EXPERIMENTAL STUDIES ON VISCOSITY OF ASPHALT CEMENTS AT 77 F

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This paper provides the background for the direct application of capillary rheometry to asphalt cements. It is this background that has lead to the development of a practical rapid method for evaluating the apparent consistency in poises and the shear susceptibility of asphalt cements at 77 F and other ambient temperatures. The apparatus and studies employed in the original experimental work are presented. The theoretical complications in capillary measurements and their effect on the results for asphalt cements are discussed. Comments on the magnitude of the effects and measures to allow for them are considered. Experimental data on a number of asphalts from different sources are shown. It is concluded that the capillary type of apparatus is feasible and merits consideration by asphalt technologists as a first satisfactory answer to the problem of measuring asphalt viscosity at service temperatures.

•A MAJOR problem in asphalt technology is an evaluation of the apparent viscosity and shear susceptibility of asphalt cements at 25 C (77 F). These properties are important in highway construction and service of asphalts. The objective of this paper is to describe experiments with a rheological evaluation technique for asphalt cements at ambient temperatures. This technique is different from those previously proposed or currently in use.

Numerous procedures (1) are described in the literature, and the ASTM has proposed several methods (2) for measuring the consistency of asphalt cements in the range of high consistency. The cone and plate method and the sliding plate microviscometer are utilized to a considerable extent for research studies. The latter can be used for routine work and is useful for small samples and aging studies. However, this apparatus has some limitations (3).

Attention in this laboratory was directed to tubular rheometry application for asphalt that was investigated earlier by Traxler and Schweyer (4). Pezzin (5) has discussed capillary rheometry extensively where it is applied to elastomers and polymers at high temperatures to study their flow behavior.

The capillary rheometer is adaptable for asphalt consistency measurements over wide temperature and consistency ranges. It consists of a simple barrel and piston arrangement with a capillary through which the asphalt is extruded. The geometry may be varied to accommodate different consistencies, and suitable air temperature control equipment can be employed.

THEORY

The deformation of asphalts under stress may be considered to follow the classical power law concept for relating flow parameters.

$$\tau = \eta \dot{\gamma}^{C} \tag{1}$$

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where

τ = shearing stress at the wall, dynes/cm^2;
$\mathring{\gamma}$ = average rate of shear, sec^{-1};
C = power function parameter;
η = correlation coefficient [when C has a value of unity (Newtonian fluid), η is the viscosity in poises].

For non-Newtonian flow, the average rate of shear as normally computed must be changed by the Rabinowitsch correction to give the proper value, $\mathring{\gamma}_W$, at the wall in capillary flow as noted by Pezzin (5). The apparent viscosity, η_a, is then computed as follows at a selected point:

$$\eta_a = \tau/\mathring{\gamma}_W \text{ poises} \tag{2}$$

where all values are based on calculations at the wall.

The value of $\mathring{\gamma}_W$ is computed from the average $\mathring{\gamma}$ by use of the Rabinowitsch correction as stated by Pezzin (5) as well as others. The corrected value for $\mathring{\gamma}_W$ is as follows:

$$\mathring{\gamma}_W = \mathring{\gamma}(0.75 + 0.25/C) \tag{3}$$

where C is the slope $(\partial \log \tau)/(\partial \log \tau)$ of a plot of

$$\log \tau = C \log \mathring{\gamma} + \log \eta \tag{4}$$

Such a plot is shown in Figure 1 for a typical asphalt cement of 89 penetration. The evaluation of C, sometimes called the complex flow index, provides a method for evaluating the shear susceptibility of asphalt cements. If C is greater than one, the material is considered to be dilatant; whereas, if C is less than one, the material is pseudoplastic. If C is equal to one, the material is a Newtonian fluid with a line having a slope of 45 deg (Fig. 1). Thus, the greater the divergence of C from unity is, the greater is the shear susceptibility.

When η varies with the rate of shear, it cannot be used to evaluate the viscosity except at some selected rate of shear or shearing stress. It is proposed that viscosity be evaluated at a constant power input per unit volume of $\tau\mathring{\gamma}$ = 100,000 ergs/(sec-cm^3). As shown in Figure 1, the line of constant power input falls close to the region of experimental data for asphalt cements used in paving construction. Traxler (6) suggested that measurements at a power input of 1,000 ergs/sec per unit volume be used, but the higher value appears more appropriate because it requires less extrapolation of the experimental data. The apparent viscosity is reported in poises according to Eq. 5 using the value for τ_p and $\mathring{\gamma}_p$ read at the intersection of best straight line (by inspection or by numerical methods) for the experimental data and the constant power input line at 100,000 ergs/(sec-cm^3).

$$\eta_a = \tau_p/[\mathring{\gamma}_p(0.75 + 0.25/C)] \text{ poises} \tag{5}$$

It will be noted that the correction for the value of C = 0.5 causes a 25 percent decrease in viscosity, whereas a value of C = 1.5 results in about an 8 percent increase compared to the uncorrected average value for $\mathring{\gamma}$.

