FIG. 4—AUTOCLAVE 0.25-IN. HIGH PRESSURE SWIVEL JOINTS.
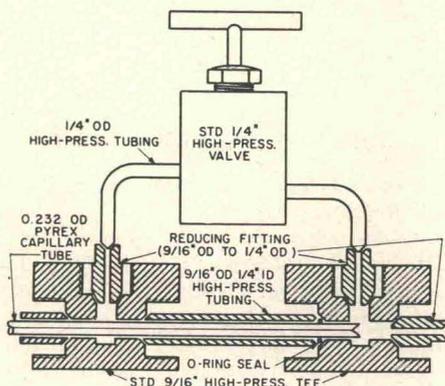
FIG. 5—DETAILS OF CAPILLARY TUBE SEAL AND BY-PASS VALVE ASSEMBLY.

A. CONDITION FOR CHARGING TEST FLUID

B. EXCESS MERCURY WITHDRAWN, VISCOMETER AT REFERENCE LEVEL

C. BYPASS VALVE OPEN, RESERVOIR LOWERED TO FILL WITH MERCURY
D. BYPASS VALVE CLOSED, RESERVOIR RAISED TO TEST POSITION
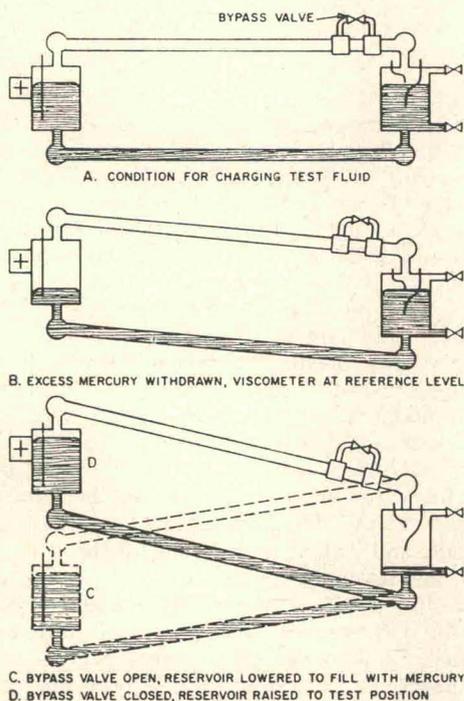
FIG. 6—RELATIVE RESERVOIR AND RECEIVER POSITIONS DURING CHARGING AND TESTS.

TABLE 1—SUMMARY OF CAPILLARY TUBE CALIBRATION DATA

	Tube No. 13	Tube No. 14
Length, L (cm)	90.070	91.165
r_1 by Fisher[7] method, cm	0.010876	0.012088
r_1 by gravimetric method, cm	0.010877	0.012092
$\int_o^L dL/r_1^4$ by Fisher[7] method, cm^{-3}	64.380×10^8	42.695×10^8
$\int_o^L dL/r_1^4$ by gravimetric method, cm^{-3}	64.359×10^8	42.648×10^8

The Pyrex capillary tubes were obtained as a special draw. The tubes are from 0.021- to 0.025-cm bore by 0.5- to 0.6-cm OD, and from 90 to 95 cm in length. The ends of the capillary tubes are flared slightly to provide a streamlined entrance and exit with consequent reduction in end-effects. Average internal radii of the capillary tubes were determined gravimetrically, and the variation of radius along the tubes was determined by a graphical integration technique proposed by Fisher[7] and described in detail by Carr.[2] A summary of calibration data for two of the capillary tubes which have been used is given in Table 1.

EXPERIMENTAL PROCEDURE

Prior to assembly of the viscometer all parts were thoroughly cleaned and dried. The capillary tube was cleaned with chromic acid solution followed by flushing with water, acetone and ether. The metal parts were degreased and rinsed in acetone.

The receiver electrode spacing was measured and the viscometer assembled, except for the top of the mercury reservoir. Clean, dry mercury was added to the system until approximately 1/3- in. of mercury remained in the reservoir when the mercury surface in the receiver just made contact with the short electrode, the top was then bolted in place. The technique of charging the mercury to the viscometer ensured that sufficient mercury was present in the system for proper operation.

