelohde Semi-Micro Dilution suspended level viscometers.

<i>Size No.</i>	<i>Kinematic Viscosity Range, mm<sup>2</sup>/s (cSt)</i>	<i>Diameter of Capillary, mm (<math>\pm 2\%</math>)</i>
<b>Cannon-Ubbelohde and Cannon-Ubbelohde Dilution viscometers</b>		
25	0.5 – 2	0.31
50	0.8 – 4.0	0.44
75	1.6 – 8.0	0.54
100	3 – 15	0.63
150	7 – 35	0.78
200	20 – 100	1.01
300	50 – 250	1.26
350	100 – 500	1.48
400	240 – 1,200	1.88
450	500 – 2,500	2.25
500	1,600 – 8,000	3.00
600	4,000 – 20,000	3.75
650	9,000 – 45,000	4.60
700	20,000 – 100,000	5.60
<b>Cannon-Ubbelohde Semi-Micro and Cannon-Ubbelohde Semi-Micro Dilution viscometers</b>		
25	0.4 – 1.0	0.22
50	0.8 – 4	0.25
75	1.6 – 8	0.30
100	3 – 15	0.36
150	7 – 35	0.47
200	20 – 100	0.61
300	50 – 250	0.76
350	100 – 500	0.90
400	240 – 1,200	1.13
450	500 – 2,500	1.40
500	1,600 – 8,000	1.85
600	4,000 – 20,000	2.35



capillary, and the time is noted. The size number and corresponding kinematic viscosity range is given in Table 2.11.

Table 2.11. Various sizes of BS/IP/SL, BS/IP/SL(S), BS/IP/MSL viscometers.

<i>Size No.</i>	<i>Kinematic Viscosity Range, mm<sup>2</sup>/s (cSt)</i>	<i>Capillary Diameter, mm (± 2%)</i>
<b>BS/IP/SL</b>		
1	3.5 – 10	
1A	6 – 30	
2	20 – 100	
2A	60 – 300	
3	200 – 1,000	
3A	600 – 3,000	
4	2,000 – 10,000	
4A	6,000 – 30,000	
5	20,000 – 100,000	
<b>BS/IP/SL(S)</b>		
1	1.05 minimum	0.36
2	2.1 – 3	0.49
3	3.8 – 10	0.66
4	6 – 30	0.87
5	20 – 100	1.18
6	60 – 300	1.55
7	200 – 1,000	2.10
8	600 – 3,000	2.76
9	2,000 – 10,000	3.80
<b>BS/IP/MSL(S)</b>		
1	0.6 – 3	0.36
2	2 – 10	0.45
3	6 – 30	0.62
4	20 – 100	0.81
5	60 – 300	1.10
6	200 – 1,000	1.45
7	600 – 3,000	1.98

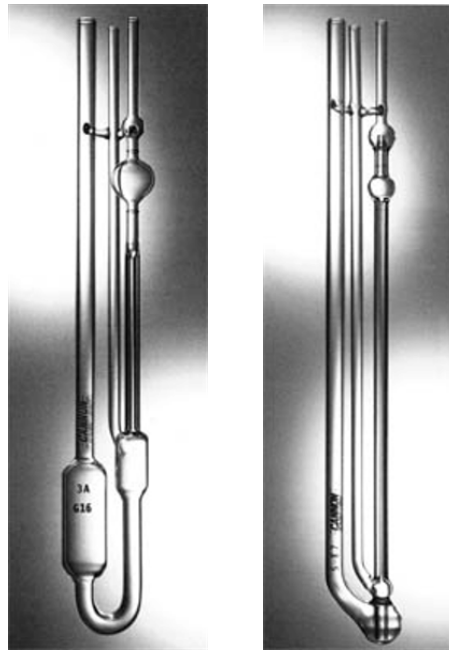


Figure 2.15a. BS/IP/SL, BS/IP/MSL viscometers for measuring viscosity of transparent liquids (Courtesy of Cannon Instrument Co., State College, PA, USA.)

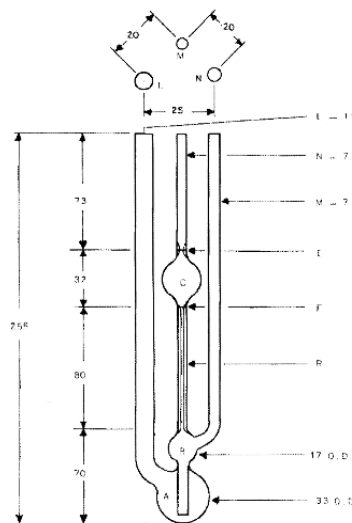


Figure 2.15b. BS/IP/SL(S) viscometer for measuring viscosity of transparent liquids (Reproduced from ASTM D446-04 with permission of American Society for Testing and Materials International, West Conshohocken, PA, USA).

### 2.1.5 REVERSE FLOW VISCOMETERS

Special arrangement is necessary when measuring viscosity of opaque liquids. Viscometers used for this purpose are generally known as Reverse Flow Viscometer. They can be used for both transparent and opaque liquids, specially for measurement of kinematic viscosity of dark Newtonian liquids according to ASTM D 445 and ISO 3104. These reverse flow type viscometers wet the timing section of the viscometer capillary only during the actual measurement. The liquid sample flows into a timing bulb not previously wetted by sample, thus allowing the timing of liquids whose thin films are opaque. Reverse flow viscometers must be cleaned, dried, and refilled prior to each measurement. By contrast, other viscometer types commonly used to measure transparent liquids allow the same sample to be repeatedly drawn up into the capillary, permitting multiple measurements for verification. The reverse flow viscometers for transparent and opaque liquid include the following types:

- a. Cannon-Fenske Opaque
- b. Zeitfuchs cross-arm
- c. Lantz-Zeitfuchs
- d. BS/IP/RF

#### 2.1.5.1 Cannon-Fenske Opaque Viscometer

The most common type of reverse flow viscometer is a Cannon-Fenske Opaque Viscometer<sup>15</sup>. A schematic diagram of the apparatus is shown in Fig. 2.16. The sample is drawn in the same manner as described for the transparent liquids through bulb *B*, up to the mark *C*. The viscometer is turned to the normal position, wiped and cleaned. When the meniscus travels through *D* and fills the bulb *E* up to its half, a small rubber tube connected to a pinch cock is used to close the limb *A* to stop the flow of the liquid. The viscometer is placed in a thermostat by means of a proper holder and is allowed to attain the required temperature. Subsequently the pinch-cock is removed and the efflux time for the liquid to pass through the bulbs *G* and *J*, is recorded by measuring the time required by the liquid to pass between the markings *F*, *H* and *K*. The viscosities calculated from the efflux times for the two bulbs are compared. It should be noted that any reverse flow viscometers must be cleaned, dried, and refilled before a repeat measurement can be made. By contrast, Cannon-Fenske Opaque Viscometer or similar types commonly used to measure transparent liquids allow the same sample to be repeatedly drawn up into the capillary, permitting multiple measurements for verification. Cannon-Fenske Opaque viscometers are





### 2.1.5.2 Zeitfuchs Cross-Arm Viscometer

The Zeitfuchs Cross-arm viscometer<sup>16</sup> is useful for the determination of the kinematic viscosity in the range of 0.3 to 10,000 cSt for both transparent and opaque liquids. Fig. 2.17 is a schematic drawing of the apparatus. Two positions of the bulb are shown. Position *A* requires a larger capillary for a fixed volume of the bulb and the diameter thereby increases. Position *A* is normally used for viscometer constants of 24 or less, because an increase in diameter is desirable for relatively small bore capillaries to avoid clogging by foreign matter and provides a free passage for cleaning. Position *B* for the larger constants makes it possible to use a smaller capillary and a bulb. Capillary diameters range from 0.28 mm (for 0.6 to 3.0 cSt) to 3.06 mm (for 20,000 to 100,000 cSt) and the length of the capillary tube is either 210 or 165 mm. The lower bulb volume is 0.3 or 0.35 mL and the horizontal tube diameter is 3.8 or 4.3 mm.

The viscometer is mounted in the vertical position in a thermostat by means of a metal holder. The sample is introduced into the clean dry viscometer through the filling tube 1. The sample flows into the horizontal cross arm 6, until the meniscus stands at the line 5, on the siphon 4. The sample is allowed to reach equilibrium temperature. Suction is applied to start the flow of the sample through the siphon and into the capillary 11. The efflux time is measured. Advantages<sup>17</sup> include – small sample size, high length/diameter ratio, and application to both opaque and transparent liquids and wide range. Table 2.13 provides the size number and the corresponding viscosity range of Zeitfuchs Cross-arm viscometer.

Table 2.13. Size number and viscosity range of Zeitfuchs Cross-arm viscometer.

<i>Size No.</i>	<i>Kinematic Viscosity Range, mm<sup>2</sup>/s (cSt)</i>	<i>Capillary Diameter, mm (± 2%)</i>
1	0.6-3	0.27
2	2-10	0.35
3	6-30	0.46
4	20-100	0.64
5	60-300	0.84
6	200-1,000	1.15
7	600-3,000	1.42
8	2,000-10,000	1.93
9	6,000-30,000	2.52
10	20,000 -100,000	3.06

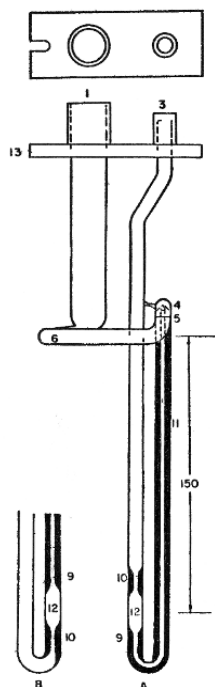


Figure 2.17. A Zeifuchs Cross-arm viscometer (Adopted from J. F. Johnson, R. L. LeTourneua, and R. Matteson, All-purpose capillary viscometer, *Anal. Chem.* **24**(9), 1505-1508, 1952).

### 2.1.5.3 Lantz-Zeifuchs Reverse Flow Viscometer

The kinematic viscosity of opaque Newtonian liquids in the range of 60 to 120,000 cSt, at temperatures between 50 to 250°C can be measured conveniently by Lantz-Zeifuchs reverse flow viscometer, shown schematically in Fig. 2.18. The capillary diameters vary in the range of  $1.62 \pm 0.03$  mm (for 60 to 360 cSt) to  $5.60 \pm 0.08$  mm (for 20,000 to 120,000 cSt). The siphon diameters are in the range of  $3.00 \pm 0.03$  mm to  $5.60 \pm 0.08$  mm and the bulb volumes range from  $5.0 \pm 0.4$  mL to  $0.9 \pm 0.06$  mL. The lengths of the capillary range between 200 and 490 mm. Various sizes of commercial units are listed in Table 2.14.

The sample is charged into the apparatus through a filter screen into the filling tube 1 until the sample overflows the weir into the trap. Sufficient time is allowed to bring the sample to the required temperature. The sample flow is started by applying a slight vacuum at the vent 3 or if the application of vacuum is undesirable (due to the presence of volatile matter in the sample) the flow may be started by the application of slight pressure to the

filling tube until the sample flows over the siphon to a point opposite to the timing mark *A*. The sample is allowed to flow under its own head around the bend in the capillary and the efflux time is measured.

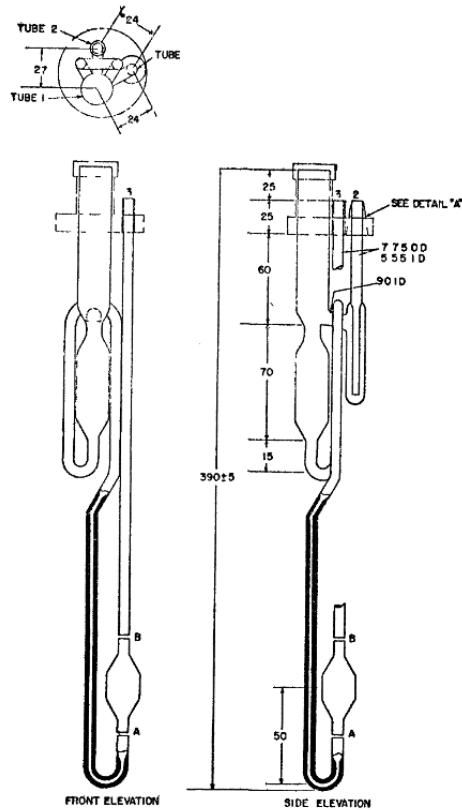


Figure 2.18. Lantz-Zeitfuchs reverse flow viscometer for opaque liquids (Reproduced from ASTM D446-04 with permission of American Society for Testing and Materials International, West Conshohocken, PA, USA).

Table 2.14. Various commercially available Lantz-Zeitfuchs reverse flow viscometers.

Size No.	Kinematic Viscosity Range, $\text{mm}^2/\text{s}$ (cSt)	Capillary Diameter, mm ( $\pm 2\%$ )
5	60-300	1.65
6	200-1,000	2.25
7	600-3,000	3.00
8	2,000-10,000	4.10
9	6,000-30,000	5.20
10	20,000-100,000	5.20

### 2.1.5.4 BS/IP/RF U - Tube Reverse Flow

BS/IP/RF U-Tube reverse flow viscometer is useful for opaque liquids for the determination of kinematic viscosity in the range of 2.0 to 10,000 cSt. The viscometer is made of clear borosilicate glass or other heat resistant glass free of visible defects. The apparatus shown in Fig. 2.19, typically has a capillary diameter from  $0.51 \pm 0.02$  mm (1.7 cSt) to  $10 \pm 0.3$  mm (60,000 cSt). Inside diameters at the marks *B*, *C* and *D* range from 3 to 10 mm. The lengths of the capillaries are between  $185 \pm 2$  and  $210 \pm 2$  mm. A viscometer giving at least 200 s efflux time is selected. Filtered sample is introduced into the viscometer. The viscometer is placed in the thermostat in such a manner that the upper filling mark is about 3 cm below the surface of the bath liquid and the capillary is vertical. After temperature equilibrium is reached, the liquid is allowed to flow freely through the capillary, taking care that the liquid column remains unbroken until it reaches a position about 5 mm below the lower filling mark. The flow is arrested by closing the timing tube with a cork or rubber bung. If necessary, more liquid can be added. The viscometer should be allowed to attain the desired temperature. Adjustments are made to coincide the top of the upper meniscus of the liquid to the upper filling mark. The stopper is removed and the time required for the liquid to flow from the top mark to the bottom mark is recorded.

Table 2.15. Specifications for BS/IP/RF-U tube Reverse flow viscometers.

<i>Size</i>	<i>Approximate Instrument Constant, mm<sup>2</sup>/s<sup>2</sup> (cSt/s)</i>	<i>Viscosity Range, mm<sup>2</sup>/s (cSt)</i>
1	0.003	0.6 – 3
2	0.01	2 – 10
3	0.03	6 – 30
4	0.1	20 – 100
5	0.3	60 – 300
6	1.0	200 – 1000
7	3.0	600 – 3000
8	10	2000 – 10000
9	30	6000 – 30000
10	100	20000 – 100000
11	300	60000 – 300000



volume of sample through an orifice and efflux time is taken as an arbitrary measure of viscosity. Conversion formulas or tables must be used for comparing results which are generally not very accurate. Absolute measurements cannot be carried out using this type of equipment and also the viscosity of non-Newtonian fluid cannot be determined.