PROCEDURE AND APPARATUS

The capillary procedure is quite simple and consists of measuring the force required to extrude the sample at essentially a constant volume flow rate through a capillary by using different fixed piston speeds. From these machine data, the calculated shearing stress and rates of shear are plotted as shown in Figure 1. The apparent viscosity is calculated at a constant power input of 100,000 ergs/sec per unit volume, and the shear susceptibility is indicated by the value of C.

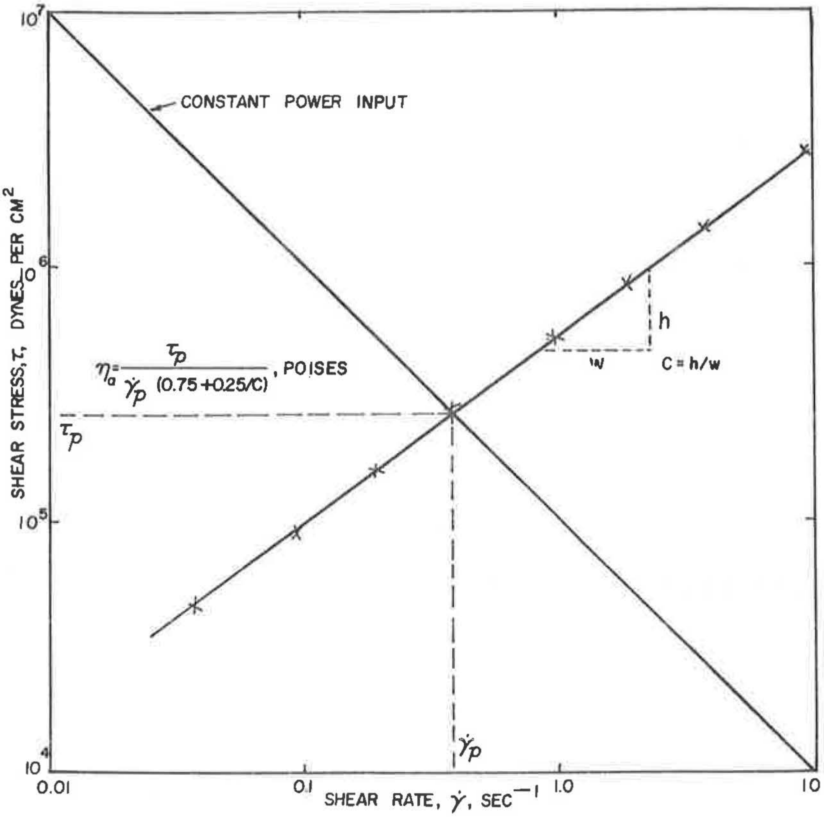

Figure 1. Rheogram for an asphalt cement.

A diagram of the barrel assembly is shown in Figure 2; the components are shown in Figure 3. The Instron machine used is shown in Figure 4, and Figure 5 shows a close-up of the environmental chamber that contains the cooling and heating coils and the ducts for circulating the air by means of a blower behind the chamber. Typical chart data obtained at different crosshead speeds are shown in Figure 6. The load scale multipliers (10X, 5X, and 2X) are shown for corresponding crosshead speeds with changes in the multiplier indicated for each change. (The multipliers are changed to increase the sensitivity at low developed loads.)

Typical results for several asphalt cements are given in Table 1, and typical plots are shown in Figure 7 for 3 samples. It is not the intent to discuss the significance of these data at this time. These preliminary data were sufficiently encouraging to warrant further study.

RHEOLOGICAL CONSIDERATIONS

The following factors affecting flow through a capillary from a reservoir must be considered in applying this technique to asphalt cements.

Sample Reservoir

Results of studies on the influence of a sample charge in the barrel that acts as a reservoir are given in Table 2 for several asphalts. These data demonstrate that the reservoir pressure drop must be considered. The effect decreases as the ratio of length to diameter (L/D) increases. Pezzin (5) indicated that the effect amounted to 0.5