After the assembled viscometer had been evacuated, the system was pressured to about 1,000 psi for a leak test. A 5,000-psi deadweight gauge measured pressure to the nearest pound. Oil in the deadweight gauge was separated from the test fluid in the viscometer by a diaphragm-type pressure-balance indicator. The viscometer system was considered pressure tight when no pressure change could be detected with the diaphragm indicator in one hour.

After any leaks were sealed the nitrogen was vented and the unit evacuated and charged with test fluid. The remaining operations are best described by reference to Fig. 6. Prior to charging the viscometer with test fluid the reservoir level was varied until the mercury in the receiver made contact with the short electrode as in A, Fig. 6. This provided a means for monitoring the position of the mercury in the unit during the charging operation. The by-pass valve was open during evacuation and charging.

The volume of mercury in the viscometer was adjusted by slowly draining mercury from the pressurized system. As mercury was removed the reservoir was raised to keep the mercury in contact with the short electrode. Withdrawal was stopped when the reservoir electrode tip was still submerged 2 to 4 mm and the short electrode just made contact with the mercury in the receiver, as in B, Fig. 6. The by-pass valve was open during mercury withdrawal and determination of the reference level.

The cathetometer reading for the scribed crosshair of the reservoir target is taken at this point and represents the reference level for calculating mercury driving forces used in the tests.

With the quantity of mercury in the system properly adjusted and the reservoir target reference level measured, the instrument was ready for viscosity measurements. The reservoir was lowered to permit most of the mercury in the system to accumulate there, as in C, Fig. 6, and the by-pass valve closed. The reservoir was raised to run position, as in D, Fig. 6. The cathetometer reading was set to provide a pre-selected pressure differential across the capillary tube, and the reservoir height carefully adjusted each time until the target was aligned with the cathetometer cross hairs. In this manner multiple runs could be made at the same driving force, which resulted in the variation in flow times being a direct comparison of data reproducibility.

The electrical circuit utilized to control the timer and indicate mercury levels is shown in Fig. 7. Switch S_5, a five-pole rotary gang switch, has three positions: off, test and run. With S_5 in test position and toggle switches S_1 and S_2 closed, green lights on the controller indicate when mercury is in contact with the reservoir electrode or the short receiver electrode. With S_5 in the run position and S_2 and S_3 closed, the controller will close R_3 when mercury contacts the long receiver electrode, starting the timer, and will close R_2 when mercury contacts the short electrode, stopping the timer. Relay R_3 can be by-passed to insure dependable timer operation by closing S_1 after the timer has started, then R_3 can be released by opening S_3.

Typical experimental data for nitrogen are given in Table 2. The reproducibility of the flow time proved an excellent indication of condition of the viscometer. Whenever flow time variations consistently exceeded 0.5 per cent, it was time to disassemble and clean the apparatus. Occasionally erratic timer readings were obtained (as illustrated in Table 2) far removed from the average of three or more repeat runs; these points were discarded before averaging. Generally, variation of the measurements was ± 0.2 per cent or less from the average value.

S₁, S₂, S₃ & S₄ - TOGGLE SWITCHES
R₁, R₂ & R₃ - 6-VOLT SPDT RELAYS
S₅ - 3-POSITION, 5-POLE ROTARY GANG SWITCH
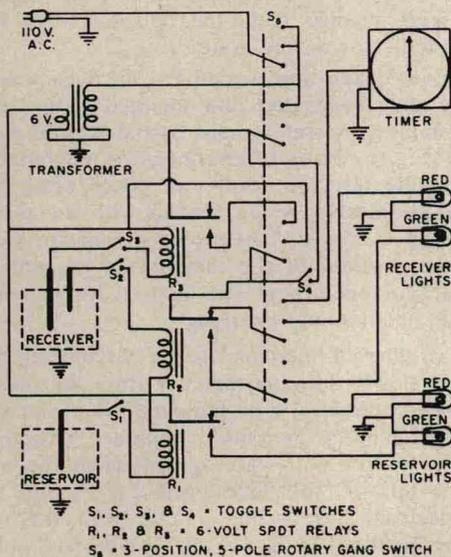
FIG. 7—WIRING DIAGRAM FOR TIMER CONTROL AND REFERENCE LEVEL CHECK.

