

Methods for

Determination of the viscosity of liquids

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- British Resin Manufacturers Association
- Chemical Industries Association Ltd.
- Chemical Society
- Institute of Petroleum
- Ministry of Defence
- National Physical Laboratory (Department of Industry)
- Oil and Colour Chemists Association
- Standardization of Tar Products Tests Committee

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Foreword

BS 188 was first published in 1923 and revised editions were issued in 1929, 1937 and 1957. After nearly two decades it was timely to prepare this revision to take account of developments that have led to changes in emphasis, though not of principle. Three particular changes have been made to bring the standard as a whole up-to-date. First, precedence is given to SI units over CGS units. Second, the definitions given are based on those in BS 5168. Third, the recommended test temperatures are taken from ISO 3205.

The techniques of viscosity measurement owe much to the work of the petroleum industry, and two International Standards have been prepared by ISO/TC 28, Petroleum Products. They are ISO 3104, on the method of measuring viscosity of petroleum products, and ISO 3105, on (design of) glass capillary viscometers for the same purpose. Taking them into account, the content of BS 188:1957 has been rearranged so that the method using glass capillary viscometers and associated material is presented as an entity, section 2. Other industries are also active in the field of viscosity measurement, and besides those relying upon the glass capillary method there is a continuing call for the falling sphere method, which is retained as section 3 with minor revisions such as reference to spheres of common metric sizes and to materials additional to hardened steel.

In section 2 (glass capillary viscometers), the basis of the practical scale remains unchanged, namely the value adopted in 1953 for the dynamic and kinematic viscosities of freshly distilled water at 20.00 °C, together with a step-by-step comparison procedure for calibrating viscometers through the range. In the previous 1957 revision a reduced expression $v = Ct - B/t$ was well recognized, where v is kinematic viscosity; C , the viscometer factor; t , flow time; B , the kinetic energy factor of significance at rapid flows, i.e. short flow times, particularly in fine capillaries. The variability of B between viscometers apparently alike in other respects had been a long-standing problem, and the 1957 edition gave special attention to B . Since then the trend has been to require calibrating laboratories to state the minimum flow time for which the simpler expression $v = Ct$ is of sufficient precision. Certain refinements in design in 1957 also helped to reduce the magnitude of B . The necessary safeguards with respect to flow time having been established, the B factor has been eliminated from the calculations in the present edition. Another change relates to the specification of detailed designs of particular glass capillary viscometers. In the 1957 edition dimensional details were specified for seven designs. As, however, full information about accepted designs is now available in ISO 3105, the information given in this revision is concentrated on the salient features of use and design of representative viscometers from five well-known families.

Particular attention has been given in the details of the method to amplifying ISO 3104 in ways considered likely to be helpful to the user.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 20, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Section 1. General

1 Scope

This British Standard describes the determination of the viscosity of liquids by the method using glass capillary viscometers (section 2) and by the falling sphere method (section 3).

The standard applies only to liquids exhibiting Newtonian flow under the conditions of measurement. A test for Newtonian flow is described in each section.

2 References

The titles of the standards publications referred to in this standard are listed on the inside back cover.

3 Definitions

For the purposes of this British Standard the following definitions apply.

3.1

viscosity

the property of a liquid to resist shear deformation increasingly with increasing rate of deformation

shear is the orderly movement of layers of liquid relative to parallel adjacent layers. In a steady shear the rate of relative movement of layers is known as the **rate of shear**¹⁾ and the tangential force per unit area applied to the layers to sustain the motion is the **shear stress**

thus viscosity is the property that resists orderly laminar flow of liquid through a capillary; in this case the layers involved are concentric cylinders

3.2

dynamic viscosity, η

the shear stress divided by the rate of shear for steady flow of the liquid

3.3

kinematic viscosity, ν

the dynamic viscosity divided by the density of the liquid, both measured at the same temperature

NOTE to 3.1 to 3.3 The word “viscosity” is used also as a contraction for “coefficient of viscosity”, and a distinction is made between (coefficient of) dynamic viscosity and (coefficient of) kinematic viscosity.

3.4

newtonian flow

a liquid is said to exhibit Newtonian flow when the shear stress and the rate of shear are directly proportional; that is, at constant temperature and pressure the viscosity is independent of the rate of shear

4 Units

The SI unit of dynamic viscosity is the pascal second (symbol Pa s). The SI unit of kinematic viscosity is the metre squared per second (m^2/s).

