Low Temperature Measurement of Asphalts for Viscosity and Ductility

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ABSTRACT

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The main purpose of this research was to search for a method of characterizing asphalt viscosity at temperatures of 77 F and below.

A cone-plate viscometer was found to be useful for measuring viscosities down to about 23 F and up to 140 F. This, combined with measurement procedure for glass transition temperature of asphalts (which was developed by Tons and Funazaki in 1972-73 studies), provides a procedure for showing asphalt viscosity between 140 F and temperatures below 0 F (at the glass transition point). Once such viscosity curves are available, they can be correlated with field performance and specified limits for different grades of asphalts can be established.

The work also involved preliminary viscosity-penetration studies at 77 F for establishing criteria for changing to viscosity grading of asphalts in Michigan.

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DISCLAIMER

The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the Michigan State Highway Commission or The University of Michigan.

LOW TEMPERATURE MEASUREMENT
OF ASPHALTS FOR VISCOSITY AND DUCTILITY

INTRODUCTION

Asphalts used in pavements are subjected to a wide range of temperatures. During mixing and placing operations of bituminous mixes temperatures above 300 F may be encountered, while on the road in Michigan 0 F and below is not uncommon.

At present, the viscosity of road asphalts is often measured at one or more of the following temperatures:

(1) At 275 F when the asphalt is in a relatively liquid state.

(2) At 140 F which may be assumed as the highest temperature under service conditions in a road.

(3) At 77 F (25C) or "room temperature" which also coincides with the standard temperature for penetraion test.

There are no standard methods for viscosity measurement at temperatures below 77 F and no agreed-upon method even for 77 F. Since average temperatures in the compacted bituminous mix on roads in Michigan are below 77 F, the need for a method to make practical viscosity measurements at 77 F and below is apparent. Since previous work had resulted in a method for measuring a glass transition temperature for various asphalts used in Michigan, the connection between the asphalt flow characteristics at warm temperatures and the glassy state is of interest.

PURPOSE AND SCOPE

The main purpose of this research was to find a method (or methods) for practical measurement of flow characteristics for road asphalts in the low temperature range. The work done included:

(1) Literature review and search for relatively simple methods and equipment which are presently available for measuring low temperature characteristics of asphalts.

(2) Adaptation and development of procedures for performing physical low temperature measurements on asphalts.

(3) Designing experimental procedures for testing and analysis of data.

(4) Performing of tests on original and available recovered asphalts.

(5) Using available literature and experience, attempted to outline criteria for desired physical characteristics of asphalt so as to reduce destructive cold weather influences on compacted bituminous mixtures.

(6) Write final report, including procedure for practical applications of the results.

The above six goals were outlined originally. Work was done in all of the above areas, but due to practical needs for viscosity grading of asphalts in Michigan, a considerable amount of time was spent on viscosity-penetration characteristics of asphalts at 77 F.

LITERATURE REVIEW

There are a number of investigators who have attempted to make viscosity measurements with various instruments and at different temperatures. One of the most extensive surveys on viscosity and consistency measurements was done by Neppe (1).* The various methods and types of apparatus employed are summarized in Table 1. Neppe among other things pointed out the importance of shear stress in viscosity determination. In extreme cases one viscometer employing a certain shearing stress with two given asphalts may show that asphalt A is more viscous than asphalt B; by changing to another instrument and shearing stress asphalt B may show a higher viscosity than asphalt A. The approximate values of shearing stress in some well known tests are (in dynes per square cm):

Thin Film Flow Test	100-500
Redwood II Viscometer	200
Sinker Viscometer	1,000

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*Numbers in parentheses denote numbers in Bibliography

R & B Softening Point	1,000
Penetration 300	100,000
Penetration 30	1,000,000
Penetration 3	10,000,000

Neppe also discussed the graphical representation for viscosity-temperature relationship and found that there are some six different graphical ways with a main goal to obtain a straight line relationship between the two above variables. From these methods, the log log viscosity versus log absolute temperature was found to be quite accurate for a wide range of temperatures. This graphical method has gained an official acceptance by the American Society for Testing and Materials and is standardized under ASTM D 2493-68.

Neppe also made a comprehensive survey of various aspects of the standard ring and ball softening point, penetration, and ductility tests and their interrelationship. Especially the relationship between penetration measurements and absolute viscosity is of great interest. He cited various researchers with the following examples being quite well known:

$$\eta = 5.13 \times 10^{9}/\text{Pen}^{1.93} \quad \text{(Saal and Koens)}$$

$$\eta = 6.25 \times 10^{9}/\text{Pen}\ (\text{Pen}-K^{1}) \quad \text{(Pendleton)}$$

where:

η = absolute viscosity in poises

Pen = penetration at 77 F

K_1 = Pen (0.849-0.00788 Pen) - 0.0562
(for penetration values below 54)

K_1 = 23 (for penetration values above 54)

One of the practical problems with the viscometers listed in Table 1 is the limited ranges of viscosity (and temperature) at which they can be operated. For instance, to measure asphalt viscosity between say 20 and 100 F no instrumentation can be found in Table 1. This fact has kept researchers looking for other ways of measuring viscosity of asphalts.

Sliding plate viscometer has been investigated by a number of researchers (3)(4)(5)(6). Griffin et al. (3) claimed that the instrument was sufficiently accurate and simple to permit bringing the measurement of viscosity in fundamental units into the testing laboratory and eventually into asphalt specifications. The practical range of viscosity that can be determined using the instrument was suggested between 10^2 to 10^{11} poises. An example of the repeatability of the instrument is presented in Table 2.

Another viscometer which has gained popularity is so-called cone and plate viscometer developed by Markowitz (2). The sample was sheared in a gap between a stationary truncated cone and a rotating flat plate. A known torque was applied to the plate and the resulting angular velocity was measured. Liquids with viscosities from 1 to 5×10^4 poises were investigated with rates of shear from 2×10^{-4} to 9×10^3 $\sec^{-1}$.

A relatively simple and convenient cone-plate viscometer which can be used for asphalt was developed by Sisko (9). According to Sisko the instrument can be used for determining viscocities between 10^2 to 10^{10} poises at shear rates from 10^{-3} to 10^2 sec^{-1}. This permits to measure asphalt viscosities from around 32 F or below to 150 F. Using this apparatus viscosity of any asphalt can be determined at a given temperature and shear rate and within the capabilities of the instrument. To obtain a more detailed picture, tests at different temperatures and shear rates were performed and analyzed using two different methods.

Method 1

Step 1. Measure asphalt viscosity at various temperatures and shear rates. Plot a log log shear rate versus log viscosity as shown in Figure 1. Each asphalt will form a family of curves the same general shape. These curves usually extrapolate to limiting viscosities at low shear rates and to a common slope at high shear rates.

Step 2. Make one composite curve (master curve) by shifting the flow curves in Figure 1 down or up (vertically) and to the left or to the right (horizontally). Vertical shifts are needed to obtain the overlap or coincidence of the limiting viscosities at a selected reference temperature. As seen from Figure 2, the coordinates of the master curve are: (a) reduced viscosity, derived from the vertical shift made to obtain coincidence of the limiting viscosities at a selected reference temperature, and (b) reduced shear rate, which is the product of the horizontal shift factor and the shear rate. Horizontal shifts are needed after application of the vertical shifts to obtain coincidence of the individual curves at high shear rates.

Step 3. Plot the amounts of horizontal shift required during operations in Step 2 (see Figure 3).

Step 4. Plot the limiting viscosities in Walther coordinates, i.e., log log viscosity (centipoises) versus log absolute temperature (absolute temperature = F + 459.7). Limiting viscosities at other temperatures can be found from this plot (see Figure 4).

Using Figures 2, 3 and 4 viscosities can be calculated at any desired shear rate and temperature. An actual numerical example of how this is done will be given for one Michigan asphalt in the Analysis of Data Section.

Method 2 (Steps 1 to 4 the same as in Method 1)

Step 5. Calculate two constants, "b" and "n" from the master curve (Figure 2) using the equation:

$$\varphi = \varphi_0 + b\ D^n$$

where:

$\varphi = 1/\eta$ (fluidity, reciprocal of viscosity)

φ_0 = limiting fluidity at low shear rate

D = shear rate

b and n are constants

The above equation generally represents the flow curves in Figure 1. For the master curve the following equation is used:

$$\varphi^* = \varphi^* + b^* D^n \qquad (1)$$

Where the values with * imply values for the master curve, the constants b^* and n are calculated from the limiting slope of the master curve at high shear rates.

Equation (1) can be also written as:

$$\log (\varphi^* - \varphi_0^*) = \log b^* + n \log D$$

In terms of viscosity the equation becomes:

$$\log \frac{1}{\eta^*} - \frac{1}{\eta_0^*} = \log b^* + n \log D \qquad (2)$$

If the coordinates of two points located on the master curve (limiting slope part) are used, an equation can be set up for the two points:

$$\log (\frac{1}{\eta_1^*} - \frac{1}{\eta_0^*} = \log b^* + n \log D_1$$

$$\log (\frac{1}{\eta_2^*} - \frac{1}{\eta_0^*} = \log b^* + n \log D_2$$

where (D_1, η_1^*) and (D_2, η_2^*) are coordinates of the two points.

Solving for n and log b*

$$n = \frac{\log\left(\frac{1}{\eta_1^*} - \frac{1}{\eta_0^*}\right) - \log\left(\frac{1}{\eta_2^*} - \frac{1}{\eta_0^*}\right)}{\log D_1 - \log D_2}$$

$$\log b^* = \log\left(\frac{1}{\eta_1^*} - \frac{1}{\eta_0^*}\right) - n \log D_1 =$$

$$= \log\left(\frac{1}{\eta_2^*} - \frac{1}{\eta_0^*}\right) - n \log D_2$$

Next, the Walther constants m and a can be obtained:

$$m = \frac{\log \log 100\, \eta_{01} - \log \log 100\, \eta_{02}}{\log T_2 - \log T_1}$$

$$a = \log \log 100\, \eta_{01} + m \log T_1 =$$

$$= \log \log 100\, \eta_{02} + m \log T_2,$$

where (T_1, η_{01}) and (T_2, η_{02}) are the coordinates of two points on the Walther curve.

Step 6. Calculate the viscosity (knowing that it varies with temperature and shear rate) using the following equation:

$$\log \log 100\, \eta\, (1 + c\, D^n) = a - m \log T,$$

where $c = \eta_0^*\, b^*\, \alpha^n$

and η_0^* = limiting viscosity at low shear rate

α = horizontal shift factor for the temperature under consideration,

b^* and n are constants calculated in Step 5,

D = shear rate,

a and m are Walther constants, and

T = absolute temperature.

The equation in Step 6 yields a family of curves, one for each temperature, similar to those in Figure 1, or a family of curves, one for each shear rate on Walther plots as shown in Figure 5. The calculations here are best done by using a computer.

Both of the above methods can describe the flow characteristics of asphalts over a range of shear rates and temperatures which include most conditions found in stability tests, viscosity measurements for specification purposes and other service conditions.

During the initial planning and review stage consideration was also given to methods other than viscosity measurements. Ductility testing at lower temperatures was considered, but the indications were that interpretation of ductility data for practical meaning and use would be difficult.

A brief look was also taken at the dynamic methods of measuring asphalt properties. Work in this area has been done by a number of investigators (10) (11) (12) (13) (14). Dynamic experiments are geared to obtain information on the behavior of asphalt subjected to short-time tests. Since asphalts are viscoelastic liquids with short-time behavior similar to elastic solids, such as glass, long-time behavior is similar to a viscous liquid, while at intermediate times their behavior includes elements of both extremes. If the loading time is fixed, low temperatures lead to short-time behavior and high temperatures are associated with long-time response. Dynamic measurements can be used to investigate the low temperature behavior of asphalt and the end product usually is shear modulus (equivalent to stiffness).

Viscosity and shear modulus can be interrelated under limited conditions. Only in the case of linear viscoelastic behavior (13)(14)(15) the relationship between viscosity and shear modulus is given:

$$\eta^* = G^* \omega,$$

where η^* = complex viscosity

G^* = complex shear modulus

ω = angular velocity, reciprocal of loading time

It has been shown that this closely resembles the steady flow viscosity as a function of shear rate (15)(16):

$$\eta = \tau \dot{\gamma}$$

where η = steady flow viscosity

τ = shear stress

$\dot{\gamma}$ = shear rate

Thus one can construct log G versus log ω graph from Log τ versus log $\dot{\gamma}$ plot (13).

Finally, the lowest point which may be considered on the asphalt consistency curve is so-called glass transition temperature. The glass transition point is defined as the temperature at which asphalts change from a "fluid" to a glassy condition. At temperature below the glass transition, asphalt is assumed to behave like an elastic material.

It has been proposed that the viscosity of asphalt at any temperature can be calculated if the glass transition point and viscosity at another higher convenient temperature is known (17).

So-called Williams, Landel and Ferry (WLF) equation is used for this purpose:

$$\log \frac{\eta_1}{\eta_2} = \frac{c_1 (T_2 - T_g)}{c_2 + T_2 - T_g} - \frac{c_1 (T_1 - T_g)}{c_2 + T_1 - T_g}$$

where η_1 = viscosity to be calculated at temperature T_1

η_2 = given viscosity at temperature T_2

T_g = experimentally determined glass transition temperature

c_1 = 17.44 (constant)

c_2 = 92.88 (constant)

The constants, c_1 and c_2 were established empirically by Williams, Landel and Ferry (21). They appear to be satisfactory for a wide range of organic polymers and some inorganic glasses.

Viscosity at glass transition temperature can be calculated by substituting T_g for T_1 :

$$\log \frac{\eta_g}{\eta_2} = \frac{c_1 (T_2 - T_g)}{c_2 + T_2 - T_g}$$

where η_g = viscosity at T_g.

The application of knowledge of rheological properties of asphalts to performance in the field is in a development stage. Sisko and Brunstrum (12) investigated the correlation between pavement performance and rheological properties of asphalt binder. They found that large increase in hardness of asphalt with aging was associated with pavement cracking.

McLeod (18) has reported that low temperature transverse BC pavement cracking is likely to occur whenever the modulus of stiffness of a pavement attains a value of 1,000,000 psi at a pavement depth of 2 inches due to any critical combination of chilling to a low temperature, hardness of the asphalt cement, and other controlling factors. The factors that contribute to the attainment of the critical modulus value are:

(a) The penetration of asphalt at 77 F affects the modulus. When all other factors are equal, the higher the penetration at 77 F of the asphalt cement, the less is the low temperature cracking.

(b) The penetration-viscosity number (or penetration index) of the asphalt cement also affects the modulus. When all other factors are equal, the higher the penetration-viscosity number of the asphalt cement the less transverse cracking is expected.

EQUIPMENT SELECTED

One of the main difficulties with viscosity measurement and prediction for asphalts is that a wide range of shear rates and temperatures are encountered during service life of an asphalt pavement. To duplicate this in the laboratory several different instruments may be necessary. Presently at higher temperatures, say 140 and above, capillary viscometers (ASTM D 2170, D 2171) are used. However, at temperatures below 140 F no popular method for viscosity measurement has been established. The sliding plate viscometer, cone and plate viscometer, the dynamic methods, or other procedures can be used. After the review of the literature and private discussions with several leading personalities in the field of viscosity measurement, the cone-plate viscometer was decided upon as the most promising piece of equipment to measure viscosities below 140 F in this investigation. Two reasons may be added:

(1) The goal was to look for or develop an instrument which is simple enough to use for "routine" laboratory testing. The cone-plate appeared to approach this condition.

(2) The cone-plate procedure also appeared to be suitable for a wide range of temperatures and shear rates (using asphalt).

(3) The cone-plate procedure does not require measurement of sample density and weight.

ASPHALTS USED IN THIS INVESTIGATION

The asphalts used in this investigation were obtained from the MDSHT Bituminous Testing Laboratory. These asphalts were collected as samples during the years 1973 and 1974 for testing purposes. Altogether 43 different asphalts ranging from low to high penetrations were included in the various tests at different temperatures and shear rates. For the 1973 samples glass transition temperature (T_g) was investigated during a previous study (19) and the properties of these asphalts are also given here in Table 11.

LABORATORY MEASUREMENTS

Calibration of Equipment

The cone-plate viscometer was first calibrated using viscosity standard N 190000 with viscosity of 9194 poises at 68 F (see ASTM D445, D88, and D2161). The angular velocity in degrees per second was measured with different weights. Measurements were repeated 4 times without changing the sample.

The instrument comes with 3 sizes of cones and 2 different sizes of strings for applying the torque weights. Calibration was made also for the thick and thin string and each size of the cones.

The viscosity constant K_η was determined by first plotting weight (grams) versus angular velocity (degrees per sec. x 10^{-1}); then the slope of the line is determined:

$$K_\eta = b \times \eta$$

b = slope of weight - angular velocity line

η = 9194 poises.

Using this information, K_η was computed.

The shear stress constants were determined by measuring the radii of the cones and the drum and the thickness of the strings:

$$K_s = \frac{3R_g}{2\pi r^3}$$

where K_s = shear stress constant

R = sum of radii of the drum and the string

r = radius of cone

g = 980 cm/sec^2

The shear rate constants were calculated as follows:

$$K_D = \frac{K_s}{K_\eta}$$

where K_D = shear rate constant

K_s = shear stress constant

K_η = viscosity constant

The results of the calibration are summarized in Table 3.

Using the above constants viscosity, shear stress and shear rate can be calculated as follows:

$$\eta = K_\eta \; t/\theta \quad L$$

where η = viscosity (of asphalt) in poises

K_η = viscosity constant

t/θ = reciprocal of angular velocity (sec/deg)

L = weight on string, in grams

t = time in seconds

Shear stress calculation:

$$\tau = K_s \; L$$

where τ = shear stress (dynes/cm^2)

K_s = shear stress constant

L = weight on string, in grams

Shear rate calculation:

$$D = K_D\ \theta/t$$

where D = shear rate in $\sec^{-1}$

K_D = shear rate constant

θ/t = angular velocity (deg/sec)

Selection of Cone Size

The selection of cone size is based on the viscosity of the sample. Approximate ranges for each size of cone at shear rate of 2×10^{-2} $\sec^{-1}$:

Large cone - viscosity $\eta < 1.25 \times 10^{6}$ poises

Medium cone - $1.25 \times 10^{6} < \eta < 1.0 \times 10^{7}$

Small cone - viscosity $\eta > 10^{7}$ poises

The shear rate of 2×10^{-2} $\sec^{-1}$ as a "standard" was estimated to be convenient from a practical point of view. The above ranges were set so that while using a weight of 200 grams the angular velocity is around 1/100 degrees per second. This velocity was convenient for two reasons: (a) if the velocity is too fast, the accuracy of the measurement is poor; (b) if the velocity is too slow, it takes too long to run a test, especially for the lighter weights. Generally, the large cone was used for higher temperatures or asphalts with low viscosities and the small cone for temperatures below 40 F and viscous materials.

Sample Preparation and Testing

The detailed description of sample preparation and testing is given in Appendix A. Briefly, the sample is placed between the cone and plate assembly which is then brought to test temperature. Weights acting through a pulley apply torque to the cone and the angular velocity of the cone is measured. Viscosity in poises and shear rate in reciprocal seconds (sec^{-1}) are calculated from the angular velocity, torque and calibration constants.

A typical run is started with a heavy weight, before going to lighter weights to reduce thixotropic effects. Weights are removed after each reading to minimize rotation and give the operator time to record the data. Results for a typical viscosity run on a 64-penetration asphalt (73B-1) are shown in Table 4.

Analysis of Low Temperature Viscosity Data

All together more than 43 asphalts of various hardness were investigated. The test temperatures were: 23, 32, 41, 50, 59, 68 and 77 F, the largest number of tests being run at 77 F. The lowest practical shear rate was set at 2×10^{-2} sec^{-1} and could be designated as the "standard" rate for those experiments.

The first part of the laboratory work involved extensive measurements on two asphalts: (a) 60/70 pen (73B-1) and

(b) 120/150 pen (73B-23). The goal of this was to establish a characteristic viscosity curve for an asphalt between 140 F and a glass transition point T_g. This would approximate very well the whole range of temperatures to which asphalt is subjected during service on the road. In the laboratory viscosities of an asphalt were obtained at different temperatures and shear rates as shown in Figure 7. The data was then analyzed in accordance with methods used by Sisko (9) as discussed in the literature review.

Method 1

Flow curves for the two asphalts are shown by Figures 7 and 8 (Step 1).

Master curves were obtained by shifting the flow curves and they are shown in Figure 9. (Step 2). Horizontal shifts were made using 77 F as the reference temperature.

The amount of shift required for coincidence of the individual curves is shown in Figure 10 as the horizontal shift factor (Step 3).

Limiting viscosities at low shear rates are plotted on the Walther coordinates as shown in Figure 11 (Step 4). Using Figures 9, 10, 11 viscosities can be calculated at any desired shear rate and temperature as shown in the following calculations.

Example:

Calculate the viscosity of asphalt 73 B-1 at 50 F and shear rate of 10^{-2} $\sec^{-1}$.

Solution: Since there is no test data at 50 F, it is impossible to read the viscosity directly from Figure 7. Therefore, Figures 9, 10 and 11 will be used.

(1) Find the horizontal shift factor α at 50 F.

From Figure 10, $\alpha = 30$.

(2) Calculate the reduced shear rate. This is equal to α x desired shear rate $= 30 \times 10^{-2}$ $\sec^{-1} = 0.3$ $\sec^{-1}$.

(3) Find the reduced viscosity corresponding to the reduced shear rate using Figure 9. The reduced viscosity is read as 1.9×10^{6} poises.

(4) Find the ratio of limiting viscosity at low shear rates between viscosity at 50 F (desired temperature) and the reference temperature (77 F) from Figure 11.

$$\text{Ratio} = \frac{\text{Viscosity at 50 F}}{\text{Viscosity at 77 F}} \doteq \frac{3 \times 10^{8}}{3 \times 10^{6}} = 100 .$$

(5) The viscosity at 50 F and shear rate of 10^{-2} $\sec^{-1}$ can now be calculated:

$$\eta = \text{Reduced viscosity} \times \text{Ratio}$$
$$= 1.9 \times 10^{6} \times 100 = 1.9 \times 10^{8} \text{ poises.}$$

Method 2 (See literature review)

Viscosities were calculated using the equations proposed by Sisko (9). A computer program was developed for these calculations using data from

Figures 9, 10 and 11. The results for the two asphalts are shown in Figures 12 and 13. Viscosities at the glass transition temperature were calculated using 140 F for T_2 values, as discussed in the literature review (Reference 17).

In Figures 12 and 13, the straight line limiting the upper boundary of the curves may be close to Newtonian flow. It must be emphasized that at low temperatures the shear rate has to be very slow to realize a Newtonian behavior. For example, in Figure 12 at 10 F a 60/70 pen asphalt is already non-Newtonian at a shear rate of 10^{-4} sec $^{-1}$, which is very low for practical test runs. This problem is further illustrated in Figures 20 and 21 at 32 F, while Figure 22 with tests at 77 F indicates Newtonian behavior at shear rates of 2 x 10^{-2} or lower.

Since it is extremely difficult if not impossible to measure viscosities at high shear rates (such as 100 sec $^{-1}$) and low temperatures (say 32 F), Figures 12 and 13 were constructed using actual measurements and well established theories. The lowest temperature to which the curves at various shear rates were drawn is 20 F, which is close to some of the measured values using the cone-plate viscometer. The points from various shear rates were then connected with the viscosity values at the glass transition temperature. As the result of this procedure a very complete viscosity-temperature-shear rate relationship for any asphalt can be obtained. The temperature range can start at the mixing and end at the glass transition point. Below this point, the asphalt is assumed to behave elastically. The difference between the 60/70 pen asphalt in Figure 12 and the 120/150 pen asphalt in Figure 13 is in: (a) the starting points T_g on the left side, (b) the vertical coordinates, (c) the spread of the curves, and (d) the slopes of the main curves. These features could be used for practical classification of asphalts and will be discussed later under Practical Applications.

To obtain the curves in Figures 12 and 13 the tests were run at various shear rates. From this experience it was concluded that for practical applications of the cone-plate viscometer a shear rate of 2×10^{-2} sec^{-1} was convenient. To illustrate the applicability of this rate, the viscosity of six asphalts at different temperatures was measured and the results are shown in Table 6 and Figures 15 to 15-D on ASTM D 2493 paper. From this data it is concluded that the softer asphalts are close to Newtonian behavior (straight line) down to 23 F using the 2×10^{-2} sec^{-1} shear rate, while the harder asphalts show deviations below 40 F or so.

Sometimes it is desirable to represent the characteristic of asphalt in terms of stiffness rather than viscosity. This is true especially when the temperature is low and the asphalt behaves more or less like an elastic body.

When the linear viscoelastic behavior as discussed in the literature review (14) is considered, the shear modulus G can be obtained from a log τ vs log $\dot{\varepsilon}$ plot. This type of plot is shown in Figures 16 and 17 for the same two asphalts as in Figures 12 and 13. Using Figures 16 and 17 shear modulus and temperature relationship can be obtained as shown in Figures 18 and 19.

As shown in Figures 16 and 17 the linearity is represented by the straight line representing the slope of unity and any departure shows non-linear behavior. For the non-linear zone there is no established relationship between viscosity and shear modulus.

The limits for linear stress-strain relationship are rather low as shown in Table 4.

Analysis of Viscosity-Penetration Data at 77 F

Although the plan was to perform experimental measurements at 77 F, 68 F, 59 F, 50 F, 41 F, 32 F and 23 F (the lowest possible with the cone-plate viscometer) a large number of measurements were taken at 77 F. The primary reason for this was the relative urgency to consider switching to viscosity grading of asphalts in Michigan specifications. Thus, initial work was undertaken to study viscosities at 77 F for various asphalts used in Michigan. Since the standard measurement for asphalt consistency to date was penetration, first a correlation between viscosities measured at 77 F and 2 x 10^{-2} $\sec^{-1}$ shear rate and a standard penetration was checked. This work was expanded to include two other penetrations:

(a) Penetration at 77 F where the starting point (zero point) was not the tip of the needle, but 70 dmm of the needle was submerged in order to negate the effects of the truncated cone. With the truncated cone there is a tendency for erratic results with asphalts of lower penetration value due to the variance of the cross-sectional area of the truncated cone. This test is referred to as submerged penetration.

(b) The second revised penetration measurement was done using a needle with the truncated cone cut off and rounded with a radius equal to 1/2 of the needle's diameter. Reasons for this test are the same as for submerged penetration. This test is referred to as stub penetration.

The results on viscosity tests for 43 asphalts at 77 F are tabulated in Table 7. On each asphalt two or more replications were made. In a similar manner the data obtained on the various types of penetrations is given in Table 8. A regression analysis was run to compare the viscosity with various types of penetration and the basic results are shown in Figures 23 to 27 and tabulated in Table 9. For the viscosity-penetration comparisons in Table 8 two equations are given. The first one was obtained using all data as gathered during testing. The second equation is based on 40 samples, with three asphalts determined as highly shear rate dependent and not considered representative of the other 40 asphalts. From the correlation coefficients alone, the submerged penetration versus the viscosity gives the highest correlation coefficient. This is also indicated by bar graphs in Figure 30.

