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 [(°)·ml·dm^{-1}·g^{-1}] are understood.

EQUIPMENT

The polarimeter typically consists of:

- a light source, for example a sodium discharge lamp, a light-emitting diode (LED) or another light source capable of providing radiation at the desired wavelength (589 nm unless otherwise prescribed in the monograph); if the light source is polychromatic, a means of isolating the required wavelength is necessary, e.g. an optical filter;
- a polariser and an analyser;
- a sample cell with a path length of 1.00 dm, unless otherwise specified in the monograph;
- a detection system to measure the angle of optical rotation, which must be capable of giving readings to at least the nearest 0.01°, unless otherwise specified in the monograph;
- a temperature control system that indicates the temperature with a readability of 0.1 °C; it may be embedded in the polarimeter (e.g. a Peltier system) or be an external unit (e.g. a cycle-cryostat), and must be able to maintain the temperature of the liquid to within ± 0.5 °C of that prescribed.

EQUIPMENT PERFORMANCE

The accuracy of the scale is checked near the value to be measured or over an appropriate range, usually by means of certified quartz plates. Other certified reference materials may also be suitable (e.g. sucrose solutions).

Optical rotation measurements may be used to quantify the amount of an enantiomer or the ratio of enantiomers present in a sample. For that purpose, the linearity must be checked, for example using sucrose solutions.

PROCEDURE

Determine the zero of the polarimeter and the angle of rotation of the liquid at a wavelength of 589 nm and a temperature of 20 ± 0.5 °C, unless otherwise prescribed. The zero of the polarimeter is determined with the sample cell closed.

For neat liquids, the zero is determined with an empty sample cell.

For solutions, the zero is determined with the sample cell filled with the same solvent as that used for the solution to be examined and measured at the same temperature. The sample preparation procedure is prescribed in the monograph.

Calculate the specific optical rotation at temperature t and wavelength λ using the following formulae.

For neat liquids, the density of the liquid is taken into account:

$$[\alpha]_{\lambda}^{t} = \frac{\alpha}{l \cdot \rho_t}$$

For solutions:

$$[\alpha]_{\lambda}^{t} = \frac{1000\alpha}{l \cdot c}$$

α = angle of rotation measured at temperature t and wavelength λ, in degrees;

l = path length of the polarimeter sample cell, in decimetres;

ρ_t = density determined at the temperature of measurement t, in grams per cubic centimetre; for the purposes of the Pharmacopoeia, density is replaced by relative density (*2.2.5*);

c = concentration of the solution, in grams per litre.

When the limits for optical rotation or specific optical rotation are expressed as the dried substance, the anhydrous substance or the solvent-free substance, the result must be corrected for loss on drying (*2.2.32*), water content (*2.5.12* or *2.5.32*) or content of solvent as appropriate.

04/2018:20208

2.2.8. VISCOSITY

The *dynamic* viscosity or *viscosity coefficient* η is the tangential force per unit surface, known as *shearing stress* τ 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 (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* ν, expressed in square metres per second, is obtained by dividing the dynamic viscosity η by the density ρ 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 are at least as satisfactory as those obtained with the viscometers described in the related chapters.

01/2019:20209

2.2.9. CAPILLARY VISCOMETER METHOD

PRINCIPLE

The determination of viscosity is carried out using a suspended-level (Ubbelohde-type) capillary viscometer of appropriate size at a temperature of 20.0 ± 0.1 °C, unless otherwise prescribed. The time required for the level of the liquid to drop from one mark to the other is measured.

EQUIPMENT

The principal components of an Ubbelohde-type capillary viscometer(1). are shown in Figure 2.2.9.-1.

(1) The European Pharmacopoeia describes the system proposed by the International Organization for Standardization (ISO)

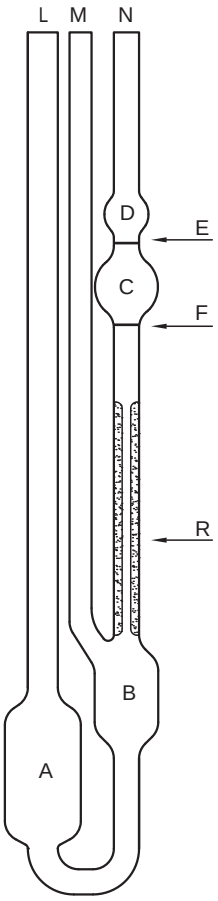

Figure 2.2.9.-1. – *Suspended-level (Ubbelohde-type) capillary viscometer*

PROCEDURE

Select a capillary viscometer of appropriate size to obtain a minimum flow time of 200 s.

Calibration

Capillary viscometers are calibrated at regular intervals as defined in the quality management system and dictated by the frequency of use of the equipment and the application.

Calibrate the instrument at the temperature used for the measurement by using at least 2 certified reference materials matching the viscosity range of the viscometer.

Calculate the viscometer constant (k) in square millimetres per second squared, using the following expression:

$$k = \frac{\eta}{\rho t}$$

η = dynamic viscosity of the certified reference material, in millipascal seconds;

ρ = density of the certified reference material, in milligrams per cubic millimetre;

t = flow time for the certified reference material to drop from the upper mark to the lower mark, in seconds.

Calculate the mean of the values obtained.

Method

Charge the viscometer (Figure 2.2.9.-1) 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* while ensuring that the level of liquid in bulb *B* is below the exit to ventilation tube *M*. Immerse the viscometer in the upright position in a water-bath at 20.0 ± 0.1 °C (unless otherwise prescribed) and allow to stand for not less than 30 min to allow the temperature to reach equilibrium. Close tube *M* and draw 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, using a stopwatch, measure the time required, to at least the nearest 1/5 of a second, for the level of the liquid to drop from mark *E* to mark *F*.

The flow time of the liquid to be examined is the mean of 3 consecutive measurements. The result is valid if the relative standard deviation of the 3 measurements is not more than 2.0 per cent.

Calculation

Calculate the kinematic viscosity (ν) (*2.2.8*), in square millimetres per second, using the following expression:

$$\nu = kt$$

k = viscometer constant, in square millimetres per second squared;

t = flow time of the liquid to be examined, in seconds.

Calculate the dynamic viscosity (η) (*2.2.8*), in millipascal seconds, using the following expression:

$$\eta = k\rho t$$

ρ = density of the liquid to be examined at the temperature used for the viscosity measurement, in milligrams per cubic millimetre.

The density may be obtained by multiplying the relative density of the liquid to be examined by 0.99820 (measurement at 20 °C) or 0.99704 (measurement at 25 °C).

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) η expressed in pascal-seconds is given by the following formula:

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