The general method of operation of most of the orifice viscometers is basically the same. The sample liquid is poured into a cup which is maintained at a constant temperature by a water or oil bath. The level of the liquid in the cup is adjusted to a definite height, and the liquid is allowed to attain the temperature of the bath. Once the desired temperature is reached, a valve at the base of the cup is opened and the time required for a specific volume of liquid to discharge through the orifice is measured.

The efflux time measured in seconds is a totally arbitrary expression of the viscosity and is usually designated as *viscometer seconds* corresponding to the type of viscometer used. The viscosity is calculated using an empirical expression that is specific to each instrument and has the general form as follows.

$$\nu = \frac{\eta}{\rho} = kt - \frac{K}{t} \quad (2.25)$$

where  $t$  is the viscometer second (such as Redwood seconds, Saybolt seconds, or Engler degrees), and  $k$  and  $K$  are instrument specific constants and must be determined for each instrument.

Most common orifice viscometers used by oil industry are listed below. A brief description of these instruments is given below.

1. Redwood viscometer
2. Engler viscometer
3. Saybolt viscometer
4. Ford viscosity cup viscometer
5. Shell viscosity cup viscometer
6. Zahn cup viscometer

### 2.2.1 REDWOOD VISCOMETER

The Redwood viscometer may be considered as the first orifice meter designed and described by Redwood<sup>18</sup> in his address before the Society of the Chemical Industry in 1886. The Redwood viscometer was primarily developed for the determination of the viscosity of petroleum products which follow Newton's law exhibiting a linear relationship between shearing

stress and the rate of shear under the test conditions. Redwood viscometer is mainly used in the United Kingdom and follows the British Standard IP 70/62. The method measures the viscosity of oil as a time of flow in seconds. The apparatus is illustrated in Fig. 2.20. The important components of the viscometer are—oil cup, jet, heating bath, stirrer, valve, thermometer support, oil cup cover and screen.

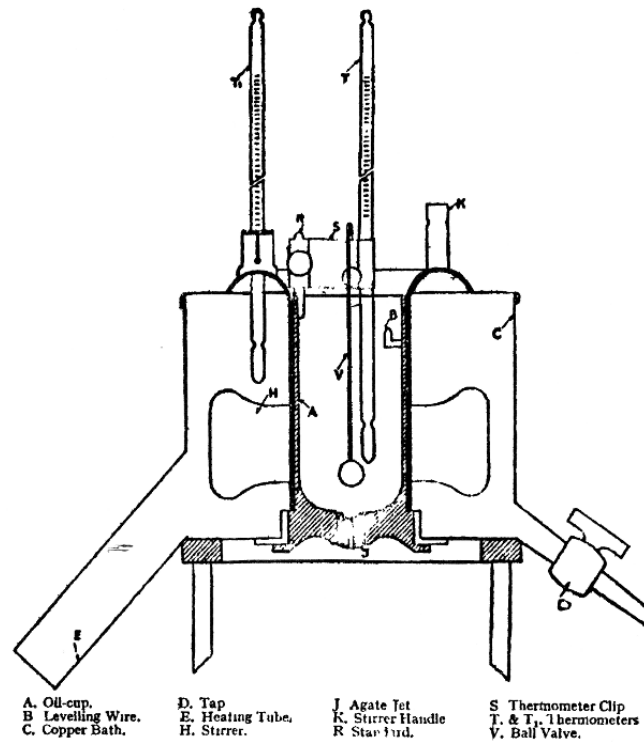


Figure 2.20. Redwood viscometer (Adapted from A. W. Nash and A. R. Bowen, *The Principles and Practice of Lubrication*, Chapman and Hall, London, 1929).

A cylindrical brass vessel *A* of 3 mm wall thickness and a base for the cup are provided with a tapering and a central hole into which the jet is fitted and cemented with its bore along the axis of the cup. The level (to which the oil is to be filled) is indicated by a stout wire *B*. This wire is turned upwards at right angles and tapered to a sharp point at the precise level for the oil surface. The point is at  $7 \pm 1$  mm from the inner wall of the cup. The oil cup is internally silver plated to reduce corrosion. The internal diameter of the oil cup is  $46.5 \pm 0.5$  mm. The vertical distance from the rim is  $46 \pm 1$  mm, the height of the cylindrical portion of the oil cup is  $86 \pm 1$  mm, and the



height of the filling point above the bore is  $82.5 \pm 2$  mm. The jet is constructed of agate and the central hole is drilled and polished to the maximum possible precision.

The upper end of the jet is provided with a concave depression, into which a ball valve for starting and stopping the flow of the oil is fitted. The standard specifications are: internal length of the jet:  $10 \pm 0.05$  mm, minimum internal diameter of the jet: 1.62 mm. The heating bath is of 4 cm diameter and is 9.5 cm deep and spun from a sheet of copper that surrounds the oil cup and is provided with a tap for emptying. The bath is externally heated by means of a suitable electrical resistance wire. Stirring of the oil is provided by means of a cylinder with four vanes surrounding the oil cup.

The valve  $V$ , for starting and stopping of the flow of the liquid from the oil cup consists of a metal ball approximately 11 mm diameter, carried on a stiff wire (1.63 mm diameter). The wire and the ball are both heavily silver plated. The fit of the ball valve in the socket is such that, when the cup is filled with an oil of viscosity 300 to 400 seconds, the leakage is not more than 2 drops per minute. A spring clamp, approximately 20 mm in width is provided to support the oil cup thermometer  $T$ , as illustrated in Fig. 2.21. The instrument is provided with a brass cover, fitted with an insulated handle as illustrated in Fig. 2.21. A suitable screen is attached to the stand to prevent undue cooling of the lower side of the instrument. With this instrument, the flow time should be more than 30 seconds. Measurements for flow times above 2000 seconds should be carried out in the instrument illustrated in Fig. 2.22. The filtered sample is filled in the oil cup up to the level mark and is brought to the desired temperature. The efflux time for 50 mL of the sample flow out of the viscometer is noted. Redwood viscometer is available in two sizes; Redwood viscometer 1 or Redwood viscometer 2. Redwood viscometer 1 is used when the time of flow of the oil at the desired temperature is less than 2000 seconds. The difference between the two viscometers is the diameter of the orifice. The capillary diameter of Redwood viscometer 1 is 1.62 mm and its length is 10.0 mm, whereas that of Redwood viscometer 2 is 3.5 mm and 5.0 mm, respectively.

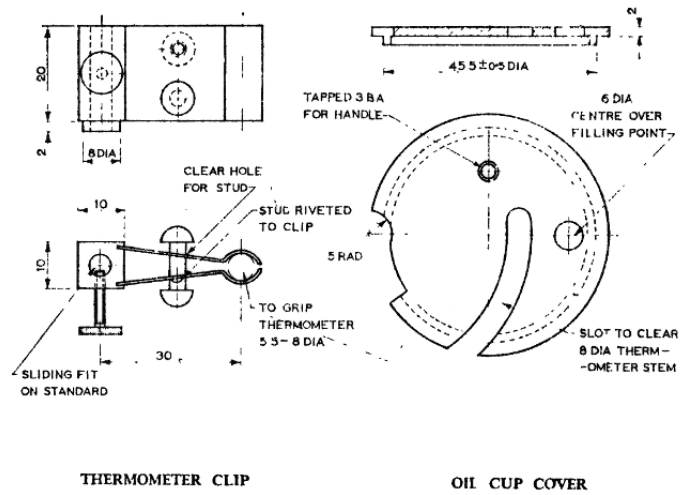


Figure 2.21. Details of the Redwood viscometers (Adapted from A. W. Nash and A. R. Bowen, *The Principles and Practice of Lubrication*, Chapman and Hall, London, 1929).

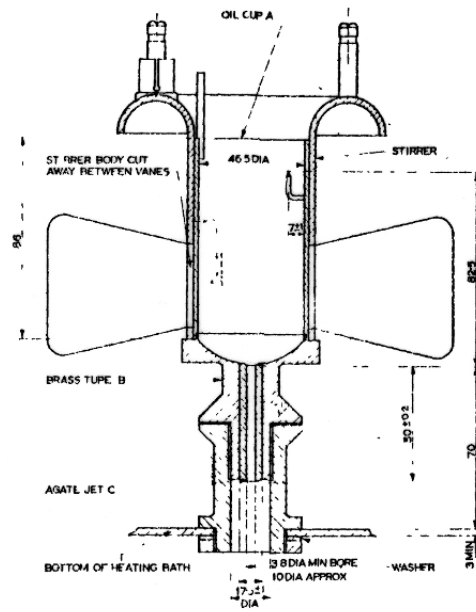


Figure 2.22. Details of the Redwood viscometer 2 used for measuring flow times greater than 2000 s (Adapted from A. W. Nash and A. R. Bowen, *The Principles and Practice of Lubrication*, Chapman and Hall, London, 1929).

### 2.2.2 ENGLER VISCOMETER

Engler viscometer is mainly used in former Eastern Europe for measuring viscosities of lubricating oils. Engler recommended that all viscosities be compared with water. By comparison with standards, lubricating oils may be rated as to its viscosity thus giving one of the values required for a lubricant. A full description of the instrument is given by Stillman<sup>19</sup>. It is also used for determination of specific viscosity of tars and their fluid products according to ASTM D1665-98 standard. The constructional features of the viscometer are shown in Fig. 2.23. The principle of operation is similar to that of Redwood viscometer. The jet is made of platinum instead of agate. The oil cup of this type of viscometer is made in such a manner that the time for the out flow of 200 mL of water at 20°C is 52 s. However for standardization and calibration of viscometer, the efflux time for 200 mL of distilled water at 20°C is first determined and then the factor representing the efflux time for 50 mL of water at 25°C. The oil container is surrounded by a thermostat and is closed with a lid through which a thermometer, a valve plug and a hand stirrer are introduced. The height of the oil is noted by means of three inverted point gauges (which also indicate the level of the apparatus). The measuring flask is graduated at 100 mL and 200 mL for checking purposes. Engler specific viscosity which is usually expressed as Engler degrees is defined as the ratio of the time of outflow of 50 mL of sample at the selected temperature to the time of out flow of the same volume of water at 25°C.

### 2.2.3 SAYBOLT VISCOMETER

Saybolt viscometer was used as a standard viscometer by the chemists of the Standard Oil Co. in the USA for determining viscosity of oil. A detailed description of the instrument was first described by Gill<sup>20</sup>. Saybolt viscometer is available in two types: Saybolt universal viscometer and Saybolt–Furol viscometer. The viscosity of lubricating oils is generally measured by means of a Saybolt universal viscometer while the viscosity of fuel oils is often measured by means of a Saybolt–Furol viscometer. Standard test methods using Saybolt viscometer is described in ASTM D88-94 standard. Saybolt universal viscometer should not be used for the liquids whose outflow time is less than 32 s. The apparatus is shown in Fig. 2.24. It consists of an oil tube fitted at the top with an over flow cup, the tube is surrounded by a bath. The outflow capillary tube is fitted to the bottom of

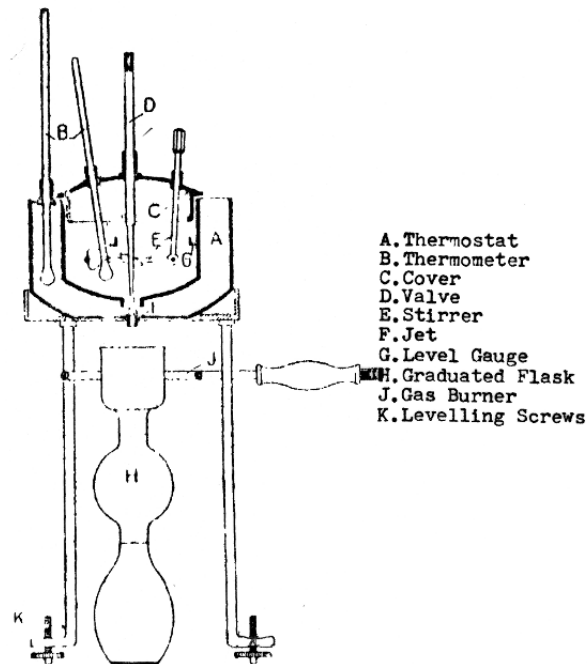


Figure 2.23. Engler viscometer (Adapted from A. W. Nash and A. R. Bowen, *The Principles and Practice of Lubrication*, Chapman and Hall, London, 1929).

the oil tube and is made of stainless steel or of hard and corrosion resistant materials. The receiving flask is marked to hold 60 mL ( $\pm 0.15$  mL) at 20°C.

The lower end of the outflow tube is enclosed by a larger tube, sealed by means of a cork, which acts as a closed air chamber and prevents the flow of oil until the cork is removed. The use of a looped string to withdraw this cork is recommended. Two thermometers are installed to measure the temperature of the oil as well of the bath. The bath is heated to the required temperature using an electrical heater. Oil used in this instrument should first be strained through a 100-mesh sieve and the excess oil over flow at the top should be removed by means of a suitable pipette. The outflow time is recorded by making use of a stop-watch. The results are normally expressed as Saybolt universal seconds.

There are several precautions to be observed in connection with this instrument. The level of the bath liquid must be above the over flow rim of the oil tube and any oil used for test or cleaning should be strained before its use. The oil is heated to the required temperature and some of it poured through the clean tube. The cork is inserted tightly enough to prevent air

leakage but not to reach the outflow tube. The oil under test is heated outside the viscometer to a temperature about  $1.5^{\circ}\text{C}$  or  $3^{\circ}\text{F}$  above the temperature of the test and is poured into the oil tube until it ceases to over flow into the over flow cup. Stirring of the oil in the container and also of the bath should be constant during the entire time of the test. Adequate stirring and control should be provided for the bath so that the temperature of a test sample in the viscometer will not vary more than  $\pm 0.03^{\circ}\text{C}$  ( $\pm 0.05^{\circ}\text{F}$ ). After the bath and the oil in the container have reached the required temperature, the oil tube thermometer is withdrawn, the excess oil in the over flow cup is pipetted out, so that the level of the oil in the over flow cup is below the level of the oil in the tube. The cork is then dexterously withdrawn and the time of flow of 60 mL of the oil is recorded. The liquid in the surrounding bath is kept under constant stirring during the entire test.

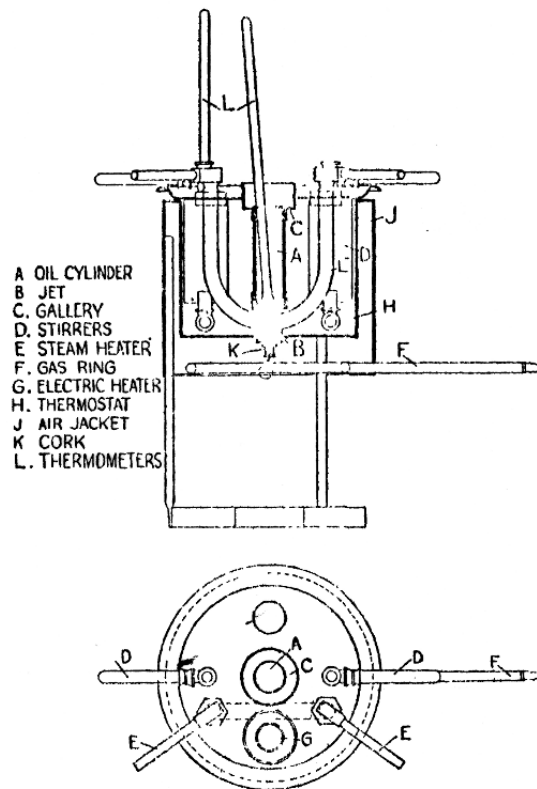


Figure 2.24. Saybolt viscometer (Adapted from A. W. Nash and A. R. Bowen, *The Principles and Practice of Lubrication*, Chapman and Hall, London, 1929).