DISCUSSION

The work with the cone-plate viscometer has indicated that it is a useful tool for viscosity measurements between 23 and 140 F. At temperatures lower than 23 F the asphalts get easily fractured and repeatable data at shear rate of 2×10^{-2} is difficult to obtain. The viscometer was checked up to 140 F and it worked well. The flexibility in shear rates is also evident and range between 10^{-4} to 10^{2} sec^{-1} was tried. As seen from Figures 12 and 13, the higher shear rates can be used only at high temperatures. It would take a large force and the asphalt may fracture if high shear rates are used at low temperatures. If the viscosity values obtained at different shear rates and temperatures are used in conjunction with analysis such as illustrated by Sisko (9) viscosities over a wide range of temperature and shear rates can be achieved as shown in Figures 12 and 13. In addition, if the glass transition temperature point for the asphalt is known and the 275 F viscosity has been measured, a complete picture of asphalt consistency between mixing temperatures and cold weather service temperatures can be presented in one graph as shown in Figures 12 and 13. This procedure could be very useful to compare and classify various asphalts and a suggested practical approach will be presented later. It is realized that three types of testing is needed to obtain the complete range of viscosities:

(a) The glass transition temperature for which a measurement technique has been developed (19).

(b) The 275 F viscosity measurement using a capillary viscometer and 140 F.

(c) The intermediate viscosities using the cone-plate viscometer.

Since the 275 F viscosity is available routinely for every asphalt, the intermediate and low temperature areas are the ones where measurements are to be taken.

As mentioned before, a large number of viscosity and different penetration tests were performed at 77 F. Although one of the main reasons for emphasizing the 77 F area was the need for developing viscosity grading procedure for asphalts, it was also realized that the 77 F viscosity measurements may be the first stepping stone towards characterizing asphalt consistency at lower temperatures. Once a technique for measurement has been established and the technical personnel is familiar with it, further expansion is possible.

The measurement of viscosity at 77 F and shear rate of 2×10^{-2} appears to present no great problems. As shown by Figure 22 at shear rates of 2×10^{-2} or lower the asphalts show Newtonian behavior.

At present, the penetration test at 77 F is rather simple and maybe as many as 50 samples or more can be tested by 2 technicians during an 8-hour work day. The cone-plate viscometer efficiency in mass production has not been rigorously tested yet.

If a number of cone-plate sets were available in the laboratory the average test time per sample probably could compete quite favorably with regular penetration, because the waiting times between operations in the viscosity test are short.

The literature review showed that Saal with Koens and Pendleton also tried to correlate viscosities with standard penetration values. Table 10 shows how these two investigations compare with the present authors' investigation. Saal and Koens' equation was based on variations in penetration-viscosity data obtained on only two related samples simply by changing the temperature of the determinations (1). Pendleton used a large number of samples and a capillary viscometer (1). Figure 6 shows how viscosity measurements can differ with the type of viscometer. These factors may explain why there was some difference in the results of the three investigations.

While 40 of the 43 asphalts tested appear to be close to the regression line shown in Figure 23, three asphalts are "visually" different. These 3 asphalts, when tested with the cone-plate viscometer, were unusual when compared to the other 40 asphalts. At low shear rates, the viscosities were higher than other comparable grade asphalts, but at higher shear rates the viscosities dropped below that of other comparable grade asphalts. The switch to stub and submerged penetrations does change the position of the three points on the regression line with the lowest penetration asphalt changing most.

From empirical data a penetration reading of 25 dmm seems to correspond roughly to 2×10^{-2} $\sec^{-1}$. With the slower shear rate found in the stub penetrations, the correlation improves and in the slowest penetration test (submerged) the correlation is best.

If estimate of viscosity from penetration values is attempted, Table 9 shows that the standard penetration curve in Figure 23 will give an estimate with a 10% or higher error about 50% of time (48.9%). As shown in Table 9 for the submerged penetration this is decreased to about 33 percent (32.6). If viscosity estimate is desired from penetration, more revisions and refinements are needed in the penetration procedure.

PRACTICAL APPLICATIONS

Figures 12 and 13 give a very comprehensive picture of asphalt consistency between mixing and low winter service temperatures on the road. Every asphalt tested should have different T_g value, different viscosity at 275 F and different viscosities (for a given temperature) in between these two values. Thus, it should be possible to establish limits within which a standard series of viscosity points for a given grade asphalt should lay, as illustrated in Figure 14. It is apparent, that the high temperature (275 F) viscosity should have limits; also the slope of the temperature-viscosity curve should be regulated. In addition, the shear rate sensitivity could be regulated by using a standard 10^{-4} to 10^{2} sec^{-1} shear rate plots. The low temperature end could be left open allowing as low T_g values as they can be, as long as the rest of the curve system fits the limits. A minimum T_g could be specified for different climates. In summary, the T_g point should be generally outside the limit area while all other points are inside.

The actual limits for the glass transition temperature could be set at say -10 F. The viscosity limits for 275 F would depend on the grade of asphalt and should not be too difficult to establish. The rest of the limits need comparisons with field performance. Curves similar to those in Figures 12 and 13 should be compared with asphalts having good field performance and those associated with failures.

CONCLUSIONS

These conclusions pertain to a method of characterizing asphalt viscosity over the entire range of service temperatures. Initial conclusions on classifying asphalts according to viscosity at 77 F are also given.

(1) The cone-plate viscometer can be used for determining viscosities of asphalts at approximate temperatures between 23 F and 140 F. For practical laboratory use, a reasonably fast standard shear rate is desired. From this work a shear rate of 2×10^{-2} $\sec^{-1}$ is suggested. The asphalts tested showed Newtonian behavior, at the above shear rate, down to 40 F and below.

(2) The method of data treatment in the mid-range temperatures as used by Sisko give a good picture of the asphalt characteristics. If this is combined with viscosities at the glass transition temperature and a viscosity of 275 F, a complete picture of asphalt consistency is obtained (Figures 12 and 13). This could lead to practical performance limits as shown in Figure 14.

(3) If the glass transition temperature and the viscosity of a given asphalt at another temperature (say 77 F) is known, a limiting viscosity curve can be constructed between 77 F and the glass transition temperature for any asphalt using simple calculations and Walther graph paper. This may be a starting point for specifications on low temperature properties of asphalts, since to get such a curve relatively little time

is required (Figures 15E to 15J).

(4) The standardization of asphalts according to their viscosities at 77 F appears to be desirable for various reasons. Preliminary work so far indicates that viscosity measurements at 77 F and 2 x 10^{-2} sec^{-1} shear rate is practical using the cone-plate viscometer.

(5) Additional studies comparing viscosity of Michigan asphalts (at 77 F) with 3 types of penetration (standard, submerged and stub penetrations) suggest that the main problem in these correlations is the shear rate. Thus, the submerged penetration with the lowest shear rate during the test gave the best correlation with viscosity.

RECOMMENDATIONS

(1) Viscosity at low temperatures should be measured for as many asphalts as time permits during the winter months in the laboratory.

(2) Each asphalt under study should be observed for performance on the road so that data can be gathered to set up wide range temperature-viscosity limits.

(3) For routine laboratory tests at least three cone-plate viscometer sets are needed.

(4) Further studies to facilitate the conversion of penetration grading to viscosity grading should be undertaken.

(5) The crudes which had large deviations from the normal when penetration versus viscosity was studied, should be investigated further.

(6) A possible project would be to develop a penetration test where instead of a constant weight being applied, a constant rate of penetration would be applied (equivalent to the chosen standard viscometer shear rate) and the force applied would be measured with respect to time. The area under the force-time plot would be energy and could be correlated to the viscosity, hopefully with greater precision. This should eliminate oddities encountered with shear rate dependent asphalts.

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BIBLIOGRAPHY

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TABLES

TABLE 1

INSTRUMENTS IN USE AS BITUMEN VISCOMETERS

Type or description of viscometer	Method	Approx temperature range, °F	Approx viscosity range, poises
1. Rod	Saal		10^7–10^{13}
2. Penetrometer	ASTM D5		10^4–10^{13}
3. Disk	Obermayer		10^7–10^{12}
4. Metro plastimeter	Evans–Pickard	60–90	10^8–10^{10}
5. Falling coaxial cylinder	Pochettino	32–100	10^7–10^{10}
6. Alternating stress	Bingham–Stephens	60–95	10^5–10^8
7. Conicylindrical rotation	Couette	77–(150)	0·01 –10^9
8. Twisting point	Frankland Taylor		10^3–4×10^7
9. Capillary tube	Modified Bingham–Murray	110–270	10–10^6
10. Capillary tube	Modified Ostwald	60–140	Up to 10^6
11. Rotating cylinder	Csagoly		1–10^6
12. Plastometer	Bingham–Murray	(212)–270	5–10^5
13. Sinker	Saal		0·1–10^5
14. Rising-column capillary tube	Rhodes–Volkmann–Barker	110–200	60–6×10^4
15. Modified BRTA Trough	Solvey	85–195	60–10^4
16. Rotating cylinder	Volarovich	110–212	25–$2{\cdot}1 \times 10^4$
17. Falling sphere	Höppler		Up to $1{\cdot}2 \times 10^4$
18. Orifice efflux	BRTA (STPTC RT2)		80–1000
19. Trough	Evans–Pickard	60–200	30–1000
20. Torsion	Brookfield Synchro–Lectric		0·1–1000
21. Torsion	Fuidge		Up to 100
22. Ball and bucket	Beale–Docksey		Up to 100
23. Orifice efflux	Saybolt–Furol (ASTM D88)		0·5–50
24. Orifice efflux	Redwood II (IP 70)		0·5–50
25. Orifice efflux	Engler	350–400	0·1–10
26. Conicylindrical rotation	Stormer	325–450	0·1–20
27. Conicylindrical rotation	MacMichael	325–450	0·1–20

(Taken from Reference 1)

TABLE 2

REPEATABILITY OF VISCOSITY MEASUREMENTS
BY THE
SLIDING PLATE MICROVISCOMETER

Sample	Test. Temp. (C)	No. of Operators	No. of Tests	Mean $\bar{x}$ (poises)	Coeff. of Dev. D, %	95% Confidence Limits for $\bar{x}$
85/100 pen.	4	1	18	6.03×10^8	2.69	± 1.4%
	25	3	45	7.81×10^5	2.86	± 0.86%
	60	1	17	8.86×10^2	4.80	± 2.5%
SC-3 cut back	25	1	14	2.54×10^2	4.4	± 2.6%

TABLE 3

RESULTS OF CONE-PLATE VISCOMETER CALIBRATION

K_η , poise - deg/gm - sec - Viscosity calibration constant

$\eta = K_\eta \cdot t/\theta \cdot L$ L : Load in grams

Cone	Thin string	Thick string
Large	15.177	15.546
Medium	118.78	122.88
Small	1,059.9	1,096.5

K_s , dynes/cm^2/gm -- Shear stress constant

Cone	Thin string	Thick string
Large	31.114	32.190
Medium	248.52	257.11
Small	1,988.1	2,056.8

K_D , deg^{-1} - Shear rate constant $K_D = \frac{K_s}{K_\eta}$

(reciprocal of the angle between the plate and cone)

Shear rate = $K_D \quad \theta/t$ (sec^{-1})

Cone	Thin string	Thick string
Large	2.050	2.071
Medium	2.092	2.092
Small	1.876	1.876

TABLE 4

LIMITS FOR LINEAR STRESS-STRAIN RELATIONSHIP

Sample 73 B-1 (60/70 Pen)

Temperature (F)	Limit of Shear-Rate ($sec.^{-1}$)	Limit of Stress (psi)
59	3×10^{-3}	2.9
77	7×10^{-2}	2.9
104	1×10^{-1}	0.15
140	1	0.051

Sample 73 B-23 (120/150 Pen)

Temperature (F)	Limit of Shear-Rate ($sec.^{-1}$)	Limit of Stress (psi)
41	1.5×10^{-3}	2.9
59	4×10^{-2}	2.9
77	1×10^{-1}	0.77
104	1×10^{-1}	0.029
140	2	0.029

(1 psi = 6.89×10^4 dynes/cm^2)

TABLE 5

TYPICAL VISCOSITY RUN DATA

Sample 74B-9 (85-100) #3
Temperature 77 F
Large Cone - Thick String

Weight (g)	Deg.	Time (sec)	t/θ (sec./deg.)	η (Poises)	(dynes/cm^2)	(sec^{-1})
1000	1	70.6	70.6	1.10×10^6	3.22×10^4	2.93×10^{-2}
500	1	145.1	145.1	1.13×10^6	1.61×10^4	1.43×10^{-2}
1000	1	70.1	70.1	1.09×10^6	3.22×10^4	2.95×10^{-2}
3000	3	67.8	22.6	1.05×10^6	9.66×10^4	9.16×10^{-2}
5000	5	67.0	13.4	1.04×10^6	1.61×10^5	1.55×10^{-1}
10000	10	64.1	6.41	9.96×10^5	3.22×10^5	3.23×10^{-1}
15000	15	59.7	3.98	9.28×10^5	4.83×10^5	5.20×10^{-1}
20000	20	55.1	2.75	8.55×10^5	6.44×10^5	7.53×10^{-1}

TABLE 6

VISCOSITIES AT DIFFERENT TEMPERATURES

Sample Identification				Viscosity @ 2 x 10^{-2} sec^{-1}							
No.	MDSHT Code No.	Pen. Grade	Sample Source	1	2	3	4	5	6	$\bar{x}$	σ
				Viscosities @ 100 F							
1	73B-5	60-70	Amoc-'72	1.18×10^{5}						1.18×10^{5}	
2	73B-17	200-250	Arco-'72	1.53×10^{4}						1.53×10^{4}	
3	73B-21	60-70	Trum-'72	2.45×10^{5}						2.45×10^{5}	
				Viscosities @ 68 F							
1	73B-19	120-150	Leon-'72	1.67×10^{6}						1.67×10^{6}	
2	73B-21	60-70	Trum-'72	8.07×10^{7}						8.07×10^{7}	
3	74B-1	60-70	Leon-'74	9.13×10^{6}						9.13×10^{6}	
4	74B-12	60-70	Lion-'74	1.04×10^{7}	1.14×10^{7}	1.05×10^{7}	1.02×10^{7}	1.03×10^{7}		1.06×10^{7}	4.83×10^{5}
5	74B-18	120-150	Amer-'74 (S C)	1.68×10^{6}						1.68×10^{6}	
6	74B-19	200-300	Amer-'74 (S C)	6.48×10^{5}						6.48×10^{5}	
7	74B-23	85-100	Amer-'74 (Whit)	4.54×10^{6}	4.59×10^{6}					4.56×10^{6}	3.54×10^{4}
8	74B-29	200-300	Atl-'74	7.22×10^{5}	7.33×10^{5}	7.39×10^{5}	7.28×10^{5}	7.63×10^{5}		7.37×10^{5}	1.58×10^{4}

TABLE 6 (continued)

Sample Identification				Viscosity @ 2×10^{-2} sec^{-1}							
No.	MDSHT Code No.	Pen. Grade	Sample Source	1	2	3	4	5	6	$\bar{x}$	σ
				Viscosities @ 59 F							
1	73B-21	60-70	Trum-'72	$3.55x10^{7}$						$3.55x10^{7}$	
2	74B-12	60-70	Lion-'74	$3.60x10^{7}$	$3.75x10^{7}$	$3.58x10^{7}$	$3.86x10^{7}$	$3.76x10^{7}$	$3.84x10^{7}$	$3.73x10^{7}$	$1.18x10^{6}$
3	74B-18	120-150	Amer-'74 (S C)	$6.59x10^{6}$						$6.59x10^{6}$	
4	74B-19	200-300	Amer-'74 (S C)	$1.86x10^{6}$	$2.05x10^{6}$					$1.95x10^{6}$	$1.34x10^{5}$
5	74B-23	85-100	Amer-'74 (Whit)	$1.90x10^{7}$	$1.73x10^{7}$	$1.82x10^{7}$	$1.85x10^{7}$	$1.85x10^{7}$		$1.83x10^{7}$	$6.28x10^{5}$
6	74B-29	200-300	Atl-'74	$3.45x10^{6}$	$3.47x10^{6}$	$3.38x10^{6}$	$3.45x10^{6}$	$4.03x10^{6*}$		$3.44x10^{6}$	$3.95x10^{4}$
				Viscosities @ 50 F							
1	73B-21	60-70	Trum-'72	$9.76x10^{7}$						$9.76x10^{7}$	
2	74B-12	60-70	Lion-'74	$1.04x10^{8}$	$9.55x10^{7}$	$1.03x10^{8}$	$1.11x10^{8}$	$9.66x10^{7}$		$1.02x10^{8}$	$6.27x10^{6}$
3	74B-18	120-150	Amer-'74 (S C)	$2.59x10^{7}$						$2.59x10^{7}$	
4	74B-19	200-300	Amer-'74 (S C)	$4.97x10^{6}$						$4.97x10^{6}$	
5	74B-23	85-100	Amer-'74 (Whit)	$6.62x10^{7}$						$6.62x10^{7}$	
6	74B-29	200-300	Atl-'74	$1.48x10^{7}$						$1.48x10^{7}$	

TABLE 6 (continued)

Sample Identification				Viscosity @ 2 x 10^{-2} sec^{-1}							
No.	MDSHT Code No.	Pen. Grade	Sample Source	1	2	3	4	5	6	$\bar{x}$	σ
				Viscosities @ 41 F							
1	73B-21	60-70	Trum-'72	$2.26x10^{8}$						$2.26x10^{8}$	
2	74B-12	60-70	Lion-'74	$2.18x10^{8}$	$2.49x10^{8}$	$2.77x10^{8}$	$2.80x10^{8}$	$2.73x10^{8}$	$2.57x10^{8}$	$2.59x10^{8}$	$2.34x10^{7}$
3	74B-18	120-150	Amer-'74 (S C)	$1.42x10^{8}$	$1.07x10^{8}$					$1.24x10^{8}$	$2.47x10^{7}$
4	74B-19	200-300	Amer-'74 (S C)	$2.72x10^{7}$	$3.97x10^{7}$					$3.35x10^{8}$	$8.84x10^{6}$
5	74B-23	85-100	Amer-'74 (Whit)	$1.55x10^{8}$						$1.55x10^{8}$	
6	74B-29	200-300	Atl-'74	$5.50x10^{7}$						$5.50x10^{7}$	
				Viscosities @ 32 F							
1	73B-21	60-70	Trum-'72	$6.05x10^{8}$						$6.05x10^{8}$	
2	74B-12	60-70	Lion-'74	$5.53x10^{8}$	$4.44x10^{8}$	$3.69x10^{8}$	$4.84x10^{8}$			$4.63x10^{8}$	$7.69x10^{7}$
3	74B-18	120-150	Amer-'74 (S C)	$3.97x10^{8}$	$1.19x10^{9}$*	$3.98x10^{8}$				$3.97x10^{8}$	$6.36x10^{6}$
4	74B-19	200-300	Amer-'74 (S C)	$1.46x10^{8}$						$1.46x10^{8}$	
5	74B-23	85-100	Amer-'74 (Whit)	$5.18x10^{8}$						$5.18x10^{8}$	
6	74B-29	200-300	Atl-'74	$1.70x10^{8}$	$1.13x10^{8}$	$1.47x10^{8}$	$1.59x10^{8}$			$1.47x10^{8}$	$2.47x10^{7}$

TABLE 6 (continued)

Sample Identification No.	MDSHT Code No.	Pen. Grade	Sample Source	Viscosity @ 2 x 10^{-2} sec^{-1} 1	2	3	4	5	6	$\bar{x}$	σ
				Viscosities @ 23 F							
1	74B-12	60-70	Lion-'74	8.37x10^8						8.37x10^8	

*Determined as outlying point by Student's test and omitted.

TABLE 7

VISCOSITY TEST DATA AT 77 F AND 2 x 10^{-2} sec.$^{-1}$ SHEAR RATE

Sample Identification				Viscosity, Poises, for Samples							
No.	MDSHT Code No.	Pen. Grade	Sample Source	1	2	3	4	5	6	$\bar{x}$	σ
1	73B-1	60-70	Lion-'72	2.55×10^{6}	2.32×10^{6}					2.44×10^{6}	1.63×10^{5}
2	73B-2	85-100	Lion-'72	9.25×10^{5}	$1.45 \times 10^{6*}$	9.39×10^{5}				9.32×10^{5}	9.90×10^{3}
3	73B-3	120-150	Lion-'72	4.71×10^{5}	4.20×10^{5}	4.41×10^{5}	5.39×10^{5}	4.55×10^{5}		4.65×10^{5}	4.53×10^{4}
4	73B-4	200-250	Lion-'72	1.50×10^{5}	1.37×10^{5}					1.44×10^{5}	9.19×10^{3}
5	73B-5	60-70	Amoc-'72	$3.52 \times 10^{6*}$	2.53×10^{6}	2.31×10^{6}	2.50×10^{6}			2.45×10^{6}	1.19×10^{5}
6	73B-6	85-100	Amoc-'72	$1.20 \times 10^{6*}$	9.35×10^{5}	9.22×10^{5}	9.17×10^{5}	8.99×10^{5}	9.06×10^{5}	9.16×10^{5}	1.40×10^{4}
7	73B-7	120-150	Amoc-'72	$5.51 \times 10^{5*}$	4.62×10^{5}	4.49×10^{5}				4.56×10^{5}	9.19×10^{3}
8	73B-11	120-150	Petro-'72	$8.08 \times 10^{5*}$	4.84×10^{5}	4.84×10^{5}				4.84×10^{5}	0.00
9	73B-12	200-250	Petro-'72	1.62×10^{5}	1.55×10^{5}	1.51×10^{5}				1.56×10^{5}	5.57×10^{3}
10	73B-13	85-100	Ashl-'72	$1.48 \times 10^{6*}$	1.20×10^{6}	$2.31 \times 10^{6*}$	1.17×10^{6}	1.27×10^{6}	1.28×10^{6}	1.23×10^{6}	5.35×10^{4}
11	73B-17	200-250	Arco-'72	1.79×10^{5}	1.89×10^{5}	1.97×10^{5}				1.88×10^{5}	9.02×10^{3}
12	73B-18	85-100	Leon-'72	$1.36 \times 10^{6*}$	$1.47 \times 10^{6*}$	1.06×10^{6}	9.89×10^{5}	1.06×10^{5}		1.04×10^{6}	4.10×10^{4}
13	73B-19	120-150	Leon-'72	5.05×10^{5}	4.51×10^{5}	4.78×10^{5}	4.53×10^{5}			4.72×10^{5}	2.53×10^{4}
14	73B-21	60-70	Trum-'72	3.07×10^{6}	3.42×10^{6}	3.12×10^{6}	3.39×10^{6}	3.27×10^{6}		3.25×10^{6}	1.57×10^{5}
15	73B-22	85-100	Trum-'72	1.30×10^{6}	9.68×10^{5}	1.08×10^{6}	9.33×10^{5}	1.11×10^{6}	1.33×10^{6}	1.12×10^{6}	1.65×10^{5}
16	73B-23	120-150	Trum-'72	$4.85 \times 10^{5*}$	3.57×10^{5}	3.58×10^{5}	3.56×10^{5}	3.58×10^{5}		3.57×10^{5}	9.57×10^{2}

TABLE 7 (continued)

Sample Identification				Viscosity, Poises, for Samples							
No.	MDSHT Code No.	Pen. Grade	Sample Source	1	2	3	4	5	6	$\bar{x}$	σ
17	74B-1	60-70	Leon-'74	$1.89x10^6$	$1.92x10^6$					$1.91x10^6$	$2.12x10^4$
18	74B-2	85-100	Leon-'74	$9.19x10^5$	$8.36x10^5$	$9.14x10^5$	$9.13x10^5$	$8.35x10^5$	$8.13x10^5$	$8.72x10^5$	$4.86x10^4$
19	74B-3	120-150	Leon-'74	$4.94x10^5$	$4.82x10^5$					$4.88x10^5$	$8.49x10^3$
20	74B-4	200-250	Leon-'74	$1.93x10^5$	$1.96x10^5$					$1.95x10^5$	$2.12x10^3$
21	74B-5	60-70	Trum-'74	$1.93x10^6$	$1.94x10^6$					$1.94x10^6$	$7.07x10^3$
22	74B-6	85-100	Trum-'74	$8.03x10^5$	$8.21x10^5$					$8.12x10^5$	$1.27x10^4$
23	74B-7	120-150	Trum-'74	$4.58x10^5$	$4.68x10^5$					$4.63x10^5$	$7.07x10^3$
24	74B-8	200-250	Trum-'74	$1.74x10^5$	$1.74x10^5$					$1.74x10^5$	0.00
25	74B-9	85-100	Murp-'74	$1.02x10^6$	$1.05x10^6$	$1.11x10^6$	$1.09x10^6$			$1.07x10^6$	$4.03x10^4$
26	74B-10	120-150	Murp-'74	$4.98x10^5$	$4.78x10^5$					$4.88x10^5$	$1.41x10^4$
27	74B-11	200-250	Murp-'74	$1.61x10^5$	$1.60x10^5$					$1.61x10^5$	$7.07x10^2$
28	74B-12	60-70	Lion-'74	$2.51x10^6$	$2.47x10^6$	$2.63x10^6$	$2.80x10^6$			$2.60x10^6$	$1.48x10^5$
29	74B-13	85-100	Lion-'74	$9.79x10^5$	$9.87x10^5$					$9.83x10^5$	$5.66x10^3$
30	74B-14	120-150	Lion-'74	$5.61x10^5$	$4.70x10^5$	$4.54x10^5$	$4.11x10^5$			$4.74x10^5$	$6.31x10^4$
31	75B-15	200-250	Lion-'74	$1.58x10^5$	$1.64x10^5$	$1.64x10^5$	$1.65x10^5$			$1.63x10^5$	$3.20x10^3$
32	74B-18	120-150	Amer-'74 (S C)	$5.40x10^5$	$5.60x10^5$					$5.50x10^5$	$1.41x10^4$
33	74B-19	200-300	Amer-'74 (S C)	$2.16x10^5$	$2.10x10^5$	$1.99x10^5$	$2.86x10^{5*}$	$2.96x10^{5*}$		$2.08x10^5$	$8.62x10^3$

TABLE 7 (continued)

Sample Identification				Viscosity, Poises, for Samples							
No.	MDSHT Code No.	Pen. Grade	Sample Source	1	2	3	4	5	6	$\bar{x}$	σ
34	74B-21	85-100	Amer-'74 (S C)	8.46×10^5	7.43×10^5	8.23×10^5				8.04×10^5	5.41×10^4
35	74B-22	60-70	Amer-'74 (Whit)	2.36×10^6	2.30×10^6					2.33×10^6	4.24×10^4
36	74B-23	85-100	Amer-'74 (Whit)	1.07×10^6	9.90×10^5					1.03×10^6	5.66×10^4
37	74B-24	120-150	Amer-'74 (Whit)	5.45×10^5	5.54×10^5	5.13×10^5	5.91×10^5	5.97×10^5		5.60×10^5	3.46×10^4
38	74B-25	200-250	Amer-'74 (Whit)	1.68×10^5	1.69×10^5					1.69×10^5	7.07×10^2
39	74B-26	60-70	Atl-'74	2.41×10^6	2.43×10^6					2.42×10^6	1.41×10^4
40	74B-27	85-100	Atl-'74	1.33×10^6	1.33×10^6					1.33×10^6	0.00
41	74B-28	120-150	Atl-'74	5.47×10^5	5.60×10^5					5.54×10^5	9.19×10^3
42	74B-29	200-250	Atl-'74	1.98×10^5	1.90×10^5					1.94×10^5	5.66×10^3
43	74B-30	85-100	Imp-'74	1.28×10^6	1.31×10^6					1.30×10^6	2.12×10^4

* Determined as outlying point by Student's test and omitted.