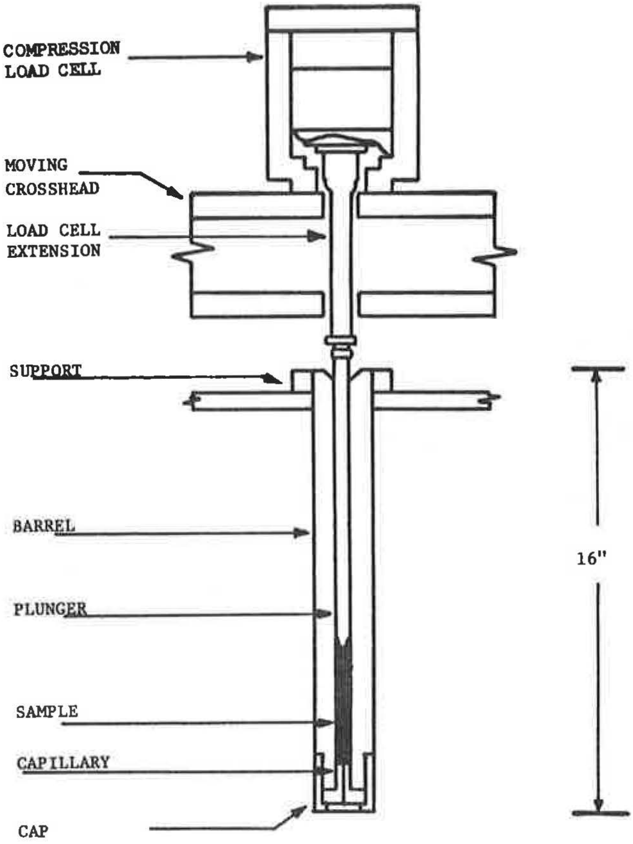

Figure 2. Barrel assembly for rheometer.

percent for polymers with an L/D ratio of the order 35. Further studies of this type are discussed later. The sample size may be a critical factor for certain materials.

Entrance Geometry

An entrance effect occurs (Couette's) where the flow accelerates from the reservoir rate to the capillary velocity. In general, a correction of equivalent length is used to account for the pressure drop due to this energy dissipation as discussed by Pezzin (5), Duvdevani and Klein (7), and Philippoff and Gaskins (8).

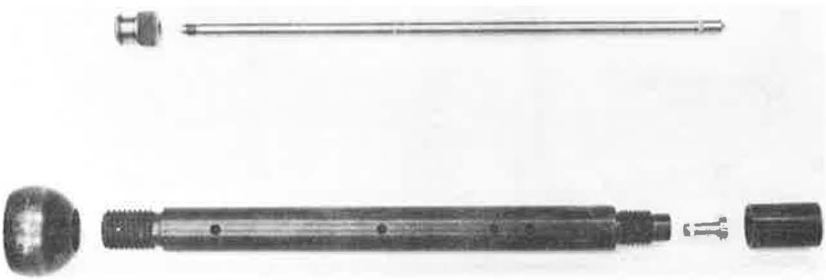

Figure 3. Rheometer components.

Figure 4. Instron machine.

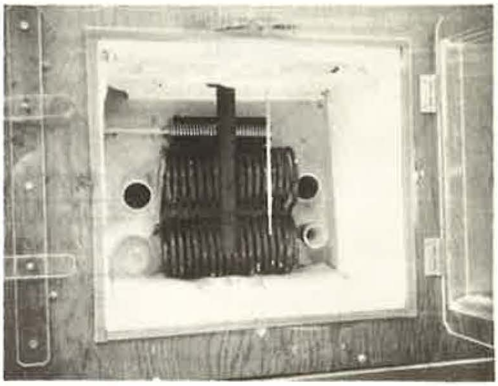

Figure 5. Environmental chamber.

A study on the practical effect of using a flat, 180-deg capillary entrance with a flat plunger was developed, and the results are given in Table 3. Variable results occur for conical or flat entry with the latter being considered as acceptable for asphalt cements at this time. Bagley (9) comments on flat entry as do Boles and Bogue (10). An extensive analysis with diagrams on this subject was given by Cook, Furno, and Eirich (11). Metzger and Knox (12) used 180- and 90-deg entrance geometry without appreciable differences.

The experimental results given in Table 3 indicate that the energy corrections required are small in comparison with flow energy loss through the capillaries for an L/D

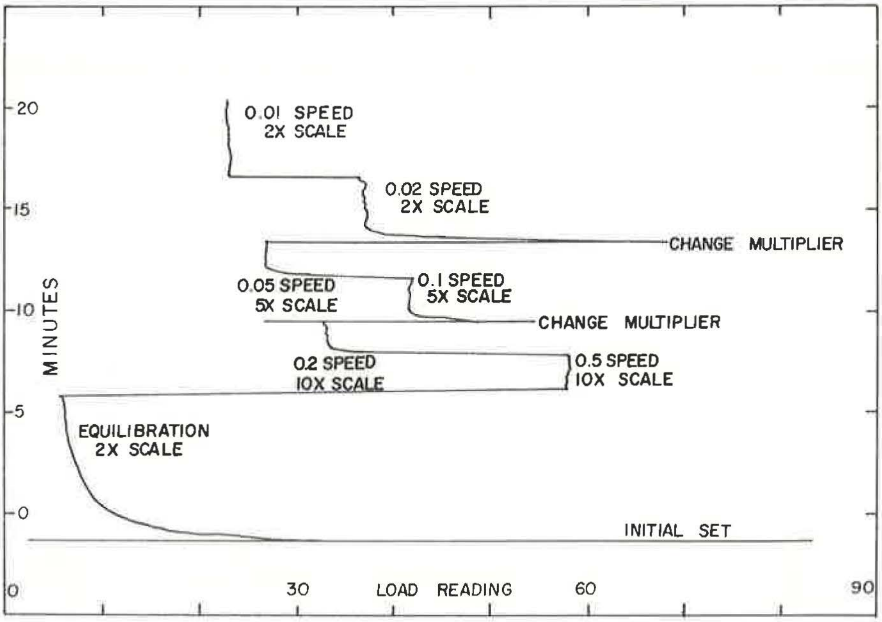

Figure 6. Tracing of actual chart data.

