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Remote Control Viscometer

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A remote control viscometer, designed for use in a high pressure bomb up to 5000 atm, is described. Operation is as follows: a solenoid lifts a tapered piston, which then drops into a cylinder. On the down stroke, needles attached rigidly to the piston dip successively into two mercury wells, the first one starting an electric timer and the second stopping it. Three insulated leads into the bomb are required; the case acts as the common ground. Relative viscosities can be reliably measured within 2%. Results at 30° are given over the pressure range for *n*-propyl ether, cumene, *n*-butyl chloride, and 1,2-dichloroethane.

A NECESSARY supplement to our study of the conductance under pressure of electrolytes in nonaqueous solvents is measurement of the dependence of their viscosities on pressure. A variety of remote control viscometers have already been described[1] but none seemed readily adaptable to the geometry of our high pressure bomb, which pressurizes a cylindrical volume 30.5 cm high and 3.8 cm in diameter.[2] The falling cylinder viscometer[3–5] appeared to be the most promising, provided it could be made to operate by remote control, through the three insulated leads into the bomb. We present here the design of a viscometer in which a tapered piston falls into a cylindrical barrel, starting and stopping an external clock during its fall by means of internal mercury switches attached to the piston. For successive drops, the piston is raised to the starting position by an internal solenoid, which lifts a permanent magnet to which the piston and mercurcy contact needles are attached. A wide range of viscosities can be covered by using pistons of various diameters in a barrel of fixed diameter.

It was found that the drop time is accurately proportional to the viscosity of the liquid in which the assembly is immersed, if a small buoyancy correction is made. Furthermore, the ratio of drop time in a given liquid at pressure p to the drop time in the same liquid at 1 atm is equal to the corresponding relative viscosity, multiplied by the buoyancy factor and another small factor which corrects for the compression of the mercury in the switch tubes. Our values for the relative viscosities of diethyl ether and of isopropanol agree with Bridgman's values[6] to better than 1% up to 5000 atm (our high pressure limit).

VISCOMETER DESIGN

The viscometer will be described in three stages: first, a functional description of the parts and of the assembled instrument; second, a shop description with dimensions and tolerances; and third, a description of assembly and use.

The parts are shown in Fig. 1; assembly is from left to right. The valve block, barrel and mercury tubes are shown in detail in Fig. 2. The tapered piston A1 screws into a flange A2 which also carries a permanent bar magnet A3 and three tungsten wires A4, 5, 6. The piston drops into the barrel B1 (M, Fig. 2), which is filled with the test liquid (because the entire assembly is immersed in the liquid). The time of fall is a measure of the viscosity and is determined by the following sequence of events: the longest tungsten wire A5 makes permanent contact with the mercury in tube B2 (N of Fig. 2); when the assembly A drops, wire A6 first makes contact with mercury in tube B3, starting the timer, and then, before the piston bottoms in the barrel, wire A4 makes contact with mercury in tube B4, stopping the time clock. The barrel and mercury tubes are carried by the valve block, P of Fig. 2, which will be described later. Tubes B3 and B4 are insulated from the valve block by Teflon washers and sleeves; tube B2 fits directly into the block and is the ground lead, because the valve block connects through a series of metal-to-metal contacts with the high pressure bomb. The ball valve P4 in the block closes on the down stroke and opens when the piston is lifted, permitting the barrel to refill with liquid through the channels shown as dotted lines at P2, Fig. 2. The piston assembly is lifted by the solenoid C1, which is wound on a stainless steel bobbin C2. The latter is held in the slotted solenoid housing E by the lock nut D. After the solenoid is inserted in the housing and locked against the flange E1 at the top, the CDE assembly is slipped over the AB assembly, where

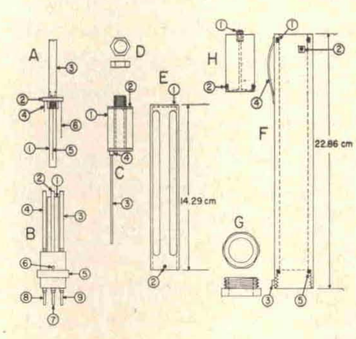

FIG. 1. Piston-cylinder viscometer, exploded view.

[1] J. R. Van Wazer, J. W. Lyons, K. Y. Kim, and R. E. Colewell, *Viscosity and Flow Measurement* (John Wiley & Sons, Inc., New York, 1963). An extensive bibliography on viscometers is also given by R. A. Horne and D. S. Johnson, J. Phys. Chem. **70**, 2183 (1966).
[2] J. F. Skinner and R. M. Fuoss, J. Phys. Chem. **69**, 1437 (1965).
[3] F. Lawaczek, Z. Ver. Deut. Ing. **63**, 677 (1919).
[4] J. J. Bickermann, J. Colloid Sci. **3**, 75 (1948).
[5] T. L. Smith, J. D. Ferry, and F. W. Schremp, J. Appl. Phys. **20**, 144 (1949).
[6] P. W. Bridgman, Proc. Am. Acad. Arts Sci. **61**, 57 (1926).

it rests on the ring B5 of the valve block. It is fixed in position by a small screw E2, which fits into the tapped hole B6. One end of the solenoid winding is soldered to the bobbin and hence is grounded through E; the other end goes to the needle C3 held in an insulated bushing C4. This wire dips into a mercury tube diametrically opposite to tube B2 (and hence invisible in Fig. 1). This tube connects to the stud B7 (Q of Fig. 2) which makes contact with the mercury in tube N through the tie rod S shown in Fig. 2. Tubes B3 and B4 connect with studs B9 and B8 in a similar fashion.

