Determination and Treatment of Asphalt Viscosity Data

A. W. SISKO
Research and Development Department, American Oil Company, Whiting, Indiana

A simple and convenient cone-plate viscometer has been developed for asphalts. It determines viscosities from 10^2 to greater than 10^{10} poises at temperatures from below 32 to 156 F and at shear rates from 10^{-3} to 10^2 sec^{-1}, requires no more than 1.5 g of sample, and may be used with or without a recorder. Three asphalts have been studied in detail. When the viscosities at various shear rates and temperatures are shifted to a selected reference temperature, they yield a master curve covering a wider range of shear rates than is possible by direct measurement. The master curve correlates shear rates and temperature encountered in service, in stability tests, and in viscosity measurements for specification purposes. A simple equation fits the master curve well and can be combined with the Walther equation to give an equation describing the dependence of viscosity on both shear rate and temperature.

•THE STRENGTH of a bituminous material, in service at a particular temperature, is dependent on the mechanical properties of the asphalt at that temperature. Relevant properties are the elasticity and viscosity, and instruments for their determination have been designed (1, 2, 3). However, these instruments are complicated and for many situations, such as in deformation by rutting (4), for creep under steadily applied loads, and for specification purposes (5), the viscosity sufficiently describes the mechanical behavior of the asphalt. A difficulty with viscosity measurements is that asphalts, in service and in stability tests, encounter shear rates and temperatures covering ranges too wide to be duplicated with a single viscometer. The difficulty can be overcome by the use of a viscometer that covers as much of these ranges as is practical and by treatment of the viscosity data with methods that in effect extend the data to cover the desired ranges of shear rates and temperatures.

The requirements for such a viscometer are that it be rugged, preferably require small samples (such as those recovered from thin film tests), and because asphalts are non-Newtonian fluids be operable over a range of shear rates. Parallel-plate (6) and cone-plate viscometers (7, 8) meet these requirements. However, cone-plate instruments have the advantages of a wider range of shear rates and of requiring neither the sample density nor weight. These advantages and the absence of a suitable commercial instrument prompted the development of a simple and rugged cone-plate viscometer that can be used with or without a recorder.

CONE-PLATE VISCOMETER

Description

Cone-plate viscometers provide means for determining the drag produced by a sample sheared between the cone and the plate. In this instrument, torques are applied to the cone by weights and angular velocities of the cone are measured.

Paper sponsored by Committee on Characteristics of Bituminous Materials and Means for Their Evaluation.

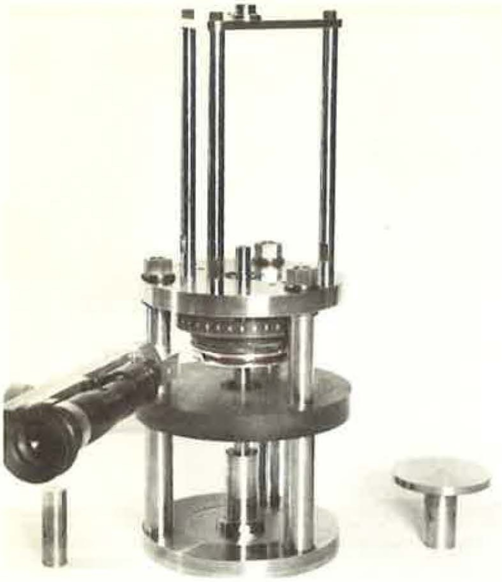

Figure 1. Viscometer.

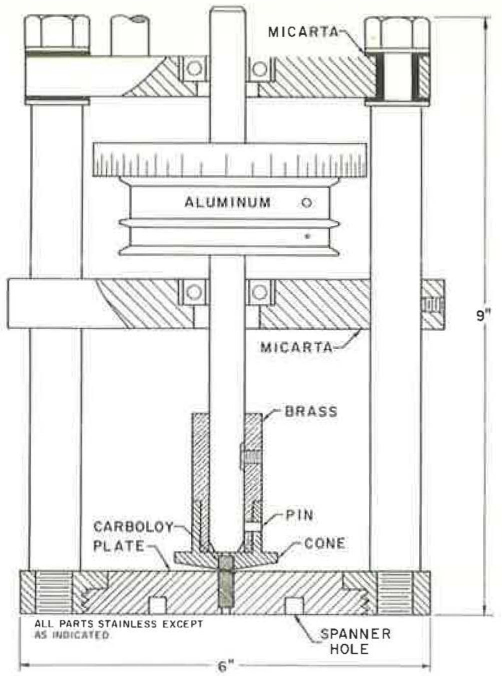

Figure 2. Assembly view of viscometer.