ratio above 15. Experiments indicate that asphalt S63-20 shows incipient dilatant flow behavior (C > 1) with anomalous flow developing above some critical pressure. Further study may elucidate this behavior.

Conditioning Time

Studies made showed that 30 minutes is necessary to cool the barrel and sample center in the regular barrel with forced convection fan cooling in an environment at 77 F. Other test temperatures might require different preparation times. The test procedure prescribes a preliminary stabilizing period at low flow rates that tends to ensure constant temperature before data are taken.

Incompressible Flow

The theoretical equations assume incompressibility for asphalt. This is not true, but in this paper such effects are considered negligible.

TABLE 1

RHEOLOGICAL DATA AT 77 F ON ASPHALT CEMENTS

Cement	Penetration at 77 F	Apparent Viscosity (mp)	Complex Flow Index C
S63-4 Smackover	89	1.48	0.84
S63-6 Florida AC-8	89	0.93	0.99
S63-13 Air Blown	89	0.62	0.64
S63-19 Panuco	90	0.98	0.85
S63-20 Los Angeles Basin	89	1.34	1.02
S63-21 Kern River	89	0.85	1.10
7171B Recovered AC-8	63	3.2	0.60
7192B Recovered AC-8	35	7.5	0.65
7442A Recovered AC-8	18	49.5	0.55
7136A Recovered AC-6	20	52.5	0.85
S62-2 Hawkins Residual	soft	0.0860	1.00
S63-2 Mid-Continent Flux	soft	0.00925	0.93

Radial Flow

Based on the literature (7), the effects of radial velocity are considered negligible because the asphalts are considered to follow the power law at the temperatures used.

Pressure Effect

The effect of pressure on viscosity is assumed to be negligible for the data reported here. This subject is discussed by Duvdevani and Klein (7). Any increased viscosity because of pressure offsets to some extent the decrease in viscosity caused by thermal effects.

TABLE 2

COMPARATIVE DATA ON 85 TO 100 PENETRATION ASPHALT CEMENTS WITH DIFFERENT RESERVOIR LENGTHS

Capillary[a]	Cement	8-in. Reservoir		3-in. Reservoir	
		Apparent Viscosity (mp)	Complex Flow Index C	Apparent Viscosity (mp)	Complex Flow Index C
No. 8	S63-13	1.09	0.73	0.72	0.71
Diameter = 0.147 in.	S63-19	2.31	0.81	2.21	0.79
L/D = 7.24	S63-20	2.35	1.19	2.31	1.03
No. 6	S63-13	–	–	0.78	0.60
Diameter = 0.097 in.	S63-19	–	–	1.20	0.83
L/D = 11.4	S63-20	–	–	1.20	0.99
No. 4 and No. 5	S63-13	0.71	0.66	0.62[b]	0.64[b]
Diameter = 0.060 in.	S63-19	1.10	0.91	0.98[b]	0.85[b]
L/D = 16.7	S63-20	1.14	0.89	1.34[b]	1.02[b]

Note: Data for 77 C with power input of 100,000 ergs/(sec-cm^3).

[a] L/D is ratio of length to diameter.

[b] From Table 5.

