# VISCOSITY AND ITS MEASUREMENT

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# SYNOPSIS

INSTRUMENTS of many different designs have been and are used for the measurement of viscosity. Throughout this Monograph emphasis has been placed on the principles of these methods rather than on experimental detail, though some notes on experimental techniques have been included.

Successive chapters are devoted to capillary, rotational and falling body types of viscometer. A special feature is the inclusion in each chapter of a section on non-Newtonian materials. This arises in part from the authors' particular interest in clay and other ceramic materials, but they also believe that in the industrial uses of viscometry some understanding of this type of material is of great practical importance.

An extensive list of references provides suggestions for further reading. In the preparation of this Monograph the authors have drawn on standard works on the subject, but they would especially like to mention their indebtedness to Dr. G. Barr's Monograph on Viscometry.

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# INTRODUCTION

Viscosity is the property of a fluid—liquid or gas—that mainly characterizes its flow behaviour. By 'flow' is understood the continuous deformation process occurring under the action of gravity or of an externally applied force. The concept of viscosity embraces the idea of the internal friction between the molecules of the fluid for, whenever any part of a fluid is caused to move, neighbouring parts tend to be carried along too. This resistance to the development of velocity differences within a fluid is the essential feature of viscosity and it forms the basis of the quantitative assessment of viscosity.

The measurement of viscosity is of considerable importance in both industrial production and fundamental science. Viscosity is the quantity that determines the forces to be overcome when fluids are used in pipelines, bearings, etc., and it controls the flow of liquid in such processes as spraying, injection moulding, extrusion and surface coating. In so far as, under given conditions, it determines whether flow will be streamline or turbulent, viscosity has an important bearing on the mixing and heat transfer characteristics of fluids. In other applications the measurement of viscosity affords a convenient means of checking the constancy of a product. Viscosity measurement has also proved a valuable tool for the physical chemist since the viscosity coefficient is profoundly influenced by the size, shape and arrangement of the molecules. The relationship between the viscosity and the concentration of polymer solutions, for example, has frequently been used to estimate the average molecular weight of the polymer.

In order to give a more exact definition of viscosity, it is desirable first to introduce the terms 'shearing stress' and 'rate of shear'. For simplicity we consider the flow of a liquid contained between two parallel planes, each of area $A$ cm$^2$, and $h$ cm apart, when a force of $P$ dyn is applied to the upper plane causing it to move with velocity $v$ cm sec$^{-1}$, relative to the lower plane, as shown in figure 1.

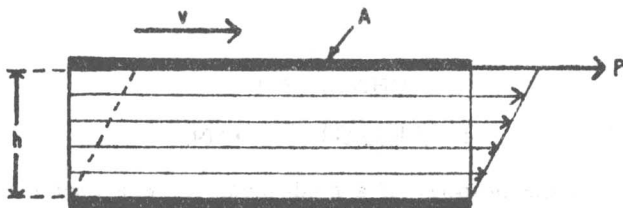The shearing stress $F$ acting throughout the liquid is defined as

FIGURE 1. Simple shear.

the shearing force $P$ divided by the area $A$ over which it acts, i.e. the shearing stress is $P/A$ dyn cm$^{-2}$. The dimensions of shearing stress are therefore $ML^{-1}T^{-2}$.

The mean rate of shear $D$ or velocity gradient equals the velocity difference $v$ divided by the distance between the planes $h$, i.e. $v/h$. Rate of shear, being a velocity divided by a length, has the dimension of $T^{-1}$; its unit is the 'reciprocal second' denoted by sec$^{-1}$. In the simple case depicted in figure 1 the velocity gradient is uniform throughout the sample. More generally it would be defined as the differential coefficient $dv/dx$, where $x$ is the distance measured perpendicular to the direction of shear. It may be noted that 'rate of shear' and 'velocity gradient' are not synonymous when the lines of shear are curved as in a rotational viscometer (see chapter 3).