The viscometer (Fig. 1) consists of a stainless steel frame, composed of three plates and support rods, containing a central vertical shaft. Above the frame are three rods and a plate with a bubble level for supporting and leveling the instrument in a thermoregulated bath. Construction details are shown in Figure 2. Precision bearings (SKF 6201 Y/C782) align the central shaft radially but allow free vertical movement. The top plate is electrically insulated from the support rods by micarta washers and sleeves. The middle plate is made of micarta. The bottom plate consists of a ring, into which is threaded a precision ground plate. A pulley and a cone are attached to the vertical shaft. The pulley is slotted to receive the knotted end of a cord. About five turns of cord are wrapped around the pulley and led over a second pulley at the edge of the laboratory bench.

Torques are produced by hanging weights (10 to 25,000 g have been used) from the free end of the cord. Angular velocity is measured by timing the displacement of a drum dial on the instrument pulley. The dial is read by sighting through either a 10-power microscope mounted on the instrument or a cathetometer. Alternatively, a galvanometer light focused on a small mirror attached to the shaft and reflected to a frosted glass scale could be used for observing motion of the cone. Angular velocity might also be determined by recording the output of a rotary variable differential transformer at the top of the shaft.

The cones have large apex angles but differ in diameter. In Figure 2 the cone angle is exaggerated. The cone tip and plate center are made of wear-resistant Carboloy. Cone diameters are approximately 1.9, 3.8, and 7.6 cm, and the respective sample requirements are approximately 0.002, 0.02, and 1.5 g. Cone selection is based on temperature and viscosity; the large cone is used at high temperatures or for materials with low viscosities, and the small cone below 40 F or for very viscous materials.

A sample is prepared for measurement by placing it on the plate beneath the raised cone, and then letting the cone settle. An ohmmeter, placed across the top and bottom plates, indicates when the cone and the plate are in contact. Applying a hot plate to the base and a kilogram weight to the top of the shaft shortens loading time. The instrument is then cooled to room temperature and finally mounted in a temperature-

regulated bath with the bottom of the micarta plate approximately 1 in. above the thermostatted liquid.

Calculations

Viscosity, η, is determined as a function of shear rate, D, or shear stress, S. These are given by:

$$\eta = \frac{S}{D} = \frac{3\,T\,\psi}{2\,\pi\,R^3\,\Omega} \tag{1}$$

$$D = \frac{\Omega}{\psi} \tag{2}$$

$$S = \frac{3T}{2\,\pi\,R^3} \tag{3}$$

in which T is torque in dyne centimeters, ψ is cone angle in radians, R is cone radius in centimeters, and Ω is angular velocity in radians per second. Viscosity, shear rate, and shear stress are in poises, reciprocal seconds, and dynes per square centimeter, respectively. In an actual run, angular velocities are measured in degrees per second for various loads in grams.

The effects of various construction errors have been analyzed (8). Errors arise from friction, drag at the edge of the cone, misalignment of cone and shaft axes, and rounding of the cone tip. Because these errors cannot be completely eliminated, the instrument is calibrated with a liquid of known viscosity. National Bureau of Standards Oil P at 30 C, with viscosity of 461.2 poises, was used. Data for Oil P with the medium cone are shown in Figure 3. Friction prevents the plotted line from passing through the origin. The equation of the line is

$$\omega = -0.490 + 0.2481\,L \tag{4}$$

in which ω is angular velocity in degrees per second and L is weight in grams. Solving for L at zero angular velocity gives the weight, f, required to overcome friction (1.98 g for the medium cone). The shear stress is

$$S = \frac{3\,rg\,(L - f)}{2\,\pi\,R^3} = k_1(L - f) \tag{5}$$

in which r is the pulley radius plus the cord radius and g is the gravitational constant.

