

01/2008:20208

The *specific optical rotation*  $[\alpha]_{\text{D}}^{20}$  of a liquid is the angle of rotation  $\alpha$ , expressed in degrees ( $^{\circ}$ ), of the plane of polarisation at the wavelength of the D-line of sodium ( $\lambda = 589.3 \text{ nm}$ ) measured at  $20^{\circ}\text{C}$  in the liquid substance to be examined, calculated with reference to a layer of 1 dm and divided by the density expressed in grams per cubic centimetre.

The *specific optical rotation*  $[\alpha]_{\text{D}}^{20}$  of a substance in solution is the angle of rotation  $\alpha$ , expressed in degrees ( $^{\circ}$ ), of the plane of polarisation at the wavelength of the D-line of sodium ( $\lambda = 589.3 \text{ nm}$ ) measured at  $20^{\circ}\text{C}$  in a solution of the substance to be examined and calculated with reference to a layer of 1 dm containing 1 g/mL of the substance. The specific optical rotation of a substance in solution is always expressed with reference to a given solvent and concentration.

In the conventional system adopted by the Pharmacopoeia the specific optical rotation is expressed by its value without units; the actual units, degree millilitres per decimetre gram [ $^{\circ}\cdot\text{mL}\cdot\text{dm}^{-1}\cdot\text{g}^{-1}$ ] are understood.

The conversion factor from the International System to the Pharmacopoeia system is the following:

$$[\alpha_m]_{\lambda}^t = [\alpha]_{\lambda}^t \times 0.1745$$

In certain cases specified in the monograph the angle of rotation may be measured at temperatures other than  $20^{\circ}\text{C}$  and at other wavelengths.

The polarimeter must be capable of giving readings to the nearest  $0.01^{\circ}$ . The scale is usually checked by means of certified quartz plates. The linearity of the scale may be checked by means of sucrose solutions.

**Method.** Determine the zero of the polarimeter and the angle of rotation of polarised light at the wavelength of the D-line of sodium ( $\lambda = 589.3 \text{ nm}$ ) at  $20 \pm 0.5^{\circ}\text{C}$ , unless otherwise prescribed. Measurements may be carried out at other temperatures only where the monograph indicates the temperature correction to be made to the measured optical rotation. Determine the zero of the apparatus with the tube closed; for liquids the zero is determined with the tube empty and for solids filled with the prescribed solvent.

Calculate the specific optical rotation using the following formulae.

For neat liquids:

$$[\alpha]_{\text{D}}^{20} = \frac{\alpha}{l \cdot \rho_{20}}$$

For substances in solution:

$$[\alpha]_{\text{D}}^{20} = \frac{1000\alpha}{l \cdot c}$$

where  $c$  is the concentration of the solution in grams per litre.

Calculate the content  $c$  in grams per litre or the content  $c'$  in per cent  $m/m$  of a dissolved substance using the following formulae:

$$c = \frac{1000\alpha}{l \cdot [\alpha]_{\text{D}}^{20}} \quad c' = \frac{100\alpha}{l \cdot [\alpha]_{\text{D}}^{20} \cdot \rho_{20}}$$

- $\alpha$  = angle of rotation in degrees read at  $20 \pm 0.5^{\circ}\text{C}$ ;  
 $l$  = length in decimetres of the polarimeter tube;  
 $\rho_{20}$  = density at  $20^{\circ}\text{C}$  in grams per cubic centimetre. For the purposes of the Pharmacopoeia, density is replaced by relative density (2.2.5).



## 2.2.8. VISCOSITY

The *dynamic viscosity* or *viscosity coefficient*  $\eta$  is the tangential force per unit surface, known as *shearing stress*  $\tau$  and expressed in pascals, necessary to move, parallel to the sliding plane, a layer of liquid of 1 square metre at a rate ( $v$ ) of 1 metre per second relative to a parallel layer at a distance ( $x$ ) of 1 metre.