TABLE 8

AVERAGE PENETRATIONS AND VISCOSITIES

Sample Identification				Penetrations @ 77 F			Viscosity (Poises)		
No.	MDSHT Code No.	Pen. Grade	Sample Source	Regular	Submerged	Stub	@ 77 F	@ 140 F	@ 275 F
1	73B-1	60-70	Lion-'72	63	21	48	2.44×10^6	2840	5.62
2	73B-2	85-100	Lion-'72	86	38	65	9.32×10^5	1270	3.71
3	73B-3	120-150	Lion-'72	128	73	111	4.65×10^5	790	2.94
4	73B-4	200-250	Lion-'72	245	183	221	1.44×10^5	387	2.14
5	73B-5	60-70	Amoc-'72	59	20	42	2.45×10^6	2140	3.89
6	73B-6	85-100	Amoc-'72	91	44	72	9.16×10^5	1200	3.06
7	73B-7	120-150	Amoc-'72	137	81	118	4.56×10^5	629	2.27
8	73B-11	120-150	Petro-'72	133	76	112	4.84×10^5	870	3.10
9	73B-12	200-250	Petro-'72	236	188	217	1.56×10^5	430	2.36
10	73B-13	85-100	Ashl-'72	79	35	63	1.23×10^6	1690	3.66
11	73B-17	200-250	Arco-'72	220	152	180	1.88×10^5	372	2.07
12	73B-18	85-100	Leon-'72	87	39	67	1.04×10^6	1590	3.87
13	73B-19	120-150	Leon-'72	134	80	118	4.72×10^5	885	2.90
14	73B-21	60-70	Trum-'72	63	15	37	3.25×10^6	2340	4.20
15	73B-22	85-100	Trum-'72	83	40	70	1.12×10^6	1460	3.42
16	73B-23	120-150	Trum-'72	145	87	125	3.57×10^5	784	2.58

TABLE 8 (Continued)

Sample Identification				Penetrations @ 77 F			Viscosity (Poises)		
No.	MDSHT Code No.	Pen. Grade	Sample Source	Regular	Submerged	Stub	@ 77 F	@ 140 F	@ 275 F
17	74B-1	60-70	Leon-'74	71	24	51	$1.91\text{x}10^{6}$	2660	4.72
18	74B-2	85-100	Leon-'74	101	45	80	$8.72\text{x}10^{5}$	1510	3.65
19	74B-3	120-150	Leon-'74	133	72	114	$4.88\text{x}10^{5}$	943	3.18
20	74B-4	200-250	Leon-'74	210	144	202	$1.95\text{x}10^{5}$	569	2.27
21	74B-5	60-70	Trum-'74	64	21	50	$1.94\text{x}10^{6}$	2460	4.27
22	74B-6	85-100	Trum-'74	99	50	83	$8.12\text{x}10^{5}$	1300	3.09
23	74B-7	120-150	Trum-'74	132	78	115	$4.63\text{x}10^{5}$	771	2.63
24	74B-8	200-250	Trum-'74	237	166	221	$1.74\text{x}10^{5}$	402	1.94
25	74B-9	85-100	Murp-'74	90	40	73	$1.07\text{x}10^{6}$	1710	3.86
26	74B-10	120-150	Murp-'74	128	72	111	$4.88\text{x}10^{5}$	1010	3.00
27	74B-11	200-250	Murp-'74	249	164	215	$1.61\text{x}10^{5}$	480	2.11
28	74B-12	60-70	Lion-'74	63	20	48	$2.60\text{x}10^{6}$	3130	5.62
29	74B-13	85-100	Lion-'74	89	40	69	$9.83\text{x}10^{5}$	1230	3.86
30	74B-14	120-150	Lion-'74	134	77	113	$4.74\text{x}10^{5}$	861	3.26
31	74B-15	200-250	Lion-'74	244	161	217	$1.63\text{x}10^{5}$	392	2.19
32	74B-18	120-150	Amer-'74 (S C)	159	85	125	$5.50\text{x}10^{5}$	387	1.68
33	74B-19	200-250	Amer-'74 (S C)	289	213	253	$2.08\text{x}10^{5}$	161	1.11

TABLE 8 (Continued)

Sample Identification				Penetrations @ 77 F			Viscosity (Poises)		
No.	MDSHT Code No.	Pen. Grade	Sample Source	Regular	Submerged	Stub	@ 77 F	@ 140 F	@ 275 F
34	74B-21	85-100	Amer-'74 (SC50-50%)	96	48	75	8.04×10^5	817	2.69
35	74B-22	60-70	Amer-'74 (Whit)	69	21	45	2.33×10^6	1840	3.51
36	74B-23	85-100	Amer-'74 (Whit)	95	41	73	1.03×10^6	1110	2.82
37	74B-24	120-150	Amer-'74 (Whit)	124	70	110	5.60×10^5	721	2.34
38	74B-25	200-250	Amer-'74 (Whit)	235	191	224	1.69×10^5	354	1.58
39	74B-26	60-70	Atl-'74	68	22	46	2.42×10^6	1930	3.99
40	74B-27	85-100	Atl-'74	82	35	64	1.33×10^6	1230	3.38
41	74B-28	120-150	Atl-'74	125	77	109	5.54×10^5	683	2.58
42	74B-29	200-250	Atl-'74	226	155	201	1.94×10^5	388	2.00
43	74B-30	85-100	Imp-'74	87	39	74	1.30×10^6	1410	3.85
44	73B-9	60-70	Petro-'72	64	20	48		2680	4.94
45	73B-16	120-150	Arco-'72	119	66	107		720	2.87
46	73B-3384	120-150	Trum-'72	139	83	122			
47	73B-3830	60-70	Trum-'72	68	26	50			
48	73B-8205	85-100	Trum-'72	94	46	75			

TABLE 9

CORRELATIONS BETWEEN PENETRATION AND VISCOSITY MEASUREMENTS AT 77 F

X vs. Y	Samples	Correlation Coefficient	Equation	% of Estimate within Range of Tested Value					
			Range	0-5%	0-10%	0-20%	0-30%	0-40%	0-50%
Regular Penetration vs. Viscosity	43	0.98198	$Y = \frac{6.322\text{x}10^{9}}{X^{1.926}}$	27.9	51.2	83.8	93.1	97.8	100.0
	40	0.99213	$Y = \frac{7.828\text{x}10^{9}}{X^{1.978}}$	37.5	60.0	92.5	100.0	100.0	100.0
Submerged Penetration vs. Viscosity	43	0.99195	$Y = \frac{9.440\text{x}10^{7}}{X^{1.220}}$	46.5	72.1	95.4	97.7	100.0	100.0
	40	0.99579	$Y = \frac{1.081\text{x}10^{8}}{X^{1.257}}$	55.0	80.0	97.5	100.0	100.0	100.0
Stub Penetration vs. Viscosity	43	0.98888	$Y = \frac{1.229\text{x}10^{9}}{X^{1.653}}$	53.5	67.5	83.8	95.4	100.0	100.0
	40	0.99275	$Y = \frac{1.482\text{x}10^{9}}{X^{1.698}}$	47.5	70.0	90.0	97.5	100.0	100.0
Regular Penetration vs. Submerged Penetration	48	0.99278	$Y = 0.8636X-36.38$	60.4	75.0	95.8	100.0	100.0	100.0
Regular Penetration vs. Stub Penetration	48	0.99549	$Y = 0.959X-14.38$	72.9	91.7	97.9	100.0	100.0	100.0

TABLE 10

PENETRATION - ABSOLUTE VISCOSITY RELATIONSHIP

Standard Penetration	Saal & Koens	Pendleton	Tons, <u>et al</u>.
60	1.90×10^{6}	2.81×10^{6}	2.38×10^{6}
70	1.41×10^{6}	1.89×10^{6}	1.75×10^{6}
80	1.09×10^{6}	1.37×10^{6}	1.35×10^{6}
90	8.68×10^{5}	1.04×10^{6}	1.07×10^{6}
100	7.08×10^{5}	8.12×10^{5}	8.66×10^{5}
110	5.89×10^{5}	6.53×10^{5}	7.17×10^{5}
120	4.98×10^{5}	5.37×10^{5}	6.04×10^{5}
130	4.27×10^{5}	4.49×10^{5}	5.15×10^{5}
140	3.70×10^{5}	3.82×10^{5}	4.45×10^{5}
150	3.24×10^{5}	3.28×10^{5}	3.88×10^{5}
160	2.86×10^{5}	2.85×10^{5}	3.42×10^{5}
170	2.54×10^{5}	2.50×10^{5}	3.03×10^{5}
180	2.28×10^{5}	2.21×10^{5}	2.71×10^{5}
190	2.05×10^{5}	1.97×10^{5}	2.43×10^{5}
200	1.86×10^{5}	1.77×10^{5}	2.20×10^{5}
210	1.69×10^{5}	1.59×10^{5}	2.00×10^{5}
220	1.55×10^{5}	1.44×10^{5}	1.82×10^{5}
230	1.42×10^{5}	1.31×10^{5}	1.67×10^{5}
240	1.31×10^{5}	1.20×10^{5}	1.53×10^{5}
250	1.21×10^{5}	1.10×10^{5}	1.41×10^{5}

TABLE 11

GLASS TRANSITION AND VISCOSITY DATA FOR 16 MICHIGAN ASPHALTS

Sample Identification				Glass Transition Temperature, T_g, F				Viscosity (Poises)
No.	MDSHT Code No.	Pen. Grade	Sample Source	Cooling Mode	Heating Mode	Average Cooling-Heating	Grand Average	Calculated at T_g (x 10^{14})
1	73B-1	60-70	Lion-'72	-22.2 -16.6 -22.0	-13.5 - 8.9 -10.3	-17.9 -12.7 -16.2	-15.6	2.36
2	73B-2	85-100	Lion-'72	-18.4 -18.0 -20.4	-13.6 -11.2 -10.3	-16.0 -14.6 -15.4	-15.3	1.03
3	73B-3	120-150	Lion-'72	-14.4	- 8.3	-11.4	-11.4	0.507
4	73B-4	200-250	Lion-'72	-30.6	-20.7	-25.7	-25.7	0.578
5	73B-5	60-70	Amoc-'72	-22.2	-13.9	-18.1	-18.1	2.06
6	73B-6	85-100	Amoc-'72	-24.0	-14.8	-19.4	-19.4	1.25
7	73B-7	120-150	Amoc-'72	-25.6	-13.5	-19.6	-19.6	0.0664
8	73B-11	120-150	Petro-'72	-31.7	-19.7	-25.7	-25.7	1.30
9	73B-12	250-300	Petro-'72	-33.5	-20.7	-27.1	-27.1	0.695
10	73B-13	85-100	Ashl-'72	-19.8	-10.8	-15.3	-15.3	1.38
11	73B-17	200-250	Arco-'72	-28.5	-17.9	-23.2	-23.2	0.483
12	73B-18	85-100	Leon-'72	-26.1	-21.3	-23.7	-23.7	2.12

TABLE 11 (continued)

Sample Identification				Glass Transition Temperature, T_g, F				Viscosity (Poises)
No.	MDSHT Code No.	Pen. Grade	Sample Source	Cooling Mode	Heating Mode	Average Cooling-Heating	Grand Average	Calculated at T_g ($\times 10^{14}$)
13	73B-19	120-150	Leon-'72	-20.2	-15.7	-18.0	-18.0	0.850
14	73B-21	60-70	Trum-'72	-30.6	-21.6	-26.1	-26.1	3.75
15	73B-22	85-100	Trum-'72	-18.0	-13.5	-15.8	-15.8	1.23
16	73B-23	120-150	Trum-'72	-27.0	-20.6	-23.8	-23.2	1.01
				-28.8	-22.2	-25.5		
				-26.9	-18.2	-22.6		
				-25.6	-17.5	-21.6		
				-24.7	-18.2	-21.5		
				-27.8	-20.7	-24.3		

(Taken from Reference 19)

FIGURES

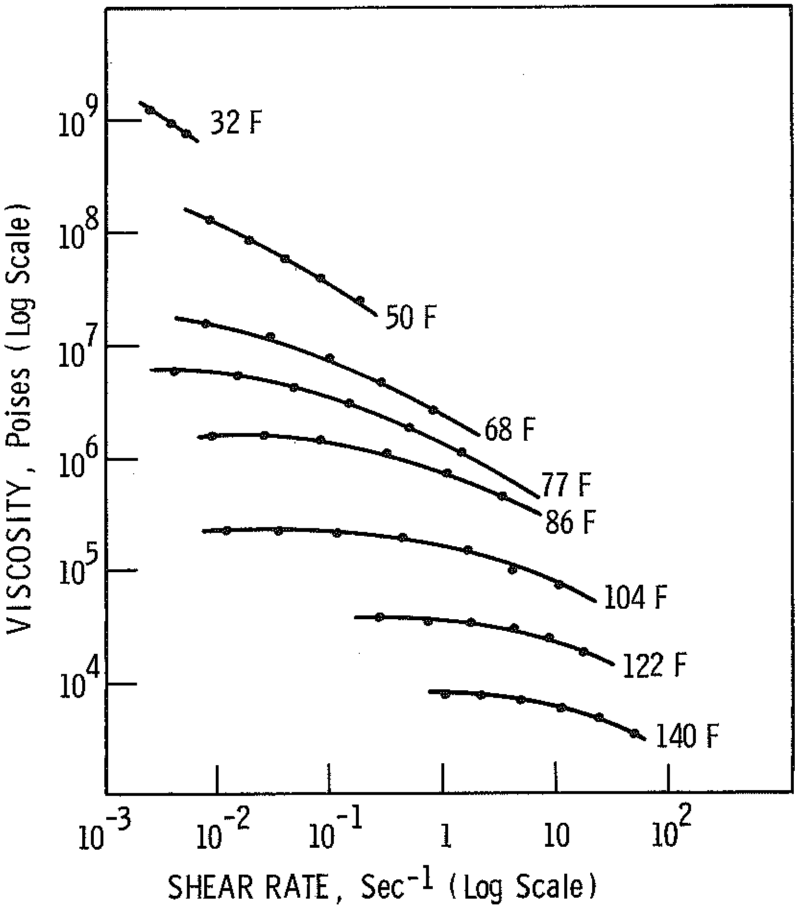

Figure 1. Flow curves at various temperatures.

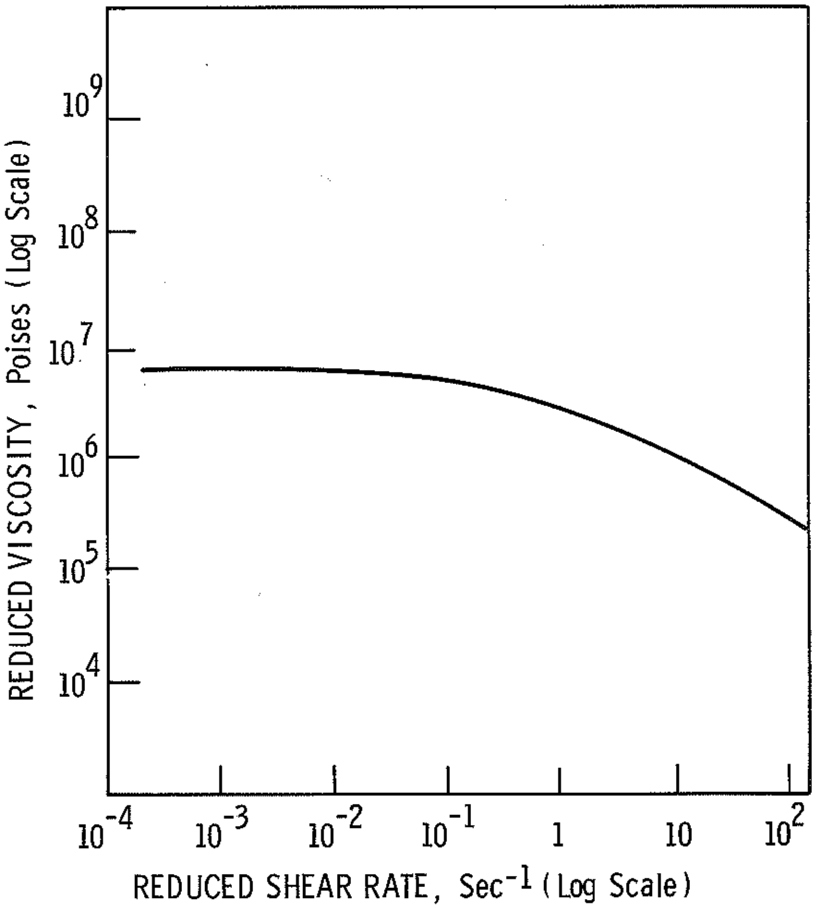

Figure 2. Master curve.

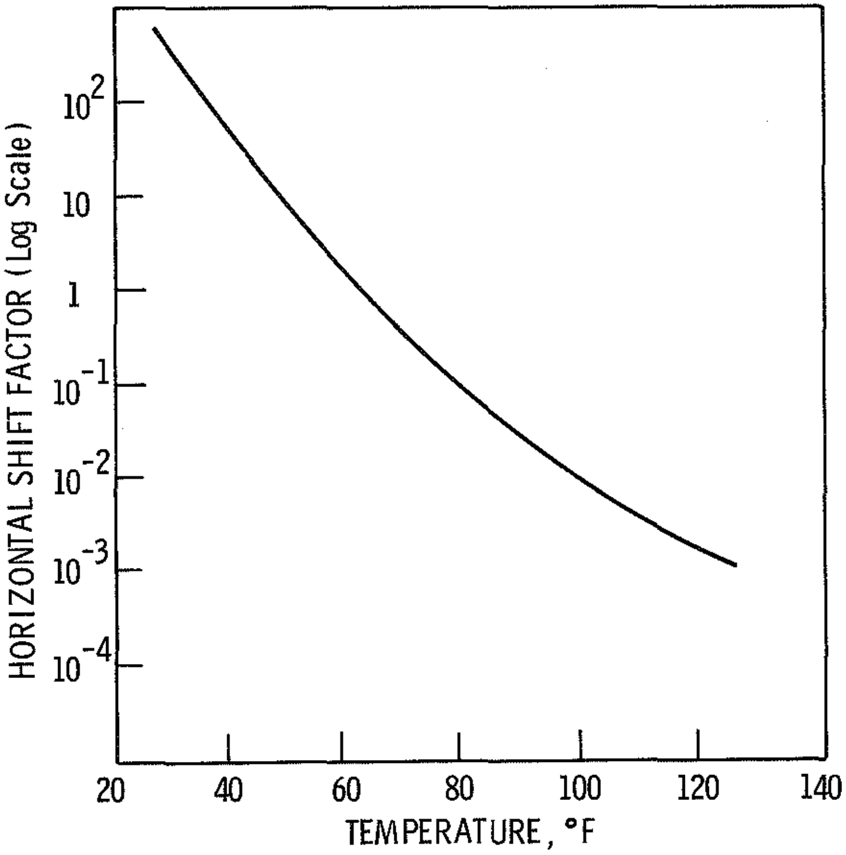

Figure 3. Horizontal shift factor.

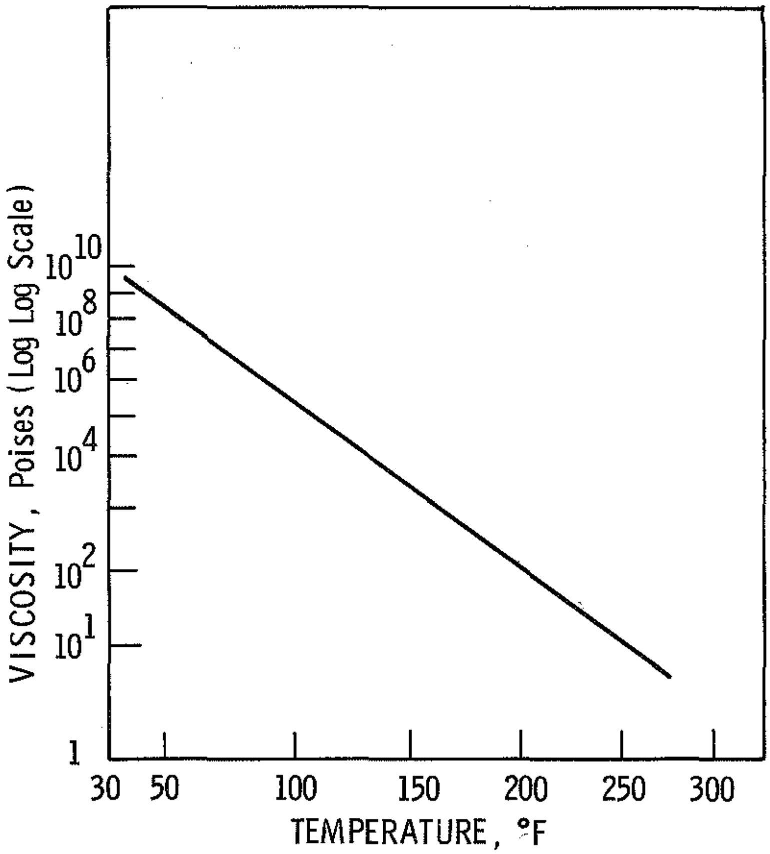

Figure 4. Viscosity in Walther coordinates.

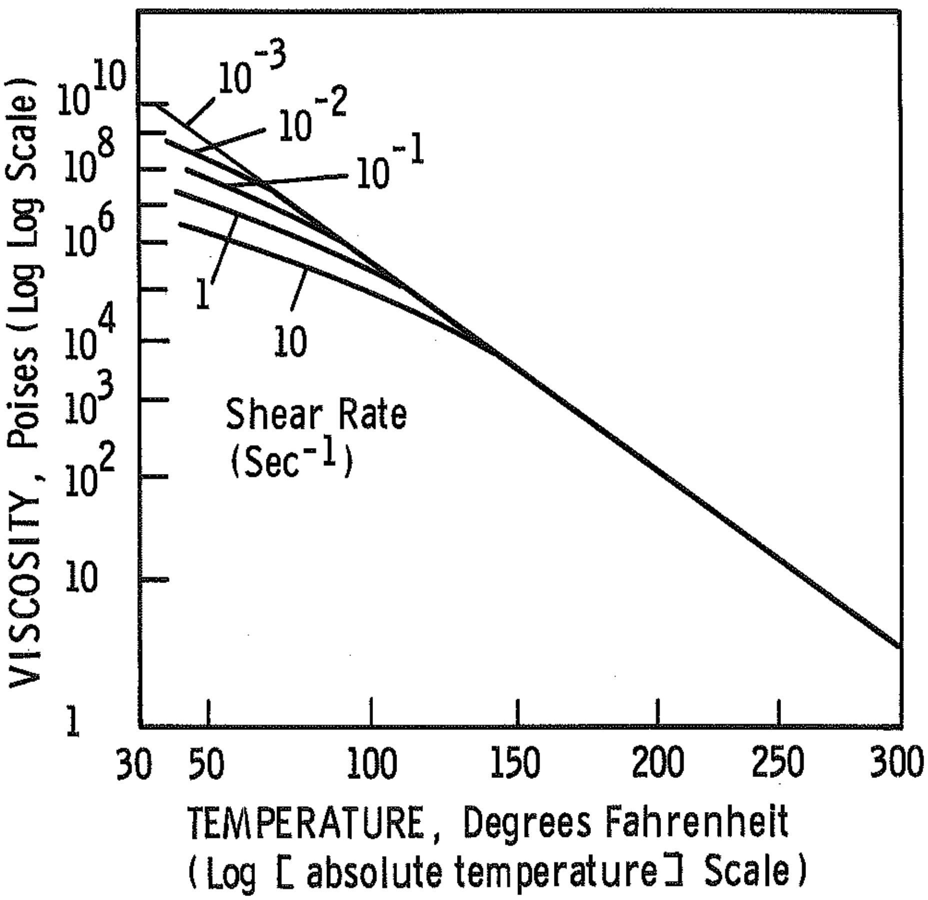

Figure 5. Effect of shear rate and temperature on viscosity.

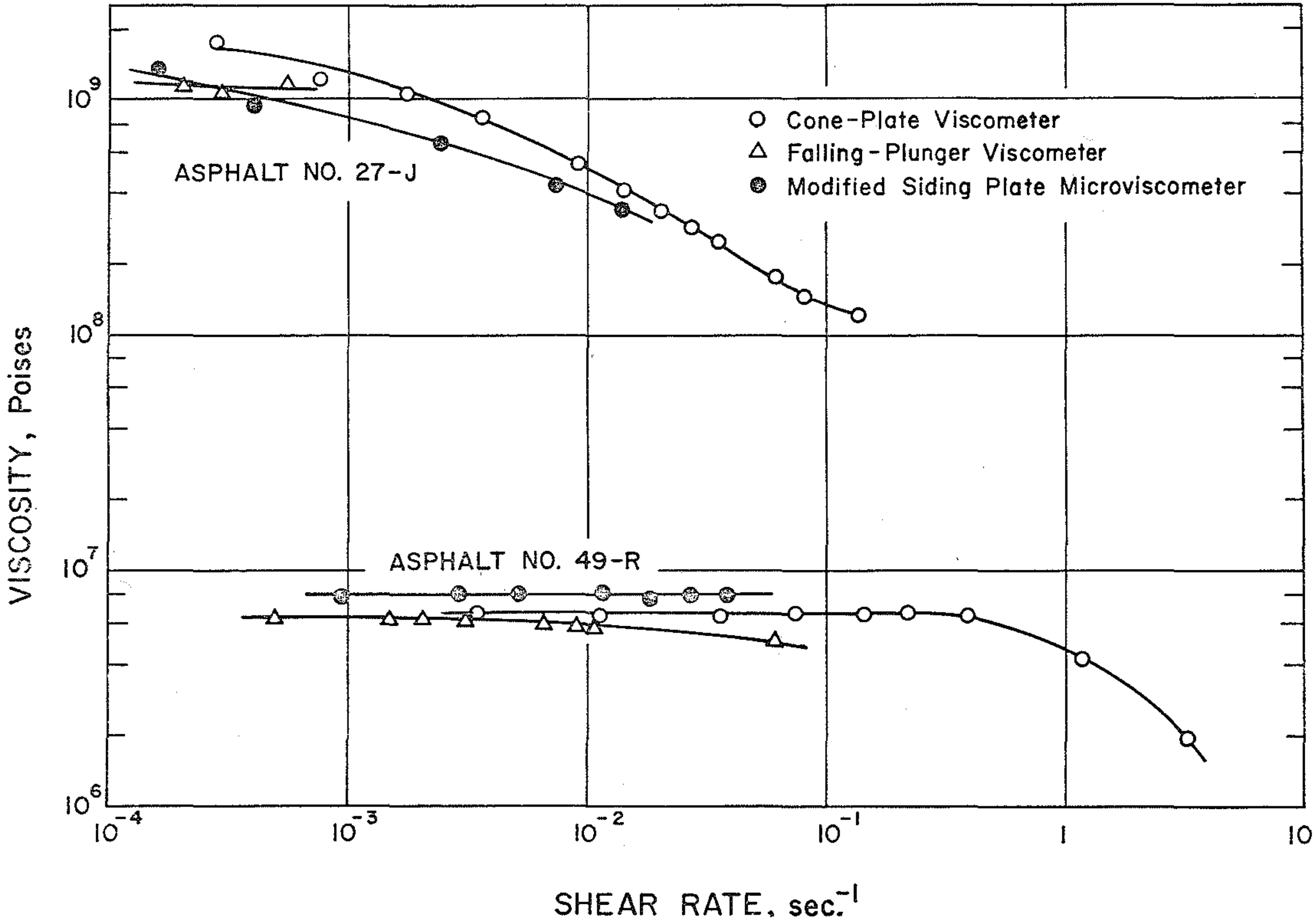

Figure 6. Viscosity measurements at 60 F with diffferent viscometers (taken from Ref. 22).