TABLE 2—TYPICAL EXPERIMENTAL DATA ON NITROGEN

June 2, 1957, data set 6 Capillary 14 Temp. = 21.7°C Press. = 1,000.5 psig Bar. = 29.66 Hg Elec. spac. = 1.375 cm			June 3, 1957, data set 7 Capillary 14 Temp. = 23.4°C Press. = 1,000.5 psig Bar. = 29.45 Hg Elec. spac. = 1.375 cm		
Run No.	Cathetometer Reading (cm)	Time (seconds)	Run No.	Cathetometer Reading (cm)	Time (seconds)
Base Line	67.770	—	Base Line	67.525	—
1	75.150	127.6	1	74.905	128.5
2	75.150	127.5	2	74.905	128.5
3	75.150	127.6	3	74.905	128.5
Avg	75.150	127.6±0.0	Avg	74.905	128.5±0.0
4	74.215	143.3	*4	73.975	145.8*
5	74.215	143.1	5	73.975	143.8
6	74.215	143.2	6	73.975	144.1
Avg	74.215	143.2±0.1	7	73.975	144.2
7	73.120	166.7	8	73.975	144.1
8	73.120	167.1	Avg	73.975	144.1±0.1
9	73.120	166.9	9	72.880	167.9
Avg	73.100	166.9±0.1	10	72.880	168.0
10	72.070	198.5	11	72.880	168.0
11	72.070	198.5	Avg	72.880	168.0±0.0
12	72.070	198.7	12	71.825	200.3
Avg	72.070	198.6±0.1	*13	71.825	171.3*
13	71.215	235.3	14	71.825	200.1
14	71.215	235.3	15	71.825	200.0
15	71.215	235.5	Avg	71.825	200.1±0.1
Avg	71.215	235.4±0.1	16	70.970	237.4
16	70.335	291.3	17	70.970	236.3
*17	70.335	298.8*	18	70.970	237.0
18	70.335	291.6	19	70.970	235.5
19	70.335	292.5	Avg	70.970	236.6±0.7
20	70.335	292.4	*20	70.090	324.3*
Avg	70.335	291.9±0.4	21	70.090	295.2
Base check	67.770	—	22	70.090	295.2
			23	70.090	294.8
			Avg	70.090	295.1±0.2
			Base check	67.540	

*Omitted from average

RESULTS AND DISCUSSION

The theory of fluid flow through capillary tubes is very well developed. Equations have been derived for calculating the viscosity from experimental measurements, based on isothermal, laminar fluid flow in the capillary tube. The correlations for end effects, establishment of parabolic velocity distribution, compressibility of the test fluid and pressure losses in the mercury flow system are described in detail in the Appendix. The final equation used to calculate the results presented is as follows:

$$\mu_f = \frac{\pi g r_c^4 \theta}{8 L_c V_D}\left[\frac{\Delta h_{1m}(\rho_m - \rho_f)g}{g_c} - \frac{\beta \rho_f V_D^2}{g \pi r_c^4 \theta^2}\right] - \frac{L_m r_c^4 \mu_m}{r_m^4 L_c} \quad \ldots \ldots \ldots \ldots \ldots (1)$$

Initial tests of the viscometer were made by use of nitrogen at pressures from 200 to 1,000 psig at room temperature. Results are presented in Fig. 8, with the viscosity data of Michels and Gibson[10] included for comparison. To determine data reproducibility, a series of eight sets of data (31 separate tests) were made at a pressure of 1,000 psig. The average of the calculated viscosities was 192.0 micropoise with a standard deviation of ± 0.6 micropoise, which gives a 95 per cent confidence limit (twice the standard deviation) of ± 0.6 per cent for the experimental data. The corresponding viscosity given by Michels and Gibson for the same conditions is 191.4 micropoise.

To demonstrate the range of the new instrument, viscosity of water was measured at 1,150 psig at room temperature. The results agreed with the data in the literature[14] within ± 0.2 per cent for 14 of the 15 tests made, and within 0.7 per cent for the other.

With the instrument's accuracy demonstrated over a range of viscosities from 180 (nitrogen) to 9,200 micropoise (water), tests were initiated with propane and ethane. The results are summarized in Table 3. The experimental measurements were made at room temperature, which varied from 24.6 to 26.0° C (76.4 to 78.8° F), and the calculated viscosities were cor-