The flow properties of all materials are defined by the relationship between shearing stress and rate of shear. We shall be mainly concerned with the important class of materials known as 'true' or 'Newtonian' liquids, for which the shearing stress is directly proportional to the rate of shear

$$F = \eta D. \qquad (1)$$

The constant of proportionality $\eta$ (the Greek letter eta) is the 'viscosity coefficient', the 'dynamic viscosity', or simply the 'viscosity' of the liquid. The dimensions of viscosity are $ML^{-1}T^{-1}$ and the c.g.s. unit, the dyn sec cm$^{-2}$, is known as the 'poise' in honour of Poiseuille, one of the great pioneers of viscometry. The viscosity of water at normal ambient temperature is approximately 0·01 poise (P) or 1 centipoise (CP).

The reciprocal of viscosity $1/\eta$ is known as 'fluidity'; it is usually denoted by $\phi$ and the unit in which it is measured is the rhe (from the Greek $\rho\varepsilon\omega$, flow).

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Some viscometers give a direct measure, not of $\eta$, but of the ratio of $\eta$ to the density $\rho$. This ratio $\eta/\rho$ is known as the 'kinematic viscosity' $\nu$, the c.g.s. unit of which is the 'stokes' (s).

Water, simple organic liquids, true solutions, dilute suspensions and emulsions, and gases are in general Newtonian in character. As we have seen, the relationship between shearing stress and rate of shear is one of direct proportionality (A, figure 2). There are
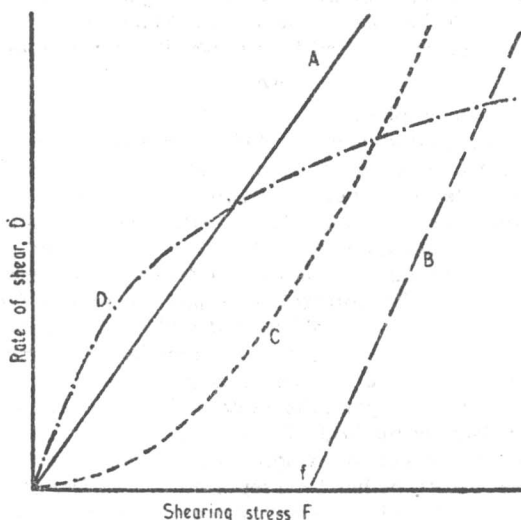


FIGURE 2. Types of consistency curve.

however many materials of great industrial importance, particularly those in the form of more concentrated suspensions and emulsions, that are non-Newtonian. These 'anomalous liquids', as they are sometimes called, are of two degrees of complexity. Firstly there are those for which the relationship between shearing stress and rate of shear can be uniquely defined by a curve or by a straight line not passing through the origin. These include the 'Bingham plastic' material (B, figure 2), a somewhat idealized representation of many real materials, for which the rate of shear is zero if the shearing

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stress is less than or equal to a 'yield stress' $f$ and is otherwise directly proportional to the shearing stress in excess of the yield stress

$$F - f = UD. \tag{2}$$

The constant $U$ is known as the 'plastic viscosity' and its reciprocal, often denoted by $\mu$, the 'mobility'. Other types of behaviour for which there are unique $(F, D)$ relationships include the 'pseudoplastic' (C, figure 2) giving a consistency curve convex to the stress axis, and the 'dilatant' type of material (D, figure 2) represented by a curve convex to the rate of shear axis. Materials of these types may sometimes be represented by a power law expression

$$D = AF^n \tag{3}$$

where $A$ and $n$ are empirical constants, the value of $n$ being positive for pseudoplastic materials and negative for dilatant ones.

The second class of anomalous materials is of a greater degree of complexity in so far as a single $(F, D)$ curve is insufficient to characterize their flow behaviour completely. Such materials, if subjected for example to a constant rate of shear, may exhibit gradually increasing or decreasing shearing stress, i.e. their fluid characteristics are time-dependent. Thixotropic materials are of this type; they gradually thicken when allowed to rest and become progressively thinner when sheared, the process being completely reversible without change of temperature. The exact specification of such materials in terms of $F$ and $D$ is beyond the scope of this book, and indeed no generally satisfactory method of doing this has yet been devised. The importance of this phenomenon must nevertheless not be underestimated, for thixotropy has been reported in a variety of materials from paints, inks and clays to honey, ice cream and chocolate, to name but a few.