Errors other than from friction are incorporated into an effective cone angle, calculated from the slopes of Figure 3:

$$\psi = \eta\,\frac{d\Omega}{dS} = \frac{461.2\,(2\pi)}{k_1\,(360)}\,\frac{d\,\omega}{d\,L} \tag{6}$$

The effective cone angle is within 6 percent of the value determined by measurement with a sine bar and precision gage blocks. This difference affects only the non-Newtonian portion of the flow curve and is negligible for the asphalts studied. Shear rate in degrees per second is given by

$$D = \frac{2\,\pi}{360\,\psi} = k_2 \tag{7}$$

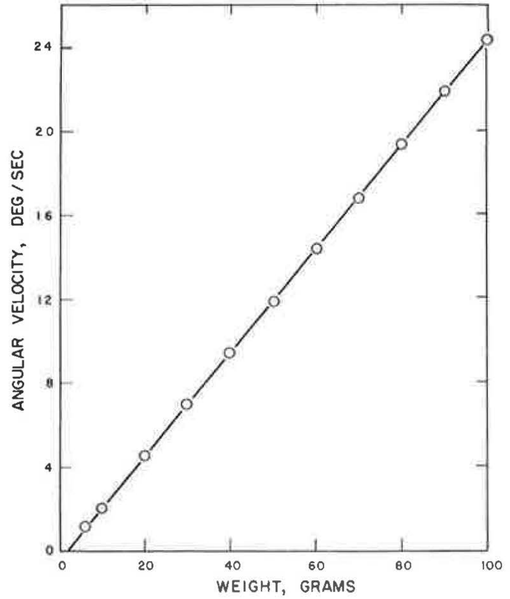

Figure 3. Calibration of instrument.

Constants for the three cones are given in Table 1. For construction purposes, angles are in the range 0.45 to 0.50 degrees. The range of the instrument may be further extended by using cones of larger angles.

TABLE 1

CONE CONSTANTS

Cone	Radius (cm)	Angle (rad)	f(g)	k_1	k_2
Small	0.850	8.622×10^{-3}	3.96	2009	2.015
Medium	1.905	8.016	1.98	249.2	2.177
Large	3.812	8.086	2.53	31.09	2.159

Results and Discussion

In viscosity determinations on asphalts, complications arise due to thixotropic, elastic, and normal stress effects. Thixotropy results in a decrease in viscosity with time of shearing. Elasticity is evidenced by sample strain without flow immediately after application of the torque. The normal stress effect causes the sample to creep out of the gap between the cone and the plate. At high temperature the three phenomena are less serious. The instrument is operated to minimize these complications. A run is started with a heavy weight, before going to lighter weights, to reduce thixotropic effects. A similar procedure is effective with the parallel-plate viscometer (9). Weights are removed after each reading to minimize rotation and give the operator time to record the data. The effect of elasticity is reduced by allowing the cone to rotate at least 1° after each weight application before angular displacement is timed. Normal stress effects are minimized by making measurements with a minimum rotation of the cone.

Data for a typical run on a 57-penetration paving asphalt at 77 F are shown in Table 2. The data cover a 660-fold range of shear rates with less than ½ rev of the cone. Fracture occurs within the asphalt, not at the asphalt-metal interface.

Comparisons were made of viscosities determined with the parallel-plate and cone-plate instruments. Figure 4 gives results obtained at two temperatures on a paving asphalt using the medium cone. The agreement between the two viscometers is good. The data demonstrate the wider range covered by the cone-plate instrument. Use of the large cone would have extended the data at 122 F to below 10^{-2} sec^{-1}.

When cooled from mixing and sampling temperatures, asphalts slowly develop an internal structure. This phenomenon, steric hardening, also occurs in samples stored in the viscometer. Figure 5 shows how the viscosity of an asphalt held in the viscometer at 77 F for 3, 18, and 65 hr before running at 77 F increases with time. At high shear rates, differences in viscosity are less than at low shear rates, indicating that shear breakdown (thixotropy) is greatest for the sample with the most developed internal structure. For routine use, standard holding times of 0.5 hr at 77 F or of 1 hr at 39.2 F have been adopted.