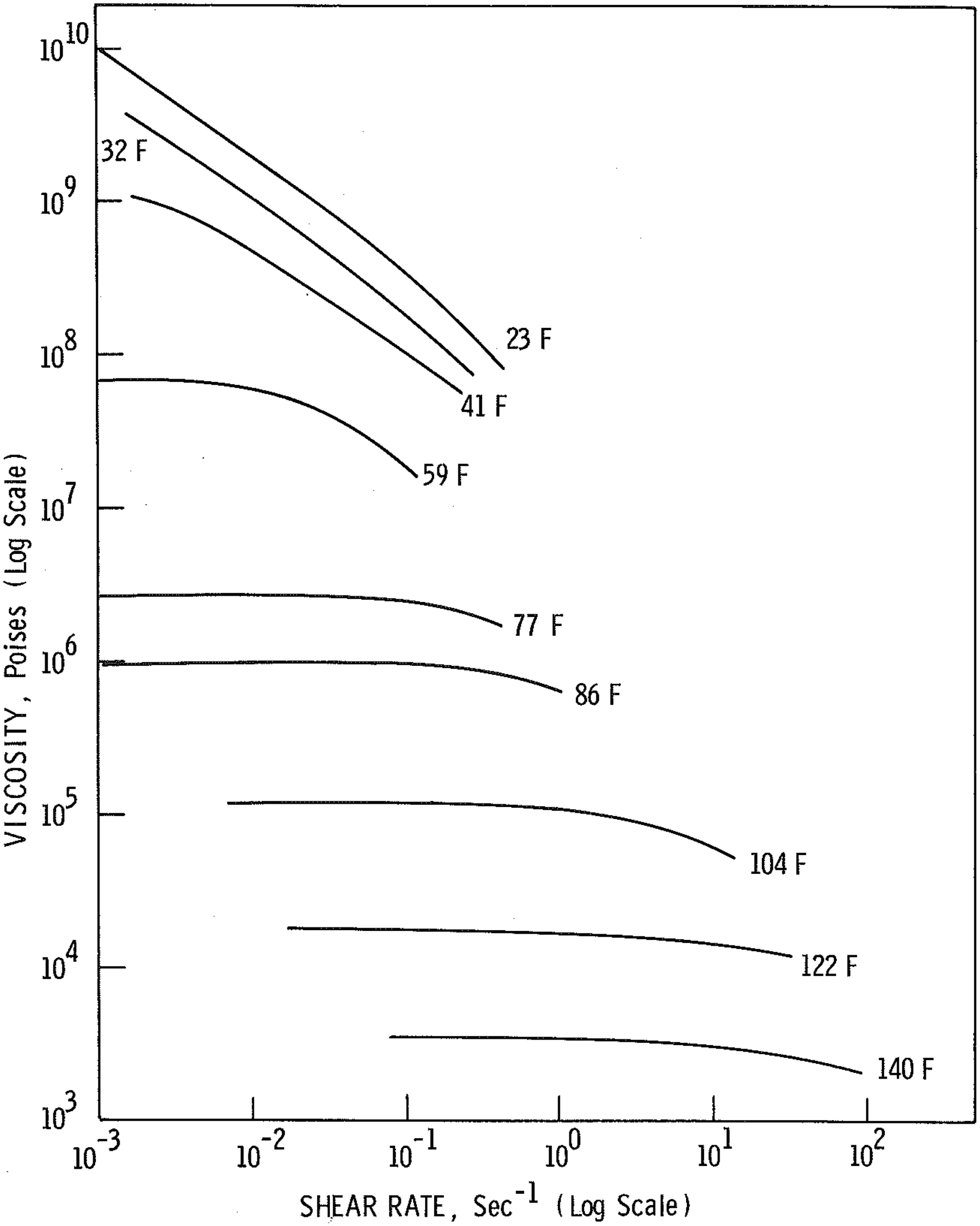

Figure 7. Flow curves for Michigan asphalt 73B-1 (60/70 pen).

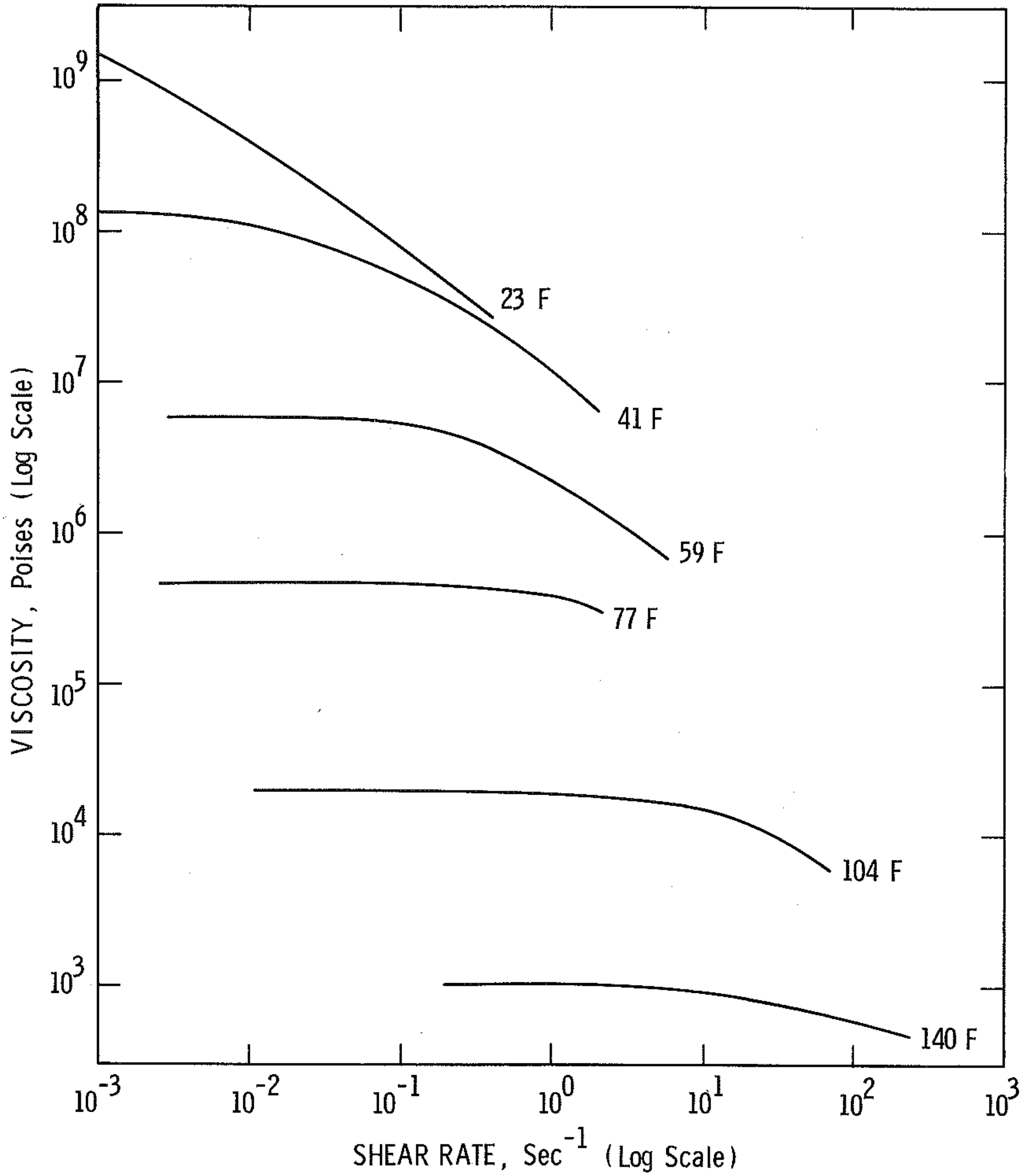

Figure 8. Flow curves for Michigan asphalt 73B-23 (120/150 pen).

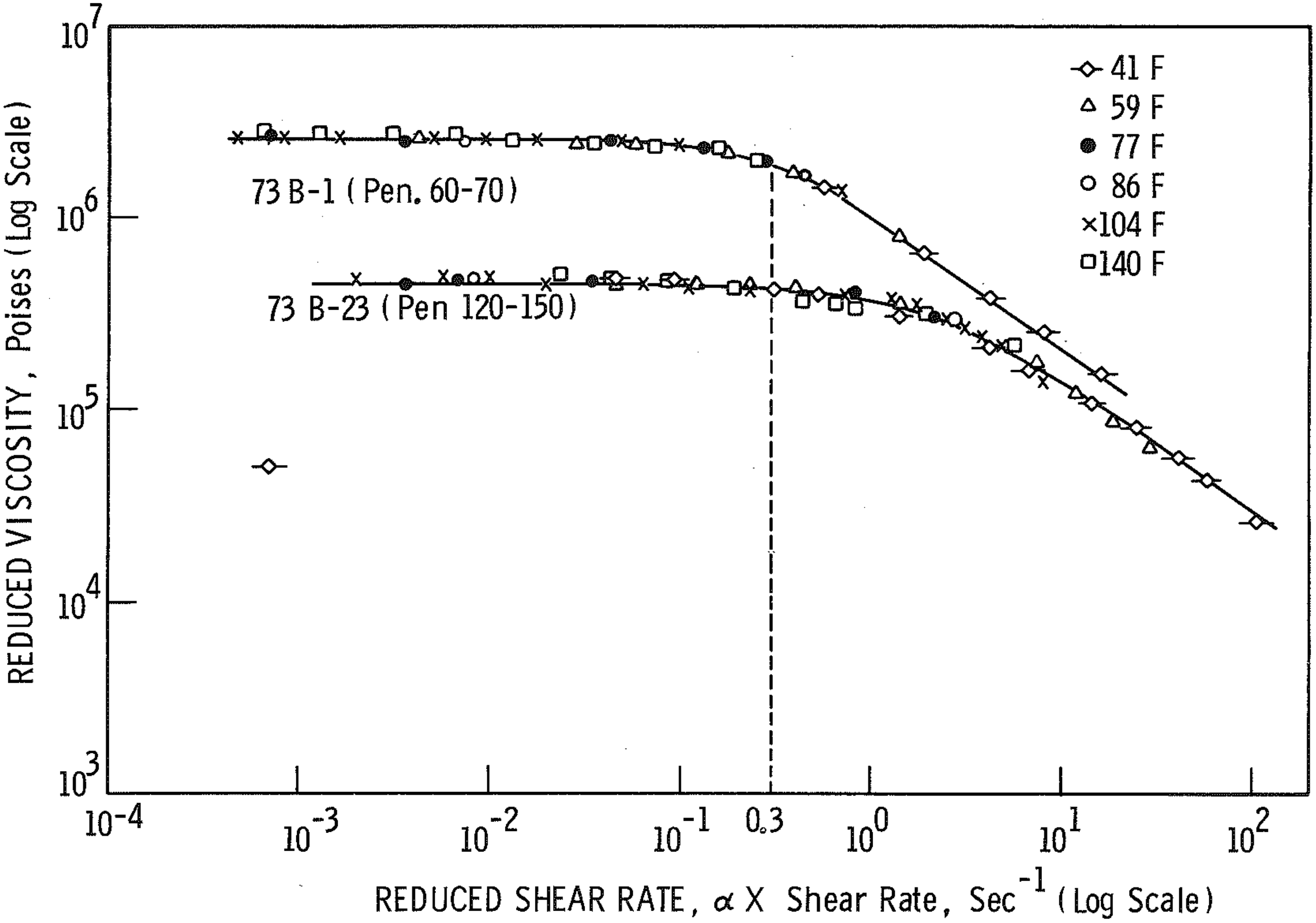

Figure 9. Master curves for two Michigan asphalts.

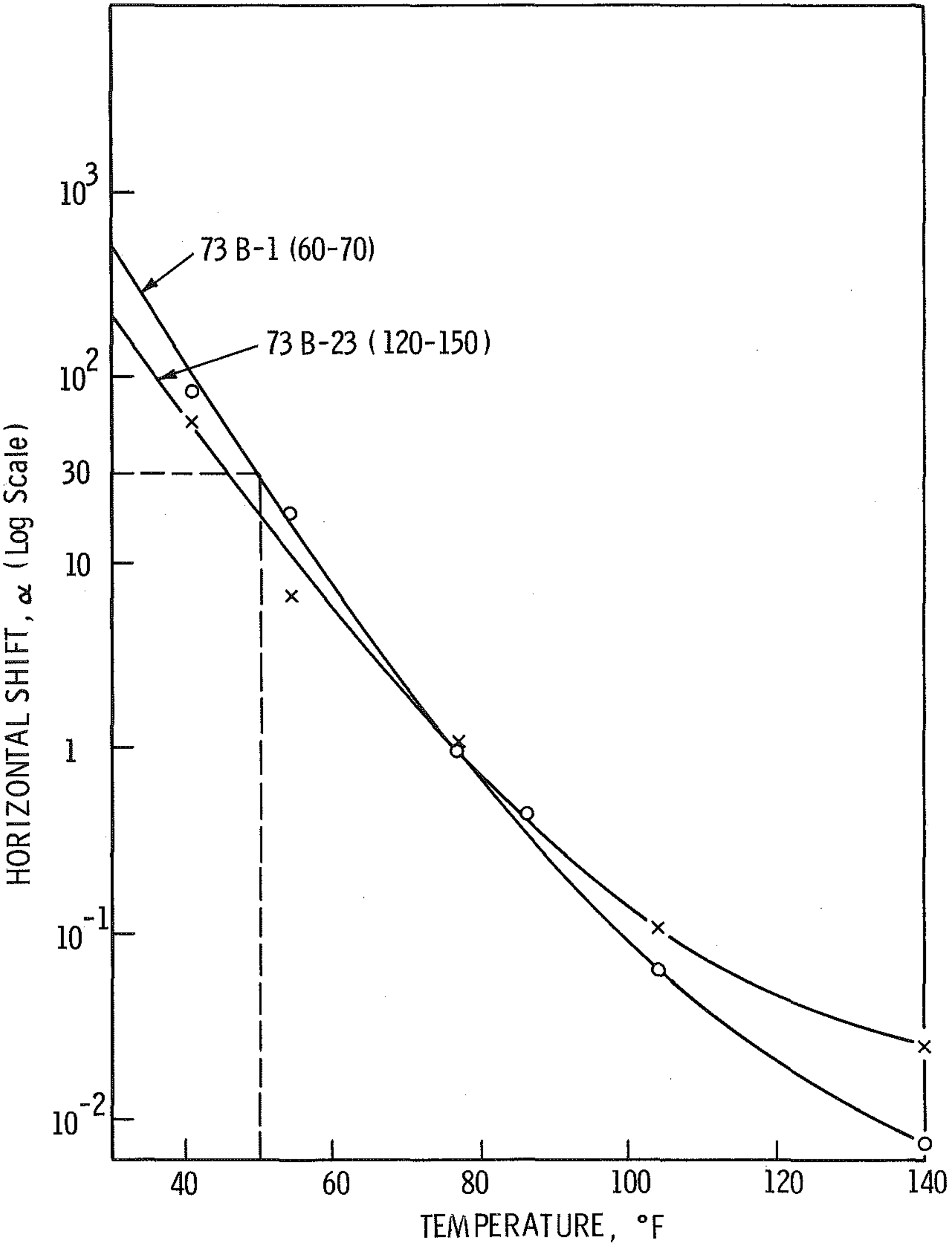

Figure 10. Horizontal shift factor.

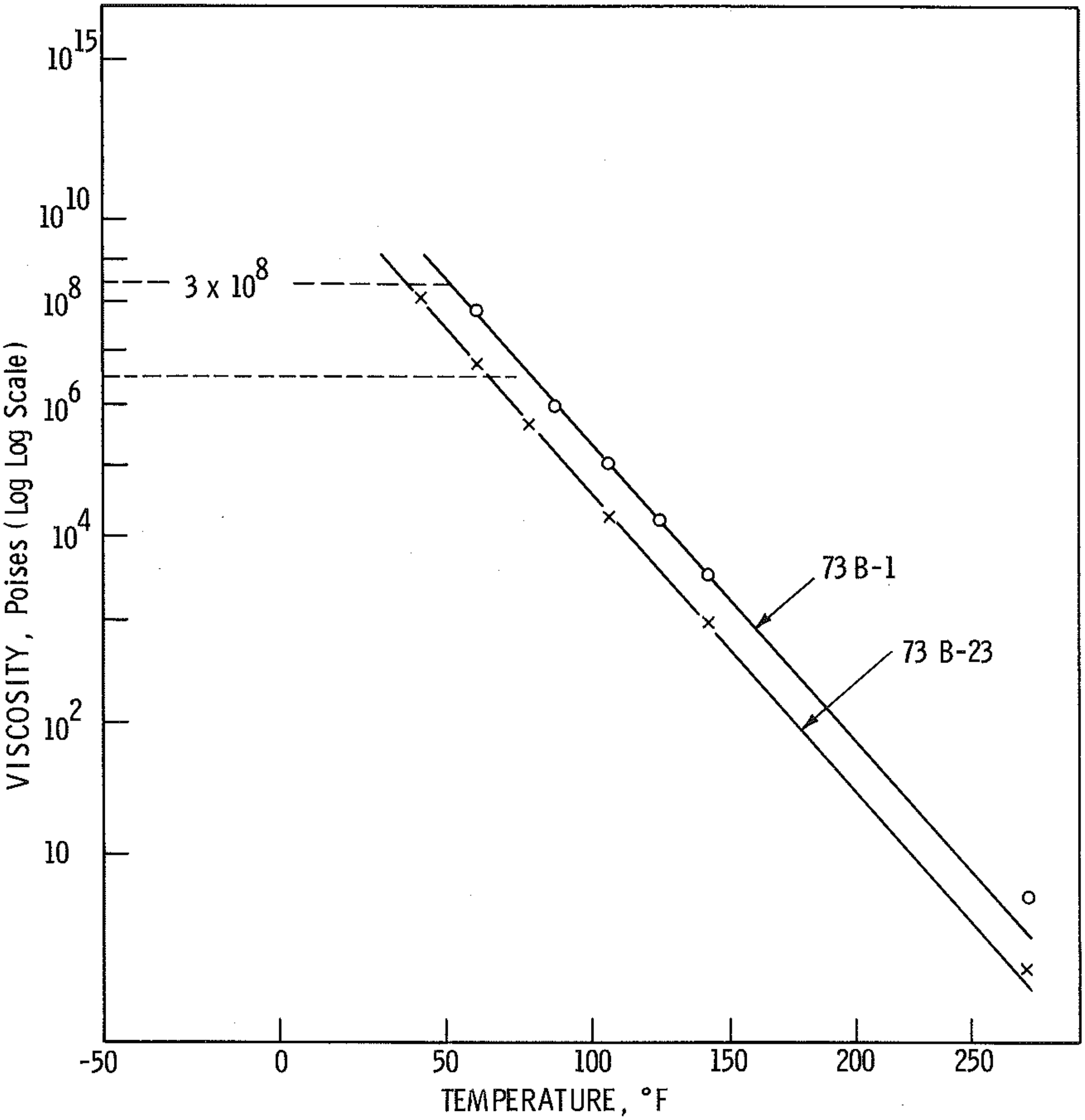

Figure 11. Limiting viscosity for two Michigan asphalts.

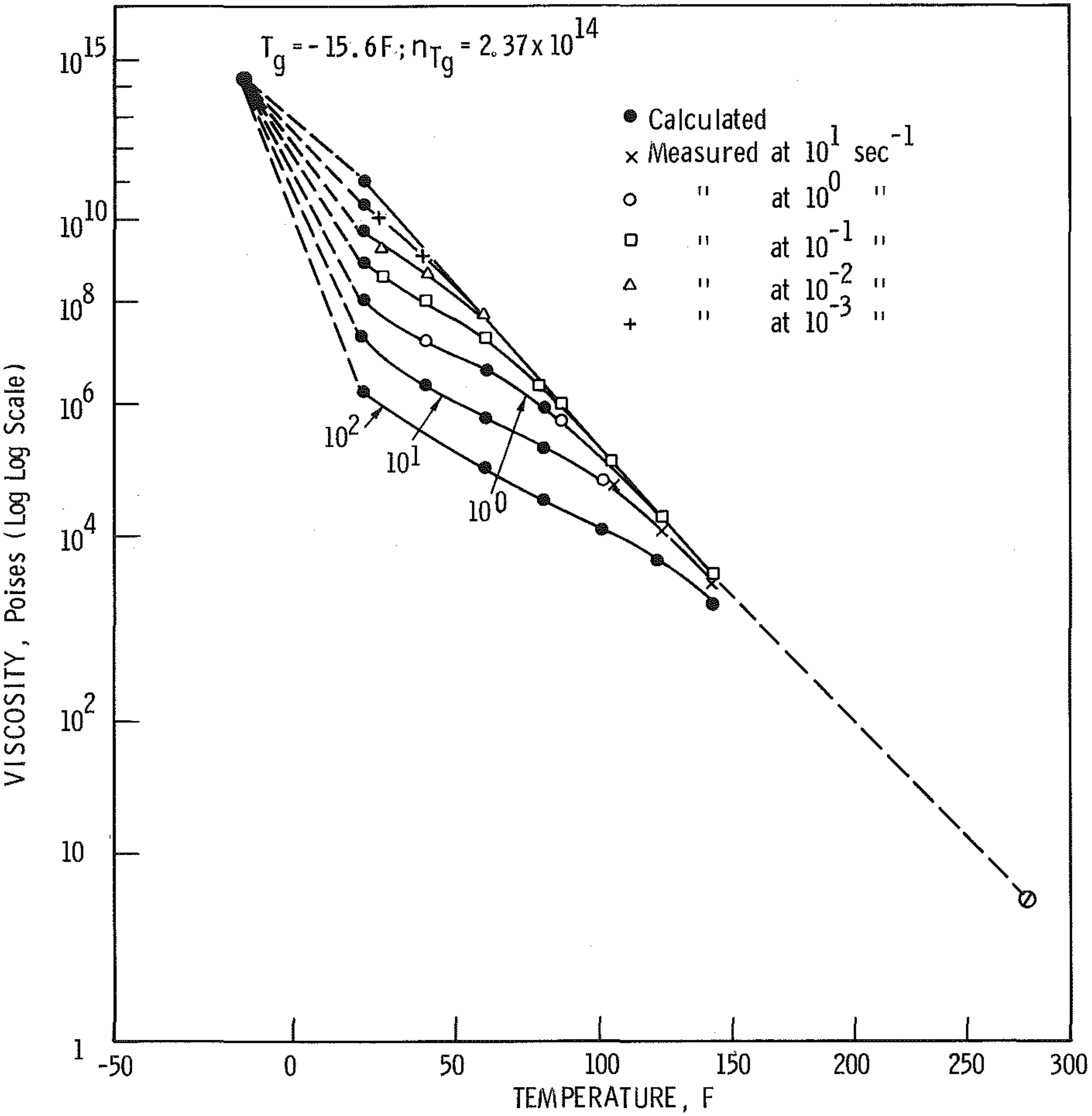

Figure 12. Viscosity versus temperature for Michigan asphalt - 73B-1 (60/70 pen asphalt).

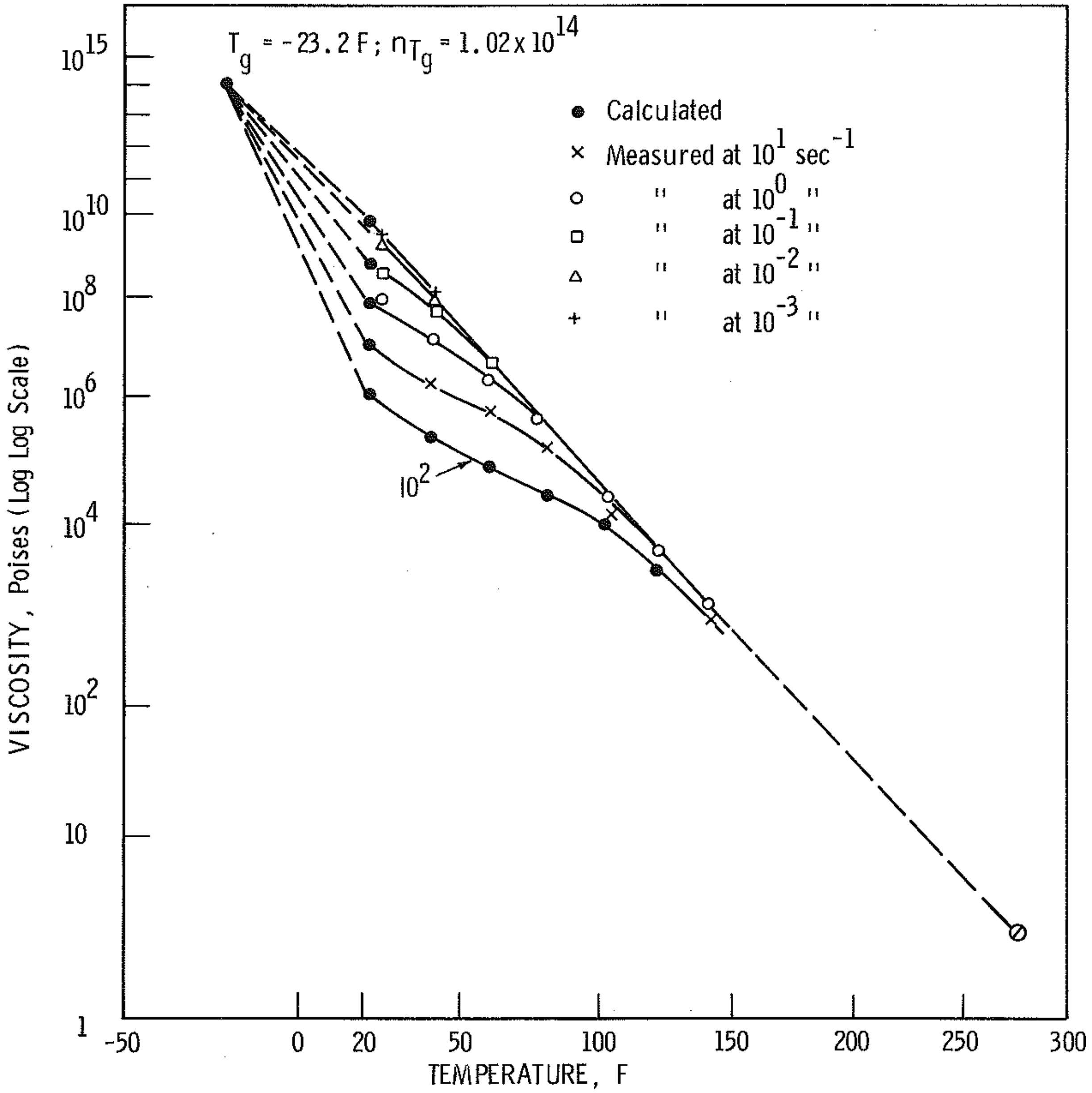

Figure 13. Temperature versus viscosity for Michigan asphalt - 73B-23 (120/150 pen asphalt).