Submultiples of these units are as follows:

millipascal second (mPa s), i.e. 10^{-3} Pa s; this is equal to the centipoise (cP);

millimetre squared per second (mm^2/s),
i.e. 10^{-6} m^2/s ; this is equal to the centistokes (cSt).

Appendix H gives information on the derivation of the SI units for dynamic and kinematic viscosity and on their relationship to the corresponding CGS units.

Section 2. Method using glass capillary viscometers

5 Field of application

This section describes the use of glass capillary viscometers for the measurement of kinematic viscosity of liquids exhibiting Newtonian flow (see note) under the measuring conditions. The method provides for the measurement of kinematic viscosity and for the calculation of dynamic viscosity from a knowledge of the kinematic viscosity and the density determined at the same temperature.

The method is described in detail for typical viscometers; sources of information about various glass capillary viscometers are indicated. The range of kinematic viscosity covered by these viscometers is $0.3 \text{ mm}^2/\text{s}$ to $300\,000 \text{ mm}^2/\text{s}$.

Additional information useful in viscometry is supplied in appendices.

NOTE A simple test for Newtonian flow is to determine the kinematic viscosity at the same temperature in glass capillary viscometers having calibration factors differing by at least three times. If the results agree within the specified reproducibility figure (see 10.2), the liquid can be considered to exhibit Newtonian flow for the purposes of this method.

¹⁾ In capillary flow, “rate of shear” is sometimes called “velocity gradient” and may be thought of as the change in velocity associated with a displacement across the layers. It has the dimensions of velocity divided by length.

6 Principle

The time is measured for a reproducible volume of the liquid to flow through the capillary viscometer under an accurately reproducible head and at a closely controlled temperature. The kinematic viscosity is then calculated from the measured flow time and the calibration factor of the viscometer. If the density is measured at the same temperature as applied to the determination of kinematic viscosity, the product of the two results is the dynamic viscosity at that temperature.

Four precepts for precise viscometry are

- a) a clean instrument,
- b) a clean liquid,
- c) constant temperature during the course of a measurement,
- d) accurate measurements of temperature and flow time.

7 Apparatus

The principal items of apparatus are as follows.

7.1 Viscometers of the glass capillary type, capable of measuring viscosity within the tolerances for repeatability and reproducibility given in clause 10 and having a certificate of calibration.

NOTE Information about viscometers is given in Appendix A.

7.2 Viscometer holders to hold the viscometers firmly in the thermostatic bath in the alignment stated in the certificate of calibration.

7.3 Temperature-measuring device. A thermometric device that, when used in the manner described in its certificate of calibration, will enable the temperature of the bath adjacent to the sample to be measured to 0.01 °C in the range 15 °C to 100 °C, and to 0.03 °C outside that range. If a liquid-in-glass thermometer is used, it shall be of the “total immersion” type with a valid calibration chart (see also Appendix E).

7.4 Thermostatic bath. A transparent liquid or vapour bath of sufficient depth that during the measurement no portion of the sample is less than 20 mm below the surface of the bath medium or less than 20 mm above the bottom of the bath.

The temperature control shall be such that within the range of 15 °C to 100 °C the temperature of the bath during the period of measurement does not vary from the desired temperature by more than 0.01 °C over the length of the viscometer, or between viscometers. For temperatures outside the range 15 °C to 100 °C, the variation shall not exceed 0.03 °C.

If there is freedom of choice, the temperatures 100, 55, 40, 25, 20, 0, – 25 and – 40 °C are recommended (see ISO 3205) as options for determination of viscosity.

7.5 Timing device. Any timing device may be used provided that its readings discriminate to 0.2 s or finer and that its rate is accurate to $\pm 0.07\%$ over 15 min. A discrimination of 0.1 s can be used with advantage by a skilled operator on flow times of the order of 200 s to 300 s.

8 Procedure

The description that follows deals with selecting equipment, preparing the sample, filling the viscometer, timing the flow and repeating observations. However, the different designs of viscometer result in some differences in the procedure for filling, mounting and starting the flow. Accordingly a general description is given followed by examples of handling particular designs. For instruction about designs not described in detail, reference should be made to the calibration certificate and/or the supplier.