The ratio  $dv/dx$  is a speed gradient giving the *rate of shear*  $D$  expressed in reciprocal seconds ( $\text{s}^{-1}$ ), so that  $\eta = \tau/D$ .

The unit of dynamic viscosity is the pascal second (Pa·s). The most commonly used submultiple is the millipascal second (mPa·s).

The *kinematic viscosity*  $\nu$ , expressed in square metres per second, is obtained by dividing the dynamic viscosity  $\eta$  by the density  $\rho$  expressed in kilograms per cubic metre, of the liquid measured at the same temperature, i.e.  $\nu = \eta/\rho$ . The kinematic viscosity is usually expressed in square millimetres per second.

A capillary viscometer may be used for determining the viscosity of Newtonian liquids and a rotating viscometer for determining the viscosity of Newtonian and non-Newtonian liquids. Other viscometers may be used provided that the accuracy and precision is not less than that obtained with the viscometers described below.

01/2008:20209



## 2.2.9. CAPILLARY VISCOMETER METHOD

The determination of viscosity using a suitable capillary viscometer is carried out at a temperature of  $20 \pm 0.1^{\circ}\text{C}$ , unless otherwise prescribed. The time required for the level of the liquid to drop from one mark to the other is measured with a stop-watch to the nearest one-fifth of a second. The result is valid only if two consecutive readings do not differ by more than 1 per cent. The average of not fewer than three readings gives the flow time of the liquid to be examined.

Calculate the dynamic viscosity  $\eta$  (2.2.8) in millipascal seconds using the formula:

$$\eta = k\rho t$$

- $k$  = constant of the viscometer, expressed in square millimetres per second squared,  
 $\rho$  = density of the liquid to be examined expressed in milligrams per cubic millimetre, obtained by multiplying its relative density ( $d_{20}^{20}$ ) by 0.9982,  
 $t$  = flow time, in seconds, of the liquid to be examined.

The constant  $k$  is determined using a suitable viscometer calibration liquid.

To calculate the kinematic viscosity ( $\text{mm}^2\cdot\text{s}^{-1}$ ), use the following formula:  $\nu = kt$ .

The determination may be carried out with an apparatus (Figure 2.2.9.-1) having the specifications described in Table 2.2.9.-1<sup>(1)</sup>:

The minimum flow time should be 350 s for size no. 1 and 200 s for all other sizes.

(1) The European Pharmacopoeia describes the system proposed by the International Organisation for Standardisation (ISO).

Table 2.2.9.-1

Size number	Nominal constant of viscometer	Kinematic viscosity range	Internal diameter of tube <i>R</i>	Volume of bulb <i>C</i>	Internal diameter of tube <i>N</i>
	mm <sup>2</sup> ·s <sup>-2</sup>	mm <sup>2</sup> ·s <sup>-1</sup>	mm (± 2 %)	mL (± 5 %)	mm
1	0.01	3.5 to 10	0.64	5.6	2.8 to 3.2
1A	0.03	6 to 30	0.84	5.6	2.8 to 3.2
2	0.1	20 to 100	1.15	5.6	2.8 to 3.2
2A	0.3	60 to 300	1.51	5.6	2.8 to 3.2
3	1.0	200 to 1000	2.06	5.6	3.7 to 4.3
3A	3.0	600 to 3000	2.74	5.6	4.6 to 5.4
4	10	2000 to 10 000	3.70	5.6	4.6 to 5.4
4A	30	6000 to 30 000	4.07	5.6	5.6 to 6.4
5	100	20 000 to 100 000	6.76	5.6	6.8 to 7.5