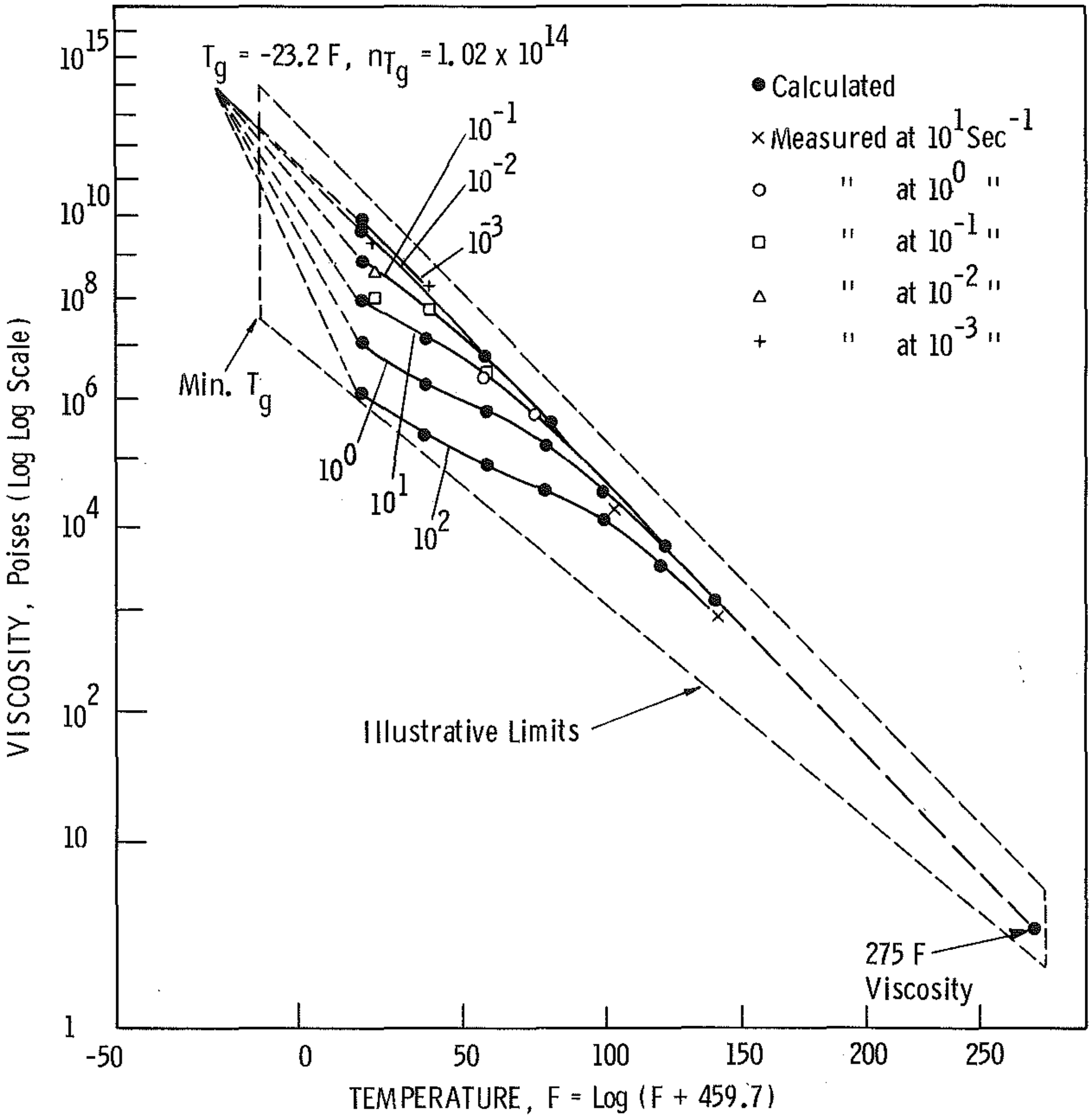

Figure 14. Temperature versus viscosity for Michigan asphalt - 73B-23 (120/150 pen).

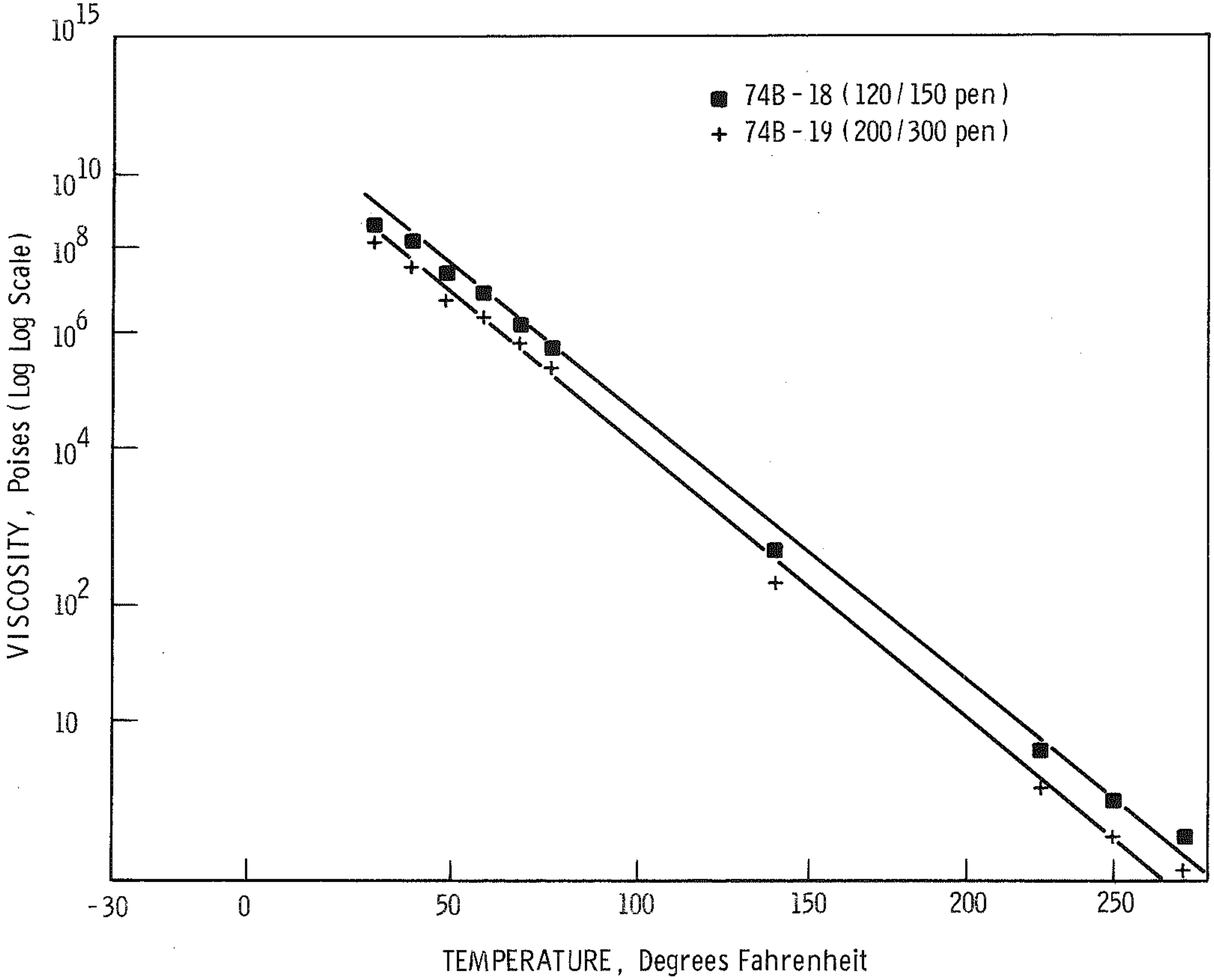

Figure 15. Viscosity-temperature chart for producer P - 74B-18 and 74B-19.

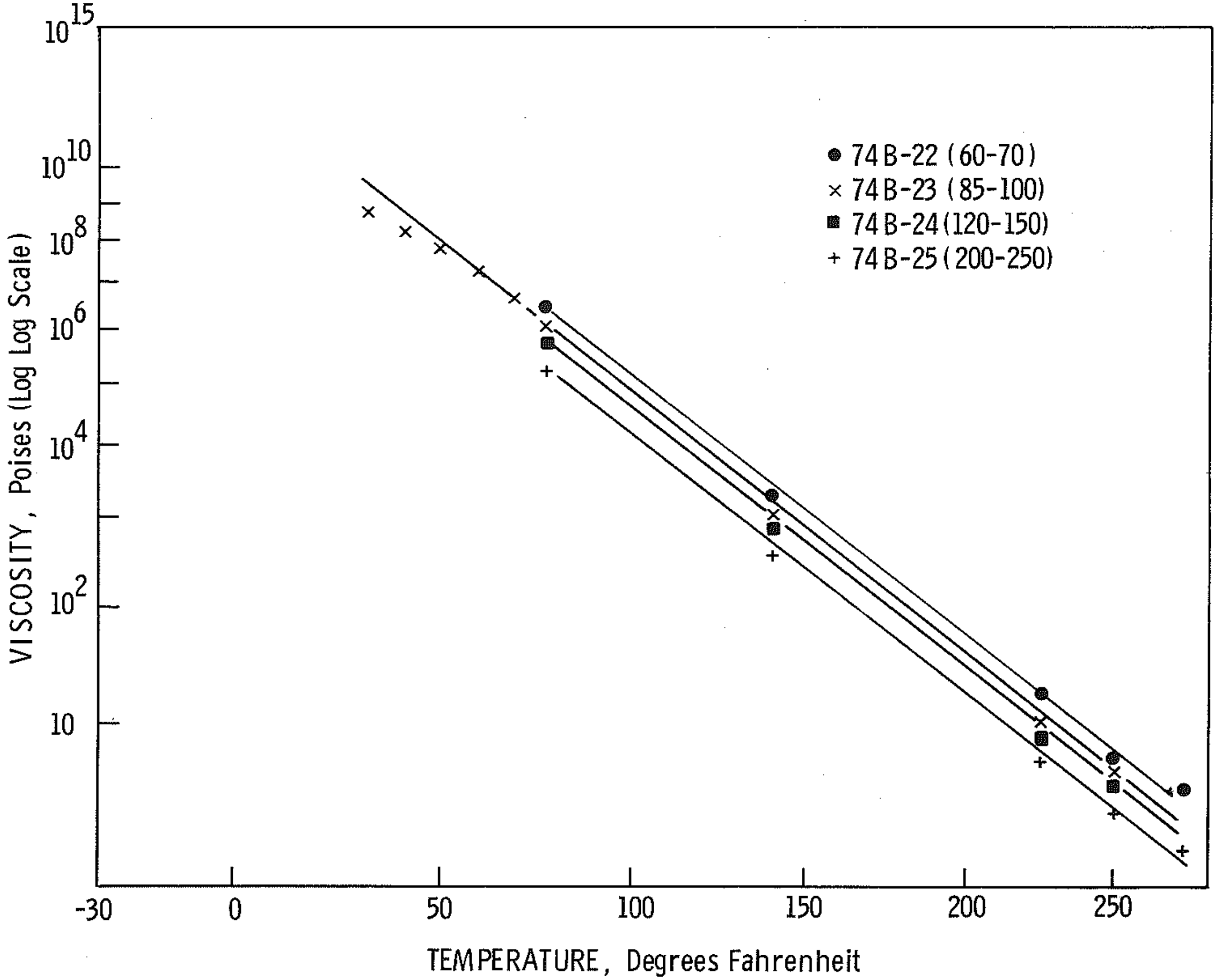

Figure 15A. Viscosity-temperature chart - American (Whiting) 74B-22, 74B-23, 74B-24, and 74B-25.

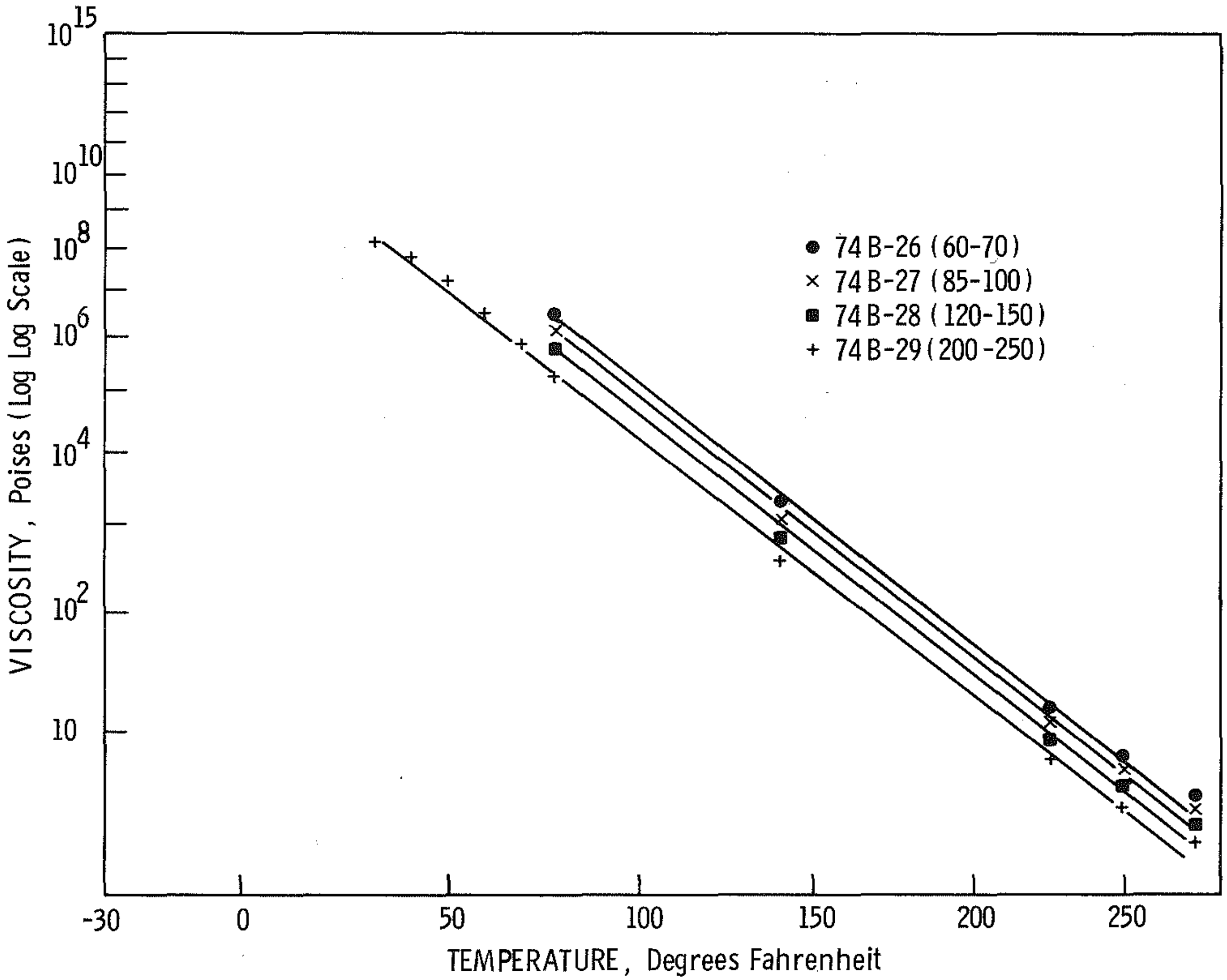

Figure 15B. Viscosity-temperature chart - Atlantic Richfield 74B-26, 74B-27, 74B-28, and 74B-29.

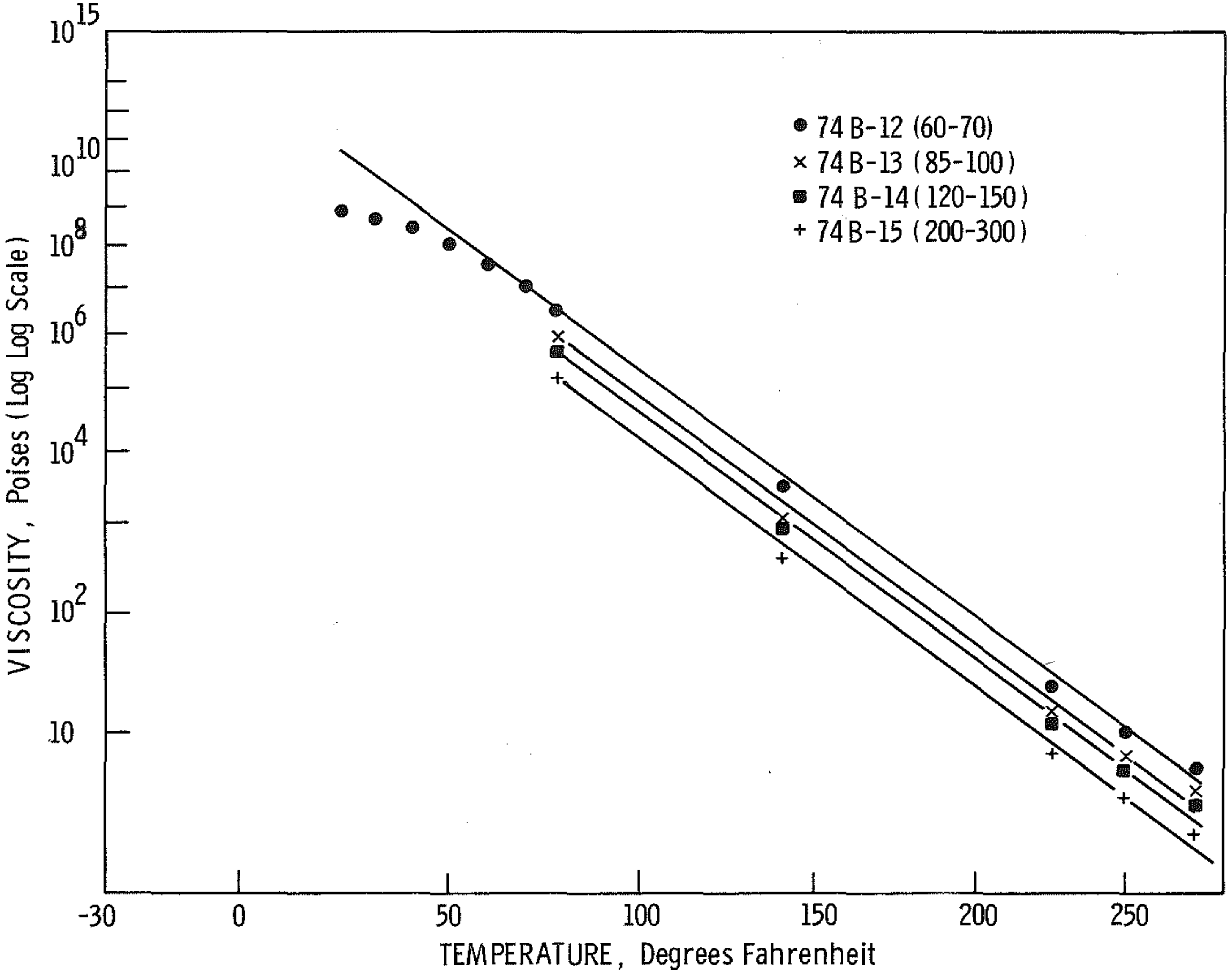

Figure 15C. Viscosity-temperature chart - Lion 74B-12, 74B-13, 74B-14, and 74B-15.

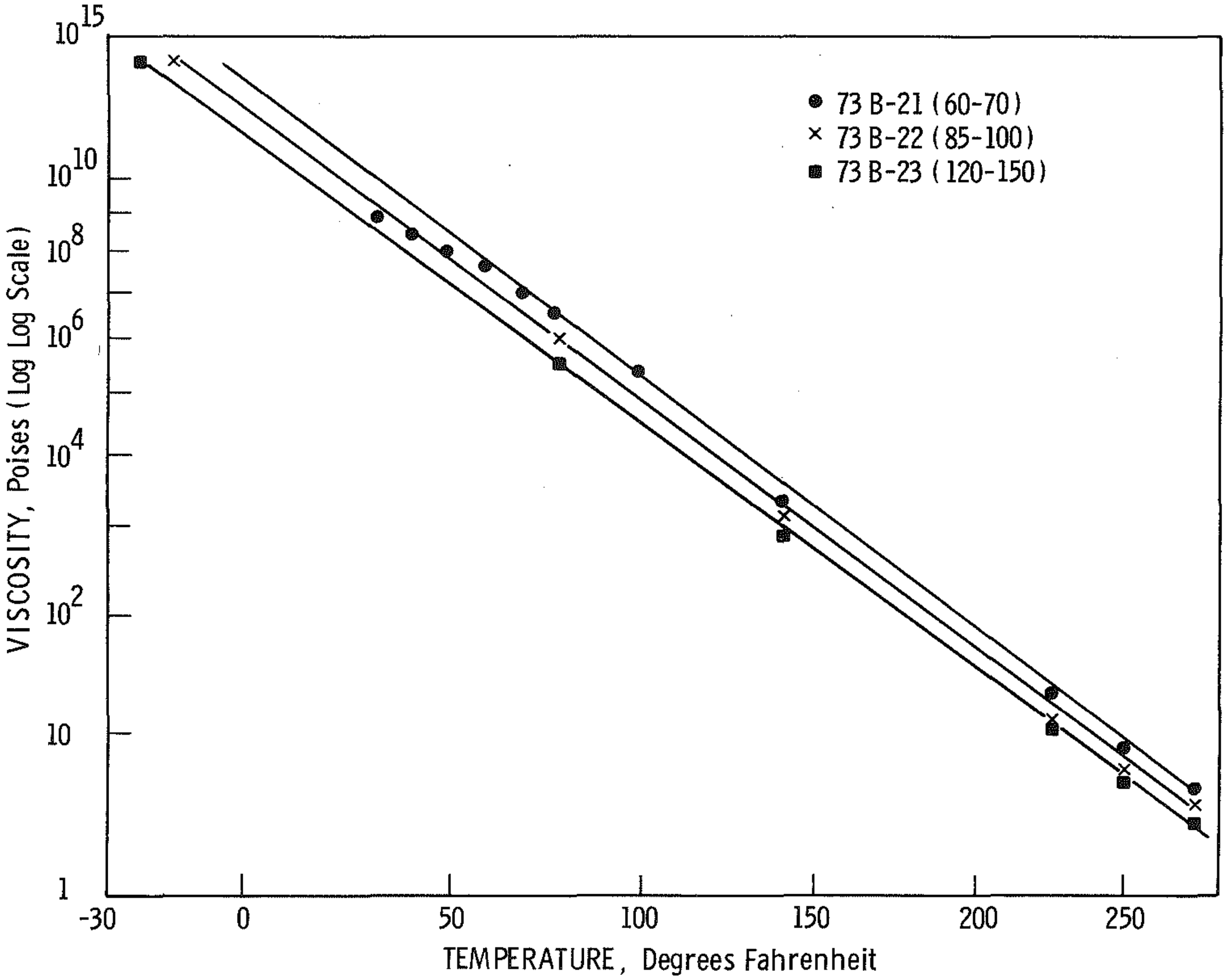

Figure 15D. Viscosity-temperature chart - Trumbull 73B-21, 73B-22, and 73B-23.