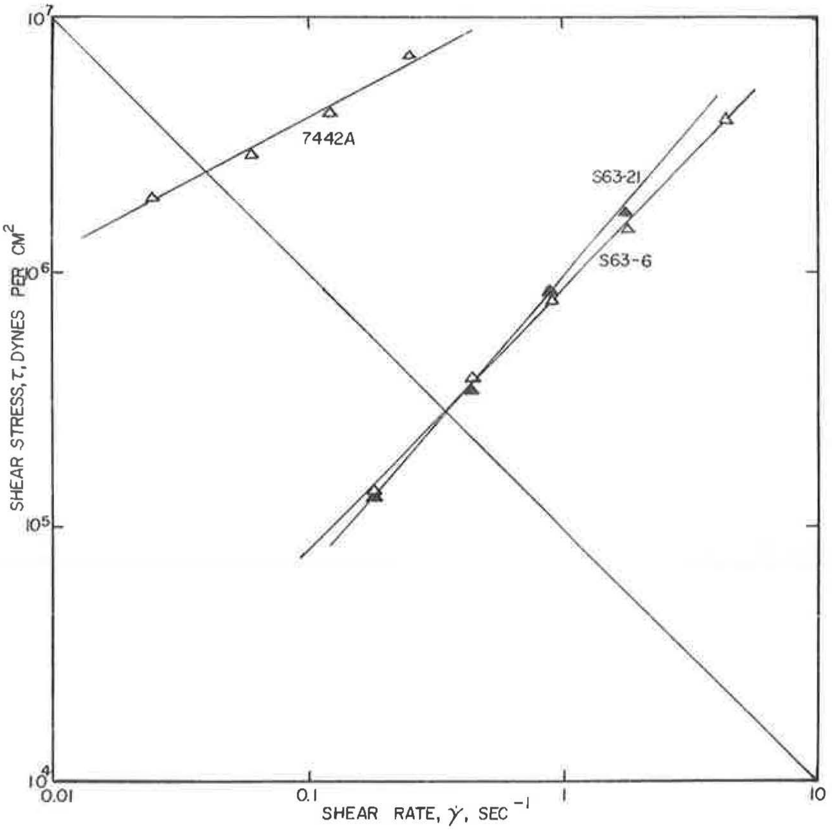

Figure 7. Example of rheograms for different asphalts.

Thermal Effects

There are two possible thermal effects in capillary viscometry. One is the rise in temperature resulting from the viscous energy dissipation as studied by Gerrard et al. (13), who showed that a 5 percent change in viscosity could be expected in so-called isothermal flow for apparent Newtonian flow at power inputs of about 1 billion ergs/(sec-cm^3). A second effect is the cooling resulting from the expansion of the fluid as the pressure decreases during the passage through the capillary (5). Both these compensating effects are not corrected for in this study because the power input of 10^7 ergs/(sec-cm^3) is 2 orders of magnitude less than the values of 10^9 noted earlier (13), and the expansion correction is considered unimportant.

TABLE 3

COMPARATIVE DATA ON 85 TO 100 PENETRATION ASPHALT CEMENTS WITH DIFFERENT ENTRANCE GEOMETRY

Cement	90-Deg Conical Entrance and Plunger[a]		180-Deg Flat Entrance and Plunger[b]	
	Apparent Viscosity (mp)	Complex Flow Index C	Apparent Viscosity (mp)	Complex Flow Index C
S63-4	1.48	0.84	1.53	0.76
S63-13	0.62	0.64	0.83	0.66
S63-19	0.98	0.85	0.88	0.87
S63-20	1.34	1.02	1.09	0.97

Note: 3-in. reservoir length for all samples.

[a] Capillaries No. 4 and No. 5.

[b] Capillary No. 7A.

Capillary Geometry

Study of the data given in Table 2 indicates that the geometry (L/D ratio) of the capillary affects the results. This was ex-

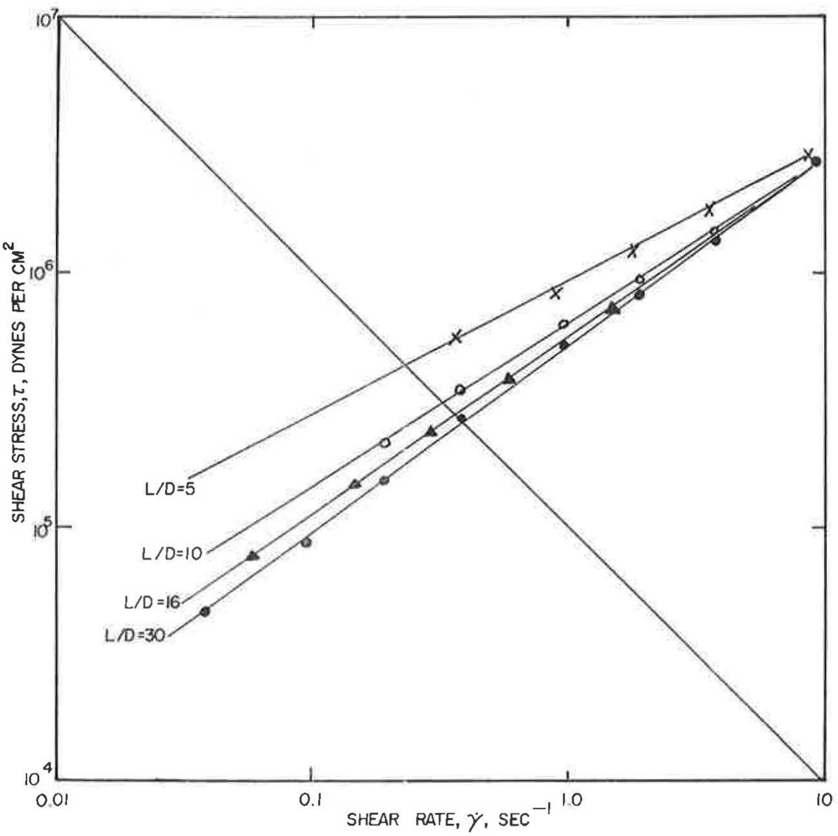

Figure 8. Rheograms for different geometry for asphalt S63-13.