### 2.2.4 FORD VISCOSITY CUP VISCOMETER

Ford viscosity cup is generally used for determination of viscosity of Newtonian paints, varnishes, lacquers, and related liquids. The protocols for the test methods are provided in ASTM D 1200-94. If the material is non-Newtonian, Ford viscosity cup may be used according to Test method ASTM D 2196. The Ford viscosity cup shown in Fig. 2.25 is filled with the liquid under test up to the top level and the time for the liquid to flow through one of the five standard orifices is measured. Based on the orifice size, five Ford viscosity cup viscometers are available: Nos. 1, 2, 3, 4, and 5. Measurement of viscosity is carried out at  $25 \pm 0.2^\circ\text{C}$  ( $77 \pm 0.4^\circ\text{F}$ ). The choice of cup depends on the efflux time and Fig. 2.26 may be used as a preliminary guide for this choice. However, all the cups should be calibrated using the procedure described in Test Method ASTM D 1200-94. Certified kinematic viscosity standards available from various companies should be used for the calibration of cups. Viscosity standards that can be used from Cannon Instrument Co., P.O. Box 16, State College, PA, 16801 for calibration of Ford viscosity cups are given in Table 2.16. The general formula to convert the time of flow in seconds,  $t$ , to kinematic viscosity,  $\nu$  is

$$\nu_i = At - B \quad (2.26)$$

where  $i$  refers to the number of the cup and  $A$  and  $B$  are constants to be determined from the standard oil.

Table 2-16. Standard oils for calibration of Ford viscosity cups.

<i>Cup Number</i>	<i>Approximate Cup Viscosity Range, cSt</i>	<i>Standard Oil Designation for Calibration</i>	<i>Approximate Viscosity Designated at 25°C, cSt</i>
1	10 – 35	S-10	20
2	25 – 120	S-20	35
3	49 – 220	S-60	120
4	70 – 370	S-60	120
5	200 – 1200	S-200	460

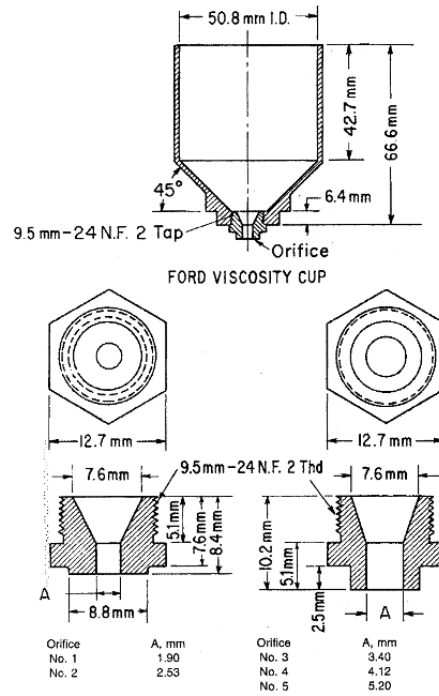


Figure 2.25. Ford viscosity cup viscometer (Reproduced from ASTM D1200-94 with permission of American Society for Testing and Materials International, West Conshohocken, PA, USA).

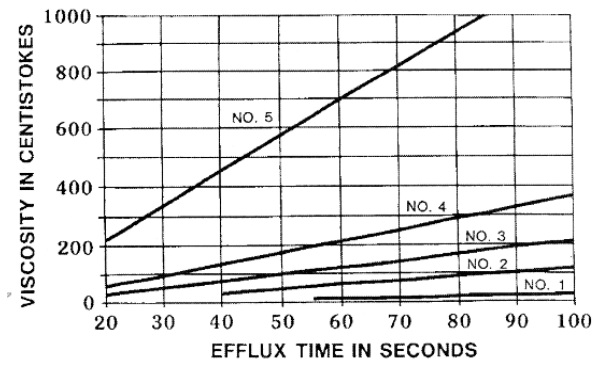


Figure 2.26. Selection of Ford viscosity cups (Reproduced from ASTM D1200-94 with permission of American Society for Testing and Materials International, West Conshohocken, PA, USA).

### 2.2.5 ZAHN VISCOSITY CUP

The General Electric (or "Zahn") viscometer consists of a bullet-shaped steel cup with a small orifice in the bottom<sup>21</sup>. Zahn viscosity cup is also known as Dip-Type viscosity cup because of the manner in which viscosity of a test sample is determined. It can be used to determine the viscosity of Newtonian paints, varnishes, lacquers, ink, and related liquids. For measuring viscosity, the cup is completely immersed in the liquid, withdrawn (that is why it is called Dip-Type), and the time for the flow through the orifice at the bottom of the cup is measured. Although, this type of cups can be used to measure viscosity directly in tanks or containers, the measurement is still approximate. Zahn viscosity cups are available in 5 sizes that can measure the oil viscosity in the range of 20 to 1600 cSt. The cups are made of corrosion and solvent resistance materials. The volume of a cup can vary from 43 to 49 mL depending on the manufacturer. The design features of Zahn cup are shown in Fig. 2.27 and cup orifice diameters are listed in Table 2.17.

Viscosity measurement using Zahn viscosity cups is generally made at 25°C. For viscosity determination at other temperature, a temperature correction curve or factor needs to be determined for each liquid. The choice of cup for measuring viscosity depends on the efflux time which should be between 20 to 80 s. The container holding the test liquid should be stirred well to provide a uniform temperature and density. The cup is immersed in the container and is kept there for 1 to 5 minutes to allow thermal equilibration. The cup is lifted vertically from the container in a quick steady motion. As the top edge of the cup breaks the surface, the timer is started and the cup is held about 6 in (15.2 cm) above the level of the liquid. When the liquid stream breaks at the base of the cup, the timer is stopped and the efflux time in seconds is noted. The efflux time in seconds is converted to kinematic viscosity using the following expression:

$$\nu = At - B \quad (2.27)$$

Zahn viscosity cup should be calibrated periodically according to Test Method ASTM 4212-99 using standard fluids. However, it should be noted that the temperature control of Zahn viscosity cups is extremely difficult and this introduces errors in the calibration.



Table 2.17. Zahn viscosity cup orifice diameter.

Cup Number	Nominal Orifice Diameter, (mm)	Approximate Viscosity Range, (cSt)	Approximate Oil Viscosity at 25° C, (cSt)
1	2.0	5 – 60	20
2	2.7	20 – 250	120
3	3.8	100 – 800	480
4	4.3	200 – 1,200	480
5	5.3	400 – 1,800	900, 1600

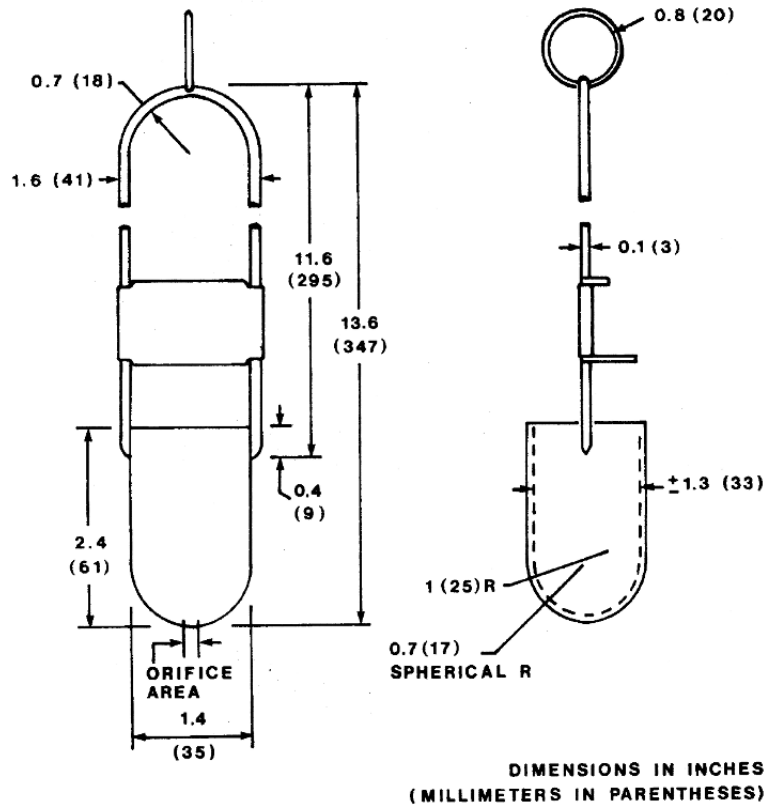


Figure 2.27. Zahn viscosity cup (Reproduced from ASTM D4212-99 with permission of American Society for Testing and Materials International, West Conshohocken, PA, USA).

## 2.2.6 SHELL VISCOSITY CUP

Shell viscosity cups are also Dip-Type viscosity cups and are available in 8 sizes. They are made of stainless steel with a capacity of 23 mL and a 25 mm long capillary in the bottom. Typical design of a Shell Viscosity cup is shown in Fig. 2.28. The orifice size and the recommended use for various viscosity ranges are given in Table 2.18. The operational procedure and calibration method is same as that of Zahn viscosity cups.

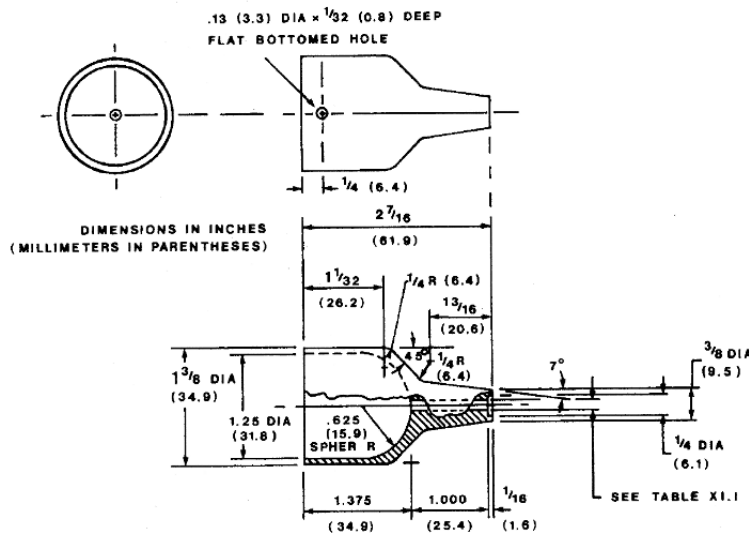


Figure 2.28. Shell viscosity cup (Reproduced from ASTM D4212-99 with permission of American Society for Testing and Materials International, West Conshohocken, PA, USA).

Table 2.18. Orifice size of Shell cup viscometer.

Cup Number	Nominal Orifice Diameter, (mm)	Approximate Viscosity Range, (cSt)	Approximate Oil Viscosity at 25°C, (cSt)
1	1.8	2 – 20	9
2	2.4	10 – 50	9, 20
2 1/2	2.7	20 – 80	35
3	3.1	30 – 120	35, 120
3 1/2	3.5	40 – 170	120
4	3.8	70 – 270	120
5	4.6	125 – 520	120, 480
6	5.8	320 – 1300	480

A number of other viscometers have been designed whose principle of operation is similar to Redwood viscometer. Various aspects of these viscometers are summarized in Table 2.19.

### **2.3 HIGH TEMPERATURE, HIGH SHEAR RATE VISCOMETERS**

Viscosities up to 1000 poise are conveniently measured using suspended-level capillary viscometers. For measuring higher viscosity, Cylinder-piston type viscometers are preferable. Cylinder-piston type viscometers are often referred to as variable pressure capillary viscometers. In the cylinder-piston type viscometers, the instrument has a cylinder as the liquid reservoir, and the fluid is displaced by a mechanically driven piston or plunger. The mechanism that activates the piston can be - a dead weight, a pneumatic device, and hydraulic pressure or mechanical devices. The equipment is well suited for the viscous non-Newtonian fluids. Several instruments in the past were designed based on cylinder-piston mechanism. These include MCER high shear rate capillary viscometer, Standard oil high pressure capillary viscometer, Plunger viscometer, and Erica-Glynn and Grunberg Viscometer. All of these viscometers were available commercially. One of the major issues with these viscometers was the temperature control of the instrument that generally led to less accurate measurement. As a consequence, most of these viscometers are now obsolete.

Several new viscometers in recent years have been developed to address the temperature control of the instrument and also the accuracy. Different configurations of capillary viscometers are available for measuring viscosity of engine oil at a single temperature and single shear rate that can provide greater uniformity and improved precision. Klaus et al.<sup>22</sup> developed a single-pass capillary viscometer that consisted of a long capillary which was necessary to meet the specifications for measuring the viscosity of oil according to the requirements of the Society of Automotive Engineers, Washington D.C., USA. However, the instrument requires a long time to run a measurement and also significant corrections to the data are necessary. Further work by Rein and Alexander<sup>23</sup> and Graham et al.<sup>24</sup> noted that the capillary approach for measuring viscosity of oil under high temperature, high shear conditions is feasible. Manning and Lloyd<sup>25</sup> designed a multicell high temperature high shear capillary viscometer that was later commercialized by the Cannon Instrument Co. PO Box 16, State College, PA, USA.

This multicell viscometer is capable of measuring viscosity of engine oil at 150°C. In this method, applied gas pressure forces a fixed volume of fluid through a small-diameter glass capillary. The rate of shear can be varied up to  $10^6 \text{ s}^{-1}$ . This technique is commonly used to simulate the viscosity of motor oils in operating crankshaft bearings. This viscosity is called high-temperature high-shear (HTHS) viscosity and is measured at 150°C and  $10^6 \text{ s}^{-1}$ . A schematic diagram of the viscosity cell is shown in Fig. 2.29.

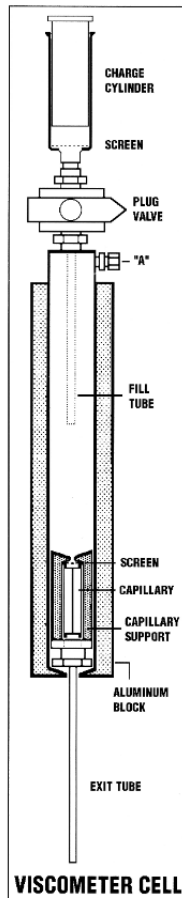


Figure 2.29. High Temperature High Shear rate viscometer (Courtesy of Cannon Instrument Co., State College, PA, USA).

## 2.4 ROTATIONAL VISCOMETERS

Rotational viscometer operates on the principle of measuring the rate of rotation of a solid shape in a viscous medium upon application of a known force or torque required to rotate the solid shape at a definite angular velocity. Although rotational viscometers are more elaborate than the capillary type during operation and often less accurate for Newtonian liquids, they have several advantages that make them attractive particularly to study the flow properties of non Newtonian materials. Some of the advantages are: measurements under steady state conditions, multiple measurements with the same sample at different shear rates, continuous measurement on materials whose properties may be function of temperature, and small or no variation in the rate of shear within the sample during a measurement.

A number of rotational viscometers with different designs are available commercially. These viscometers can be classified into three general categories based on their design configuration.