TREATMENT OF DATA

Extensive measurements were made on three paving-grade asphalts. Inspection properties of the asphalts are given in Table 3. Asphalts A and B were prepared

TABLE 2

RESULTS OF TYPICAL TEST WITH VISCOMETER[a]

Weight (g)	Deg	Time (sec)	D (sec^{-1})	η (poises)	S ($dynes/cm^2$)
300	2	825.0	5.28×10^{-3}	1.41×10^{7}	7.44×10^{4}
100	1	1,370.1	1.59	1.54	2.45
300	1	378.2	5.76	1.29	7.43
500	1	209.1	1.04×10^{-2}	1.19	1.24×10^{5}
1,000	1	83.9	2.59	9.60×10^{6}	2.49
2,000	3	81.3	8.03	6.20	4.98
4,000	6	45.7	2.86×10^{-1}	3.48	9.95
8,000	20	33.9	1.28×10^{0}	1.56	2.00×10^{6}
12,000	55	34.1	3.51	8.52×10^{5}	2.99
16,000		sample fractured			

[a]Sample of 57-penetration paving asphalt.

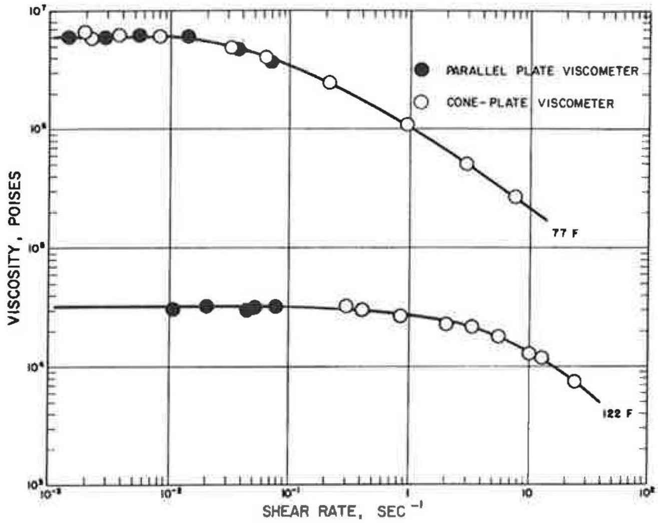

Figure 4. Comparison of parallel-plate cone-plate viscometer.

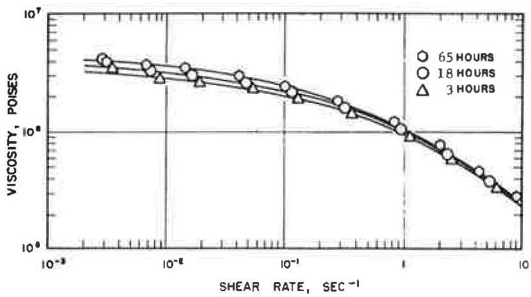

Figure 5. Dependence of viscosity on sample age.

by reduction of Midcontinent and Venezuelan crudes. Asphalt C was prepared by oxidation of a reduced Venezuelan crude. Viscosities for the asphalts are shown in Figures 6, 7, and 8.

Each asphalt forms a family of curves of the same general shape. The curves extrapolate to limiting viscosities at low shear rates and to a common slope at high shear rates. The similarity suggests that the data can be combined in single master curves, one for each asphalt, by vertical and horizontal shifts. The shifts are easily accomplished when the data are plotted on tracing paper having log-log coordinates. This is a common procedure in rheology and has been used for dynamic mechanical properties of asphalts (10, 11).

Vertical shifts are made to obtain coincidence of the limiting viscosities at a selected reference temperature. The amount of shift is determined from a plot of limiting viscosity against temperature. In Figure 6, for example, limiting viscosities are readily found from the data

TABLE 3

INSPECTION PROPERTIES OF ASPHALTS

Asphalt	Penetration[a]	Viscosity (poises) At 275 F	Viscosity (poises) At 250 F
A	55	6.99	15.2
B	60	6.48	13.6
C	154	2.91	6.70

[a]Of 100 g at 77 F in 5 sec.

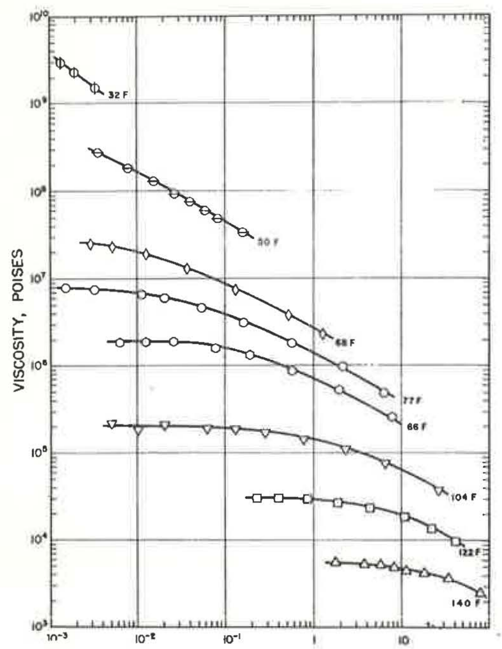

Figure 6. Flow curves for asphalt A.