A family of materials of ever-growing importance in the field of synthetic polymers is described as 'visco-elastic' because they combine some of the properties of a viscous liquid with some of the properties of an elastic solid. Such materials require specialized techniques to elucidate the relationship between stress, rate of shear and amount of shear. Detailed consideration of visco-elastic materials is outside the scope of this book.

The term 'apparent viscosity', denoted by $\eta'$, has been much used in connection with non-Newtonian materials. It is defined for any single measurement as the ratio of shearing stress to rate of shear $F/D$. It is intended to represent the viscosity of a true liquid that

would behave in the same way in the particular test. However, it is obvious that the apparent viscosity of a non-Newtonian material is not a unique quantity but one that varies in value with the rate of shear at which it is measured, e.g. for a Bingham material we have, from equation (2), $\eta' = F/D = (UD + f)/D = U + f/D$. The use of the term apparent viscosity has for this reason been deplored by some authors, but provided its limitations are fully understood there is little real objection to its continued use. It must be remembered that the apparent viscosity determined at one rate of shear may not be related, even approximately, to the behaviour of the material in conditions involving different rates of shear. For example, in figure 2, the pseudoplastic material C has a higher apparent viscosity than the dilatant material D at low shear rates, whereas the reverse applies at high shear rates. It follows that a single-point measurement on a non-Newtonian material is completely inadequate for characterizing its fluid behaviour.

While the primary emphasis of this book is on the measurement of the viscosity of true liquids, in each chapter a section has been included on the application of the methods under discussion to the characterization of non-Newtonian materials. With the possible exception of the cone-and-plate rotational viscometer, all practical viscometers subject the test material to a non-uniform distribution of shearing stress and rate of shear. Whilst this is of little consequence in the study of true liquids, it necessitates very careful consideration in the measurement of non-Newtonian materials, the problem being that of deriving the relationship between $F$ and $D$ from the observed relationship between the measured quantities such as pressure, flow rate, torque, etc.

The viscosity of a liquid decreases with increase in temperature, often by an appreciable amount, temperature coefficients of the order of from 1 to 10% per degc being quite common. It is therefore of the greatest importance that adequate temperature control should be exercised during all viscosity measurements, and that no viscosity value should ever be quoted without also quoting the temperature of measurement.

It has not been possible in a book of this size to give more than a passing reference to the measurement of the viscosity of gases. In principle the methods used are similar to those described for liquids, though the experimental techniques are of course somewhat different. In particular, the capillary, rotating cylinder and oscillating disk

viscometers have been much used for gas viscosity measurements. Since the viscosities of gases are much lower than those of liquids, use of the capillary method necessitates long capillary tubes of extremely fine bore (about 0·1 mm). The principal theoretical modification required is to allow for the compressibility of the gas, and the consequent expansion that occurs as it passes through the capillary. In addition, if the pressure of the gas is so low that the mean free path of the molecules is commensurate with the dimensions of the capillary, allowance must be made for the non-zero velocity of the gas at the wall of the capillary. According to the kinetic theory, the viscosity of a gas is independent of its pressure and is directly proportional to the square root of its absolute temperature. In practice, viscosity increases somewhat more rapidly with temperature than the theory predicts.

# CAPILLARY VISCOMETERS

## 2.1. Introduction

Capillary viscometers are the most commonly used type for measurements on Newtonian liquids. They are comparatively simple and inexpensive, they require only a small quantity of test liquid, temperature control is easy and a full mathematical treatment is possible. In general, the liquid is made to flow through a capillary tube under a known pressure difference and the rate of flow is measured, usually by noting the time taken for a given volume of liquid to pass a graduation mark. In certain types of instrument, the liquid is forced through the capillary at a predetermined rate and the pressure drop thereby produced across the capillary is measured.

Capillary viscometers include 'absolute instruments' (those permitting direct calculation of viscosity from the rate of flow, pressure and instrument dimensions) and 'relative instruments' (which must first be calibrated with one or more liquids of known viscosity). Alternatively, they may be classified as 'external pressure type' or 'gravity type' viscometers according to whether there is or is not an externally applied pressure causing the liquid to flow through the capillary.

There follows a short description of the principal designs of capillary viscometer, together with a brief note of their main disadvantages and sources of error. The various corrections necessary are discussed in greater detail in § 2.3.