1. Coaxial-Cylinder Viscometer
2. Cone and Plate Viscometer
3. Coni-Cylinder Viscometr

### 2.4.1 COAXIAL-CYLINDER VISCOMETER

The basic design of a coaxial-cylinder viscometer is shown in Fig. 2.30. It consists of an inner cylinder (which is also called a bob) of radius  $R_1$  and height  $h$ , and an outer cylinder or cup of radius  $R_2$ . The inner cylinder is stationary. The outer cylinder that contains the test sample is rotated at a constant speed ( $\Omega$  rad/sec). The resultant torque ( $T$  dyne cm) is measured by the angular deflection of the inner cylinder that is suspended by a fine wire. The velocity of the outer cylinder can be varied to obtain the data on the change in viscosity of the fluid with the shear rate. The dynamic viscosity of Newtonian fluid may be obtained from the following expression.

$$T = \frac{4\pi R_1^2 R_2^2 h \eta \Omega}{R_2^2 - R_1^2} = C \eta \Omega \quad (2.28)$$

where  $C$  is a constant specific to the instrument. Equation (2.28) actually provides the torque exerted by the liquid on the curved surface of the inner cylinder and suggests that the viscosity is directly proportional to the ratio of torque to angular velocity. However, the equation fails to account for the forces on the ends of the cylinder introducing errors in the measurement.

The end effects are generally accounted for by adjusting the instrument constant  $C$  during the calibration process using experimental data of one or more liquids of known viscosity. A general description and operating procedure of a coaxial cylinder viscometer is given below.

The ranges of operation depend on the dimensions and design of the cylinders. The outer cylinder could be about 3 in. in diameter, made of forged steel, bored to the required size of about 0.5 in. diameter, grounded and hardened. The inner cylinder is made of mild steel with a radius of about 50 microns less than that of the bore in the outer cylinder. This small clearance enables to shear the film of the same thickness as a normal paint film. The instrument functions well when housed in a constant temperature room, but in other situations the outer cylinder holds the constant temperature long enough to complete the experiment isothermally. The outer cylinder is fixed coaxially on the spindle of a motor for convenient rotation. The lower edge of the outer cylinder is cut away so that the inevitable drips of test sample fall on the flat top of the motor housing and do not run down into the apparatus. Using three springs of different gauge wire and an ordinary motor, the instrument is capable of determining the viscosities over the range of 0.2 to 100 poise.

The instrument is adjusted so that the pointer gives a zero reading on the circular scale with the motor running. The cylinder and the sample are then adjusted to the right temperature. The outer cylinder is placed in the correct position and filled with the test sample and the inner cylinder inserted. For thin fluids, the inner cylinder is just pushed in and displaces some of the sample which can be removed from the top by means of palette knife. For thicker or highly thixotropic materials, it is better to use the plug provided for the purpose of removing the excess sample from the bottom of the cylinder. This method ensures the maintenance of continuous and uniform film between the two cylinders. The motor is in a wound up condition. On commencing the experiment, the outer cylinder is rotated at a constant predetermined speed and the scale reading observed.

The inner cylinder is now displaced manually from its position and a further reading is taken with the motor in the running condition. The procedure is repeated several times and the minimum reading recorded as the correct one. If the inner cylinder is out of center, readings higher than the true value are obtained. With temperature remaining constant and the film remaining unbroken the minimum reading results. For samples containing volatile liquids, it is better to have the top of the inner cylinder about a millimeter below that of the outer cylinder and keep this small place filled with the sample. If the top of the outer cylinder is flat, the height of the inner cylinder can be adjusted by a simple gauge, resting on the top of the outer cylinder. The experiment can be repeated to ensure consistency.

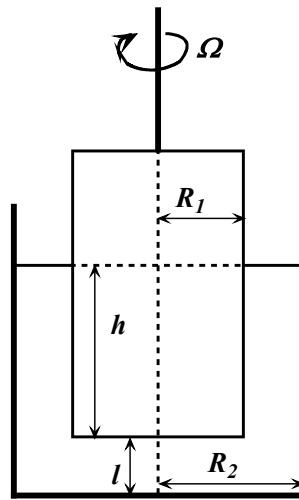


Figure 2.30. Basic structure of a coaxial cylinder viscometer.

The instrument can be calibrated for determining the torque equivalent per unit scale reading by comparison with weights operated over pulleys. Such measurements are subject to inaccuracies due to friction in the pulleys and particularly in this instrument by the fact that the cylinder radii must be determined with very great accuracy. In practice it is simpler to assemble the apparatus and calibrate the spring using a series of liquids with known Newtonian behavior. However it should be noted that the equipment is not suitable for greases and solid substances like bitumen.

The main concern with the coaxial cylinder viscometer is the end effects. Couette<sup>26</sup> and Hatschek<sup>27</sup> modified the design to eliminate end effects by the use of guard rings. Mallock<sup>28</sup> suggested that the inner cylinder can have a concave base in which a bubble of air could be trapped to reduce the drag on the base. However, the difficulty of trapping the same volume of air during each measurement prevented its wide range use. Lindsley and Fisher<sup>29</sup> found that the end effect is negligible in the range of 1 to 150 poise, but the viscosity must be corrected when it is below 1 poise. They also noted that the viscous drag on the bottom of the inner cylinder was about the same whether the bottom was open or closed. Lindsley and Fisher<sup>29</sup> and Highgate and Whorlow<sup>30</sup> suggested that modification of the design may not be adequate to account for the end effects. Therefore experimental measurements and theoretical analysis were proposed by a number of researchers to correct the viscosity for end effects. Kobayashi et al.<sup>31</sup> provided end corrections for several combinations of bobs and cup design. For rotating bob viscometer system, the end correction appears to increase

for Reynolds numbers above 10, even for low viscosity and Newtonian liquids. A conical end of the bob and a wide gap between the bob and cup give a larger end correction. According to Wein and Tovchigrechko<sup>32</sup>, liquids display both a general shear thinning behavior and an apparent slip at solid wall. They suggested a new way of correcting the viscosity data for several configurations of coaxial cylinder viscometers with different gap widths. Other concerns related to coaxial cylinder viscometer include viscous heating<sup>33</sup>, calibration procedures<sup>34</sup>, and measurement on non-Newtonian fluids<sup>35-37</sup>.

For measuring viscosity for materials of higher viscosity or at a low temperature, it is more convenient to rotate the inner cylinder keeping the outer cylinder stationary. This method is often called Cold Cranking Simulator (CCS) and follows the Test Method ASTM D2602. A schematic diagram of a CCS is given in Fig. 2.31. It should be noted that Equation 2.28 is equally applicable when the inner cylinder is rotated at angular velocity  $\Omega$  rad/sec. It also holds when torque is applied externally and the resultant speed of rotation is measured.



Figure 2.31. Cold crank simulator (Courtesy of Cannon Instrument Co., State College, PA, USA).



### 2.4.2 CONE AND PLATE VISCOMETERS

Cone and plate viscometers are probably the most popular rotational viscometers for studying rheological properties of non-Newtonian fluid. As shown in Fig. 2.32, the sample is contained in the space between a cone of large apical angle and a flat surface normal to its axis. If the angle between the cone and plate is small, less than 0.05 rad ( $3^\circ$ ), the rate of shear is essentially uniform throughout the sample. The same is also true for non-Newtonian fluids making cone and plate viscometers useful for both Newtonian and non-Newtonian fluids.

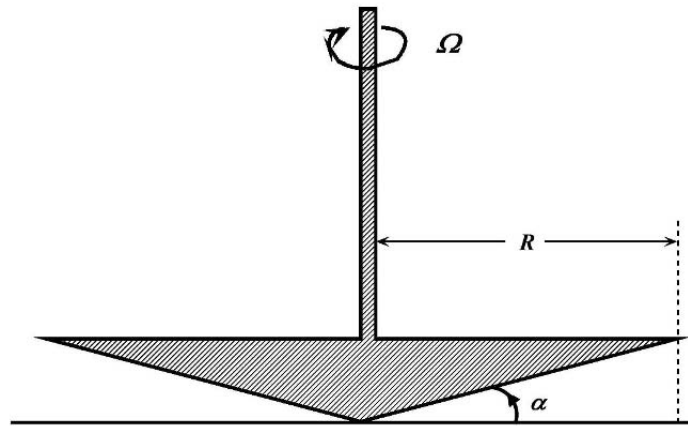


Figure 2.32. Cone and Plate viscometer.

The rate of shear  $D$  at a distance  $r$  from the axis can be written as

$$D = \frac{r \Omega}{r \alpha} = \frac{\Omega}{\alpha} \quad (2.29)$$

where,  $\alpha$  is the angle between the cone and plate in radian,  $\Omega$  is the relative angular velocity in rad/sec. Since  $D$  is independent of  $r$ , the shearing stress should be independent of  $r$  too and the total torque  $T$  can be expressed as

$$T = \int_0^R F r (2\pi r) dr = \frac{2\pi R^3 F}{3} \quad (2.30)$$

Therefore for a Newtonian liquid the viscosity may be obtained from

$$\eta D = F \quad (2.31)$$

or,

$$\eta = \frac{3T\alpha}{2\pi R^3 \Omega} = \frac{CT}{\Omega} \quad (2.32)$$

where,  $C$  is the instrument constant and is typically provided by the manufacturer. A number of possible errors associated with cone and plate viscometers have been suggested, however in normal operations these problems appear to be minimal with a well constructed and calibrated viscometer. Adams and Lodge<sup>38</sup> noted that the error in using Equation 2.32 for shear rate at the plate is less than 0.7% at 0.1 rad and 2% at 0.18 rad. A number of researchers have studied the effect of inertia and secondary flow<sup>39-42</sup>. In cone and plate viscometers, inertia forces tend to pull the plates together and generates a secondary flow that increases the torque. Turian<sup>43</sup> suggested that Equation 2.32 may be modified to take into account this extra torque as follows.

$$\frac{T}{T_0} = 1 + 6.1 \times 10^{-4} Re^2, \text{ where } Re = \frac{\rho \Omega \alpha^2 R^2}{\eta_0} \quad (2.33)$$

Other factors that can introduce errors include edge effects and shear heating. Miller and Christiansen<sup>40</sup> found that the edge effects for a 4° cone and plate viscometer were negligible. However, other researchers<sup>44-46</sup> discussed the edge problems and proposed modifications to Equation 2.32 to account them during calculations. Viscous heating in cone and plate viscometers was studied by a number of researchers<sup>47-51</sup>. However, in recent designs, the temperature of the sample is controlled during tests. This is achieved by circulating a coolant through the plate of the viscometer. Other corrections for cone and plate viscometers include secondary flow<sup>52,53</sup>, tangential stress<sup>54</sup>, non-linear flow<sup>55</sup>, migration of particles<sup>30,56</sup>, non-Newtonian fluids<sup>57,58</sup>, and slip caused by wall effects<sup>59</sup>.

Mooney and Ewart<sup>60</sup> appear to have been the first to design a cone and plate viscometer for viscosity measurements. A double cone and plate viscometer was described by Piper and Scott<sup>61</sup> in which rubber sample was subjected to increased hydrostatic pressure during viscosity measurements. Higginbotham<sup>62</sup>, and Higginbotham and Benbow<sup>63</sup> described detailed construction features of three cone and plate viscometers for which the cone angle was only 0.009 rad (0.5°). Markovitz et al.<sup>64</sup> first described a cone and plate viscometer in which cones of angle 0.5, 1 and 2° could be

interchanged. Jobling and Roberts<sup>65</sup> and Lammiman and Roberts<sup>66</sup> described the development of Weissenberg Rheogoniometer. The Ferranti Shirley viscometer was designed based on the work by Russell<sup>67</sup> who used the cone and plate geometry to measure normal stress<sup>68</sup>. Today both manual and automatic cone and plate viscometers are available commercially. A list of the manufacturers and the special features of these instruments are given in Table 2.19. Among various rotational viscometers, Rotovisco-Haake, Agfa Rotational, Brookfield, Ferranti-Shirley, Stormer, and Rheogoniometer are considered as classic one, since most of the modern viscometers are improvements of these classic ones. These viscometers are briefly described below.

#### 2.4.2.1 Haake Rotovisco

The Rotovisco originally manufactured by Gebruder-Haake in Berlin, Germany is considered one of the most versatile viscometers. It measures the viscosity in the range of  $5 \times 10^{-3}$  to  $4 \times 10^7$  poise. The range of apparent rate of shear is  $10^{-2}$  to  $10^4 \text{ s}^{-1}$ , and the range of shear stress is 10 to  $10^6 \text{ dyn.cm}^{-2}$ . The results are generally accurate within  $\pm 1\%$ . The apparatus consists of a fixed outer cup and an inner rotor. Several combinations of cups and rotors of different dimensions are available. The rotor is driven through torque dynamometer. The basic speeds ranging from 3.6 to 582 rpm, can be produced by means of gears. Rotors are some times ribbed to prevent slippage. Plastic rotors are provided for use with high temperature studies. The various torsion heads and speed reducers are interchangeable. These types are often provided with coaxial cylinder as well as plate and cone viscometers. The main disadvantage with this model is the high moment of inertia resulting in difficulties in determining the yield value.

#### 2.4.2.2 Agfa Rotational Viscometer

Viscosities in the range of  $10^{-1}$  to  $10^5$  poise with shearing rates 0.4 to  $450 \text{ s}^{-1}$  and shear stresses in the range of 10 to  $10^5 \text{ dyn.cm}^{-2}$  are the normal ranges for the use of Agfa rotational viscometers. The temperatures studied are generally  $-30$  to  $70^\circ\text{C}$ . The basic unit has an inner cylinder of 1.7 cm radius and 5.5 cm height, while the outer cylinder is of 1.82 cm radius. The torsion rod has a length of 6 cm and a diameter of 0.5 cm and a torsional spring constant of  $1.48 \times 10^7 \text{ dyn.cm} \cdot (\text{deg of deflection})^{-1}$ . The cup which is immersed in a constant temperature bath is rotated by means of a continuous variable speed drive operating in the range of 0.08 to 250 rpm. The high natural frequency of the torque sensing system produces accurate readings in intervals as short as 0.01s.

### 2.4.2.3 Rheogoniometer

Rheogoniometers are capable of measuring the normal forces and their distribution as a function of the distance and tangential stress. The range of measurable viscosities is from  $10^{-3}$  to  $10^{10}$  poise and multi point flow curves can be obtained for fluids ranging from  $10^{-2}$  to  $10^9$  poise. Normal stresses can be measured over the range of 1.3 to  $6.3 \times 10^4$  dyn.  $\text{cm}^{-2}$ . In this instrument the cone is rigidly fixed while the flat lower plate rotates. The selection of the cone angles and plate diameters can be made according to the requirements. The torsion imparted on the cone due to the rotation of the flat plate is measured. Two synchronous motors drive a sixty speed gear box. Requirement of a very small amount of the sample is the main advantage. In oscillatory tests, the plate oscillates about its axis and the oscillating motion is transmitted to the cone through the sample. The special features include – uniform shear rate throughout the sample, applicability to non-Newtonian flow. Oscillatory shear can be applied to determine the elastic and viscous components.

### 2.4.2.4 Ferranti-Shirley Cone-Plate Viscometer

The range of this instrument is from zero to 32,000 poise. It has a stationary flat plate and rotating conical disk driven by a variable speed motor through a gear train and torque spring. The torsion due to viscous drag is measured by a potentiometer on the spring which sends the signal to the recorder. This instrument is one of the most versatile types available for the study of the rheological properties of non-Newtonian fluids, especially of the pseudo-plastic type. The viscometer has an essentially constant rate of shear throughout the gap. The ranges are wide in terms of – viscosity, shear stress and shear rate. These features enable the measurement of the brush-ability of paints, etc. The temperature rise in the gap is often of the order of one degree.