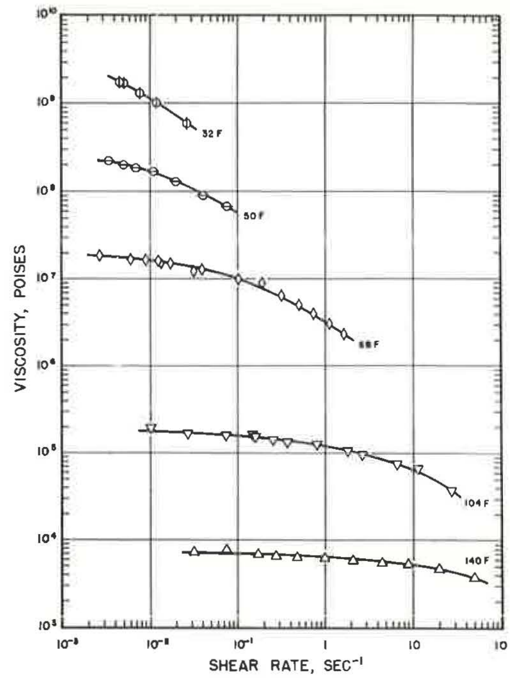

Figure 7. Flow curves for asphalt B.

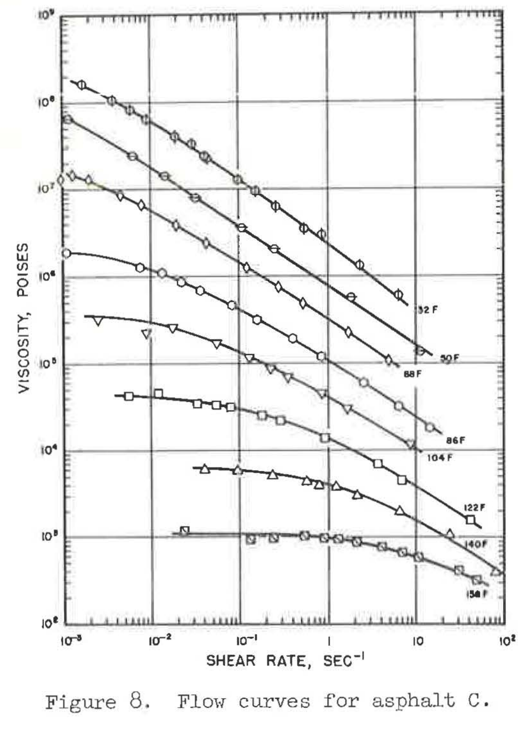

Figure 8. Flow curves for asphalt C.

at temperatures from 77 to 140 F. These limiting viscosities and capillary viscometer data at 250 and 275 F (where non-Newtonian flow is negligible) are plotted on a graph with coordinate spacings based on log log 100 η and log T (Walther coordinates), in which T is absolute Fahrenheit temperature. Such a plot is shown in Figure 9 for asphalt A. Generally these plots are straight lines or are slightly curved. Limiting viscosities at other temperatures are found by interpolation or extrapolation. Reference temperatures for asphalts A, B, and C were arbitrarily selected at 77, 68, and 86 F.

Horizontal shifts are needed after application of the vertical shifts to obtain coincidence of the individual curves at high shear rates. Figure 10 shows the amount of horizontal shift required. Curves with factors greater than 1 are shifted to the right, those with factors less than 1 are shifted to the left. The direction of the shifts shows that non-Newtonian flow begins at higher shear rates as temperature is increased.