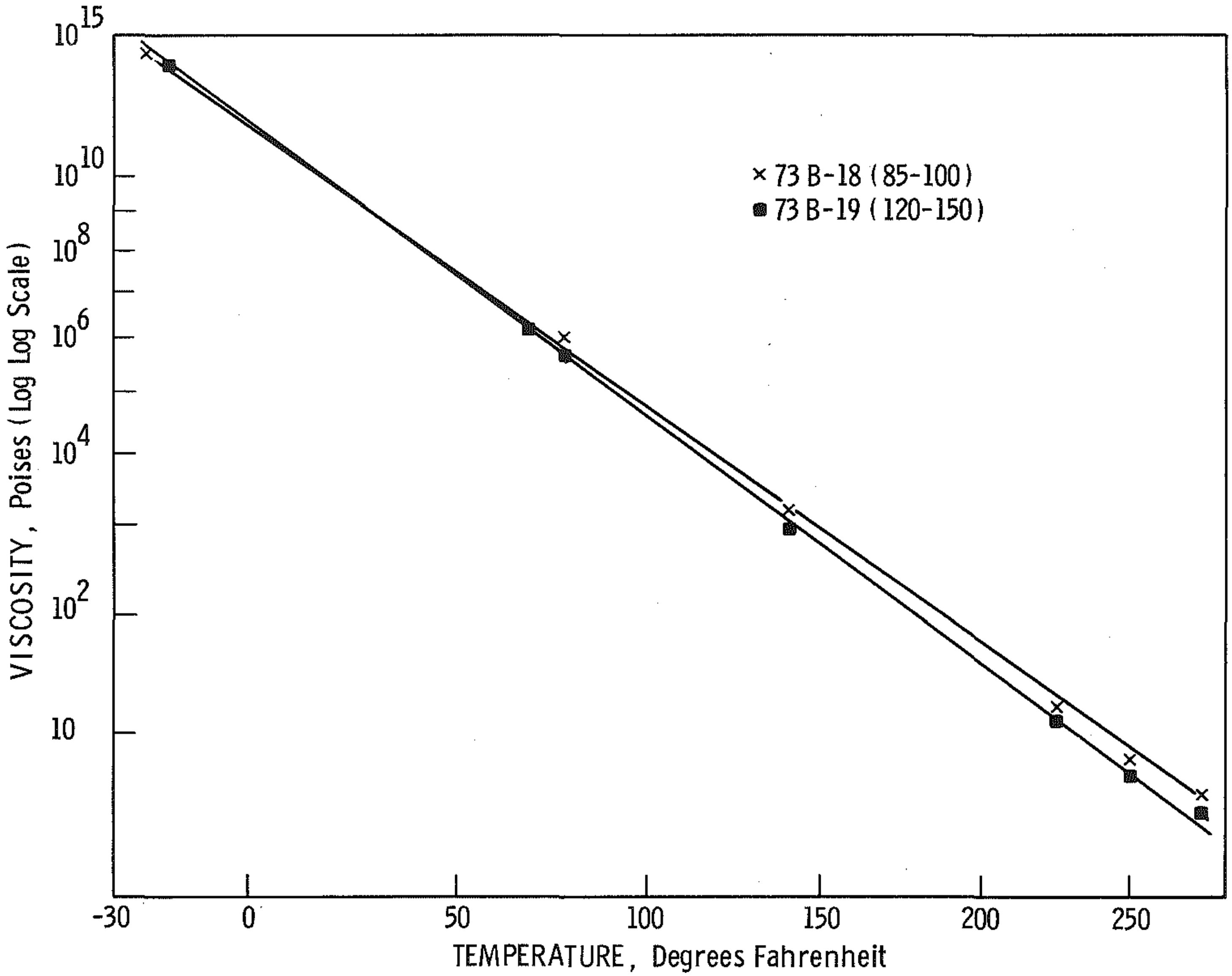

Figure 15E. Viscosity-temperature chart - Leonard 73B-18, and 73B-19

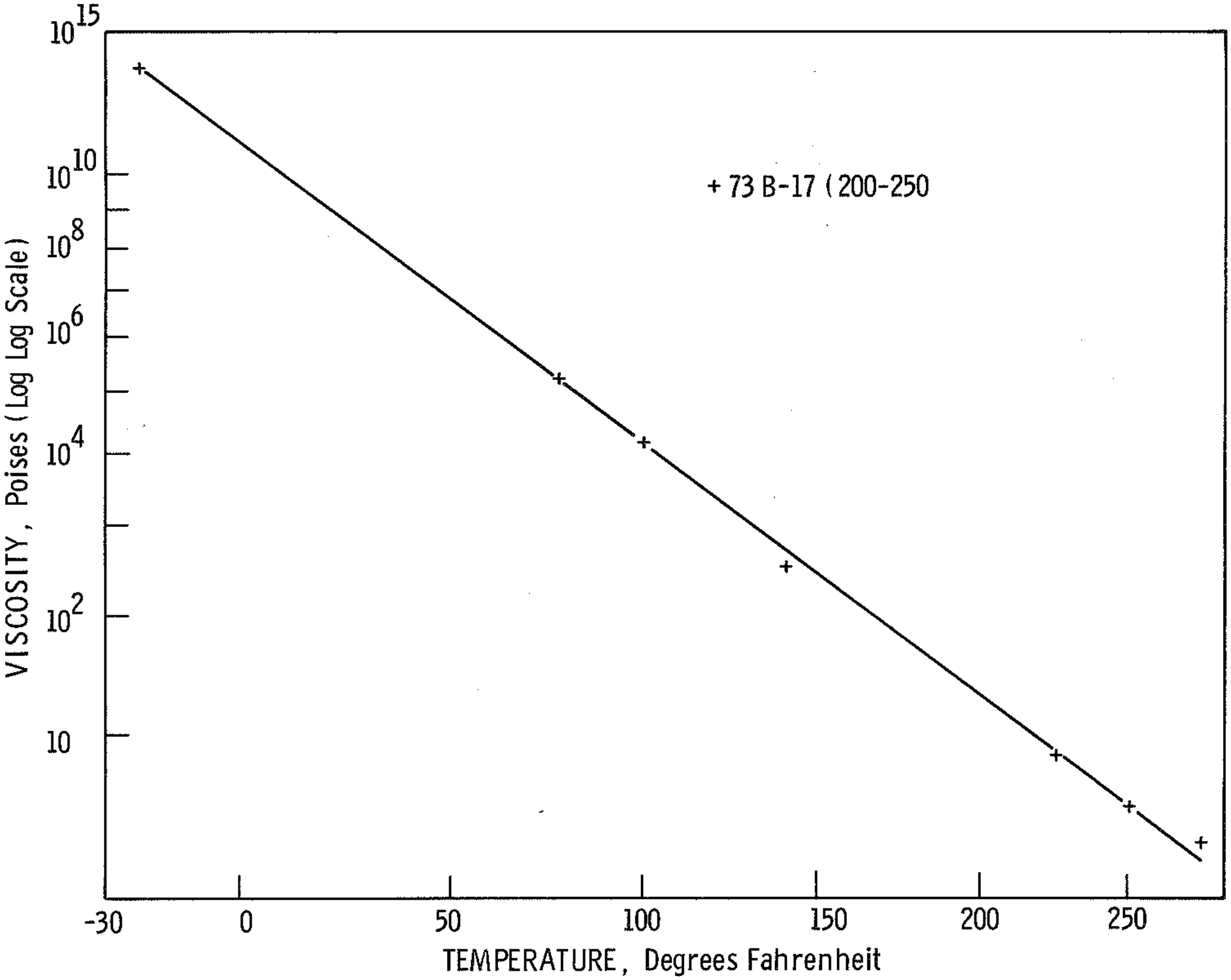

Figure 15F. Viscosity-temperature chart - Arco 73B-17.

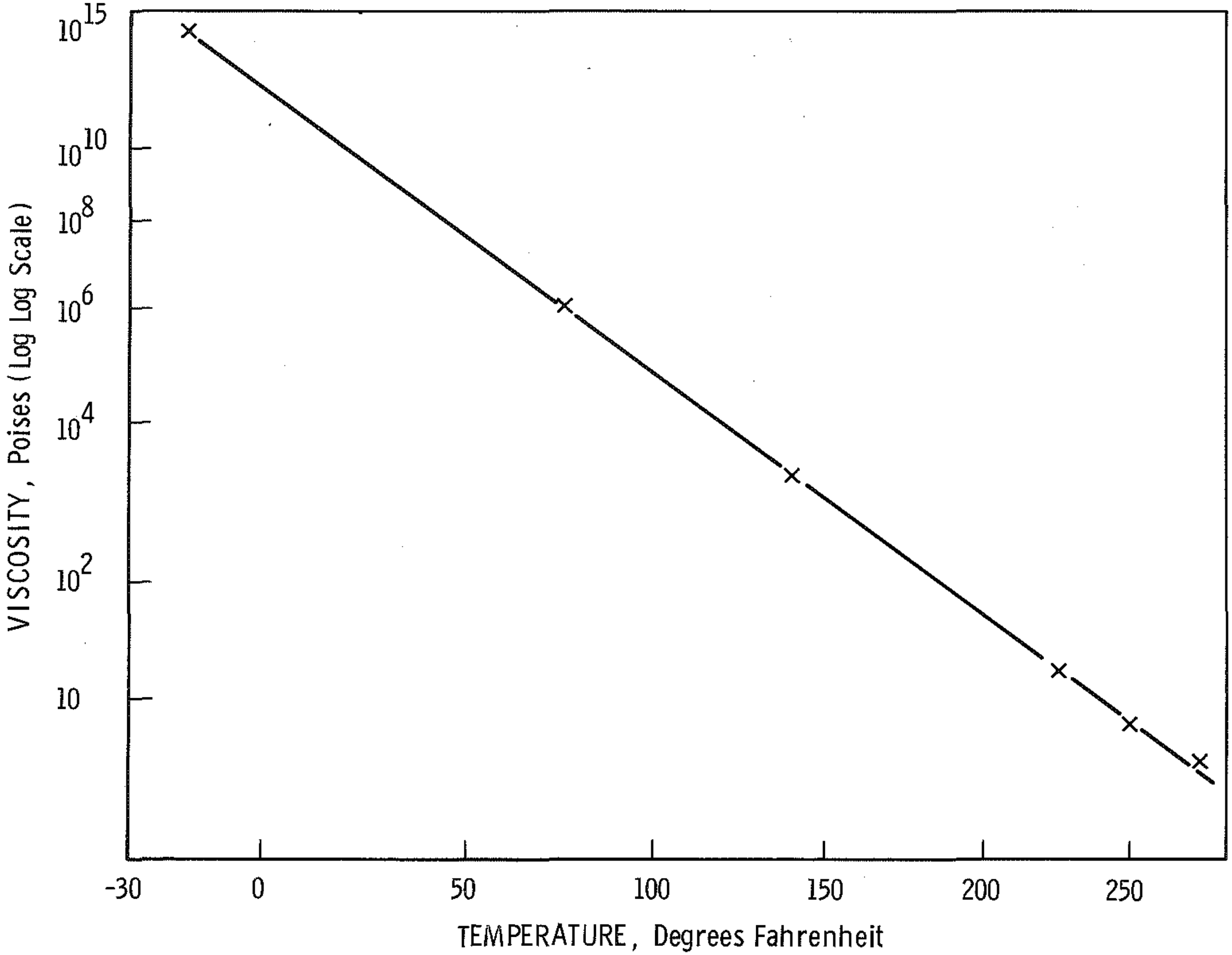

Figure 15G. Viscosity-temperature chart for producer D - 73B-13 (85/100 pen asphalt).

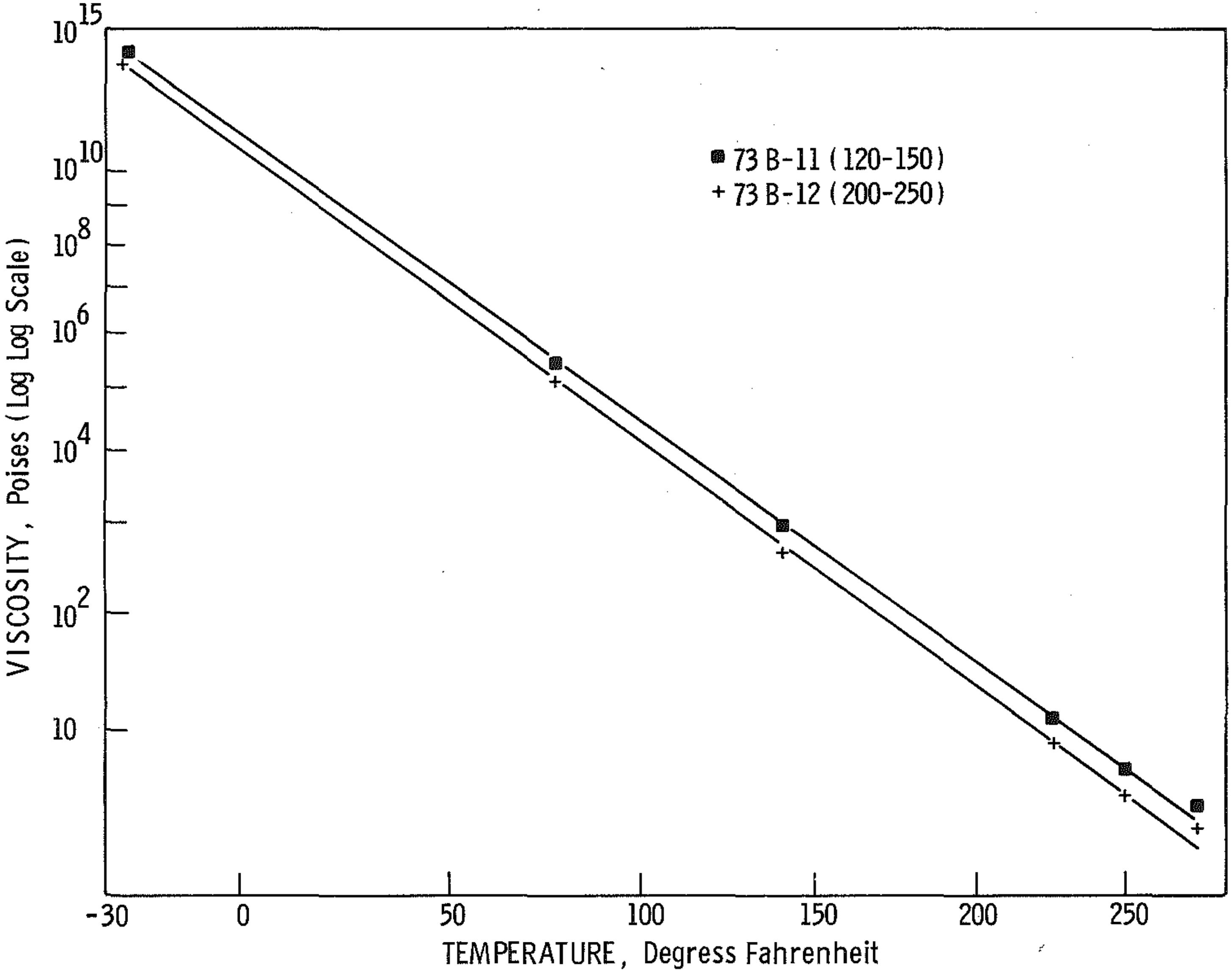

Figure 15H. Viscosity-temperature chart - Petrofina 73B-11 and 73B-12.

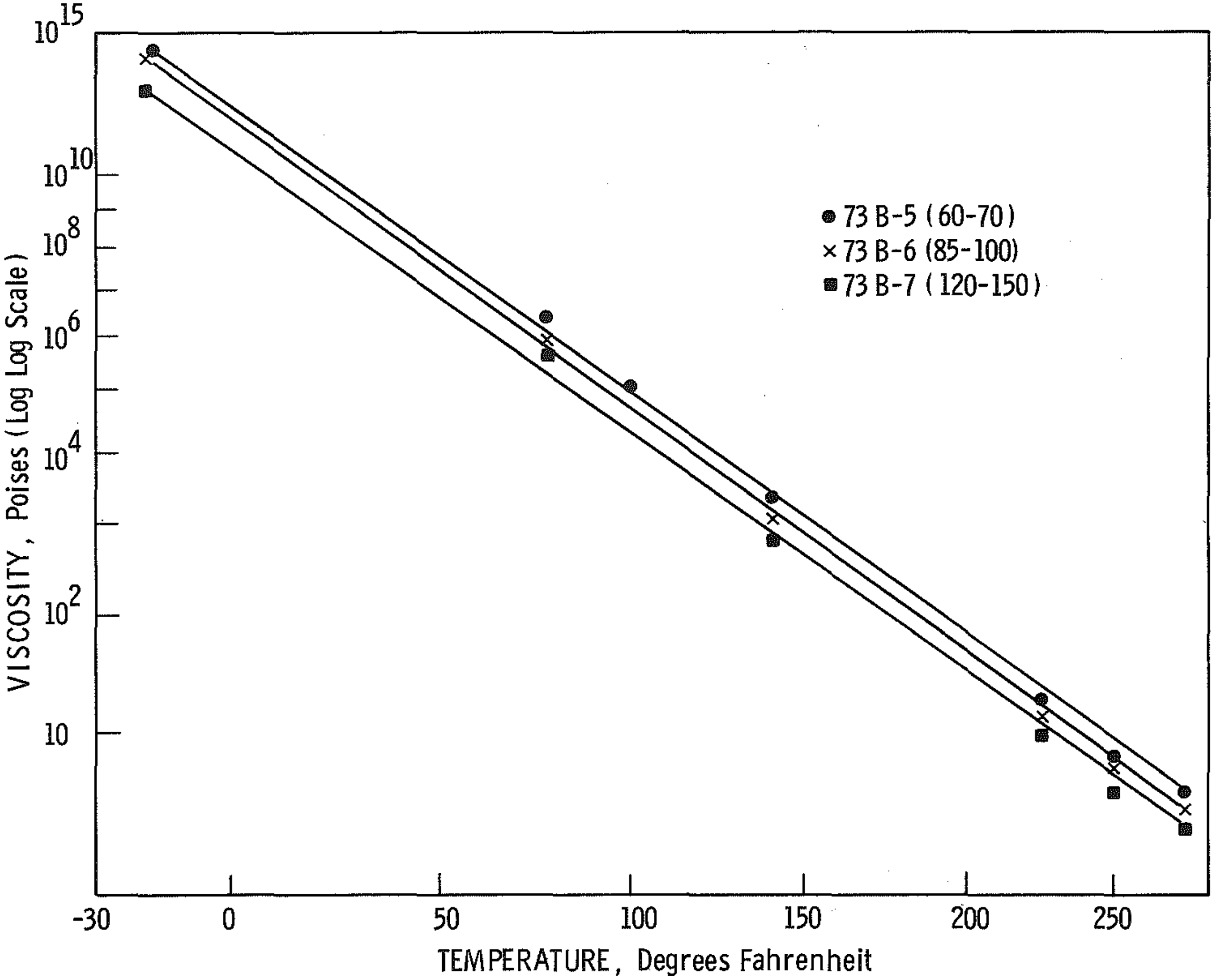

Figure 15I. Viscosity-temperature chart - Amoco 73B-5, 73B-6, and 73B-7.

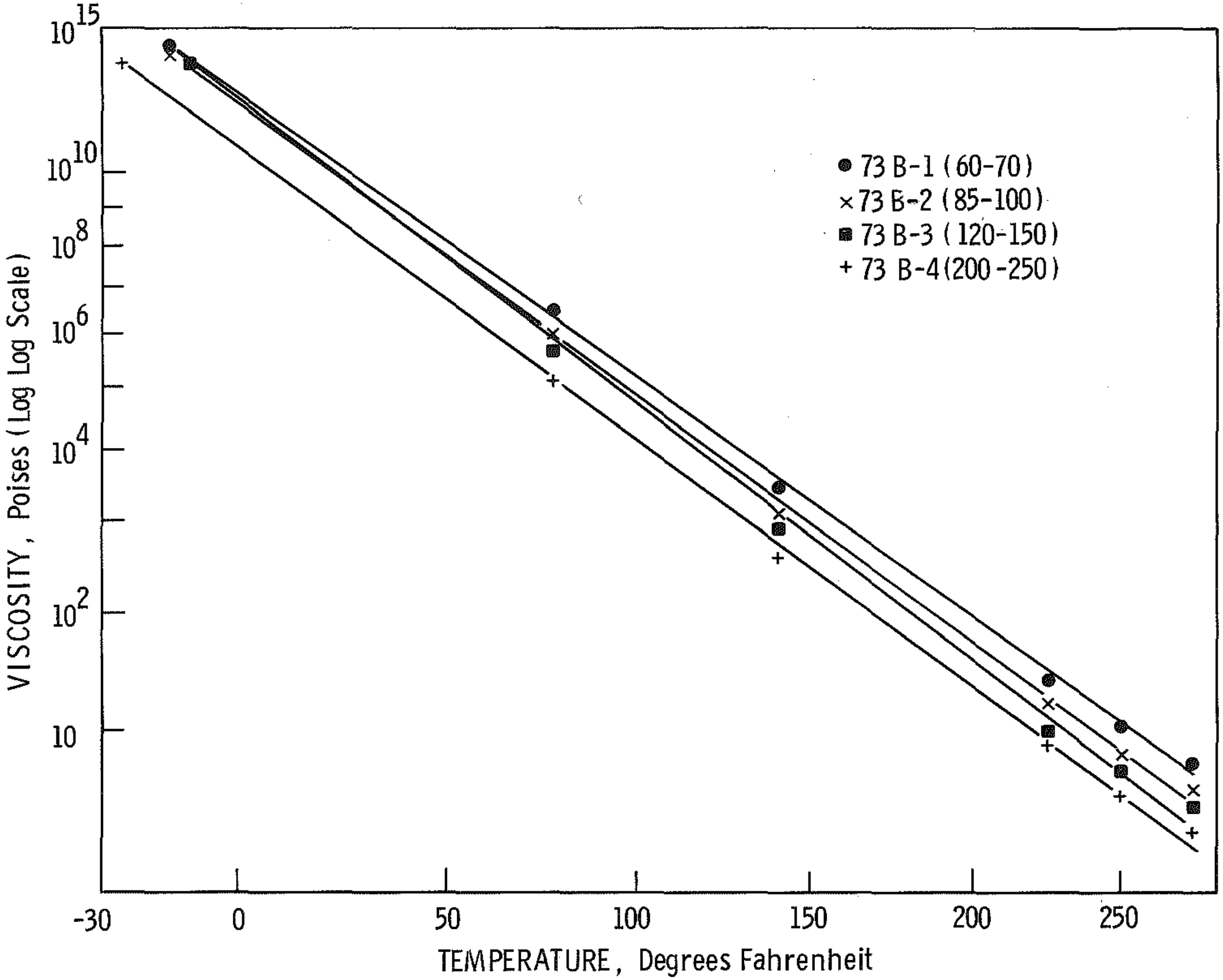

Figure 15J. Viscosity-temperature chart - Lion 13B-1, 73B-2, 73B-3, and 73B-4.

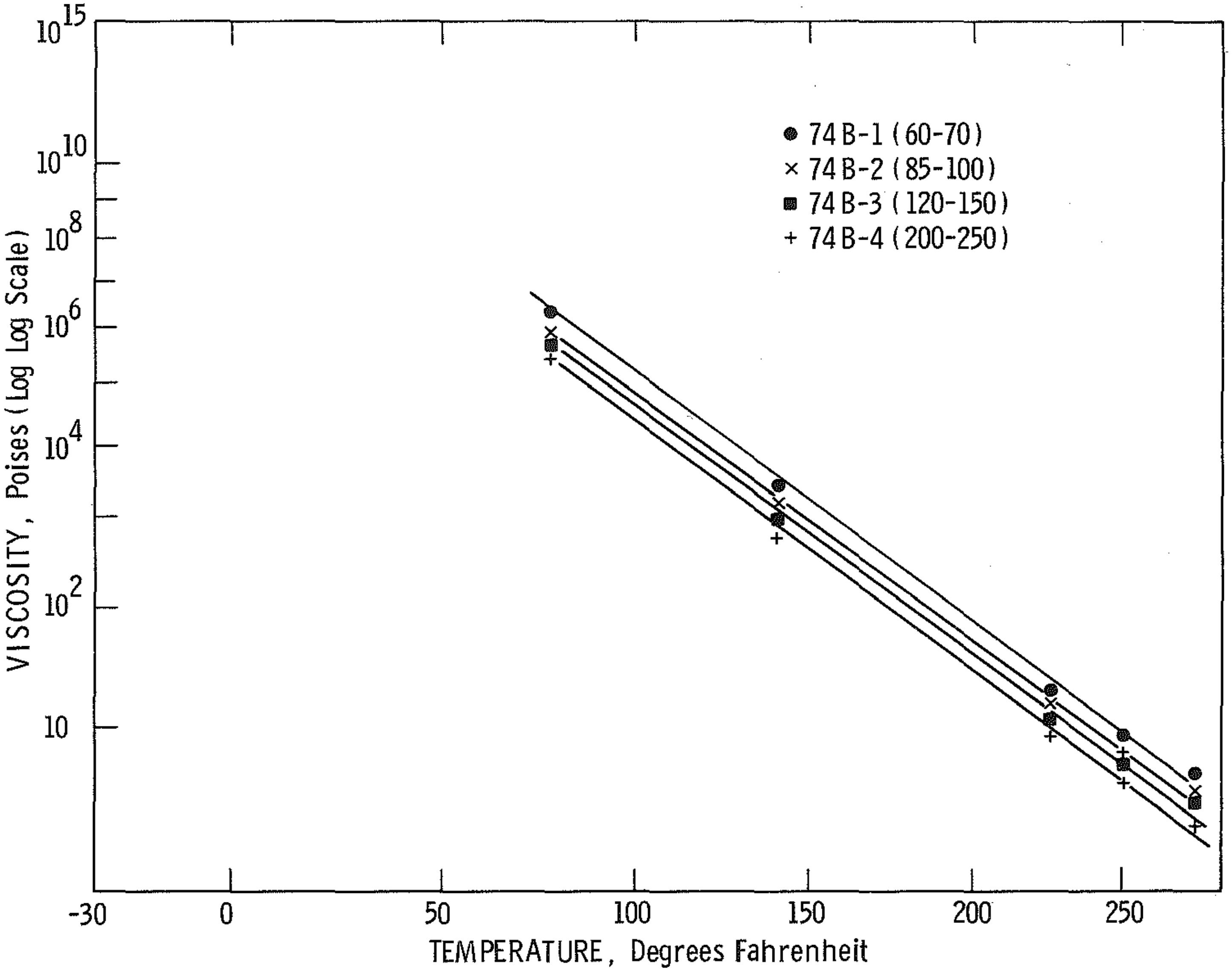

Figure 15K. Viscosity-temperature chart - Total-Leonard 74B-1, 74B-2, 74B-3, and 74B-4.

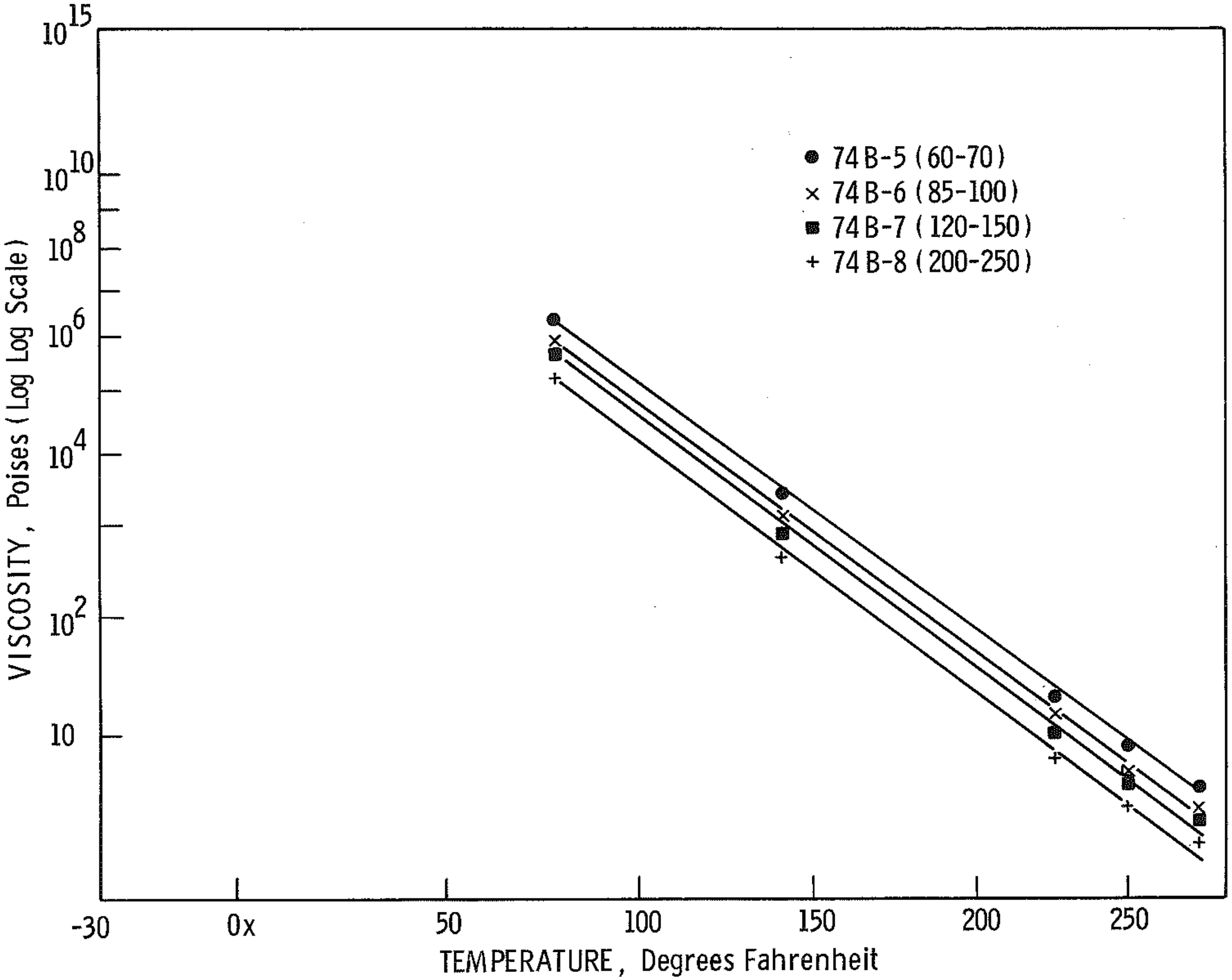

Figure 15L. Viscosity-temperature chart - Trumbull 74B-5, 74B-6, 74B-7, and 74B-8.