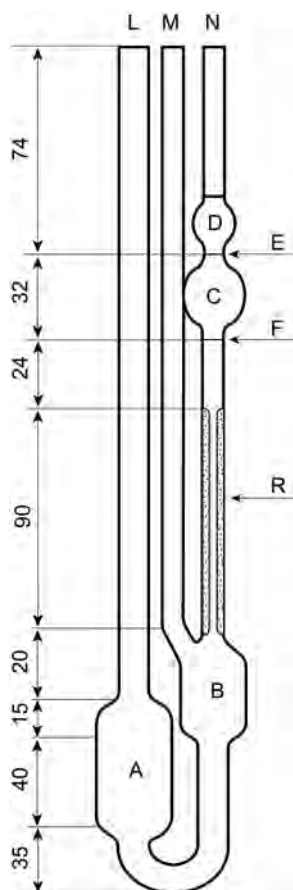


Figure 2.2.9.- 1. – Suspended level viscometer  
Dimensions in millimetres

**Method.** Fill the viscometer through tube (L) with a sufficient quantity of the liquid to be examined, previously brought to 20 °C unless otherwise prescribed, to fill bulb (A) but ensuring that the level of liquid in bulb (B) is below the exit to ventilation tube (M). Immerse the viscometer in the bath of water at 20 ± 0.1 °C, unless otherwise prescribed, maintain it in the upright position and allow to stand for not less than 30 min to allow the temperature to reach equilibrium. Close tube (M) and raise the level of the liquid in tube (N) up to a level about 8 mm above mark (E). Keep the liquid at this level

by closing tube (N) and opening tube (M). Open tube (N) and measure, with a stop-watch to the nearest one-fifth of a second, the time required for the level of the liquid to drop from mark (E) to (F).



01/2008:20210

## 2.2.10. VISCOSITY - ROTATING VISCOMETER METHOD

The principle of the method is to measure the force acting on a rotor (torque) when it rotates at a constant angular velocity (rotational speed) in a liquid. Rotating viscometers are used for measuring the viscosity of Newtonian (shear-independent viscosity) or non-Newtonian liquids (shear dependent viscosity or apparent viscosity). Rotating viscometers can be divided in 2 groups, namely absolute and relative viscometers. In absolute viscometers the flow in the measuring geometry is well defined. The measurements result in absolute viscosity values, which can be compared with any other absolute values. In relative viscometers the flow in the measuring geometry is not defined. The measurements result in relative viscosity values, which cannot be compared with absolute values or other relative values if not determined by the same relative viscometer method.

Different measuring systems are available for given viscosity ranges as well as several rotational speeds.

### APPARATUS

The following types of instruments are most common.

#### CONCENTRIC CYLINDER VISCOMETERS (ABSOLUTE VISCOMETERS)

In the concentric cylinder viscometer (coaxial double cylinder viscometer or simply coaxial cylinder viscometer), the viscosity is determined by placing the liquid in the gap between the inner cylinder and the outer cylinder. Viscosity measurement can be performed by rotating the inner cylinder (Searle type viscometer) or the outer cylinder (Couette type viscometer), as shown in Figures 2.2.10.-1 and 2.2.10.-2, respectively. For laminar flow, the viscosity (or apparent viscosity)  $\eta$  expressed in pascal-seconds is given by the following formula:

$$\eta = \frac{1}{\omega} \left( \frac{M}{4\pi h} \right) \left( \frac{1}{R_i^2} - \frac{1}{R_o^2} \right) = k \frac{M}{\omega}$$

- $M$  = torque in newton-metres acting on the cylinder surface,
- $\omega$  = angular velocity in radians per second,
- $h$  = height of immersion in metres of the inner cylinder in the liquid medium,
- $R_i$  = radius in metres of the inner cylinder,
- $R_o$  = radius in metres of the outer cylinder,
- $k$  = constant of the apparatus, expressed in radians per cubic metre.

For non-Newtonian liquids it is indispensable to specify the shear stress ( $\tau$ ) or the shear rate ( $\dot{\gamma}$ ) at which the viscosity is measured. Under narrow gap conditions (conditions satisfied in absolute viscometers), there is a proportional relationship between  $M$  and  $\tau$  and also between  $\omega$  and  $\dot{\gamma}$ :

$$\tau = AM \quad \dot{\gamma} = B\omega$$