### 2.4.2.5 Stormer Viscometers

The distinguishing feature of this instrument is that the shear rate is held constant and shear stress is measured. It consists of a stationary outer cup and inner rotor driven by weights and pulley. Stress is varied by applying different weights. The time for 10 revolutions is noted. In this model, the end and edge effects are eliminated. The model is not suitable for measuring the fast time dependant phenomena accurately and precisely.

### 2.4.3 CONI-CYLINDER VISCOMETER

The conic-cylinder viscometer is a modification of the coaxial cylinder viscometer to eliminate the end effect as much as possible. Mooney and Ewart<sup>60</sup> suggested conical extension of both inner and outer cylinders. Fig. 2.33 shows a schematic of a conic-cylinder viscometer. The advantage of conic-cylinder viscometer is that the mean rate of shear in the cylindrical annulus and in the conical portion is about the same.

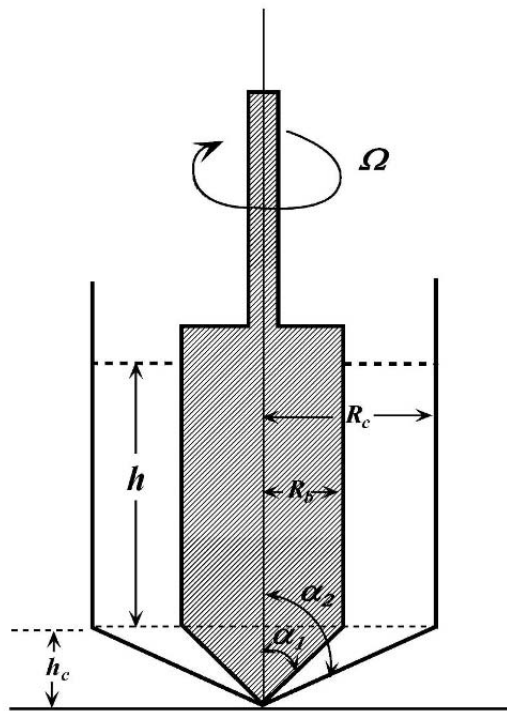


Figure 2.33. Coni-Cylinder viscometer.

The viscosity may be calculated from the following relationship.

$$\frac{T}{\Omega} = \frac{4\pi h \eta}{\left(\frac{1}{R_b^2} - \frac{1}{R_c^2}\right)} \left(1 + \frac{\Delta h}{h}\right) \quad (2.34)$$

where  $\Delta h$  is given by

$$\Delta h = \frac{h_c}{6 \cos^2 \alpha_2} \left( \frac{1}{\tan^2 \alpha_1} - \frac{1}{\tan^2 \alpha_2} \right) \left( \int_{\alpha_1}^{\alpha_2} \frac{d\alpha}{\sin^3 \alpha} \right)^{-1} \quad (2.35)$$

- and,  $T$  is the torque,  
 $\Omega$  is the angular velocity,  
 $h$  is the length of the cylindrical portion as shown in Fig. 2.33,  
 $h_c$  is the length of the conical portion as shown in Fig. 2.33,  
 $R_b$  is the radius of the inner cylinder, and  
 $R_c$  is the radius of the outer cylinder.

The conical-cylinder geometry is found to be most convenient for measuring viscosity and shear rate of slurries. Chang et al.<sup>69</sup> measured viscosity of  $\text{Al}_2\text{O}_3$  slurry using conical-cylinder geometry containing 0.10 volume fraction  $\text{Al}_2\text{O}_3$ . Wang et al.<sup>70</sup> also used a similar geometry for measuring viscosity of silicon nitride slurry containing up to 0.515 volume fractions solid.

#### 2.4.4 ROTATING/PARALLEL DISK VISCOMETERS

The rotating disk or the parallel disk geometry suggested by Mooney<sup>71</sup> consists of a disk rotating inside a cylindrical cavity formed by two dies maintained at specified conditions of temperature and die closure force. It is used extensively for measuring viscosity of rubber. Several modifications to the original design of Mooney<sup>71</sup> led to current commercial rotating disk viscometers<sup>72-75</sup>. The detailed construction features of a rotating disk viscometer, better known as the Mooney viscometer, are described in Test method ASTM D 1646. A number of Mooney viscometers are available commercially and are listed in Table 2.19. The Mooney viscometer is capable of measuring the effect of temperature and time on the viscosity of rubbers. If the stress relaxation test is to be performed, the rotation of the disk is stopped and the relaxation of stress versus time is measured. The dies and die holders forming the cavity are fabricated from a nondeforming steel, that has an unplated finish. The surfaces of the die cavity are either serrated or contain V-grooves to minimize slippage of the sample. The test procedure is given in ASTM D 3346. The viscosity of rubber is expressed as Mooney viscosity, which is an arbitrary measure of the viscosity of a rubber determined in a Mooney shearing disk viscometer, indicated by the torque required to rotate the disk embedded in a rubber specimen and enclosed in the die cavity under specified conditions.

The theory for calculating viscosity and shear stress using a rotating disk viscometer along with various correction factors has been described by a

number of researchers. Goodrich and Chatterjee<sup>76</sup> analyzed numerically the fluid flow in the neighborhood of a rotating disk inserted into a liquid interface. They concluded that the rotating disk viscometer may not be the best instrument for determination of low surface viscosity.

Cross and Kaye<sup>77</sup> obtained rubber viscosity/shear rate data ( $R$ ) with a parallel disk viscometer from measurements of viscous torque as a function of disk separation and angular velocity using a modified procedure via an equation which related the rim shear stress to the torque and the disk radius. The data appeared to be satisfactory

Perry et al.<sup>78</sup> designed a parallel-disk viscometer which can be fitted directly onto a reaction-injection molding machine and provided the criteria for isothermal operation, taking into account both conduction to the viscometer and the rapid exothermic reaction typical of reaction-injection molding polymer solutions.

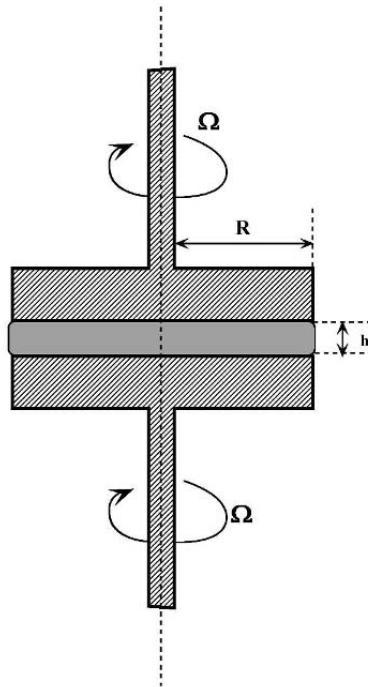


Figure 2.34. Schematic of a parallel disk viscometer (Mooney viscometer).

Other issues that were addressed by various researchers include viscometric flow under apparent wall slip<sup>79</sup>, operation for high Reynolds number<sup>73</sup> and instability of a viscoelastic fluid<sup>80</sup>.

## 2.5 FALLING BALL VISCOMETERS

In falling ball viscometers, a solid body is allowed to fall under gravity through a viscous medium. After a period of initial acceleration, the solid body attains a uniform terminal velocity when the gravitational force is balanced by the viscous resistance of the fluid. By measuring the terminal velocity of the falling body, the viscosity can be determined. Although a solid body of any shape and size can be used, a spherical geometry is preferable due to the simplicity involved in deriving the theory. Consider the system shown in Fig. 2.35 in which a sphere is falling through a homogeneous fluid. If the motion of the sphere is sufficiently slow, the inertia terms become negligible. Under this condition and assuming that the fluid medium has an infinite extension, the viscous resistance to the motion of the sphere moving with a velocity  $v$  is equal to the driving force due to the difference in density between the sphere and the fluid. This is known as the Stokes' law and is given by

$$6\pi r \eta v = \frac{4}{3} \pi r^3 (\sigma - \rho) g \quad (2.36)$$

where,  $\eta$  is viscosity of fluid,  
 $\rho$  is density of fluid  
 $\sigma$  is density of sphere  
 $r$  is radius of sphere, and  
 $g$  is acceleration due to gravity.

Equation 2.36 can be rewritten as follows to obtain the viscosity

$$\eta = \frac{2g r^2 (\sigma - \rho)}{9v\pi} \quad (2.37)$$

For a given system,  $r$ ,  $\sigma$ , and  $\rho$  are fixed. Therefore, viscosity can be obtained by measuring the velocity of the sphere through the fluid. Equation 2.37 is for ideal situation and is corrected for practical viscometers by a number of researchers. The main error is due to the wall effect. For a sphere falling in a cylinder of finite length, Ladenburg showed that the viscosity calculated from the Stokes law (Eq. 2.37) should be corrected as follows:

$$\eta_{true} = \frac{\eta_{measured}}{\left(1 + 2.4 \frac{r}{R}\right) \left(1 + 3.3 \frac{r}{h}\right)} \quad (2.38)$$



where  $r$  is the radius of the sphere,  $R$  is the radius of the cylinder containing fluid and  $h$  is the height of cylinder containing fluid. The first term in the denominator is a correction for the wall effect, where as the second term corrects for the finite depth of the fluid.

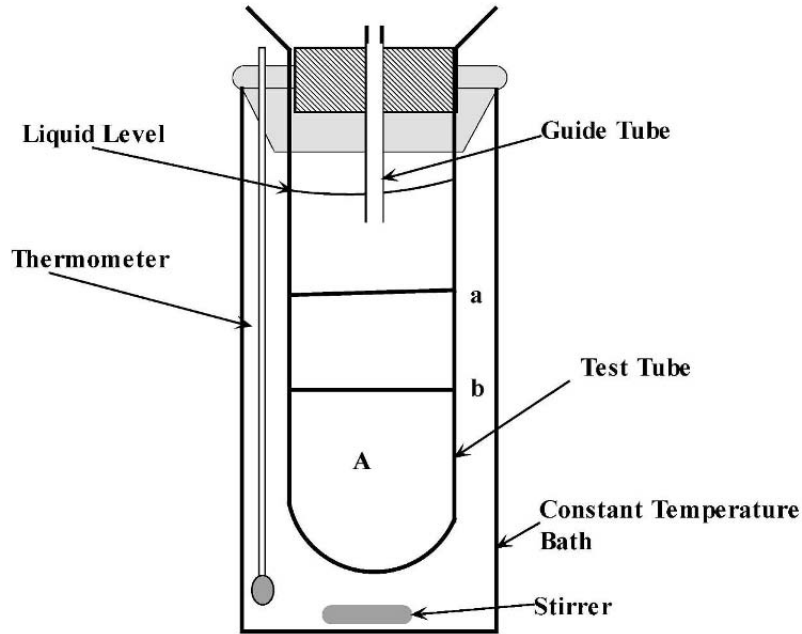


Figure 2.35. Falling ball viscometer.

Although a number of correction factors have been suggested, the Faxen expression appears to provide the best result. The Faxen correction factor is given as

$$\eta_{true} = \eta_{measured} \left[ 1 - 2.104 \frac{r}{R} + 2.09 \left( \frac{r}{R} \right)^3 - 0.95 \left( \frac{r}{R} \right)^5 \right] \quad (2.39)$$

The basic experimental system is shown in Fig. 2.35. The tube  $A$ , closed at the bottom, is filled with the sample to be tested. The tube  $A$  is placed inside a constant temperature bath that could be maintained within  $\pm 0.1^\circ\text{C}$ . The arrangement should exist to align the tube vertically inside the constant temperature bath. The tube is kept in the bath for about an hour until the test sample is at a uniform temperature and free from bubbles. Once the desired

temperature is attained, the sphere is introduced into the tube along the axis by means of a guide tube. It is advisable to clean the sphere and bring it to the temperature of the test liquid by an external means before dropping into the liquid. The position of the sphere is followed and the time at which it passes the reference marks  $a$  and  $b$  is noted. Various systems including telescope, optical device, or magnetic sensors are used to follow the sphere movement and to accurately determine the time to travel between marks  $a$  and  $b$ . The density of both the liquid and the sphere at the test temperature should be determined as accurately as possible. If the same sphere and fall tube are used, the expression for calculating viscosity may be written as

$$\eta = K t (\sigma - \rho) \quad (2.40)$$

where  $t$  is the time to fall between the reference marks  $a$  and  $b$ ,  $K$  is a constant specific to the instrument,  $\sigma$  is density of sphere, and  $\rho$  is density of fluid. In Table 2.19 is listed various manufacturers of falling ball viscometers and their sensing mechanism.

A number of researchers have studied various aspects of falling ball viscometers because of its simplicity and ease of operations. Cho<sup>81</sup> measured the viscosity of non-Newtonian flows using a falling ball viscometer. Later Cho et al.<sup>82</sup> studied the wall effect for viscoelastic fluids. They noted that with increasing elasticity, the magnitude of correction needed became smaller. The wall, inertial, and end effects in a falling ball viscometer are an interesting area of research, and several researchers in the past have addressed these issues<sup>83-86</sup>. Feng et al.<sup>87</sup> addressed these effects by obtaining both experimental data and numerically, which according to them will lead to both accuracy and reproducibility of the data. Attempts have also been made to design high precision viscometer<sup>88</sup>, computer interfaced system<sup>89</sup>, viscometer for refrigerants and mixtures<sup>90</sup> and for samples less than 1 mL<sup>91</sup>.

### 2.5.1 FALLING SPHERE VISCOMETER FOR OPAQUE LIQUIDS

Falling ball viscometers may be used to measure viscosity of opaque liquids. Since the direct observation of the sphere using an optical device is not possible, other methods such as electric and magnetic methods and x-ray photograph have been used.

### 2.5.2 ROLLING BALL VISCOMETERS

Similar to falling ball viscometers, the viscosity can also be measured using a sphere rolling down an inclined cylindrical tube filled with the test liquid. A schematic diagram of the system is shown in Fig. 2.36. Hubbard

and Brown<sup>92</sup> carried out a detailed analysis of the rolling ball viscometers for Newtonian fluid. However, for a specific instrument, for the same sphere and tube, Equation 2.40 can be used to calculate viscosity. Rolling ball viscometers offer several advantages over fall balling viscometers including (1) only a small amount of sample is required, (2) visual observation in glass apparatus is possible even with opaque liquids since the ball is in contact with the glass tube making it possible for use of optical sensing system, and (3) a number of parameters such as tube diameter, angle of inclination, and rolling distance can be varied. Flowers<sup>93</sup> first suggested the use of a rolling ball system for measuring viscosity. Hersey<sup>94</sup> developed the correlation of the variables involved in the measurement. Sage<sup>95</sup> used the system for measuring viscosity of hydrocarbons. The work of Hoespler<sup>96</sup> led to the development of a commercial instrument which is currently known as Hoespler Viscometer. Hoespler used the eccentric fall of large spheres in a tube inclined at an angle of  $80^\circ$  for his measurements. A classical Hoespler Viscometer is shown in Fig. 2.36. The modern day Hoespler Viscometer is designed in the same manner. The precision bore tube is made of heat resistant, chemically inert glass and is about 16 mm in diameter and 200 mm long. The length of the measuring distance is generally 100 mm in both running direction. The working angle is generally  $10^\circ$  vertical, but it can be extended up to  $50^\circ$  in some of the models. The tube is jacketed and using a constant temperature circulating bath, the test temperature can be controlled in the range of  $-60^\circ\text{C}$  to  $+150^\circ\text{C}$ .