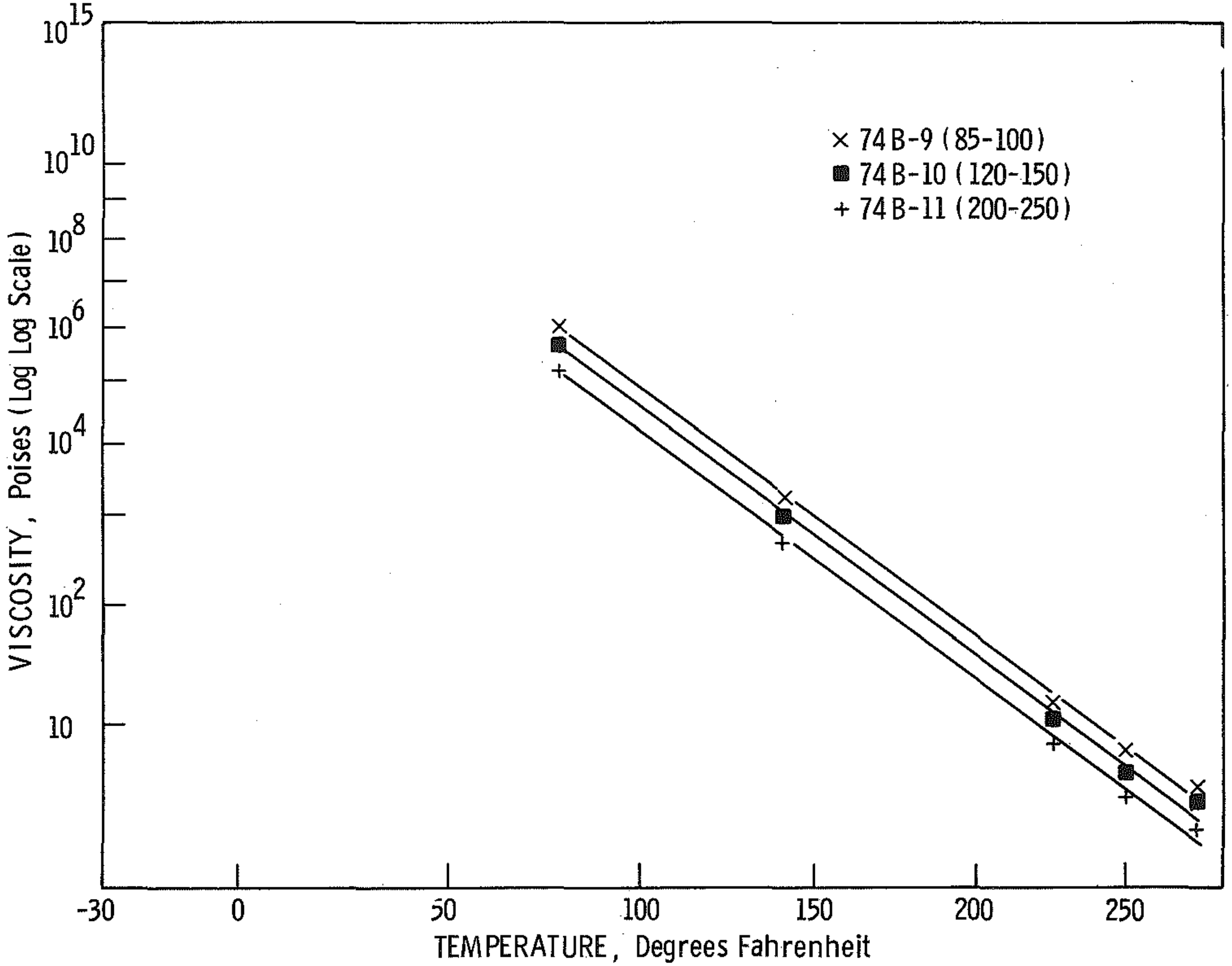

Figure 15M. Viscosity-temperature chart - Murphy 74B-9, 74B-10, and 74B-11.

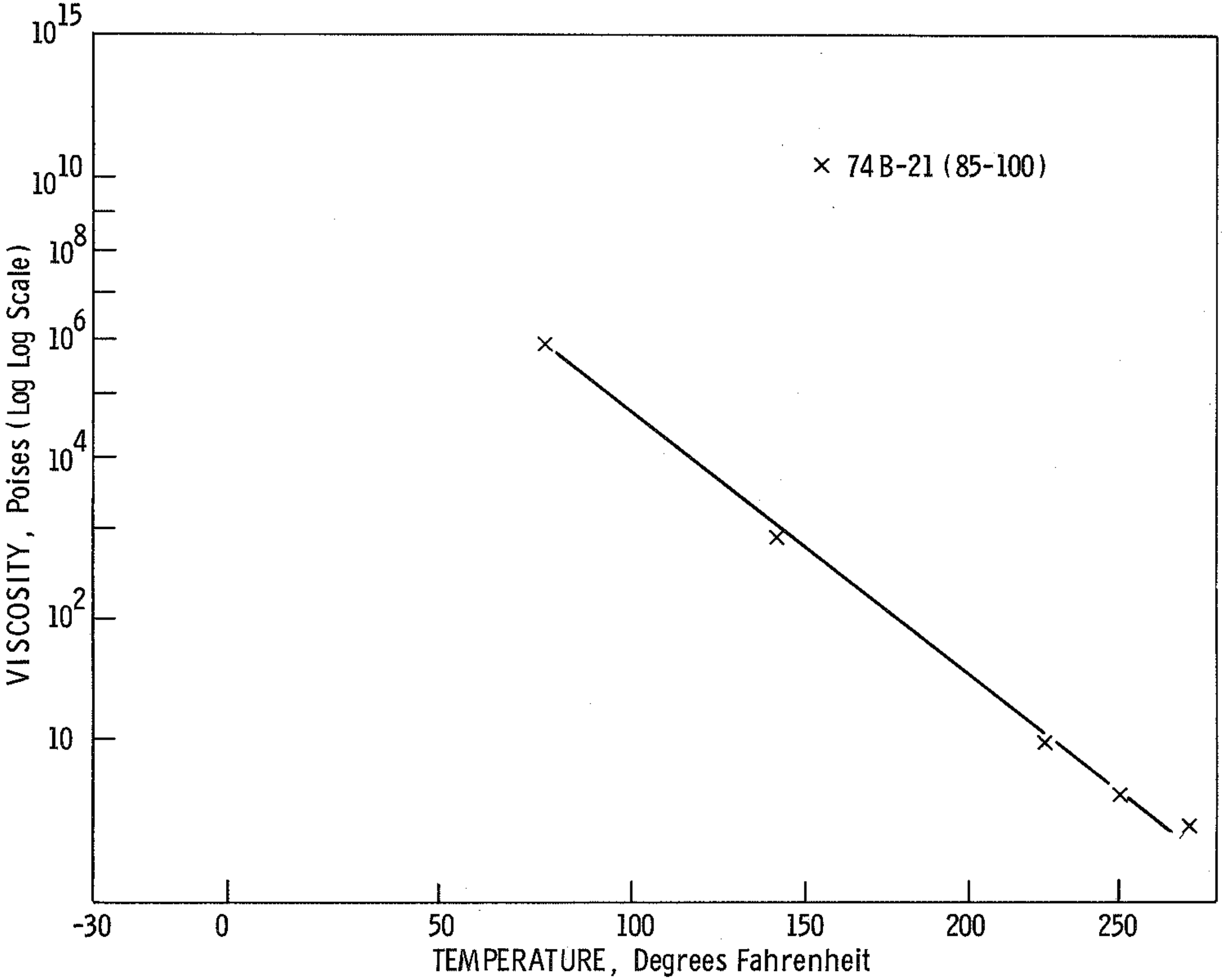

Figure 15N. Viscosity-temperature chart - American S.C. (50-50) 74B-21.

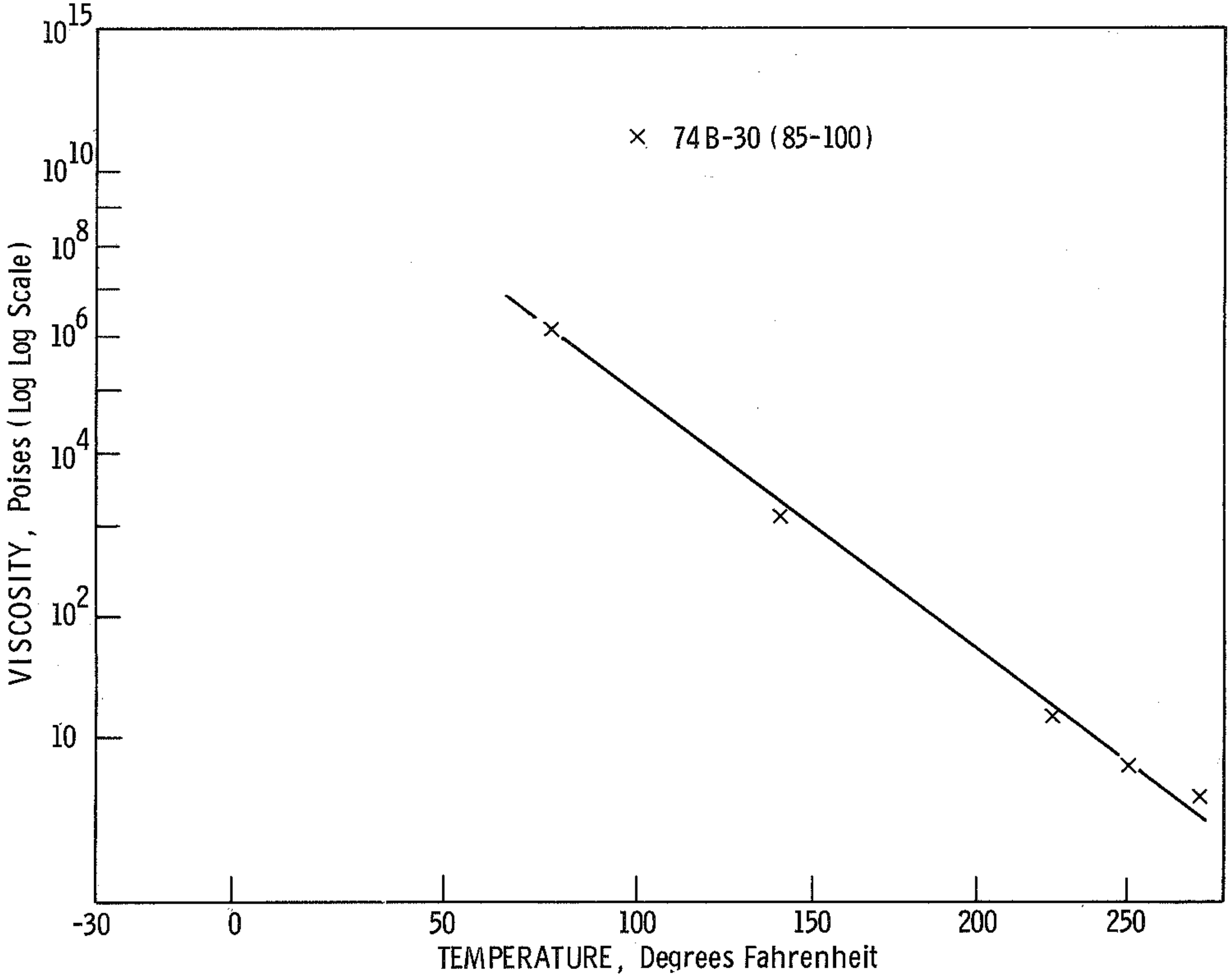

Figure 150. Viscosity-temperature chart - Imperial 74B-30.

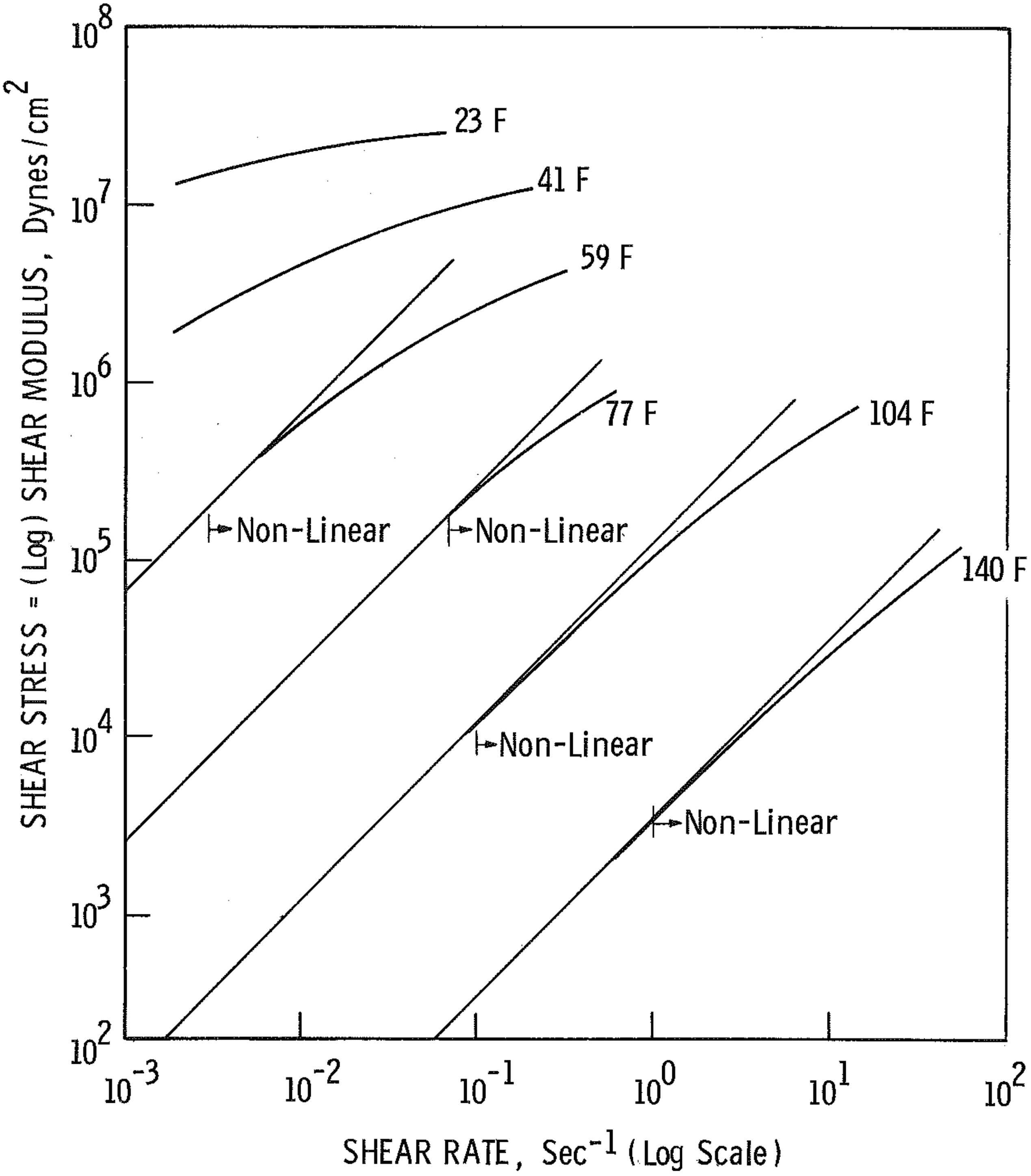

Figure 16. Shear modulus versus shear rate for asphalt 73B-1 (60/70 pen).

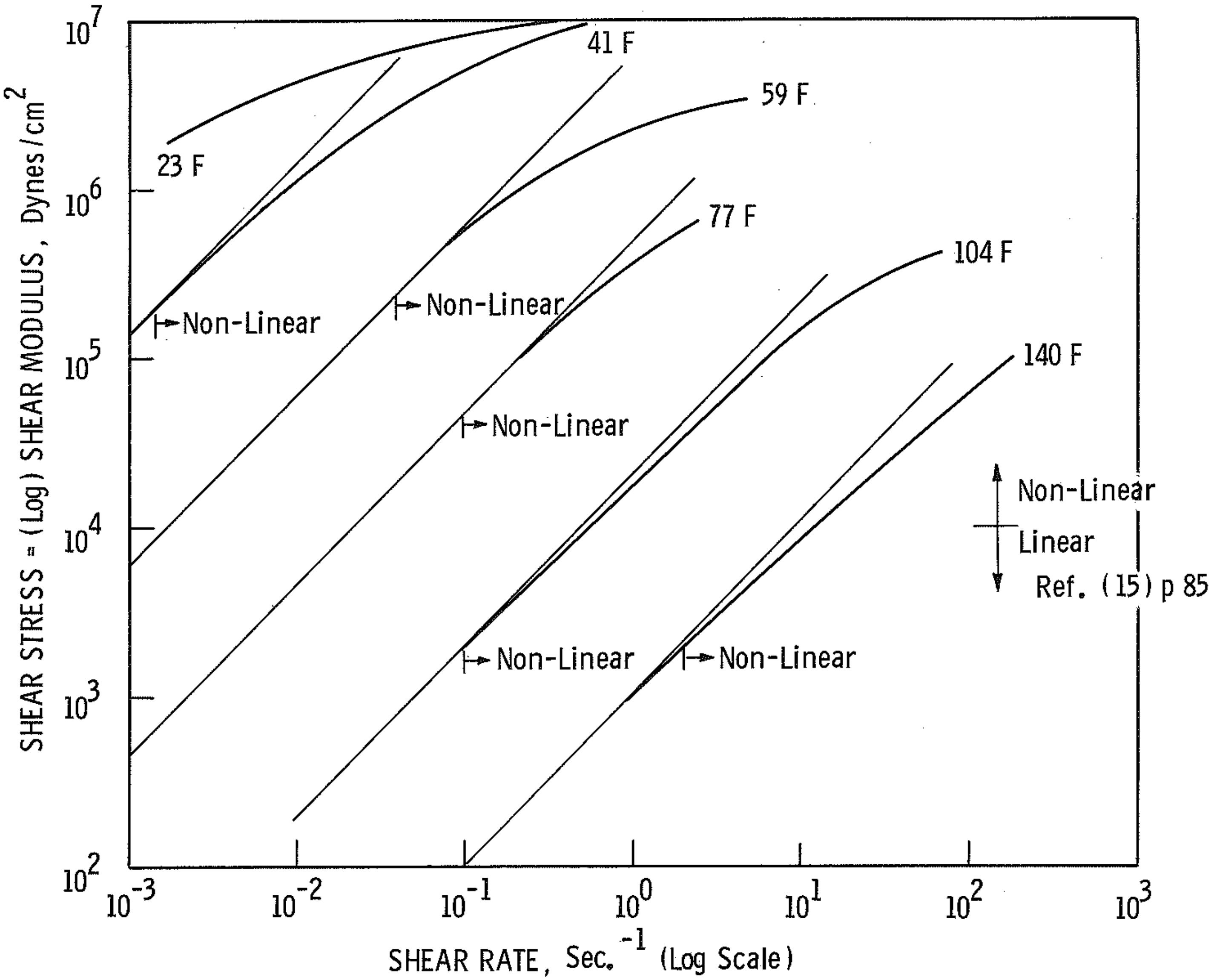

Figure 17. Shear modulus versus shear rate for asphalt 73B-23 (120/150 pen).

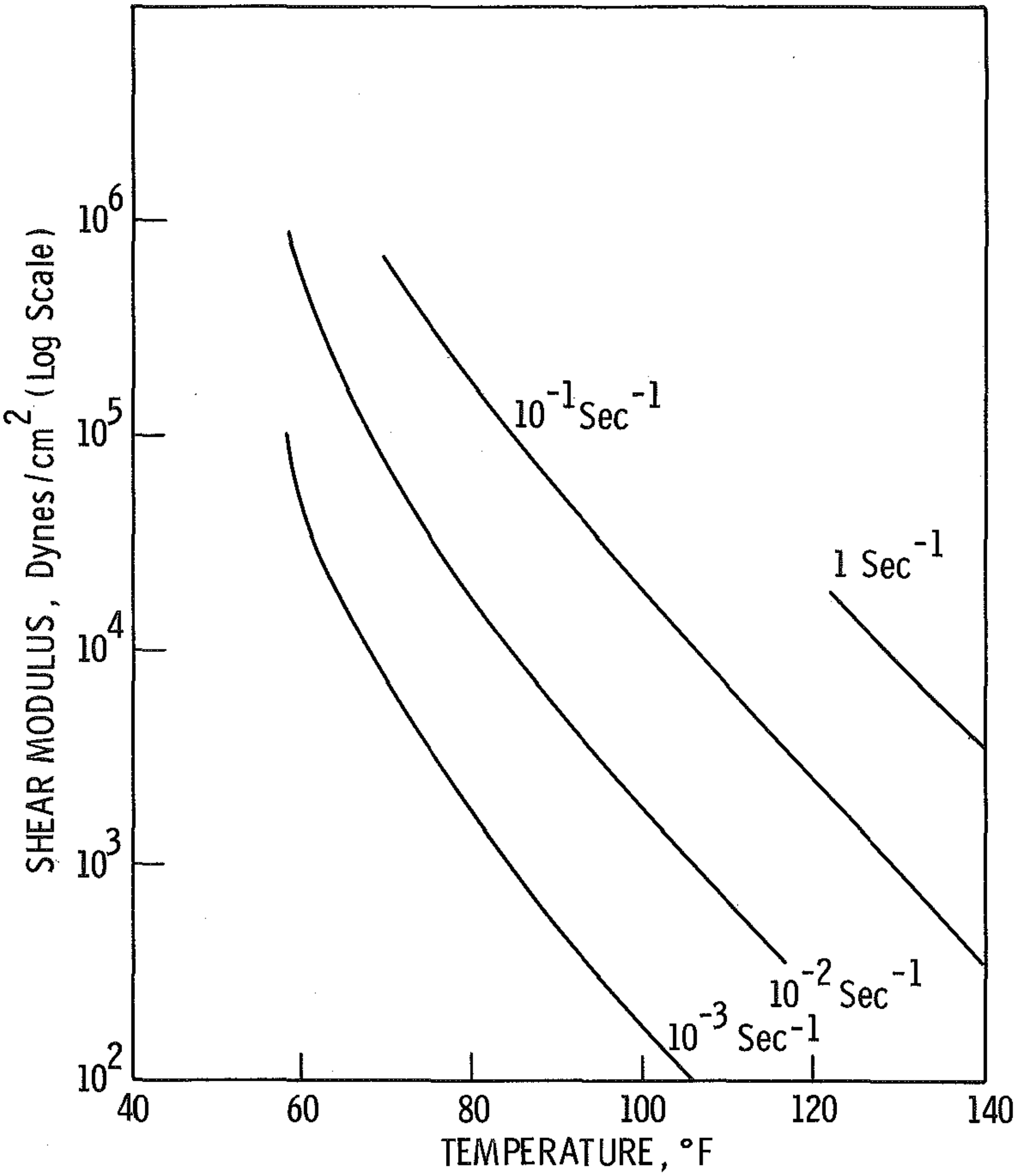

Figure 18. Shear modulus versus temperature for asphalt 73B-1 (60/70 pen).

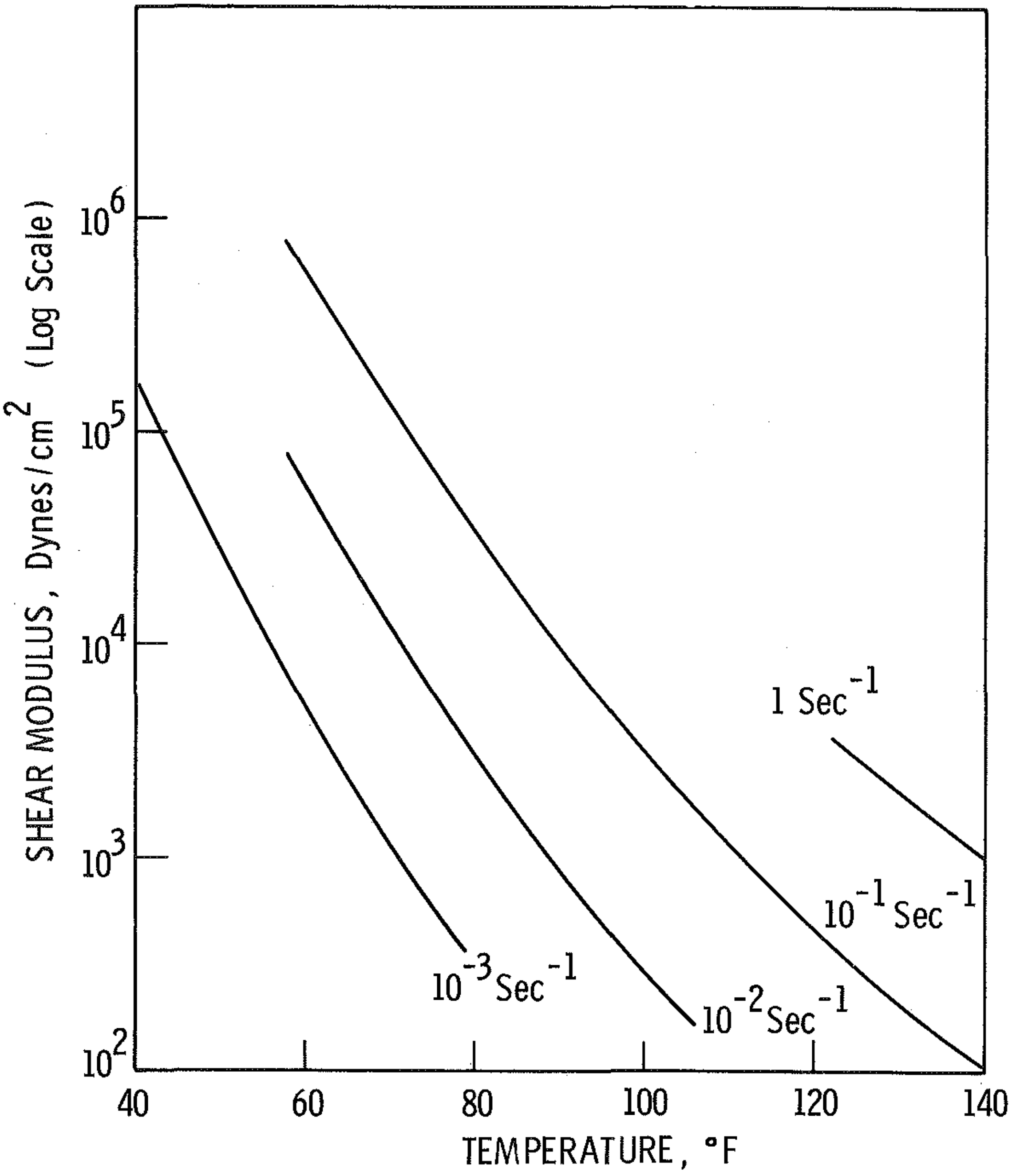

Figure 19. Shear modulus versus temperature for asphalt 73B-23 (120/150 pen).

