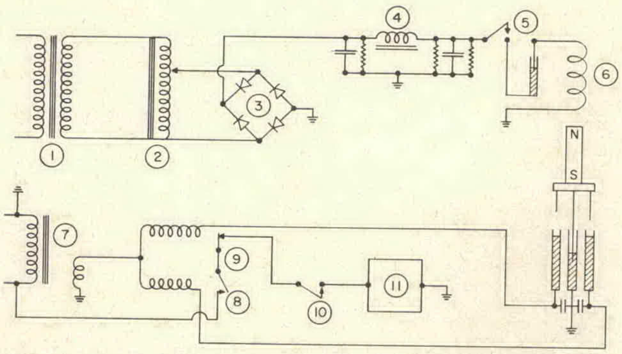

FIG. 3. Wiring diagram for remote control. 1—Isolating transformer; 2—Variac set at about 35 V; 3—full wave rectifier; 4—0.8 H choke; 5—normally open microswitch; 6—solenoid; 7—6.3 V transformer; 8—start-relay contact; 9—stop-relay contact; 10—normally closed microswitch; 11—clock.

cm and a bottom diameter of 0.6172 cm. The one used for ether measurements had a top diameter of 0.6287 cm and a bottom diameter of 0.6210 cm. The bottom edges of both pistons were gently rounded. The taper of 0.1% in the piston is essential; it provides a radial force which keeps the piston centered during its fall. Drop times with cylindrical pistons were hopelessly erratic. Three holes were drilled into the flange A2, on a radius of 0.873 cm to coincide with the centers of the holes at 10, 12 and 2 o'clock in the valve block. Tungsten rods, to project 3.175, 5.080 and 0.953 cm below the flange, ground to 0.064 cm diam were pressed into the holes in the flange. The rods had rounded ends. On assembly, the longest one is centered in the ground tube; the other two will drop into the start and stop tubes, B3 and B4.

The solenoid consists of 800 turns of 26 gauge Teflon insulated magnet wire (resistance 12 Ω), wound on the stainless steel spool C2. The hole through the center is 0.7938 cm diam, and gives ample clearance for the 0.635 cm magnet, so that the viscous drag here is negligible compared to that between piston and barrel. Successive layers of the solenoid windings are separated by Mylar film (0.008 cm). One end of the solenoid winding is hard-soldered to the spool and thereby grounded. The other end is soldered to a 7.62 cm long needle C3 carried by the lower flange of the spool and insulated from the latter by a Teflon bushing. This needle dips into the 6 o'clock tube; an external switch and power source (Fig. 3) complete the solenoid circuit. Power for the solenoid is from a full-wave rectifier (Fig. 3-3), set at about 35 V from the Variac (Fig. 3-2) which is across the output of an isolating transformer. A ballast load of 1000 Ω is across the rectifier output. Ripple is reduced by the 0.8 H choke and the 200 μF condensers and 0.15 MΩ resistors from choke to ground.

The solenoid fits into a stainless steel casing (E, Fig. 1), 14.288 cm long, and 2.54 cm o.d. Four long axial slots are milled into the casing. This casing fits snugly (tolerance 0.0025 cm) around the top of the valve block, resting on the ring B5 of the latter. A small countersunk screw E2 at the bottom of the casing positions the latter reproducibly with respect to the block. The solenoid spool is held to the casing by a nut D at the top; this nut locks the solenoid so that its needle is accurately centered in the 6 o'clock tube when the casing is locked to the block by the screw mentioned above.

The outer casing (F, Fig. 1) is a stainless steel tube 22.86 cm long, 2.540 cm i.d. and 3.493 cm o.d. The shoulder at the bottom is pressed against an O-ring of 2.543 cm i.d. and 0.159 cm thickness by the closure nut; the O-ring F1 is the one which rests on the top of the ring B5 on the valve block, and encircles the solenoid casing. At the top of the outer casing are two spacers, F2, 120° apart, of length equal to the distance between the outer casing and the inside wall of the bomb. At the third 120° position on the top circumference is located a leaf spring F4 which presses the unit into good electrical contact with the bomb. The spacers also serve as lifting pins: a tube with appropriately located L-shaped slots can be fitted around the spacers and is the tool for lowering the viscometer into the bomb and for lifting it out after a run. There is also a pin F3 at the bottom of the outer casing, which fits into a slot in the support for the viscometer in the bomb. This support is a sleeve with a shoulder on which the viscometer rests; the support in turn rests on the bottom closure of the bomb. The outer casing is reproducibly positioned in the bomb by means of the sleeve and the pin at the bottom, and by the spacers F2 and leaf spring F4 at the top. The solenoid casing fits inside the outer casing with only 0.0025 cm clearance, and the valve block fits into the solenoid casing to the same tolerance. In this way, the viscometer is always located in the same position, an essential specification for an instrument which functions through the force of gravity.

The top closure of the viscometer is the piston shown as H, in Fig. 1. Its diameter is 2.535 cm, length 5.08 cm. The seal between the test liquid and the pressurizing fluid is made by two O-rings, H2 and F1, one of which fits into a groove in the piston and the other into a groove in the outer casing, as shown in Fig. 1. After the viscometer is filled, the piston is inserted and sealed with the fillister screw H1.