The tube is filled with about 30 to 40 mL test liquid, and after the proper ball has been enclosed, it is sealed by means of a special capillary plug. This allows inversion of the tube without introducing air bubble into the system. The measurement is made by timing the passage of the ball between two marks in the glass tube using an optical device. The viscosity is calculated from Eq. (2.40) with an accuracy of 0.5 to 2% depending on the ball used. The instrument is supplied with a set of six balls. Generally two balls are made of borosilicate glass, two from nickel-iron alloy, and the other two from stainless steel.

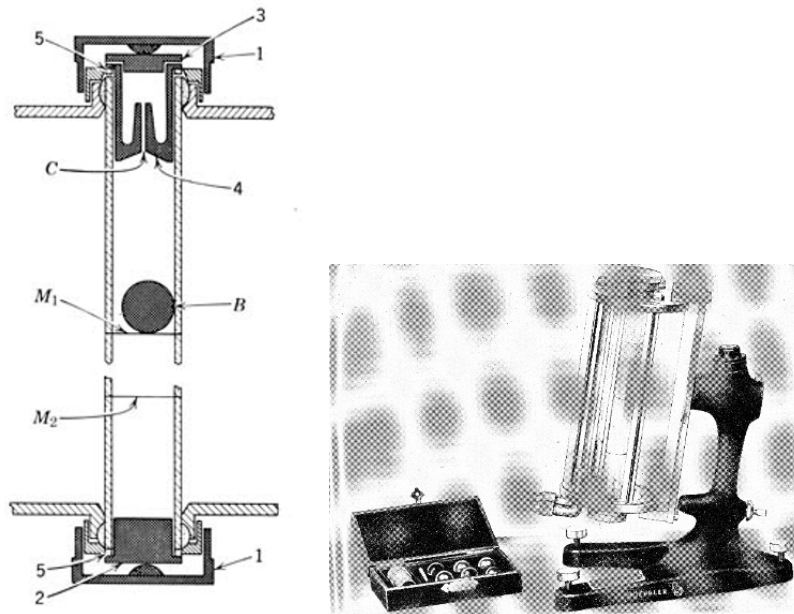


Figure 2.36. Rolling ball viscometer (Adopted from J. R. Van Wazer, J. W. Lyons, K. Y. Kim, and R. E. Colwell, *Viscosity and Flow Measurement A Laboratory Handbook of Rheology*, Interscience Publishers, New York, 1963).

Most of the current rolling ball viscometers are designed for high pressure operations<sup>97-101</sup>. A number of investigators have also addressed the theory for the improvement of the understanding of the operation particularly for non-Newtonian fluids and accuracy of the measurements<sup>102-106</sup>.

### 2.5.3 FALLING CYLINDER VISCOMETERS

Although the use of a sphere in falling ball viscometers simplifies the analysis and the calculation of viscosity, other geometry has also been explored to improve the accuracy of the measurement. Both cylinders and needles have been used by various researchers to measure the viscosity. Instruments are also available commercially using these types of geometry.

The working principle of the falling cylinder viscometer is basically the same as that of a falling ball viscometer and it can be used for both Newtonian and non-Newtonian fluids. The measuring system consists of a cylinder or a piston in a measuring tube. The measuring element is installed in a tank or in a liquid filled line. The measuring tube is completely immersed in the liquid. The piston assembly is raised by an air lifting or

other mechanisms such as magnetic lift, drawing a sample of the liquid to be measured down through the clearance between the piston and the inside of the cylinder into the space which is formed below the piston as it raised. The operation is shown in Fig. 2.37. The piston is then allowed to fall by gravity discharging the sample out through the same annulus. The time of fall is noted. The current instruments display the viscosity directly after making necessary corrections. A flow diagram of an automated a falling cylinder viscometer system using magnetic sensor is shown in Fig. 2.38.

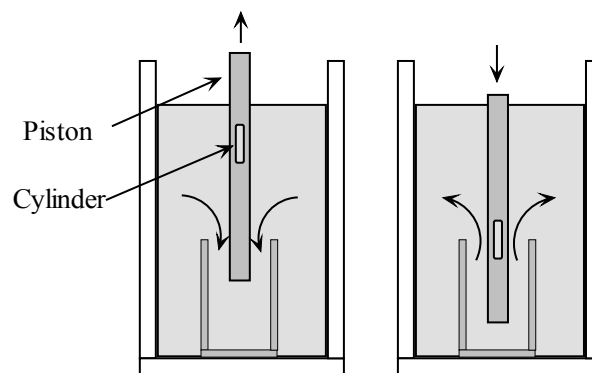


Figure 2-37. The operating principle of a falling cylinder viscometer.

Since the early work of Bridgman<sup>107</sup>, most of the work on falling cylinder viscometers concentrated on development of an accurate high pressure system<sup>108,109</sup>, studying the end effects<sup>110-116</sup>, and development of theoretical model and mathematical analysis of the system<sup>117-119</sup>.

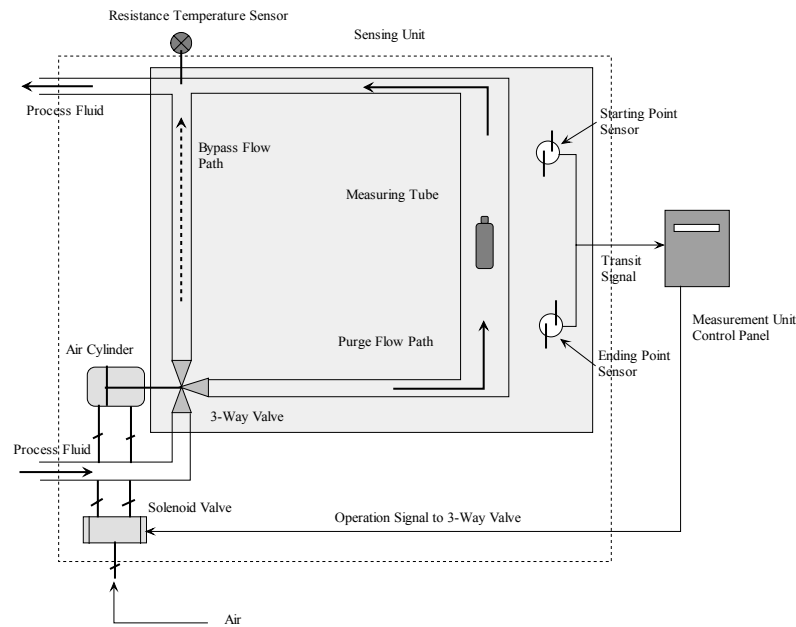


Figure 2.38. Flow diagram of a commercially available falling cylinder viscometer (Adopted from Toki Sangyo Co., Ltd. [www.tokisangyo.com](http://www.tokisangyo.com), Tokyo, Japan).

### 2.5.4 FALLING NEEDLE VISCOMETER

Falling needle viscometers offer several advantages over falling cylinder or piston viscometers and was first suggested by Park and Irvine<sup>120</sup>. It can measure absolute viscosity without instrument calibration with an accuracy of better than 1%. Also the viscosity range for falling needle viscometer is 0.1 to 10<sup>8</sup> cP. Another advantage is that it can measure density and viscosity of the test fluid simultaneously at the test temperature. A schematic diagram of the falling needle viscometer is shown in Fig. 2.39. A slender hollow cylinder, or called the needle, with hemispherical ends falls under gravity through the test liquid. The longitudinal axes of the needle and the measuring cylinder should be vertical or parallel to the gravity vector. Once the needle reaches its terminal velocity, which is generally attained after traveling a short distance, it is measured either visually or electronically. For a Newtonian fluid, the expression for calculating the dynamic viscosity for the geometry shown in Fig. 2.39 is as follows.

$$\eta = \left( \frac{g d^2 (\rho_s - \rho)}{8 v_{true}} \right) [-(1 + \ell n k)] \quad (2.41)$$

where  $g$  is acceleration due to gravity  
 $d$  is diameter of the needle  
 $\rho_s$  is density of solid (needle)  
 $\rho$  is density of fluid, and  
 $V_{true}$  is terminal velocity of needle.

Equation 2.41 is obtained assuming that the flow field around an infinitely long needle and is valid for  $k < 0.033$ , where  $k = d/D$ .  $D$  is the diameter of the cylinder. The terminal velocity must be corrected for end effects. A number of expressions have been developed for determining end correction factor. Park and Irvine<sup>121</sup> suggested following correction factor to Equation 2.41.

$$\frac{v_{measured}}{v_{true}} = 1 + \frac{2}{3L^+} \left[ 1 + \frac{3}{2C_w L^+} \left( \frac{k^2(1 - \ln k) - (1 + \ln k)}{1 + k^2} \right) \right]^{-1} \quad (2.42)$$

where

$$C_w = 1 - 2.04k + 2.09k^3 - 0.95k^5, \text{ and } L^+ = L/d.$$

$L$  is the length of the needle minus one diameter. Park and coworkers<sup>122-127</sup> studied in detail various aspects of falling needle viscometers and extended its applications to a variety of systems.

The ASTM test method D5478-98 provides the standardized protocol for measuring viscosity using a falling needle viscometer. Several improvements to the falling needle viscometer were suggested to overcome the shortcomings of the conventional instrument. Thiessen and Krantz<sup>128</sup> studied the behavior of terminal velocity of a falling needle and provided a rationale for using only small terminal velocity. Sha<sup>129</sup> suggested a new design of the needle structure, collector, and launcher and used Hall magnetic sensors and single-board computer in the system, which made the measurement automatic. Davis and Brenner<sup>130</sup> calculated drag coefficients in the presence of the tube wall for shapes varying from spheroidal toward cylindrical and suggested an alternative means of calibration. Later Davis<sup>131</sup> pointed out some of the limitations on the use of the Stokes law when applied to falling needle.

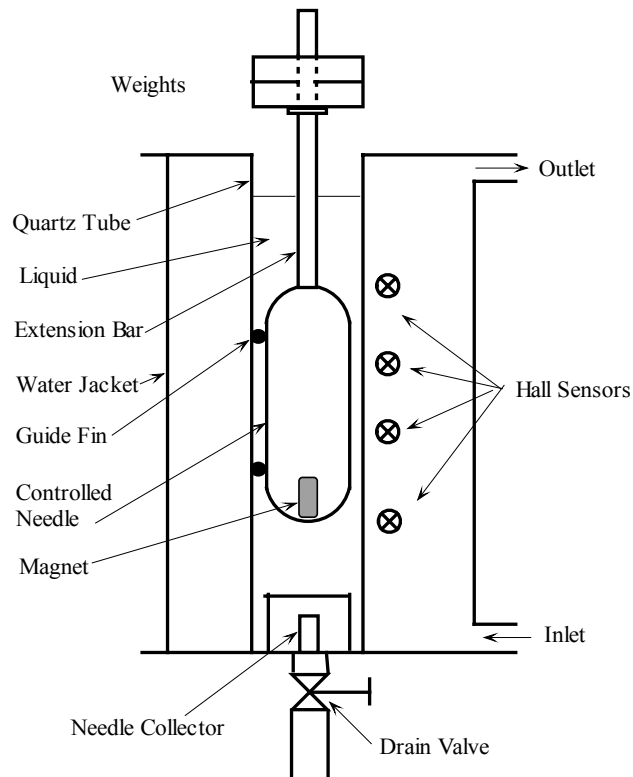


Figure 2.39. Falling needle viscometer. (Adopted from N. A. Park and T. F. Irvine, The falling needle viscometer-A new technique for viscosity measurement, *Warme Stoff.* **18**, 201-206, 1984).

## 2.6 VIBRATIONAL VISCOMETERS

In the petrochemical industry, on-line measurement of viscosity is a critical measurement parameter for maintaining the quality of the product. Vibrational viscometers are best suited for this purpose. The important features of vibrational viscometers are small sample volume requirement, high sensitivity, ease of operation, continuous readings, wide range, optional internal reference, flow through of the test liquid and consequent easy clean out and prospect of construction with easily available materials.

Vibrational viscometers measures the damping of an oscillating electromechanical resonator immersed in the test liquid. The resonator may be a cantilever beam, oscillating sphere or tuning fork which oscillates in torsion or transversely in the liquid. The resonator's damping is measured by several methods:



1. The power required to maintain the oscillator vibrating at constant, precise amplitude is measured. The higher the viscosity, the more power is needed to maintain the amplitude of oscillation. The vibrating probe accelerates the fluid and power input is proportional to product of viscosity and density.
2. The vibration of the resonator is stopped and the decay time of the oscillation is measured. The higher the viscosity, the faster the signal decays.
3. The frequency of the resonator as a function of phase angle between excitation and response waveforms is measured. The higher the viscosity, the larger the frequency change for a given phase change.

### **2.6.1 TUNING FORK TECHNOLOGY**

Vibrational viscometers designed based on tuning fork technology is capable of measuring simultaneously both the fluid viscosity and density accurately and independently. Designed specifically for hydrocarbons, continuous on line measurement is performed by determining the bandwidth and frequency of the vibrating fork resonance; the bandwidth giving the viscosity measurement whilst the frequency giving the liquid density (Fig 2.40). A temperature sensor can be easily accommodated in the instrument for temperature measurement. In addition to this, other parameters such as viscosity gravity gradients and ignition indices for fuel oils can be calculated.

### **2.6.2 OSCILLATING SPHERE**

A stainless steel sphere oscillates about its polar axis with precisely controlled amplitude (Fig. 2.41). The viscosity is calculated from the power required to maintain this predetermined amplitude of oscillation. While it is very simple in design, the oscillating sphere viscometer provides viscosity that is density dependent. Therefore, density of the test fluid should be determined independently if kinematic viscosity is required for process control.

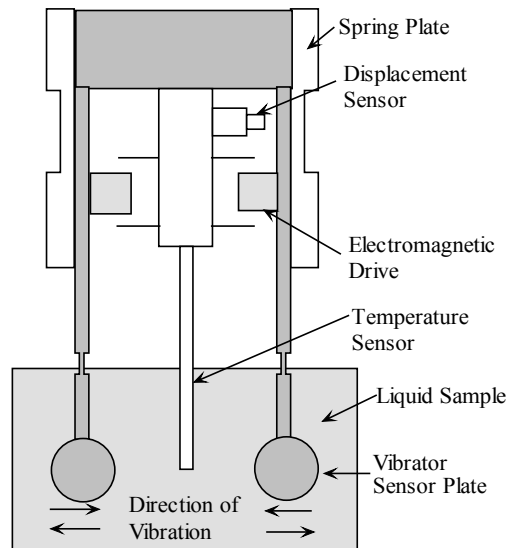


Figure. 2.40. A schematic diagram of the viscometer designed based on tuning fork technology ([http://www.gardco.com/pages/viscosity/viscometers/sv\\_10\\_100.html](http://www.gardco.com/pages/viscosity/viscometers/sv_10_100.html)).