Poiseuille's (1846) absolute viscometer (figure 3) comprised a bulb A attached to a horizontal capillary, the whole being immersed in a thermostat. The upper tube was connected to a compressed air supply fitted with a manometer and the time taken for the level of liquid to fall from B to C was noted. In addition to the need for kinetic energy, Couette and hydrostatic head corrections, the disadvantages of Poiseuille's viscometer were: the discharge of test liquid into the thermostat; uncertainty in the length of the capillary after sealing it to the bulb; the effect of varying rate of drainage

from the bulb on the volume of liquid being timed; blockage of the capillary by solid impurities.

These difficulties have been overcome in absolute viscometers devised by Thorpe and Rodger (1894) and by Bingham (1912, 1922). In Bingham's viscometer (figure 4), the capillary AA' is attached to the dust traps BB' with ground glass joints. This device allows the capillary to have plane parallel ends so that the length and diameter may be determined accurately. To reduce drainage errors the tubing just above each bulb is of similar shape to the base of the main bulb
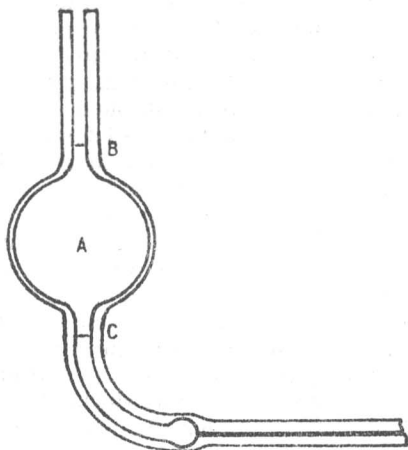


FIGURE 3. Poiseuille's viscometer.

so that drainage conditions are as nearly as possible the same when the meniscus is passing the upper and lower graduation marks. To minimize hydrostatic head corrections to the externally applied pressure, the overall length of the limbs is reduced by shortening the bulbs, and the reservoir bulb V' is made as nearly as possible of the same volume and shape as bulb V, with its centre at the same height above the horizontal capillary. The working volume of liquid is that contained between the graduation 1 and the overflow F. The rate of flow in each direction is timed between the graduations 2 and 3.

Capillary viscometers for relative measurements are usually simpler in design than absolute instruments, and for this reason and because measurement of dimensions is not required they are usually
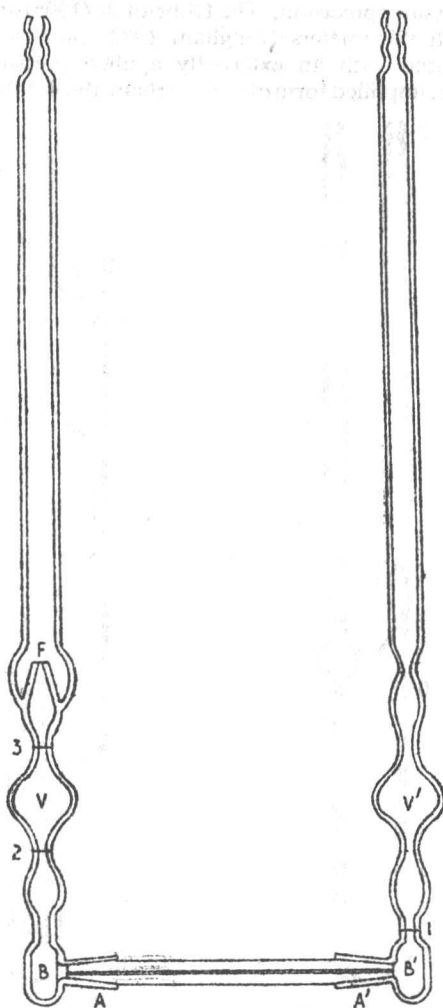
FIGURE 4. Bingham's absolute viscometer.