Master curves obtained by these shifts appear in Figures 11, 12, and 13. The

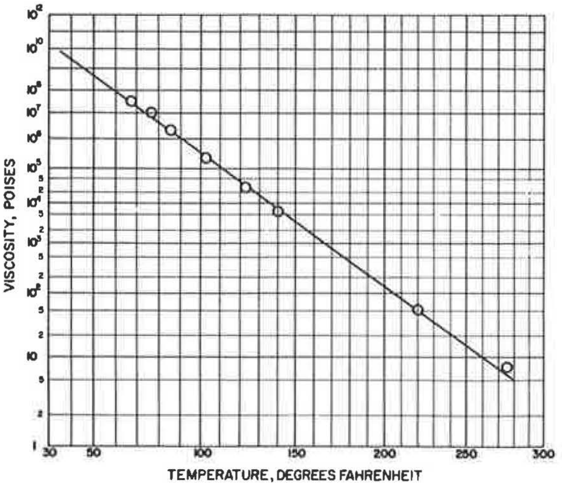

Figure 9. Effect of temperature on initial viscosity of asphalt A.

coordinates are reduced viscosity, resulting from the vertical shift, and reduced shear rate—the product of the horizontal shift factor α and the shear rate. The curves cover wider ranges of shear rates than is possible with any one viscometer. Starting with a master curve and applying the shifts yields viscosities over the complete range—from the limiting viscosity at low shear rates to the limiting slope at high shear rates—at temperatures where the limiting conditions are relatively inaccessible to experimental measurement.

An equation in good agreement with the flow curves in Figures 6, 7, and 8 has been found. At any temperature, the fluidity φ (reciprocal of the viscosity) is the sum of the limiting fluidity at low shear rates φ_0 and a fluidity that has a power dependence on shear rate:

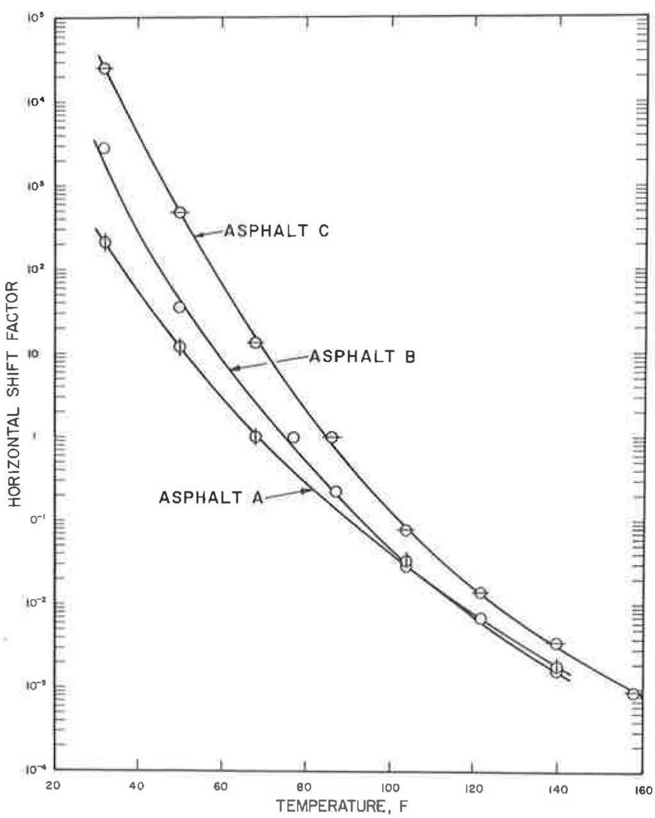

Figure 10. Horizontal shift factors for asphalts.

$$\varphi = \varphi_o + bD^n \tag{8}$$

In this equation, b and n are constants with n the limiting slope at high shear rates when log fluidity is plotted as a function of log shear rate. In terms of viscosity the equation becomes

$$\eta = \frac{\eta_o}{1 + \eta_o bD^n} \tag{9}$$

in which η_o is the limiting viscosity at low shear rates. When the constants are known for the reference temperature, flow curves at other temperatures can be calculated from the more general equation:

$$\eta = \frac{\eta_o}{1 + CD^n} \tag{10}$$

in which $C = \eta_o^* b^* \alpha^n$. Constants η_o^*, b^*, and n are those for the reference flow curve; η_o and α are values appropriate to the temperature for which the flow curve is to be calculated.

At the reference temperature, where $\alpha = 1$, the equation of the master curves in Figures 11, 12, and 13 has the constants given in Table 4. These data are insufficient for assessing how generally the narrow range of n, found here, applies to other asphalts. The agreement between the calculated master curve and the data for each asphalt, over 5 to 10 decades of shear rate, is generally good.