ASSEMBLY AND USE

Due to the small clearance between piston and barrel, and to the presence of the ball valve, it is essential that all parts of the viscometer be completely dust-free. After rinsing with alcohol and wiping with a lint-free cloth, the barrel and mercury tubes are attached to the valve block and the valve ball is dropped in. Since the fall time depends on the distance traversed by the piston between the times of contact by the start switch and the stop switch, and also on the total length of the piston within the annulus between it and the barrel, it is necessary to have the levels of the mercury in the start and stop tubes at the same height with respect to the top of the barrel in successive runs. It is also convenient to have these levels equal to each other. The stop and start tubes are filled with mercury to heights 0.100±0.012 cm below the top of the barrel. (This distance is sufficient to avoid mercury overflow when the tungsten wires enter.) About 10 drops of mercury are placed in each tube and then the unit is placed in a chamber which is evacuated. Successive portions of mercury are added, evacuating between portions, until the levels come near the top. (Evacuation is necessary to eliminate trapped air in the mercury tubes.) The final adjustment of levels in the stop and start tubes is made using a Greist microheight gauge and an ohmmeter. The gauge carries a needle which shorts the meter when it just touches the mercury surface.

Then the flange A2 carrying piston, needles and magnet is inserted, and the solenoid housing, carrying the solenoid, is slipped over the valve block and locked into place. An O-ring is placed on the ring of the valve block, and the viscometer is inserted into the outer casing. It is aligned so that the solenoid lead is directly behind the pin in the bottom of the outer casing, and then locked there by the bottom closure nut.

The assembled viscometer is now filled with the test liquid. Entrapped air and suspended dust must be avoided. The viscometer is placed in a chamber under a funnel with a sintered glass frit and a Teflon barrel stopcock. The chamber is evacuated, and then the stopcock is opened, allowing atmospheric pressure to drive the liquid into the viscometer through the filter. When full, the viscometer is removed from the vacuum chamber and the top closure piston is inserted. The latter is pushed down until liquid flows through the central channel, which is then sealed with the fillister screw. Finally, the entire assembly is lowered into the high pressure bomb and rotated until the pin at the bottom of the outer casing engages a slot in the cell support. In this position, the three studs at the bottom automatically go into the appropriate mercury cups in the bottom closure of the bomb, completing the electrical control circuit shown in Fig. 3. To prevent sparking, 0.1 μF condensers connect the start and stop tubes to ground.

The bomb is held at 30.0±0.1° by water which circulates through 1.27 cm i.d. copper tubing wrapped around it. The tubing is cemented to the bomb with Thermon T-85,[7] which has good thermal conductivity. The assembly is thermally insulated from the room by aluminum foil and rock wool.

After temperature equilibrium is reached (about 30 min), a series of drop times at 1 atm is taken. The normally closed microswitch in the clock line is opened and the solenoid switch (normally open microswitch) is closed for about $\frac{1}{2}$ sec. This raises the piston, without starting the clock on the up-stroke. The switches are released; when the long needle touches its mercury well, the normally open relay closes and starts the clock. When the short needle contacts, the normally closed relay opens, stopping the clock. For fall times less than 5 sec, 30 readings are taken and averaged; for less than 60 sec, 12 readings; and for more than 60 sec, 8 readings. Then drop times are determined at higher pressures, usually at intervals of 700 kg cm^{-2} gauge. Pressure increase should be gradual, at not more than about 35 kg cm^{-2} min^{-1}, in order to allow the heat of compression to dissipate. Too rapid compression sets up troublesome thermal gradients in the viscometer, and in extreme cases has caused the piston to seize in the barrel.

[7] Obtained from the Lars Anderson Co., So. Weymouth, Mass.

TABLE I. Determination of viscometer constant A'.

Liquid	100η	$(1-\rho\, v/w)$	t (sec)	A'
Isopropanol	1.765	0.897	5.00	254
Ethanol	1.003	0.897	2.78	249
Ethylene dichloride	0.730	0.836	2.23	255
o-xylene	0.709	0.885	2.00	250
n-butyl chloride	0.405	0.885	1.20	262
Cumene	0.725	0.887	2.07	253
Carbon tetrachloride	0.845	0.792	2.73	256
n-propyl ether	0.376	0.902	1.10	264

EXPERIMENTAL RESULTS

The drop time t of the piston-cylinder viscometer is given by the equation[5]

$$t=A\eta(h_2^2-h_1^2)/(1-\rho v/w) \tag{1}$$

where A is an instrument constant, ρ is the density of the liquid, η is its viscosity, v is the volume of the falling piston plus attached parts, w their weight, and h_2 and h_1, are, respectively, the distance from the top of the cylinder to the bottom of the piston at the stop and start of the timing period $t=t_2-t_1$. For a given piston and cylinder, with h_2 and h_1 fixed,

$$t=A'\eta/(1-\rho v/w). \tag{2}$$

For our apparatus, $v/w=0.132_3$ cc/g.

Our first test of the reproducibility of the viscometer was the determination with the piston later used for isopropanol experiments of drop times at atmospheric pressure and 30° in eight liquids of known viscosity and density. The results are summarized in Table I.

The values of the constant average to 255±5. The uncertainty of about 3% is less than half that observed by Bridgman with the falling weight viscometer.[6] It should be mentioned that the viscometer was completely disassembled between runs for cleaning; the ±3% probably is mostly due to slight differences in setting the mercury heights (which enter as the square in the viscometer equation).

The viscometer was then tested at various pressures up to about 5000 atm with isopropanol and diethyl ether.