TABLE 4

COMPARATIVE DATA ON 89 TO 90 PENETRATION ASPHALT CEMENTS WITH 180-DEG ENTRANCE GEOMETRY

Capillary	Cement	Apparent Viscosity (mp)	Complex Flow Index C
No. 15A	S63-13	1.56	0.53
Diameter = 0.10 in.	S63-19	3.09	0.73
L/D = 5	S63-20	1.01	0.95
No. 13A	S63-13	0.85	0.65
Diameter = 0.10 in.	S63-19	1.73	0.82
L/D = 10	S63-20	1.10	0.99
No. 12A	S63-13	0.64	0.75
Diameter = 0.10 in.	S63-19	1.39	0.95
L/D = 10	S63-20	1.00	0.89
No. 18A and No. 19A	S63-13	0.80	0.61
Diameter = 0.0625 in.	S63-19	0.78	0.81
L/D = 20	S63-20	1.21	1.09
No. 16A and No. 17A	S63-13	0.73	0.78
Diameter = 0.187 in.	S63-19	1.84	0.82
L/D = 16	S63-20	1.60	1.05
No. 16A and No. 17A	S63-13	0.66	0.78
Corrected by using	S63-19	1.69	0.82
Table 5 factors	S63-20	1.52	1.05
No. 4 and No. 5	S63-13	0.62	0.64
Diameter = 0.060 in.[a]	S63-19	0.98	0.85
L/D = 16.7	S63-20	1.34	1.02

Note: All samples 3 in.; at 77 F with power input of 10^5 ergs/(sec-cm^3).

[a] From Table 2 using conical entry.

plored further for different 180-deg entrance capillaries; the results are given in Table 4. Data for one asphalt are shown in Figure 8 and demonstrate for capillaries with decreasing L/D ratios that there is a trend of decreasing complex flow index with decreasing L/D ratio. It appears that, as the L/D ratio exceeds 15 to 20, the results become more consistent in accord with work of other investigators on polymers.

There is one observation that should be made regarding the data shown in Figure 8. Whenever for some reason or another the lines are not parallel indicating a variation in the complex flow index, then the relative values of the viscosities computed at different rates of shear will vary. In fact, it may be possible to select a value of $\dot{\gamma}$ at which all lines intersect at a common shear stress at the wall. However, the apparent viscosities would differ because plots with different C would have different correction factors according to Eq. 5.

Where certain geometries indicate, it is necessary to employ a modification of the basic Eq. 5. The correction is a function of the barrel geometry and the viscosity of the sample because the pressure drop is the sum of capillary pressure drop effects plus the reservoir pressure drop. Thus, for small capillaries, the pressure drop in the barrel, P_B, can be considered negligible compared to the pressure drop in the capillary, P_C, because

$$P_B/P_C = (L_B/L_C)\,(D_C/D_B)^{3C+1} \tag{6}$$

where L is length, D is diameter, and C is the complex flow index. The subscripts B and C refer to the barrel and capillary respectively. However, for other capillaries, the pressure drops in the capillary compared with those in the barrel are given in Table 5 and show a significant effect. When a fixed procedure is used with a given geometry, the capillary pressure drop, P_C, would be a corrected one from the total pressure read as $(P_C + P_B)$.

$$P_C = \frac{(P_C + P_B)}{1 + (L_B/L_C)\,(D_C/D_B)^{3C+1}} \tag{7}$$

As a first approximation, it is suggested that the apparent viscosity as calculated from Eq. 5 be corrected where indicated by dividing the apparent viscosity from Eq. 5 by 1 plus the appropriate factor given in Table 5. This is essentially the same as correcting the shear stress through Eq. 7.

Corrected values are given in Table 4. The agreement of all of the corrected values in capillaries 16A and 17A is not as good as hoped for, but additional study should result in improved techniques and better results.

Thixotropy

The effect of time on the stress-shear relation is considered negligible based on the procedure that requires reading an equilibrium stress value at different shear rates. In addition, limited data for different capillaries at L/D ratios above some critical

TABLE 5

CALCULATED PRESSURE DROP RATIOS IN CAPILLARY RHEOMETER WITH BARREL DIAMETER OF 0.375 IN. AND RESERVOIR LENGTH OF 3 IN.

Capillary						Pressure Drop Ratio, P_B/P_C			
No.	Length (in.)	Diameter (in.)	L_C/D_C	L_B/L_C	D_C/D_B	C = 0.5	C = 0.8	C = 1.0	C = 1.2
12A	3.000	0.100	30	1.0	0.267	0.037	0.011	0.005	0.002
13A	1.000	0.100	10	3.0	0.267	0.111	0.033	0.015	0.006
16A and 17A	3.004	0.1875	16	1.0	0.50	0.176	0.095	0.063	0.041
18A	1.255	0.073	20	2.4	0.195	0.040	0.009	0.003	0.001

value indicate similar values for the results. There are data available to show that in general the same results are obtained by programs for measurements at both decreasing and increasing rates of shear, but this may not be universally true.