The dependence of η_o on temperature is given by the Walther equation (12):

$$\log \log 100\,\eta_o = a - m \log T \tag{11}$$

in which a and m are constants. In the absence of phase changes the Walther equation has been found reliable for most asphalts in the temperature range 150 F and below, and for many asphalts the equation is a good approximation up to temperatures of 275 F. Walther constants are given in Table 5.

The dependence of viscosity on both shear rate and temperature is given by the equation:

$$\log \log 100\,\eta\,(1 + CD^n) = a - m \log T \tag{12}$$

This equation yields a family of curves, one for each temperature, on plots of viscosity as a function of shear rate (such as appear in Figures 6, 7, and 8) or a family of curves, one for each shear rate, on Walther plots.

Curves of the second type are shown in Figures 14, 15, and 16. The plots are easily interpolated to find viscosities at intermediate shear rates. Asphalt C is appreciably non-Newtonian at the specification temperature of 140 F and this behavior could interfere with agreement between the different vacuum capillary viscometers used at 140 F. Much of the curves in Figures 14, 15, and 16 can be found by plotting viscosities inter-

TABLE 4

CONSTANTS OF EQUATION OF MASTER CURVES

Asphalt	η_o^*	b^*	n
A	8.33×10^6	6.05×10^{-7}	0.692
B	2.00×10^7	2.70×10^{-7}	0.700
C	2.40×10^6	9.71×10^{-6}	0.691

TABLE 5

WALTHER CONSTANTS

Asphalt	a	m
A	11.0712	3.7081
B	10.9104	3.6505
C	10.3381	3.4661

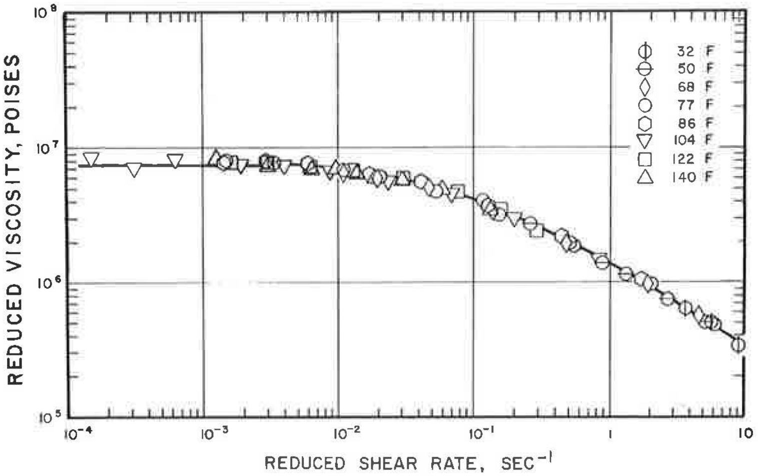

Figure 11. Master curve for asphalt A.

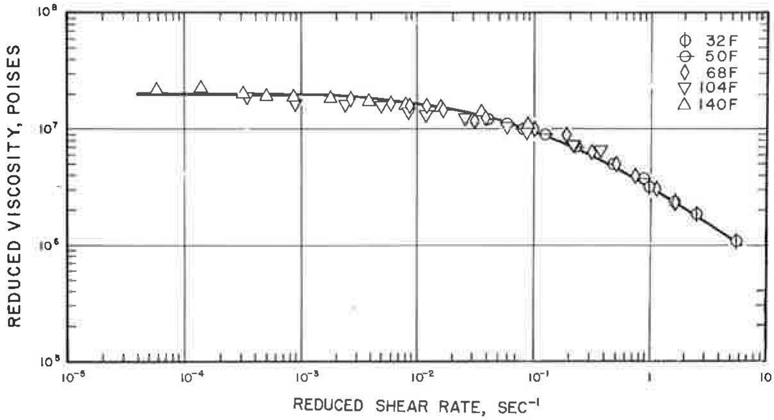

Figure 12. Master curve for asphalt B.