8.1 Selection of equipment

8.1.1 Check that there are current calibration reports for the thermometer and time-measuring device to be used.

NOTE For information see Appendix E and Appendix F.

8.1.2 If specified, condition the sample in the manner required. It is usual to filter the sample through a 75 μm aperture sieve, but a sample of low viscosity that is to be evaluated in a viscometer having a fine bore capillary may require filtering through a fritted glass filter.

At all stages protect the sample from contamination.

8.1.3 Estimate the order of viscosity of the sample and select a calibrated, dry, clean²⁾ viscometer of appropriate range for measuring the estimated viscosity.

If the sample is a silicone liquid or a fluorocarbon, which may affect the capillary by leaving a film that is difficult to remove, only viscometers reserved for such liquids should be used.

If the sample is transparent and forms a cleanly outlined meniscus, a direct flow viscometer should be used. Suspended level, direct flow Cannon-Fenske and direct flow U-tube viscometers are typical examples.

If the sample is opaque, a reverse flow viscometer should be used. Reverse flow U-tube and reverse flow Cannon-Fenske viscometers are examples.

²⁾ See Appendix G.

If resources permit, more than one viscometer and thermometer should be used.

8.2 Setting up the apparatus

8.2.1 Maintain the bath at the test temperature as given by the readings of the thermometer when used with the precautions and the corrections supplied in the certificate of calibration.

With a total immersion thermometer, immerse the entire liquid column of the thermometer in the medium so that the top of the mercury column is visible just above or just below (generally within 2 mm) the surface of the medium. The thermometer should be mounted vertically.

If the temperature of the test is below the dewpoint, loosely packed drying tubes may be fitted to the open ends of the viscometer to prevent water condensation; however, it is essential that they do not set up a pressure differential and affect the rate of flow.

8.2.2 Charge the viscometer with the sample in accordance with the recommended method (examples are given in 8.4.1 to 8.4.5). Avoid trapping air bubbles in the viscometer.

If the sample is very viscous, filling at ambient temperature is slow. Provided that the nature of the sample is not adversely affected, charging may be facilitated by warming it gently and evenly to reduce its viscosity.

8.2.3 Mount the viscometer in a holder in the bath, the aim being to repeat the criterion used when the viscometer was calibrated; i.e. the appropriate tube as stated in the certificate of calibration is to be set vertical, usually by sighting its parallelism with a plumb line.

8.2.4 Allow time for the charged viscometer to reach the same temperature as the bath. Usually 30 min suffice; it may take longer with a viscous sample from which air bubbles are slow to disperse. Unnecessary delay should be avoided, as the most consistent results are obtained shortly after reaching temperature equilibrium with a freshly charged viscometer.

Finally, adjust the volume of the charge if necessary.

8.3 Flow time measurement

8.3.1 First measurement. Adjust the level of the sample by suction or pressure to a position 5 mm above the upper timing mark of a direct flow viscometer, or 5 mm below the lower timing mark of a reverse flow viscometer. Suction may be controlled through a clean rubber tube connected to an open end of a viscometer. When using a *direct flow* viscometer, release the control, allow the sample to flow freely and measure to within 0.2 s the time for the bottom of the meniscus to pass from the first to the second timing mark. When using a *reverse flow* viscometer, release the control, allow the sample to flow freely and measure to within 0.2 s the time for the advancing ring of contact of the sample with the glass to pass from the first to the second timing mark.

If this flow time is less than the minimum stated on the certificate of calibration of the viscometer, select a viscometer of smaller factor and recommence the test.

Record the corrected thermometer readings at the beginning of, during and at the end of a timed flow.

8.3.2 Repeat measurements. When using a *direct flow* viscometer, repeat the measurement of flow time at once without emptying or recharging the instrument until two successive flow times agree within 0.2 % of their mean. When using a *reverse flow* viscometer, it is common practice and advantageous to have three timing marks spanning two timing bulbs so that the end of a first timing is the beginning of a second timing. Only in this way may a second flow time be obtained without emptying the viscometer, recharging and duplicating the flow measurement. Agreement of the values obtained from the successive timings within 0.35 % of their mean is required.

8.3.3 Duplicate determinations. It is advisable to flow the sample in two or more viscometers simultaneously but, if more than one are not available, then refilling and redetermination are desirable. Poor repeatability may be attributed to an unsuccessfully filtered sample, a contaminated viscometer or sample, unstable temperature conditions or heterogeneity of sample.