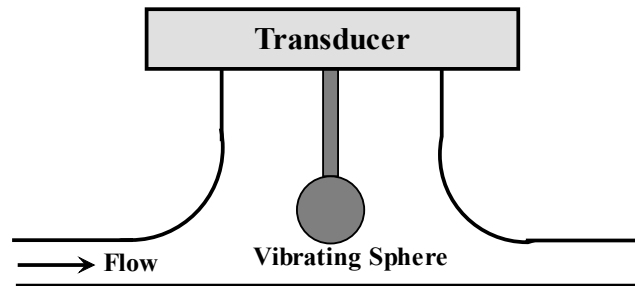


Figure. 2.41. An oscillating sphere system for creating controlled amplitude in liquid (Adopted from J. F. Steffe, *Rheological Methods in Food Process Engineering*, 2nd Ed., Freeman Press, Michigan, 1996).

### 2.6.3 VIBRATING ROD

These viscometers determine the dynamic viscosity by measuring the damping of a resonator that is excited at its natural frequency in a torsional (or twisting) vibration (Fig 2.42). A constant power source vibrates the rod, and the amplitude variations are measured to determine the viscosity. Like the oscillating sphere, the vibrating rod cannot measure kinematic viscosity.

Sieben<sup>133</sup> designed a vibrational viscometer using an oscillating capillary tube. However, recent designs employ various geometries. The solid stainless steel sensor element can be submerged in the fluid and made to twist back and forth microscopically at a high frequency. As the surface of the sensor shears through the liquid, energy is lost to the fluid because of its viscosity. Dissipated energy is accurately measured by microprocessor-controlled electronics and then equated back to viscosity (Hydramotion Ltd, York, England).

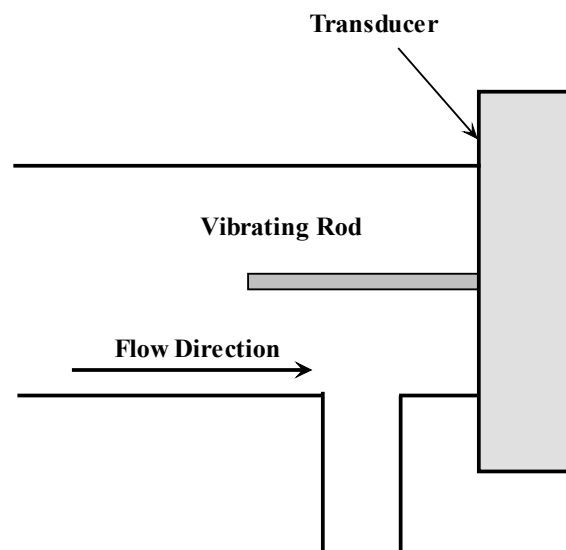


Figure. 2.42. Vibrating rod system for measuring dynamic viscosity (Adopted from J. F. Steffe, *Rheological Methods in Food Process Engineering*, 2nd Ed., Freeman Press, Michigan, 1996).

## 2.7 ULTRASONIC VISCOMETERS

Ultrasonic viscometers provide instantaneous and continuous measurement of viscosity by means of ultra high frequency sound waves. The first ultrasonic viscometer, called “Ultra-Viscoson” was developed by Rich-Roth Laboratories, Hartford, Connecticut, USA.<sup>134</sup> The basic characteristics of an ultrasonic viscometer is shown schematically in Fig. 2.43. It consists of a small sensing element or probe which is immersed in the liquid being tested. The instrument can operate over a temperature range of  $-93$  to  $315^{\circ}\text{C}$  as well as in vacuum. This instrument has high utility in connection with refinery vacuum distillation systems.

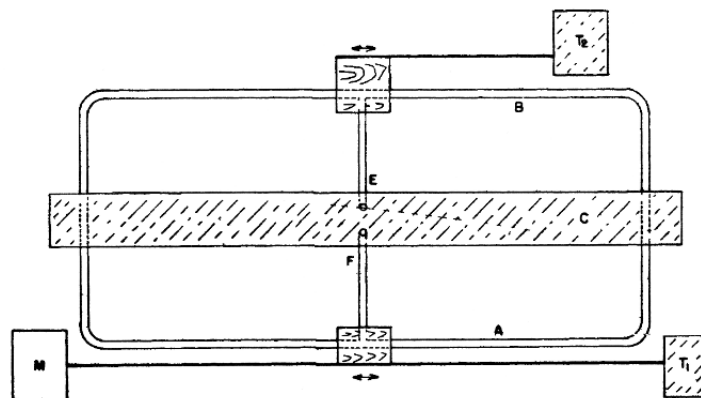


Figure 2.43. A schematic diagram of an ultrasonic viscometer.

The oscillatory motion of a sphere in a viscous liquid is utilized to measure the viscosity. The device consists of two electro dynamic transformers, placed along a common axis. The coils of both the transformers are suspended in permanent electric fields and attached to each other by means of a rod. When measurements are carried out on liquids, a sphere is attached to the rod, while the viscoelastic materials are directly fixed to the end of the rod. The emf is induced in the coil of the measuring transformer with magnitude proportional to the velocity and phase displacement. The emf is measured by means of an oscilloscope. The viscosity of the liquids over wide ranges can be determined within a few percent accuracy, applying frequencies of oscillation in the range of 0.05 to 0.07 kilocycles. Vibration is realized by switching the driving transformer.

The damping of transverse vibrations of a fine wire stretched between two rigid supports is shown to be a measure of the viscosity of liquids. The simplicity of the hydrodynamic problem and the low nuisance damping of the wire make the method attractive, particularly for the measurement of small viscosities. A number of researchers have designed and studied various types of ultrasonic viscometers<sup>135-139</sup>. Recently, Sheen et al.<sup>140</sup> of Argon National Laboratory, Il, USA designed and employed an ultrasonic viscometer for remote measurement of viscosity of radioactive liquids. Greenwood and coworkers<sup>141-144</sup> at the Pacific Northwest national Labortory, Richland, VA, US, developed an on-line sensor capable of measuring viscosity continuously. Their system is shown in Figure 2.44.

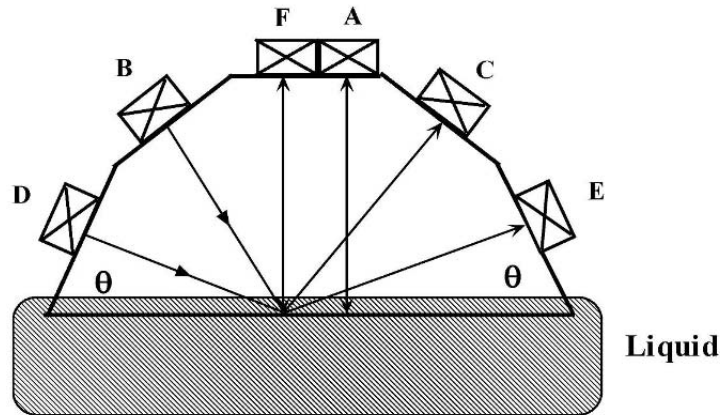


Figure 2.44. Ultrasonic wedge immersed in fluid for measuring viscosity and other properties (Adopted from M. S. Greenwood and J. A. Bamberger, Ultrasonic sensor to measure the density of a liquid or slurry during pipeline transport, *Ultrasonics*, **40**(1), 413-417, 2002).

Greenwood and Bamberger<sup>141</sup> described the working principle of the sensor as follows: The sensor consists of longitudinal (*B*, *C*, *D*, *E*, and *F*) and shear wave (*A*) transducers mounted upon a Rexolite™ wedge. The transducers have a center frequency of 2.25 MHz. When ultrasound from transducer *F* (operating in pulse-echo mode) strikes the wedge-liquid interface, part of it is reflected back toward *F* and the rest is transmitted into the liquid. Similarly, when ultrasound from transducer *D* (or *B*) (operating in pitch-catch mode) strikes the interface, some of it is reflected toward transducer *E* (or *C*), some mode converts to a shear wave in the wedge, and part is transmitted into the liquid. The reflection coefficient, that describes the amount of ultrasound reflected to the receive transducer, is dependent upon the densities and speed of sound in the liquid and the wedge material. The reflection coefficient is measured by comparing the voltage on the receive transducer when the base is immersed in the liquid with that when the base is immersed in a reference liquid, usually water. The experimental measurements are described in detail in Greenwood and Lail<sup>142</sup>, Greenwood and Harris<sup>143</sup>, and Greenwood et al.<sup>144</sup>.

## 2.8 SUMMARY

The viscosity data is necessary for the design of process equipment and maintenance of product quality, where continuous on-line viscosity data is

necessary. During processing, particularly in plastic industry, viscosity changes significantly altering both the rheological behavior and flow pattern of the liquid. Therefore no single instrument should be chosen without careful consideration of the range of measurement needed and the type of flow of the material. The change of viscosity with temperature necessitates the use of several different instruments for full characterization of the materials. Choice of measuring instruments also depends on the type of flow or if the liquid is Newtonian or non-Newtonian. Other issues that should be considered in selecting viscometers are: calibration of the instrument, ease of operation, method of accurately measuring and recording shear and shear rate variables, temperature control, and sample size. A quick guide for selecting a viscometer is given in Table 2.19.

Table 2.19 .Selection guide for viscometers, their viscosity measurement ranges and manufacturers.

<i>Instrument</i>	<i>Viscosity Range</i>	<i>Applications</i>	<i>Standard</i>	<i>Manufacturers/Suppliers</i>
<b>Glass Capillary Viscometers</b>				
Ostwald	0.3 – 10 cSt	Control testing.	ASTM D445, D446.	Cannon Instrument Co., State College, PA, USA.
Cannon-Fenske Routine	0.5 – 100 000 cSt	Petroleum products, Lubricants, Fuels, Cut back asphalt, Road oil.	ASTM D445, D446; ISO 3104, 3105.	Cole-Parmer Instrument Co., Vernon Hills, IL, USA.
Zeiffuchs Transparent	0.6 – 100 000 cSt	Newtonian liquids, Control testing.		Ertco Precision, Dubuque, IA, USA.
BS/U Tube	0.9 – 10 000 cSt	Newtonian liquids, Control testing.	ASTM D445, D446; ISO 3104, 3105; IP 71.	Rheotek -Poulten Selfe Ltd, Essex, UK. Brinkmann Instruments Inc, Westbry, NY, USA Schott Instruments GmbH, Mainz, Germany Ace Glass Inc., Vineland, NJ, USA.
<i>Suspended Level Viscometer</i>				
Ubbelohde	0.3 – 100 000 cSt		ASTM D 445, D446; ISO 3104, 3105.	DC Scientific Glass, Pasadena, CA, USA. Humboldt Mfg. Co., Norridge, IL, USA (for Zeiffuchs Cross-Arm)
Cannon-Ubbelohde	0.5 – 100 000 cSt	Low temperature applications, Lubricants, Fuels.		
BS/IP/SL	3.5 – 100 000 cSt	Transparent Newtonian liquids.		
BS/IP/SL(S)	1.05 – 10 000 cSt	Transparent Newtonian liquids.		
BS/IP/MSL	0.6 – 3 000 cSt	Transparent Newtonian liquids.		

<b>Instrument</b>	<b>Viscosity Range</b>	<b>Applications</b>	<b>Standard</b>	<b>Manufacturers/Suppliers</b>
<i>Reverse Flow Viscometer</i>				
Zeittuchs Cross-Arm	0.6 – 100 000 cSt	Lubricants, Fuels, Paints.	ASTM D 445, D446; ISO 3104, 3105.	
Cannon-Fenske Opaque	0.4 – 20 000 cSt	Lubricants, Fuels, Paints.	ASTM D 2170 and BS 188.	
BS/IP/RF-U Tube	0.6 – 300 000 cSt	Opaque Newtonian liquids.	ASTM D 445, D446; ISO 3104, 3105.	
<i>Small Volume Viscometer</i>				
Cannon Manning Semi Micro	0.5 – 100 000 cSt	Transparent Newtonian liquids, Blood and plasma, Petroleum products, Lubricants.	ASTM D 445, D446; ISO 3104, 3105.	
Cannon-Ubbelohde Semi Micro	0.3 – 100 000 cSt	Blood and plasma.		
Cannon Manning Semi Micro Extra Low Charge	0.3 – 100 000 cSt	Blood and plasma.		
<i>Dilution Viscometer</i>				
Cannon-Ubbelohde Dilution	0.5 – 100 000 cSt	Polymer solution.	ASTM D 445, D446; ISO 3104, 3105.	
Cannon-Ubbelohde Four Bulb Shear Dilution	0.5 – 100 000 cSt	Polymer.		



<b>Instrument</b>	<b>Viscosity Range</b>	<b>Applications</b>	<b>Standard</b>	<b>Manufacturers/Suppliers</b>
<i>Vacuum Viscometer</i>				
Asphalt Institute Vacuum	42 – 5 800 000 P	Asphalts.	ASTM D2171.	
Cannon Manning Vacuum	0.036 – 80 000 P	Asphalts.		
Modified Koppers Vacuum	42 – 200 000 P	Asphalts.		
<b>Efflux Type Viscometers</b>				
Saybolt	10 <sup>-1</sup> – 10 P		ASTM D88, D244.	Koehler Instrument Co., Inc., Bohemia, NY, USA.
Redwood	10 <sup>-1</sup> – 10 P			
Engler	10 <sup>-1</sup> – 10 P			
Ford Cup	10 – 1 400 cSt		ASTM D 333, D 365, and D 1200.	Cole-Parmer Instrument Co., Vernon Hills, IL, USA. BYK-Gardner GmbH Lausitzer Strasse 8 D-82538, Geretsried Germany. Elcometer Instruments Ltd., Manchester, UK. Cannon Instrument Co. State College, PA, USA Paul N. Gardner Company, Inc., Pompano Beach, FL, USA.

<i>Instrument</i>	<i>Viscosity Range</i>	<i>Applications</i>	<i>Standard</i>	<i>Manufacturers/Suppliers</i>
Zahn Cup	20 – 1 200 cps	Thin oil, Mixed paints, Lacquers.		Weschler Instruments Cleveland, OH, USA BYK-Gardner GmbH Lausitzer Strasse 8 D-82538 Geretsried Germany Cannon Instrument Co. State College, PA, USA Paul N. Gardner Company, Inc. Pompano Beach, FL, USA.
Shell Cup	1 – 7 000 cP			Norcross Corporation Newton, MA, USA. Cannon Instrument Co., State College, PA, USA.
Marsh Funnel				Fann Instrument Company, Houston, TX, USA.
EZ (Equivalent Zahn) Viscosity Cup	10 – 1 401 cSt		ASTM D 816, D 1084, and D 4212.	Cole-Parmer Instrument Co., Vernon Hills, IL, USA. BYK-Gardner GmbH Lausitzer Strasse 8 D-82538 Geretsried Germany
Gardco/Din Cup Iso Cup	38 – 545 cSt 4.6 – 2 611 cSt		DIN 53211 ISO 2431; ASTM D5125.	
Parlin Cup Fisher Cup	7 – 15 000 cSt 11 – 1 125 cSt		ASTM D 1084A. ANSI/NCSL Z540-1.	