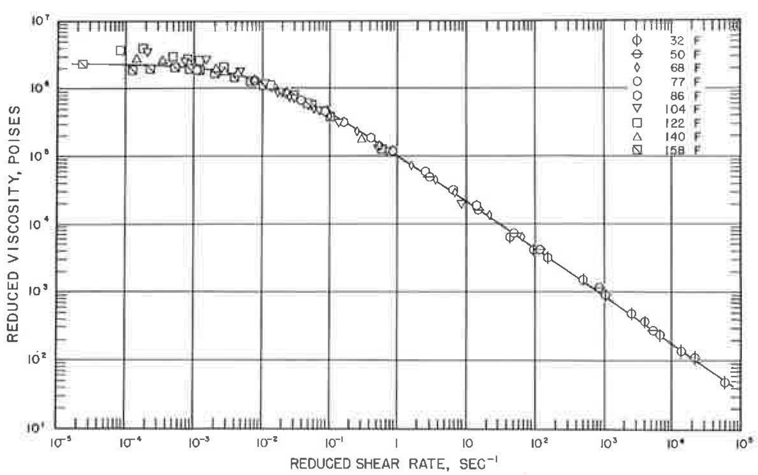

Figure 13. Master curve for asphalt C.

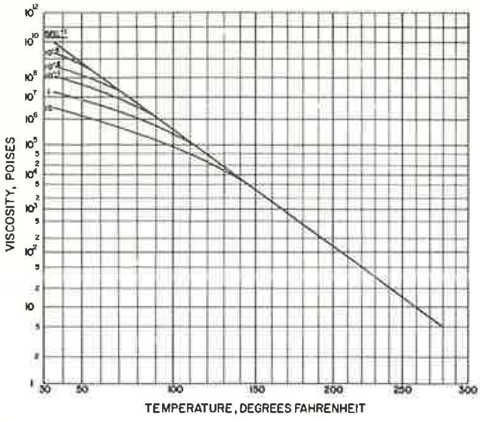

Figure 14. Effect of shear rate and temperature on viscosity, asphalt A.

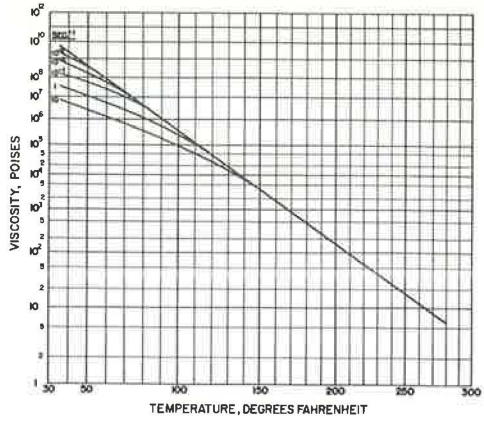

Figure 15. Effect of shear rate and temperature on viscosity, asphalt B.

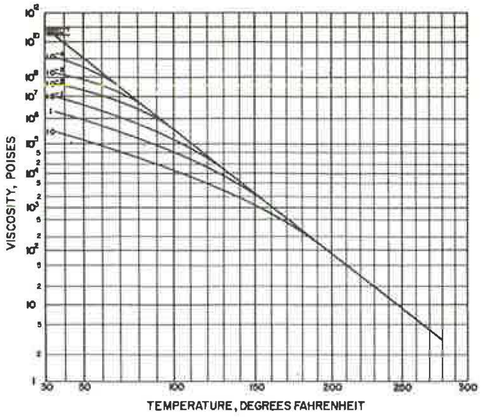

Figure 16. Effect of shear rate and temperature on viscosity, asphalt C.

polated from flow curves at different temperatures, thereby offering a simpler but limited method for portraying the effect of both shear rate and temperature on viscosity.

CONCLUSION

A cone-plate viscometer and an operating procedure for minimizing the complications of elasticity and time-dependent flow have been developed for asphalts. The instrument covers a wider range of shear rates with small samples than do other asphalt viscometers.

Two methods of treating the data are attractive. One method requires shifting viscosities determined at various shear rates and temperatures to yield a master curve at some reference temperature; the other simply displays the data on a plot of viscosity against temperature as a family of curves, one for each shear rate.

A simple equation describing asphalt flow is combined with the Walther equation to yield the dependence of viscosity on both temperatures and shear rate. Either the combined equation or the master curve technique describes asphalt flow over a range of shear rates and temperatures wide enough to include most conditions encountered

in service, in stability tests, and in viscosity measurements for specification purposes.

ACKNOWLEDGMENTS

The author wishes to thank L. C. Brunstrum for helpful suggestions and A. T. Jancosek for careful experimental work and calculations.

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