Elastic Effects

The energy imparted for elastic deformation is carried through the capillary and dissipated on exit. This portion of the energy input is not used for viscous deformation. Pezzin (5) indicates this to be of the order of 0.05 percent of total energy and can be neglected.

Melt Fracture

A possible erratic behavior at high pressures occurs for asphalts having values of $C > 1$, but in general the rheological evaluation is made at stresses below which this phenomenon occurs. At this writing, the complicating effects (entrance compression, energy dissipation, melt fracture, and others) appear to be obviated by elimination of readings if the stress exceeds 2×10^6 dynes/cm^2.

REPRODUCIBILITY

Data given in Table 6 on different runs by different operators are shown to indicate reproducibility. An example of good data for S63-13 is shown in Figure 9 for conical entry that demonstrates the validity of Eq. 1 over the range of shear rates studied.

It is believed that the reproducibility can be improved up to 40 percent by experience, improved temperature control, and improved apparatus components.

RESULTS

For data at 77 F, it will be noted that, for different non-Newtonian asphalts, the value of the apparent viscosity will vary at different power inputs (or different rates of shear). Accordingly, in a comparison of asphalts, their relative viscosities may be different. For example, Figure 10 shows relative viscosities reversed for S63-4 and S63-20 when compared at 0.1 and 10 sec^{-1}.

Comparable data for apparent viscosity at 77 F by the proposed capillary method and by the cone and plate procedure (15) are given in Table 7 where the agreement is quite good at a shear rate of 0.05 sec^{-1}.

TABLE 6

ABBREVIATED STUDY OF VARIABILITY IN FLORIDA METHOD FOR 85 TO 100 PENETRATION ASPHALT CEMENTS

Cement	Replicate	Apparent Viscosity (mp)			Complex Flow Index C		
		Amount	Avg	Deviation From Mean (percent)	Amount	Avg	Deviation From Mean (percent)
S63-4	1	1.43	1.48	3.7	0.82	0.84	5.7
	2	1.42			0.92		
	3	1.49			0.85		
	4	1.58			0.76		
S63-13	1	0.77	0.62	12.9	0.66	0.64	3.1
	2	0.56			0.67		
	3	0.60			0.63		
	4	0.54			0.62		
S63-20	1	1.39	1.34	5.2	0.97	1.02	6.8
	2	1.19			0.94		
	3	1.38			1.11		
	4	1.38			1.07		

Note: Average deviations of 3 sets of data: apparent viscosities–7.3 percent of viscosity value; shear susceptibilities–5.2 percent of index value.

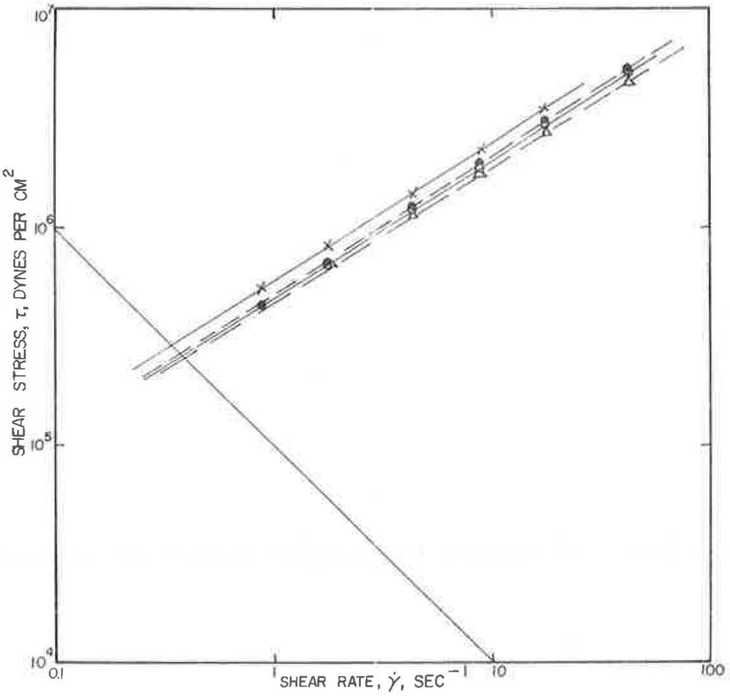

Figure 9. Reproducibility of data on asphalt S63-13.