capable of greater precision. The Ubbelohde (1908) and the Bingham and Jackson viscometers (Bingham 1922, Bingham and Jackson 1917) are used with an externally applied pressure. The latter (figure 5) is a simplified form of the Bingham absolute instrument. The
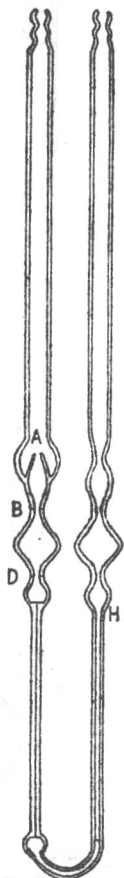


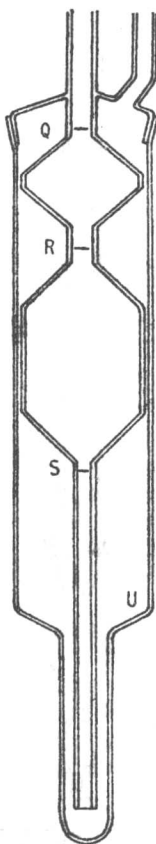FIGURE 5. Bingham and Jackson's viscometer.

FIGURE 6. Barr's pipette viscometer.

same overflow method is used for adjusting the working volume, the liquid being drawn into the left hand limb until it reaches from H to A. Air pressure is applied to the two limbs in turn, and the flow is timed from B to D or from D to B.

Gravity type viscometers depend, as their name suggests, on the flow of liquid through a capillary due to gravity, i.e. the driving pressure is that arising from the head of the liquid being tested. Since this is proportional to the density of the liquid, gravity type instruments measure kinematic viscosity ($v$, i.e. $\eta/\rho$). The ordinary 'dynamic' viscosity is obtained from the kinematic viscosity simply by multiplying by the density. The advantages of gravity type instruments are that the need for a source of constant external pressure is avoided and that wider capillaries less susceptible to blocking can be used. Gravity viscometers include pipette, U-tube, suspended level and efflux type instruments.

The pipette type consists essentially of a pipette with the jet replaced by a capillary. The lower end of the capillary is kept immersed in the liquid in order to avoid surface tension effects associated with the formation of drops. In Barr's (1924) pipette viscometer (figure 6) the 'pipette' is enclosed in an outer vessel of slightly greater diameter. The upper bulb of the pipette holds sufficient liquid to fill the outer vessel up to U. The liquid at about the required temperature is drawn up into the pipette to mark Q, the pipette is placed in position in the outer vessel in the thermostat and allowed to drain. For measurement the liquid is sucked up to just above R and the fall of the meniscus from R to S is timed.

In the Vogel–Ossag (1921) pipette type viscometer the vessel used to immerse the lower end of the capillary has an overflow device which maintains a constant level of liquid and which therefore reduces the variation in hydrostatic head during the measurement.

The most common of the gravity type instruments is the U-tube type, the original form of which is ascribed to Ostwald (1893). The U-tube instrument specified by the British Standards Institution (1937, figure 7) is filled by means of a fine pipette at h until the level of liquid reaches the mark G. (The level in the capillary is somewhat higher because of surface tension.) For measurement, the liquid is drawn into limb a and the flow is timed between marks B and C.

U-tube viscometers are particularly liable to errors arising from deviation of the instrument from the vertical (see p. 21); adjustment

to within 1° of the vertical is essential. This danger is reduced in a modification due to Cannon and Fenske (1936) in which the two bulbs lie on the same vertical axis, the capillary being disposed at an angle to the vertical to make this possible.

A second disadvantage of the U-tube viscometer is the necessity of introducing an exact volume of liquid. When measurements are required at a series of temperatures, re-adjustment of the working volume is required at each temperature. The suspended-level viscometer devised by Ubbelohde (1937) (figure 8) avoids this compli-
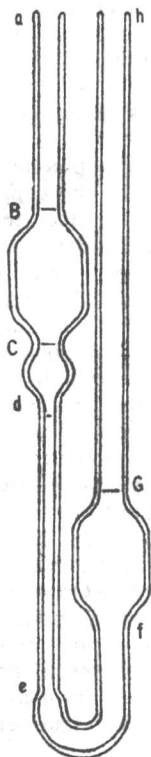


FIGURE 7. B.S.I.
U-tube viscometer.

FIGURE 8.
Ubbelohde
suspended-level
viscometer.

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