<i>Instrument</i>	<i>Viscosity Range</i>	<i>Applications</i>	<i>Standard</i>	<i>Manufacturers/Suppliers</i>
<b>Rotational Viscometers</b>				
Stabinger Viscometer SVM 3000	0.2-20 000 (mPa .s) < 1 up to 10 000 mm <sup>2</sup> /s	Diesel fuel, Engine oil, Lubricants, Asphalt and bitumen, Wax; Paraffin, Vaseline	ASTM D 7042.	Anton Paar GmbH, Graz, Austria.
Cannon Rotary Viscometer, Model 2020	1 – 13 000 000 cP			Canon Instrument Co. State College, PA, USA
Brookfield Viscometer	15 – 104 000 000 cP	Oils, Solvents, Paints, and coatings, Sauces, Adhesives, Creams, and varnishes, Epoxies, Gels, and Tars.		
<b>Cone and Plate</b>				
HAAKE ViscoTester 550	1– 1 000 000 (mPa s)	Highly viscous liquids and pastes.	ISO 3219.	Thermo Electron Corporation, Waltham, MA, USA. BYK-Gardner GmbH, Lausitzer Strasse 8 D-82538 Geretsried, Germany
Brookfield Viscometer				Ravenfield Designs Limited, Heywood Lanes, UK
Ferranti Portable Viscometer			ASTM D 3245.	Elcometer Instruments, Ltd., Manchester, UK
Elcometer Digital Viscometer	150 – 4 000 cP		ASTM D 562, D 856, D 1131; FTMS 141 M 4281.	
Toki Sangyo	0.6 – 512 000 cP			Toki Sangyo Co. Ltd., Tokyo, Japan
Brookfield CAP 1000+ Viscometer	Shear Rate: 10 s <sup>-1</sup> to 13 300 s <sup>-1</sup>		ASTM D4287; ISO 2884; BS 3900.	5. Paul N. Gardner Company, Inc., Pompano Beach, FL, USA.

<i>Instrument</i>	<i>Viscosity Range</i>	<i>Applications</i>	<i>Standard</i>	<i>Manufacturers/Suppliers</i>
<b>Coaxial Cylinder</b> Visco 88 Viscometer	5 – 10 <sup>7</sup> mPa·s	Paints and coatings, Inks, Surfactants and polymer solutions, Foods, Pharmaceuticals and cosmetics, Biochemical and medical, Plastics and polymers, Adhesives and sealants, Petrochemicals, Drilling Fluids	DIN 53019.	Malvern Instruments Ltd., Worcestershire, UK.
Direct Reading Viscometer	Shear Rate: 0.17 to 1022 s <sup>-1</sup>	Drilling fluids, Cement slurries, Fracturing fluids, and Production fluids.	API Spec 10, API RP- 13B and API RP-39.	Chandler Engineering Co., Tulsa, OK, USA
Schott Viscoeasy Rotational Viscometers	2 – 1 060 000 000 cP		ASTM D 115-96, D 789- 94, D 1076-88, D 1084- 88, D 1470-90, D 1439- 94, D 1824-95, D 2364- 89, D 2393-86, D 2556- 93a, D 2669-87, D 2983- 87, D 2994-77, D 3236- 88, D 3716-83, D 4016- 93, ISO 2555, 1652.	Schott Instruments GmbH, Mainz, Germany.

<b>Instrument</b>	<b>Viscosity Range</b>	<b>Applications</b>	<b>Standard</b>	<b>Manufacturers/Suppliers</b>
HAAKE VT550	1–1 000 000 (mPa s)	Newtonian liquids.	DIN 53019; ISO 3219.	Thermo Electron Corporation, Waltham, MA, USA.
Visco Star Plus	2–13 000 000 cP		ISO 2555 and ISO 1652.	Geneq Inc. Montreal, Canada
Viscometers RC01 / RC02	3–13 000 000 mPa s		ISO 2555.	RheoTec Messtechnik GmbH, Ottendorf-Okrilla, Germany
Visco Elite	3.2–1 060 000 P		ISO 2555 and ISO 1652.	GR Scientific Ltd, Bedfordshire, UK
<b>Falling Ball Viscometer</b>				
HAAKE Falling Ball Viscometer C	0.6–75 000 mPa s	Transparent Newtonian liquids and gases.	DIN 53015; ISO 12058.	Thermo Electron Corporation, Waltham, MA, USA.
Gilmont Falling Ball	0.2–2 000 cP		ASTM D 1343-93.	Cole-Parmer Instrument Co., Vernon Hills, IL, USA.
Falling Ball Viscometer KF 10	0.5–7 10 <sup>4</sup> mPa s	Newtonian Fluid, Fuels, Paper emulsions, Polymer solutions, Paints, Varnishes, Detergents, Food industry.	DIN 53015.	RheoTec Messtechnik GmbH, Ottendorf-Okrilla, Germany.
Minivis Micro Viscometer	0.2 to 1 500 mPa s (cSt)	Used oil, Heavy-oil, Black ink and blood.	ISO 12058; DIN 53015.	Grabner Instruments GmbH, Vienna, Austria.
<b>Rolling Ball Viscometers</b>				
Rolling Ball Viscometer		Oil reservoir fluid.		Chandler Engineering Co., Tulsa, OK, USA

<i>Instrument</i>	<i>Viscosity Range</i>	<i>Applications</i>	<i>Standard</i>	<i>Manufacturers/Suppliers</i>
<b>Falling Needle Viscometers</b>				
FNV-200	0.5 to $2.4 \times 10^6$ cP	Adhesives, Automobile fluids, Fuels, Paints, Petroleum, Polymers, Solutions, Surfactants, Suspensions, Varnish.	ASTM 5478-98.	Stony Brook Scientific, Ltd., Norristown, PA, USA
CNV-100	0.1 to $10^6$ cP			
PDV-100	5 to $10^5$ cP			APT Instruments, Farmersville, PA, USA
<b>Falling Cylinder Viscometers</b>				
Toki Sangyo Viscometer	1 – 120 000 000 cP			Toki Sangyo Co. Ltd., Tokyo, Japan.
<b>Ultrasonic Viscometers</b>				
XL7 Viscometer	0 to 1 000 000 000 cP	Polymer melt.		Hydramotion Ltd., York, England
SV-10 Vibro Viscometer	0.3 to 10 000 mPa.s			Malvern Instruments Ltd., Worcestershire, UK.
Dynatrol Viscosity System	0.3 to 10 000 cps	Newtonian, Non-Newtonian.		A&D Weighing, Milpitas, CA, USA.
Tuning Fork Vibration Viscometer	1 to 10 000 cps	Newtonian, Non-Newtonian.		Automation Products, Inc., Houston, TX, USA.
	0.3 – 100 000 cP	Ink, Paint, Food, Pharmaceutical, Chemical processing, Rubber, Oil, Paper.		Cole-Parmer Instrument Co., Vernon Hills, IL, USA.

<i>Instrument</i>	<i>Viscosity Range</i>	<i>Applications</i>	<i>Standard</i>	<i>Manufacturers/Suppliers</i>
<b>High Temperature High Shear Viscometers</b>				
Ravenfield HTHS Viscometers	Shear Rate: 100 000 to 1 500 000 s <sup>-1</sup>		ASTM D 4741; CEC L 36-A-90 and IP 370.	Ravenfield Designs Limited, Heywood, Lanes., UK
Cannon HTHS	Shear Rate: 10 <sup>6</sup> s <sup>-1</sup>	Engine oil at 150°C.	ASTM D5481; SAE J300.	
<b>Cold Cranking Simulator</b>				
CS-2 Cranking Simulator			ASTM D 5293.	Ravenfield Designs Limited, Heywood, Lanes., UK
Cannon, CCS-2050	1500 – 27 000 cP	Oil (-35°C to -5°C).	ASTM D5293; SAE J300.	Cannon Instrument Co., State College, PA, USA
<b>On-Line Viscometers</b>				
SPC/L372J Flow - Through Viscometer	0.2 – 20 000 cP			Cambridge Applied Systems, Inc., Medford, MA, USA
High Temperature, High Pressure	0-300 cP at 300 rpm			Fann Instrument Co., Houston, TX, USA.
<b>Bubble Viscometer</b>				
Cole-Parmer Bubble Viscometer	0.005 – 1 066 St		AOC method Ka 6-63, ASTM D 1131, D 1545, D 1725, and FTMS 141-a method 4271.	Cole-Parmer Instrument Co., Vernon Hills, IL, USA.
PRA Bubble Viscometer	0.7 – 225 St		ISO 3104 and 3105.	PRA Coatings Technology Center, Middlesex, UK.

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### Listing of Standards

#### American Society for Testing and Materials International, West Conshohocken, PA, USA

- ASTM D88, Standard Test Method for Saybolt Viscosity-AASHTO No.: T72, 09/15/1994 (R 2005).
- ASTM D 115-96, Standard Test Methods for Testing Solvent Containing Varnishes Used for Electrical Insulation, 09/10/2002.

- ASTM D244, Standard Test Methods and Practices for Emulsified Asphalts-AASHTO No.: T59, 12/01/2004.
- ASTM D 333, Standard Guide for Clear and Pigmented Lacquers, 05/10/2001.
- ASTM D 365, Standard Guide for Soluble Nitrocellulose Base Solutions, 05/10/2001 (R 2005).
- ASTM D445, Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)-IP Designation: 71/1/97; British Standard 2000: Part 71:1990, Doc. Date, 05/01/2004 (E 2004) (E 2005).
- ASTM D446, Standard Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers-IP Designation: 71/2/95, Doc. Date, 04/21/2004.
- ASTM D 562, Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer, 02/10/2001 (R 2005).
- ASTM D 789-94, Standard Test Method for Determination of Relative Viscosity of Polyamide (PA), 03/15/2006.
- ASTM D 816, Standard Test Methods for Rubber Cements, 05/01/2006.
- ASTM D 1076-88, Standard Specification for Rubber - Concentrated, Ammonia Preserved, Creamed, and Centrifuged Natural Latex, 06/01/2006.
- ASTM D 1084, Standard Test Methods for Viscosity of Adhesives, 03/10/1997 (R 2005).
- ASTM D 1200, Standard Test Method for Viscosity by Ford Viscosity Cup, 08/15/1994 (R 2005).
- ASTM D 1343-93., Standard Test Method for Viscosity of Cellulose Derivatives by Ball-Drop Method, 11/10/1995 (R 2006).
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- ASTM D 1545, Standard Test Method for Viscosity of Transparent Liquids by Bubble Time Method, 10/10/1998.
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- ASTM D 1824-95, Standard Test Method for Apparent Viscosity of Plastisols and Organosols at Low Shear Rates, 03/15/1995 (R 2002).
- ASTM D 2170, Standard Test Method for Kinematic Viscosity of Asphalts (Bitumens)-IP Designation: 319/84 (89), Doc. Date, 12/10/2001.
- ASTM D2171, Standard Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer-IP Designation: 222/84 (89), 08/10/2001.
- ASTM D 2364-89, Standard Test Methods for Hydroxyethylcellulose, 06/10/2001.
- ASTM D 2556-93a, Standard Test Method for Apparent Viscosity of Adhesives Having Shear-Rate-Dependent Flow Properties, 12/15/1993 (R 2005).
- ASTM D 2669-87, Standard Test Method for Apparent Viscosity of Petroleum Waxes Compounded with Additives (Hot Melts)-No Replacement, 08/30/1987 (R 1999).
- ASTM D 2983-87, Standard Test Method for Low-Temperature Viscosity of Lubricants Measured by Brookfield Viscometer, 05/01/2004.
- ASTM D 3236-88, Standard Test Method for Apparent Viscosity of Hot Melt Adhesives and Coating Materials, 10/31/1988 (R 2004).
- ASTM D 3245, Standard Test Method for Pumpability of Industrial Fuel Oils, 03/10/2003.



- ASTM D 3716-83, Standard Test Methods for Use of Emulsion Polymers in Floor Polishes, 09/10/1999 (R 2006).
- ASTM D 4016-93, Standard Test Method for Viscosity of Chemical Grouts by Brookfield Viscometer (Laboratory Method), 11/10/2002.
- ASTM D 4212, Standard Test Method for Viscosity by Dip-Type Viscosity Cups, 05/10/1999 (R 2005).
- ASTM D4287, Standard Test Method for High-Shear Viscosity Using a Cone/Plate Viscometer, 11/10/2000 (R 2005).
- ASTM D 4741; Standard Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer, 05/01/2006.
- ASTM D5125, Standard Test Method for Viscosity of Paints and Related Materials by ISO Flow Cups, 03/10/1997 (R 2005).
- ASTM D5293; Standard Test Method for Apparent Viscosity of Engine Oils Between -5 and -35 Degrees C Using the Cold-Cranking Simulator, 05/01/2004.
- ASTM 5478-98. Standard Test Methods for Viscosity of Materials by a Falling Needle Viscometer, 06/10/1998 (R 2003).
- ASTM D5481; Standard Test Method for Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multicell Capillary Viscometer, 05/01/2004.
- ASTM D 7042, Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity), 07/01/2004.

**International Organization for Standardization (ISO is the short name), Geneva, Switzerland.**

- ISO 1652. Rubber latex -- Determination of apparent viscosity by the Brookfield test method, 2004.
- ISO 2431, Paints and varnishes -- Determination of flow time by use of flow cups, 1993.
- ISO 2555, Plastics -- Resins in the liquid state or as emulsions or dispersions -- Determination of apparent viscosity by the Brookfield Test method, 1989.
- ISO 2884, Paints and varnishes -- Determination of viscosity using rotary viscometers -- Part 1: Cone-and-plate viscometer operated at a high rate of shear, 1999; Paints and varnishes -- Determination of viscosity using rotary viscometers -- Part 2: Disc or ball viscometer operated at a specified speed, 2003.
- ISO 3104, Petroleum products -- Transparent and opaque liquids -- Determination of kinematic viscosity and calculation of dynamic viscosity, 1994.
- ISO 3105, Glass capillary kinematic viscometers -- Specifications and operating instructions, 1994.
- ISO 3219, Plastics -- Polymers/resins in the liquid state or as emulsions or dispersions -- Determination of viscosity using a rotational viscometer with defined shear rate, 1993.
- ISO 3219, Plastics -- Polymers/resins in the liquid state or as emulsions or dispersions -- Determination of viscosity using a rotational viscometer with defined shear rate, 1993.
- ISO 12058, Plastics -- Determination of viscosity using a falling-ball viscometer -- Part 1: Inclined-tube method, 1997.

**Deutsches Institut für Normung (DIN), Germany.**

DIN 53015, Viscometry - Measurement of viscosity using the Hoeppler falling-ball viscometer (FOREIGN STANDARD).

DIN 53019-1, Viscometry; Determination of viscosities and flow curves using standard design rotary viscometers with a standard geometry measuring system.

DIN 53019-2, Viscosimetry - Determination of viscosity and flow curves with rotational viscosimeters - Part 2: Viscosimeter calibration and determination of the uncertainty of measurement.

**Association des Constructeurs Européens d'Automobiles/ European Automobile Manufacturers Association.**

CEC L 36-A-90, Viscosity at High-Temperature and High-Shear Rate Test.

**Institute of Petroleum (IP Test Methods)**

IP 71, Kinematic Viscosity

IP 370 is technically identical to ASTM D 4741 and CEC L-36 A-90.

**The British Standards Institution**

BS 188, Methods for determination of the viscosity of liquids, 31/12/76.

BS 3900, Methods of test for paints. General introduction, 28/02/89.

**American National Standard Institute**

ANSI/NCSL Z540-1, ANSI/NCSL Z540-1-1994: Calibration Laboratories and Measuring and Test Equipment - General Requirements.

**American Petroleum Institute**

API-RP-13B, Recommended practice for field testing oil-based drilling fluids.

API-RP-39, Measuring the viscous properties of a cross-linked water-based fracturing fluid.

**The Society of Automotive Engineers**

SAE J300, Engine Oil Viscosity Classification, May 2004.

**Federal Test Method Standard (FTMS)**

FTMS 141-a method 4271, Viscosity

FTMS 141 M 4281, Determining consistency (viscosity).