For use, the piston-barrel-solenoid assembly ABCDE is inserted into the outer casing F and locked in by the bottom closure nut G; the O-ring F5, which rests on the ring B5, seals the test liquid from the pressurizing fluid in the bomb. The viscometer is next filled (under vacuum) with the liquid whose viscosity is to be measured. The piston H is inserted into the top of F; the O-rings H2 and F1, and the screw H1 seal the viscometer assembly at the top; pressure is transmitted through the piston H. The entire unit is lowered into the bomb by a cylindrical tool which hooks onto studs F2 (one shown) at the top of the outer casing. The stud F3 fits into a slot in the viscometer support in the bomb, which guides the contacts B7, 8, 9 into three mercury cups on the bottom closure of the bomb. Insulated leads from the mercury cups go through the closure to the external circuits.

The parts shown in Figs. 1 and 2 will now be described in detail, starting with the valve block, B in Fig. 1 and P in Fig. 2. It is made from a piece of stainless steel (alloy 304), 3.016 cm diam and 3.175 cm long, which is turned down to 2.223 cm on each end, to leave a ring 0.635 cm thick 0.953 from the bottom, B5 and P1. Figure 2(b) shows the final cross section of the valve block through the ring. Two 0.3175 cm holes (b1 and b2) at 120° to each other are drilled radially and then plugged (with shrink-fit pins) to a depth of 0.48 cm to form the bottom of the U-tube P2. The ends of this horizontal V-tube connect with the vertical holes at 4 and 8 o'clock shown in Fig. 2(a), and with the hole in the center of the block, P3. The center hole is drilled and tapped to take the cylindrical barrel M into which the piston drops. The center hole has a conical (60°) valve seat, on which a ball bearing P4 (0.476 cm diam) rests. The barrel (5.385 cm long, 1.111 cm o.d., 0.635 cm i.d.) screws into the block. In operation, on the up-stroke when the solenoid C lifts the piston assembly A, liquid is drawn in through the vertical holes and the V-tube, past the ball valve, to fill the barrel. The ball then drops by gravity, closing the bottom exit, and when the piston drops, liquid can only flow up through the annular space between it and the barrel. (This valve is essential; a solenoid will pull a piston from a closed-end barrel, but the long time and large current needed produce far too much heat. With the ball valve, the piston can be lifted without appreciable heating, as shown by exact agreement between drop-times taken at 3 min intervals with those taken at 5 sec intervals.)

The other four holes shown in the top view, Fig. 2(a), are for electrical leads. The one at 12 o'clock is 0.635 cm deep; it receives a stainless steel tube, 5.398 cm long 0.3175 cm o.d., and 0.032 cm wall thickness, which is partly filled with mercury. This is the ground tube; the longest needle A5 dips into it, and thereby connects the flange A2 electrically to the outer casing (F, Fig. 1), which in turn makes electrical contact with the high pressure bomb through the leaf spring contact F4 shown at the top of the outer casing. The holes (0.3175 cm diam) at 2, 6 and 10 o'clock go the whole way through the valve block, as shown in sections a, b, c of Fig. 2. Through them pass the three contact assemblies, shown at the left in Fig. 2. Stainless steel tubes, N (4.763 cm long, 0.318 cm o.d., 0.032 cm wall), are pressed onto threaded studs, R, which receive the 3.81 cm long, 0.159 cm diam rods S, which pass through Teflon tubes in the valve block. The bottom ends of the rods screw into the longer studs Q, which are shown projecting below the valve block (B 7,8,9). Teflon washers, T, at top and bottom, between the corresponding studs and the block, together with the Teflon tubes in the vertical holes, insulate these three mercury tubes from the rest of the viscometer. The lower studs dip into mercury cups, carried on ceramic-insulated nickel wires[2] which pass through the bottom closure of the bomb. The tube at 6 o'clock receives the contact needle C3 from solenoid. The tube at 10 o'clock, (start tube) receives the medium length needle A6 carried by the flange A2; when the needle makes contact with the mercury in this tube, a relay is activated, which starts the timing clock. The tube at 2 o'clock (stop tube) receives the shortest needle A4 (i.e., the last one to make contact when the piston drops); it activates the relay which opens the timing circuit.

The flange A2 is turned from a piece of stainless steel 0.953 cm diam and 1.588 cm long. Cups are turned on top and bottom as shown. Into the top one is pressed the stud on the end of an Alnico bar magnet A3 (5.08 cm long, 0.635 cm diam), ground to a smooth finish. The bottom one is threaded to receive the stud on the top of the piston A1. Two interchangeable pistons, both 5.08 cm long, were used in this work. The one used for isopropanol measurements had a top diameter of 0.6248

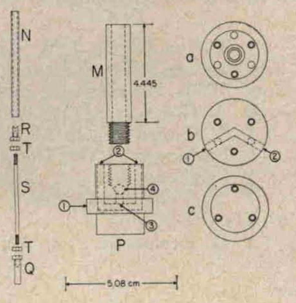

FIG. 2. Mercury tubes, cylinder, and valve block.