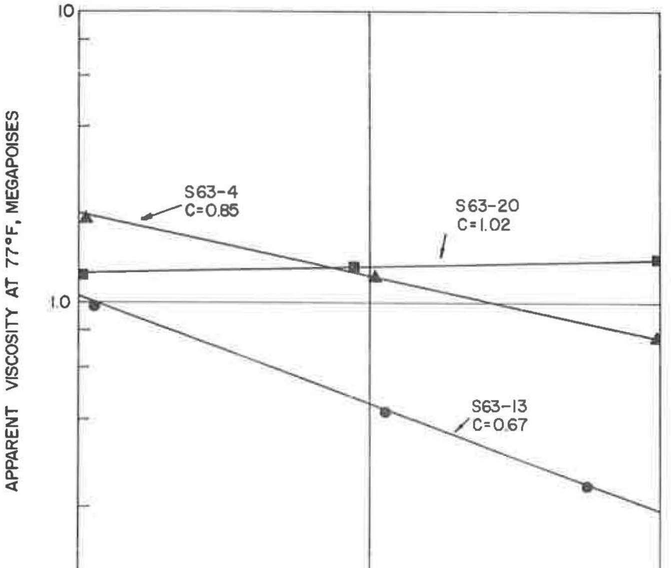

Figure 10. Variation of viscosity with shear rate.

TABLE 7

COMPARABLE VISCOSITY DATA AT 77 F

Asphalt	Penetration at 77 F	Florida Method			Asphalt Institute Method, Viscosity (mp), 0.05 sec^{-1}
		Complex Flow Index C	Viscosity (mp)		
			10^5 ergs/(sec-cm^3)	0.05 sec^{-1}	
8533	60	0.80	2.24	3.06	2.78
8621	60	0.64	2.31	3.72	4.00
8581	59	0.92	2.68	2.94	2.60
8534	90	0.73	0.97	1.50	1.19
8692	89	0.92	1.00	1.16	0.90

TABLE 8

RHEOLOGICAL DATA AT 60 F ON 89 TO 90 PENETRATION ASPHALT CEMENTS

Cement	Observed			Computed[a]		
	Apparent Viscosity (mp)		Complex Flow Index C	Penetration at 60 F, 100g/5 sec	Viscosity (mp)	Range[b]
	10^5 ergs/(sec-cm^3)	0.05 sec^{-1}				
S63-13	4.4	7.3	0.72	40	7.5	5.5 - 14
S63-19	17.1	20.0	0.90	32	11.0	8 - 18
S63-20	49.5	49.6	0.96	24	17.9	13 - 35

Note: Capillaries No. 16A and No. 17A, 0.187 × 3.0 in.; data corrected for reservoir pressure effect.

[a] Calculated from measured penetration at 60 F by using the following equation for viscosity at 60 F at a rate of shear equal to 0.05 reciprocal seconds (14): $\log \eta = 3.6 - 1.7 \log \text{pen}$; $\eta = 3{,}980\ \text{pen}^{-1.7}$ mp.

[b] Allowable range shown by Welborn and Griffith (14).

For data at 60 F, the general applicability of the procedure was demonstrated by measurements using capillaries with diameters of 0.187 in. and a length of 3 in.; results are given in Table 8. The influence of temperature is shown by comparing the results with those given in Table 1.

The results at 60 F are reported both at a power input of 10^5 ergs/(sec-cm^3) and at a rate of shear equal to 0.05 sec^{-1} for comparison with the values computed by the formula of Welborn and Griffith (14). The measured data are of the same order of magnitude as the range for computed values.

Data on 3 asphalt cements at 140 F by the method in this paper compared to results by the Cannon-Manning efflux method (also a capillary method) are given in Table 9. It is shown that low viscosity does not ensure Newtonian flow at either 140 or 77 F (Table 1). Each asphalt at any temperature is a separate entity, and its composition will determine its flow characteristics.

DEVELOPMENT OF A TEST METHOD

The philosophy observed in considering the applicability of capillary rheometry to asphalt cements as a standard test method has been to develop a satisfactory procedure even though it may have limitations. There is a great need for apparent viscosity measurements at ambient temperatures; the proposed method (or modifications of it) could be an acceptable one. Development work on modifications for a rapid measurement is now in progress, and it is expected that a final recommended procedure will soon be available. It now takes about 40 minutes to make preparations, equilibrate the temperature, and run a sample. This total elapsed time could be reduced with multiple samples and fewer points for control testing.

TABLE 9

RHEOLOGICAL DATA AT 140 F ON 89 TO 90 PENETRATION ASPHALT CEMENTS

Cement	Capillary		Cannon-Manning Viscosity (poise)
	Apparent Viscosity (poise)	Complex Flow Index C	
S63-13	1.670[a]	0.94[a]	1,726
S63-19	4.680	0.93	3,468
S63-20	1.370	1.11	1,109

[a] Average of three.

There remain certain other details such as establishment of optimal capillary dimensions and tolerances and development of an inexpensive constant rate piston machine. These are being investigated.

ACKNOWLEDGMENT

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