8.4 Detailed procedure for typical glass capillary viscometers

8.4.1 U-tube viscometers for direct flow (type BS/U) (see Figure 1). Charge the dry, clean viscometer through tube L with a slight excess of the sample, using a long pipette to minimize any wetting of the tube above the mark G.

Mount the viscometer in the bath, taking particular care that the capillary tube is set vertical.

When temperature equilibrium with the bath has been attained, adjust the volume of the sample so that the bottom of the meniscus settles at the mark G.

Apply suction or pressure to bring the liquid level to a point about 5 mm above the timing mark E. Release the suction or pressure and record the flow time taken for the bottom of the meniscus to pass from the top edge of mark E to the top edge of mark F. Record the corrected thermometer readings at the beginning of, during and at the end of the timed flow.

Repeat the measurement of flow time immediately, without emptying or recharging, until two successive flow times agree within 0.2 %.

8.4.2 Suspended-level viscometers (type BS/IP/SL) (see Figure 2). Charge the dry, clean viscometer with the sample through tube L, the amount charged being such that ventilating tube M will not be blocked during use. The initial filling may be done away from the bath.

Mount the viscometer in the bath with the capillary tube vertical.

When temperature equilibrium has been attained, close tube M and apply suction to capillary tube N until the liquid reaches a level about 5 mm above mark E. Hold the liquid at this level by closing tube N. Open tube M so that the sample drops away from the lower end of the capillary tube. (With very viscous samples, a little pressure can be applied to tube M to expedite this.)

When the sample is clear of the capillary end and the lower end of tube M, open tube N. The flow time is the time taken for the bottom of the meniscus to pass from the top edge of mark E to the top edge of mark F. Record the corrected thermometer readings at the beginning of, during and at the end of the timed flow.

If the end of tube M becomes blocked by the sample at any time while the flow is being measured, that measurement is unsatisfactory. The opening and closing of the tubes may conveniently be done by applying finger pressure, or by clips on clean flexible tubing attached to the end of the tubes, making sure that no fluff or other contaminant enters the tube.

Repeat the measurement of flow time immediately, without emptying or recharging, until two successive flow times agree within 0.2 %.

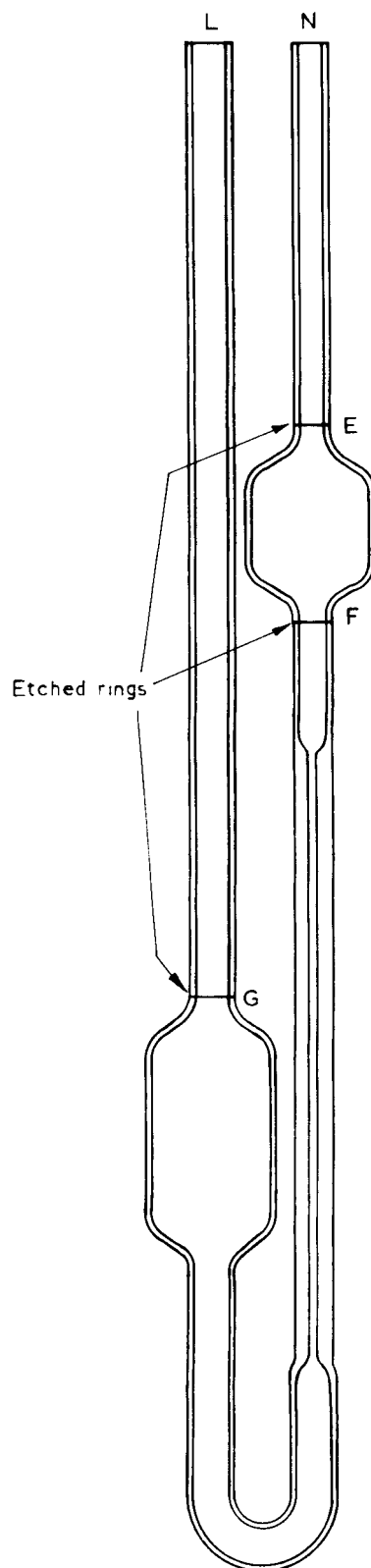


Figure 1 — BS/U viscometer