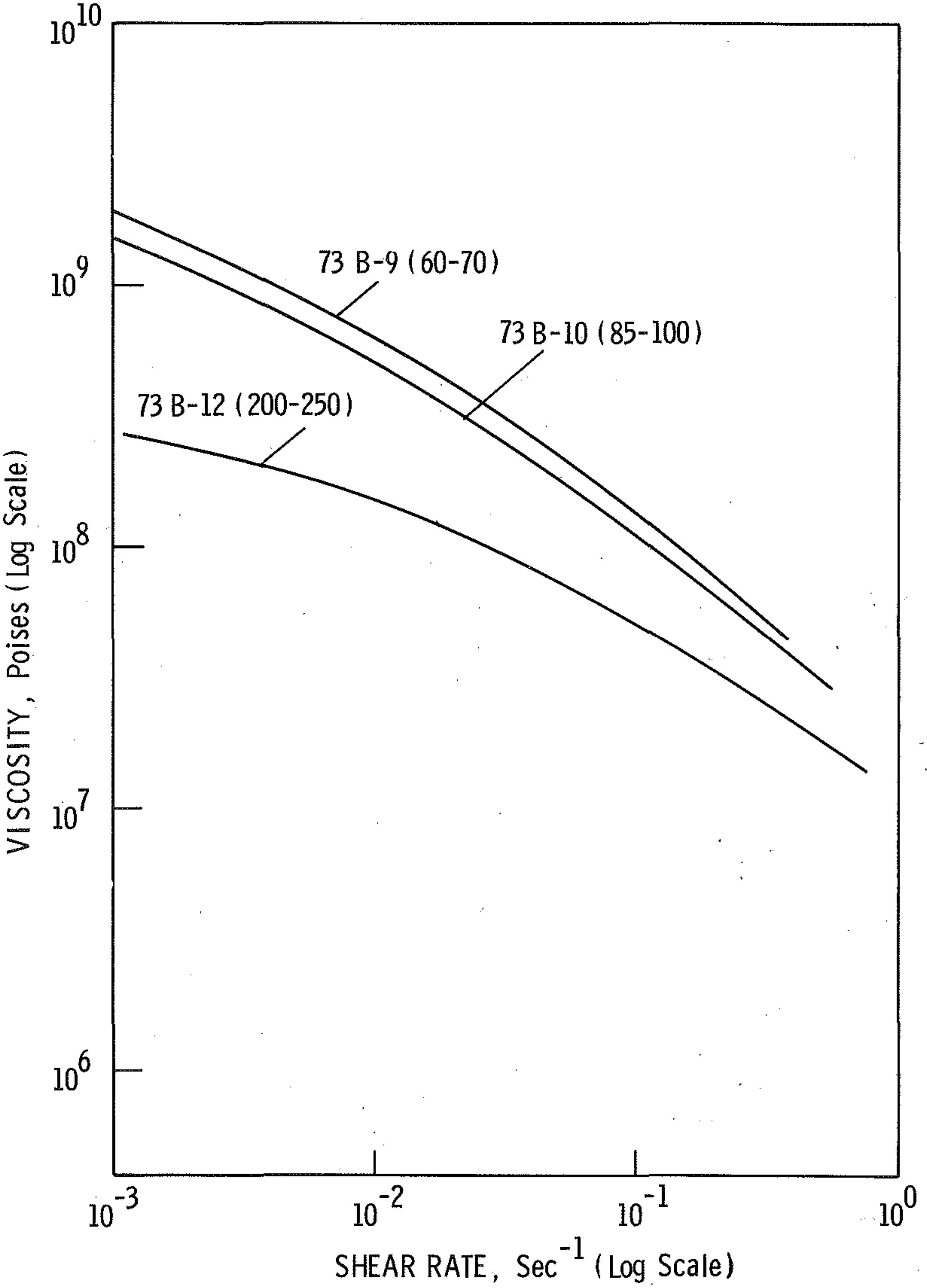

Figure 20. Viscosity versus shear rate at 32 F for three Michigan asphalts.

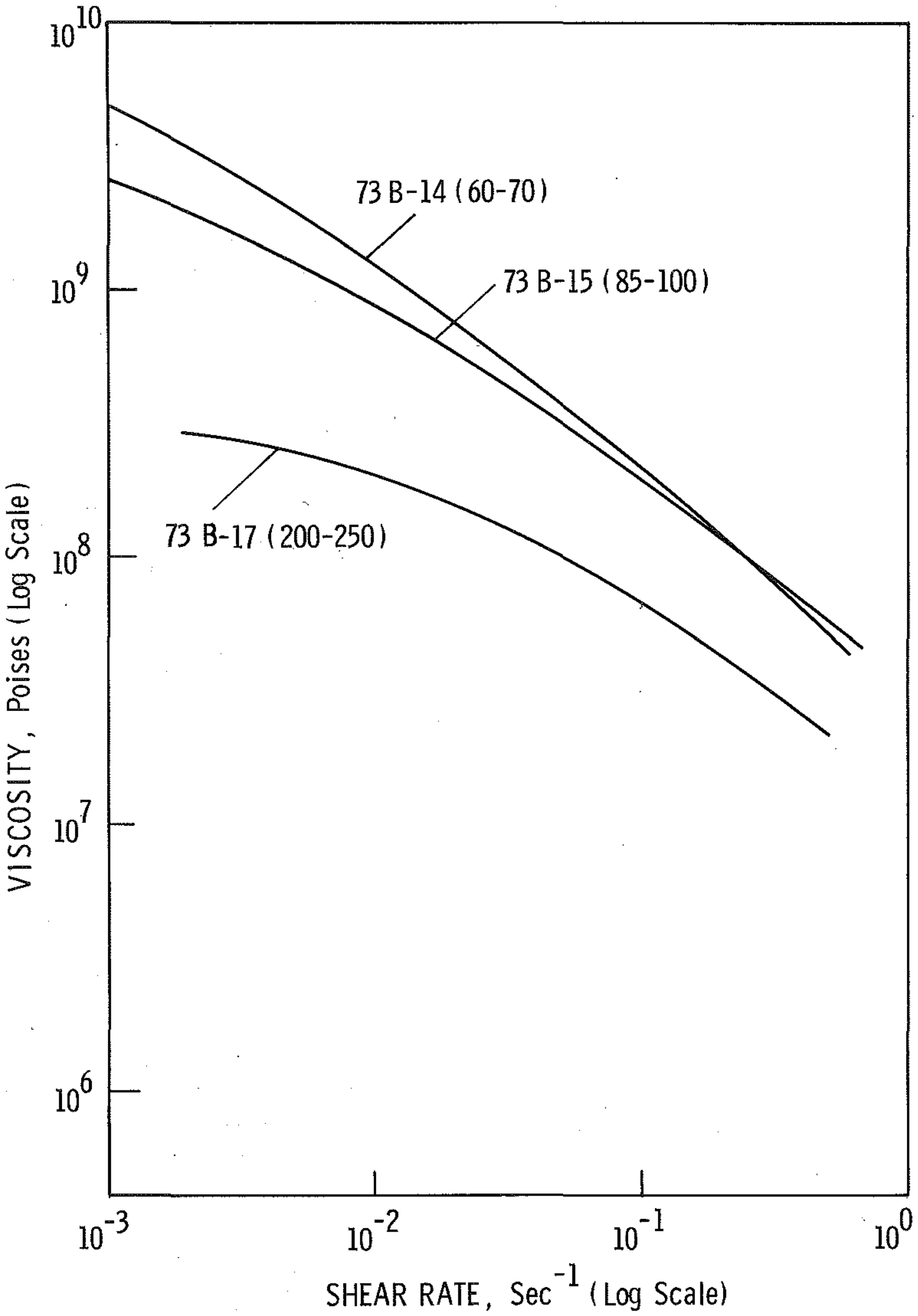

Figure 21. Viscosity versus shear rate at 32 F for three Michigan asphalts.

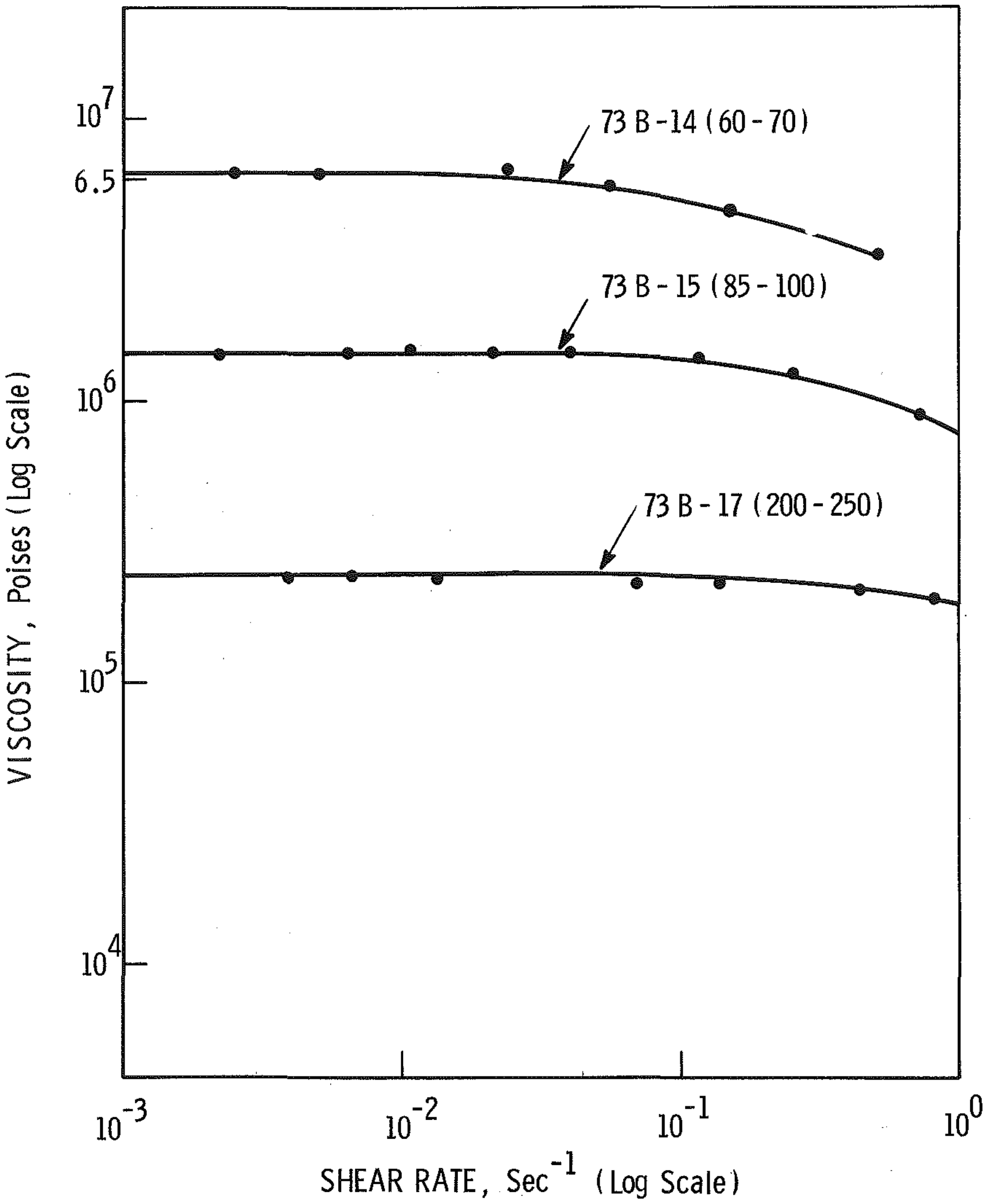

Figure 22. Viscosity versus shear rate at 25 C for three Michigan asphalts.

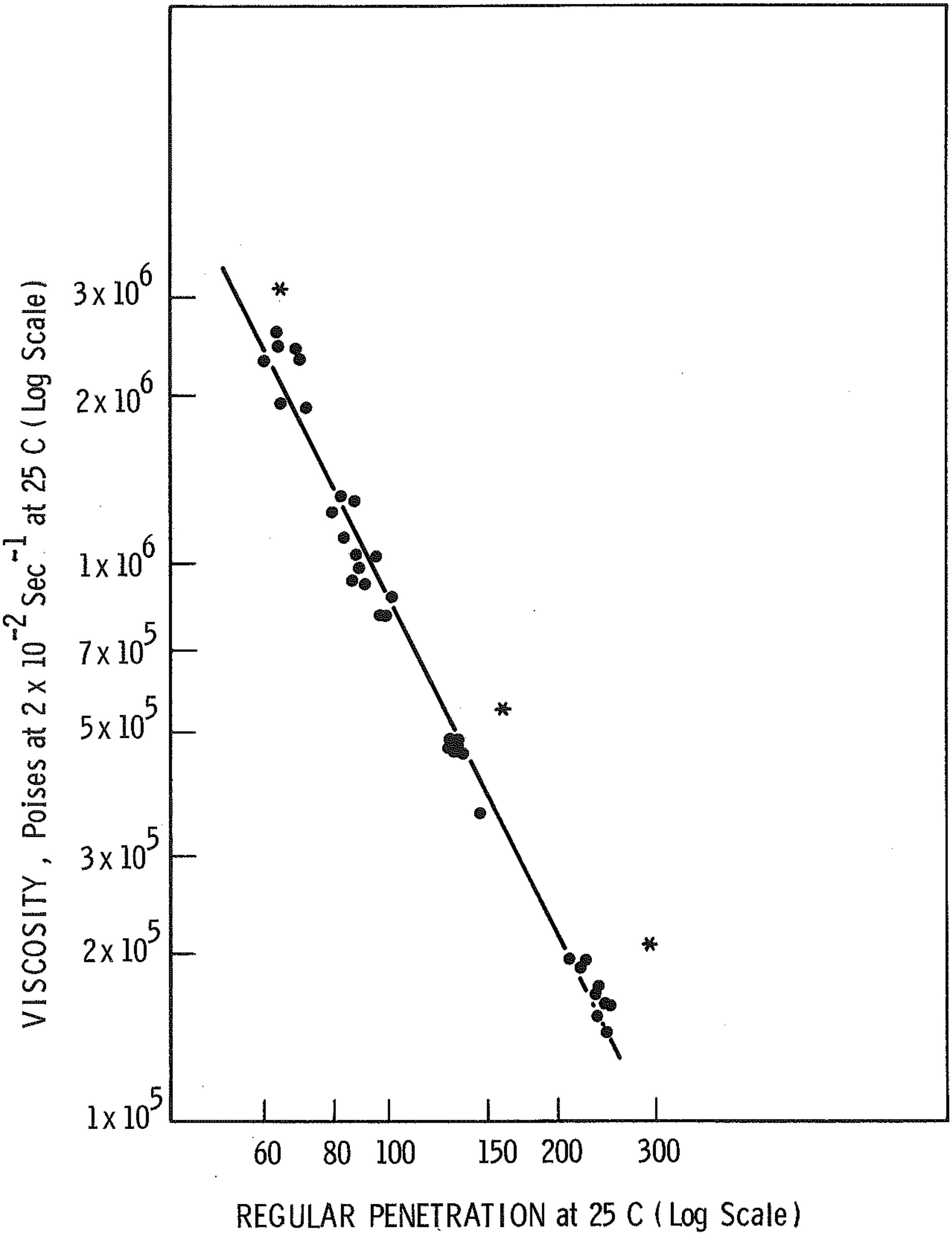

Figure 23. Viscosity versus standard penetration for 43 Michigan asphalts.

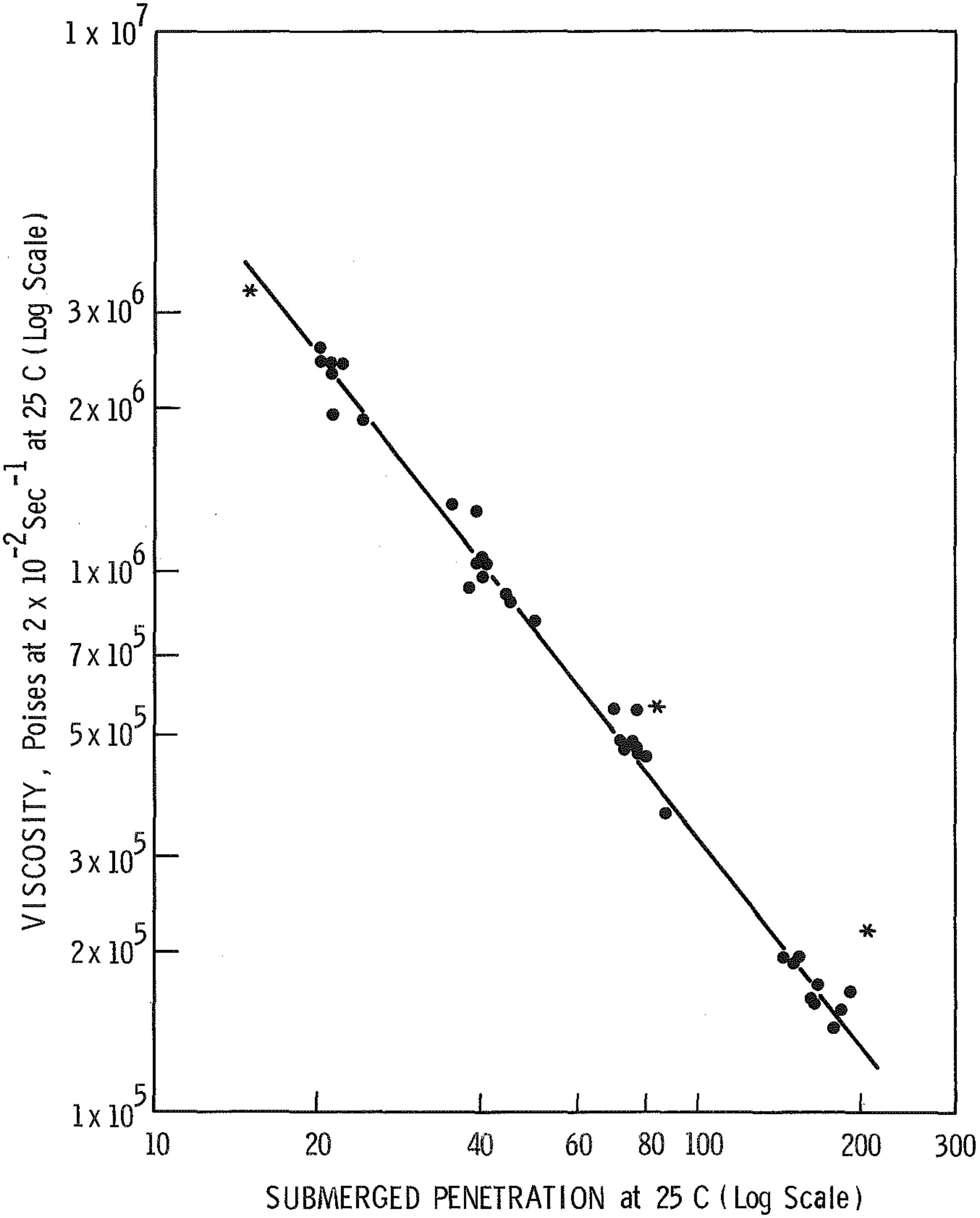

Figure 24. Viscosity versus submerged penetration for 43 Michigan asphalts.

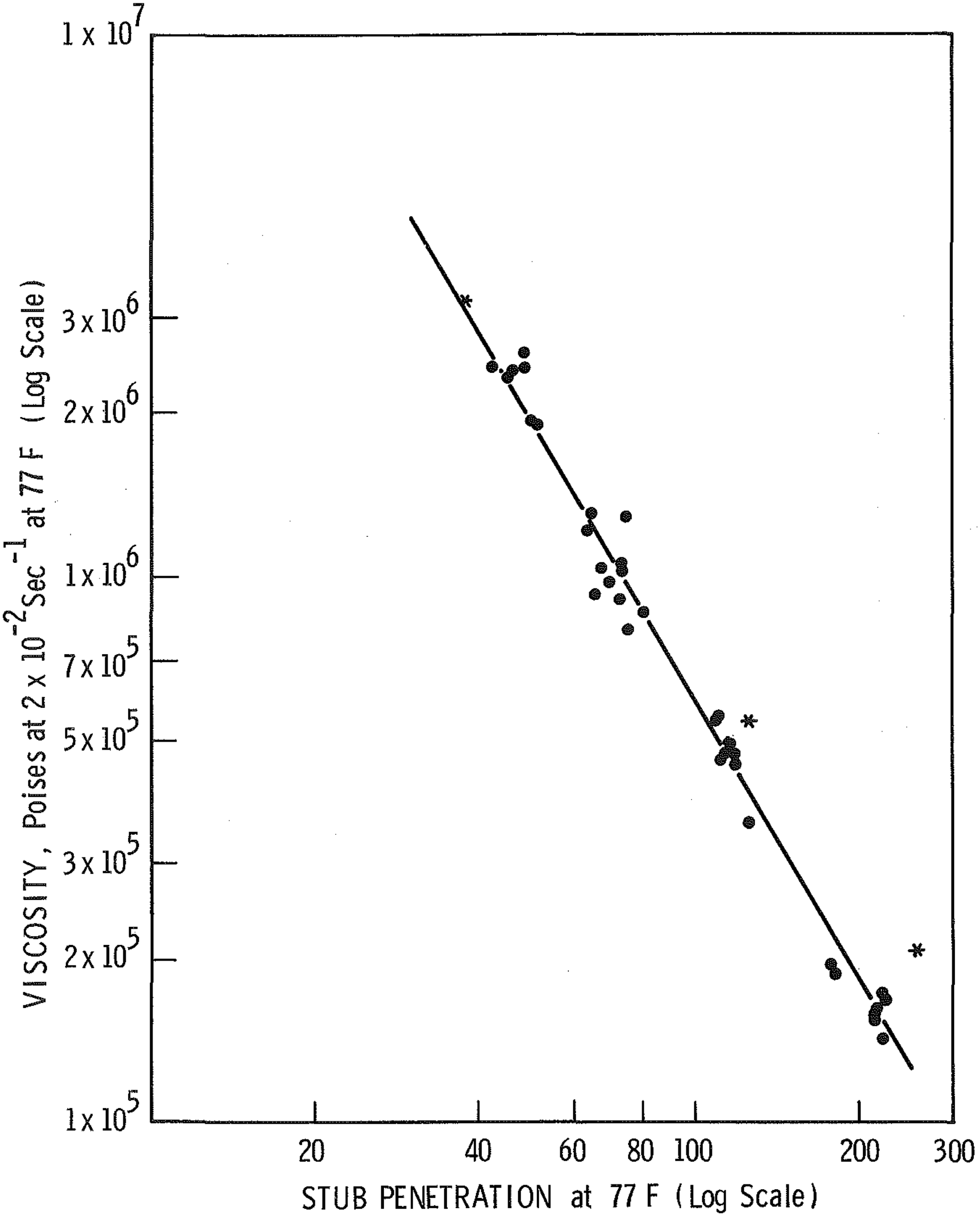

Figure 25. Viscosity versus stub penetration for 43 Michigan asphalts.

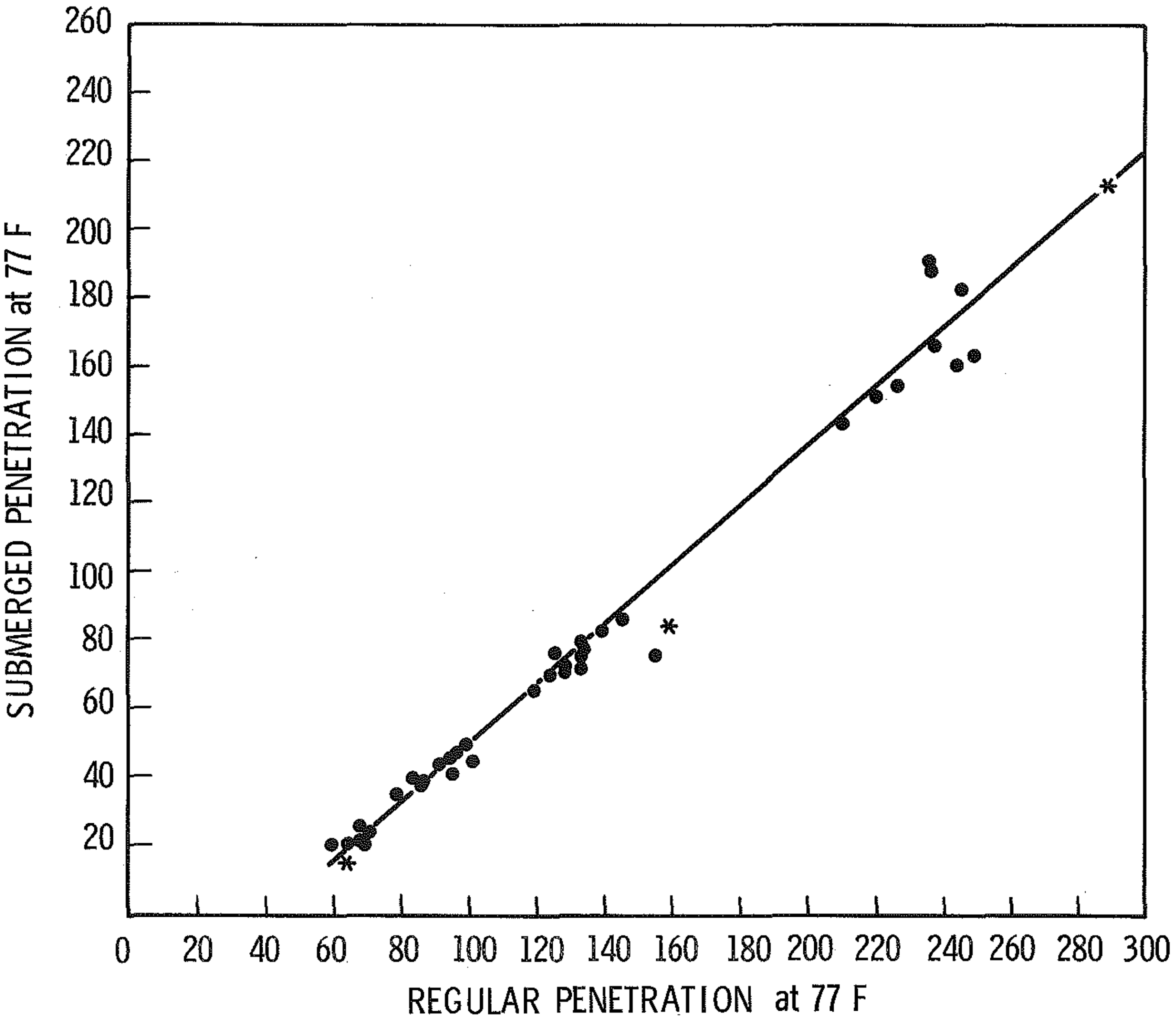

Figure 26. Regular penetration versus submerged penetration for 43 Michigan asphalts.

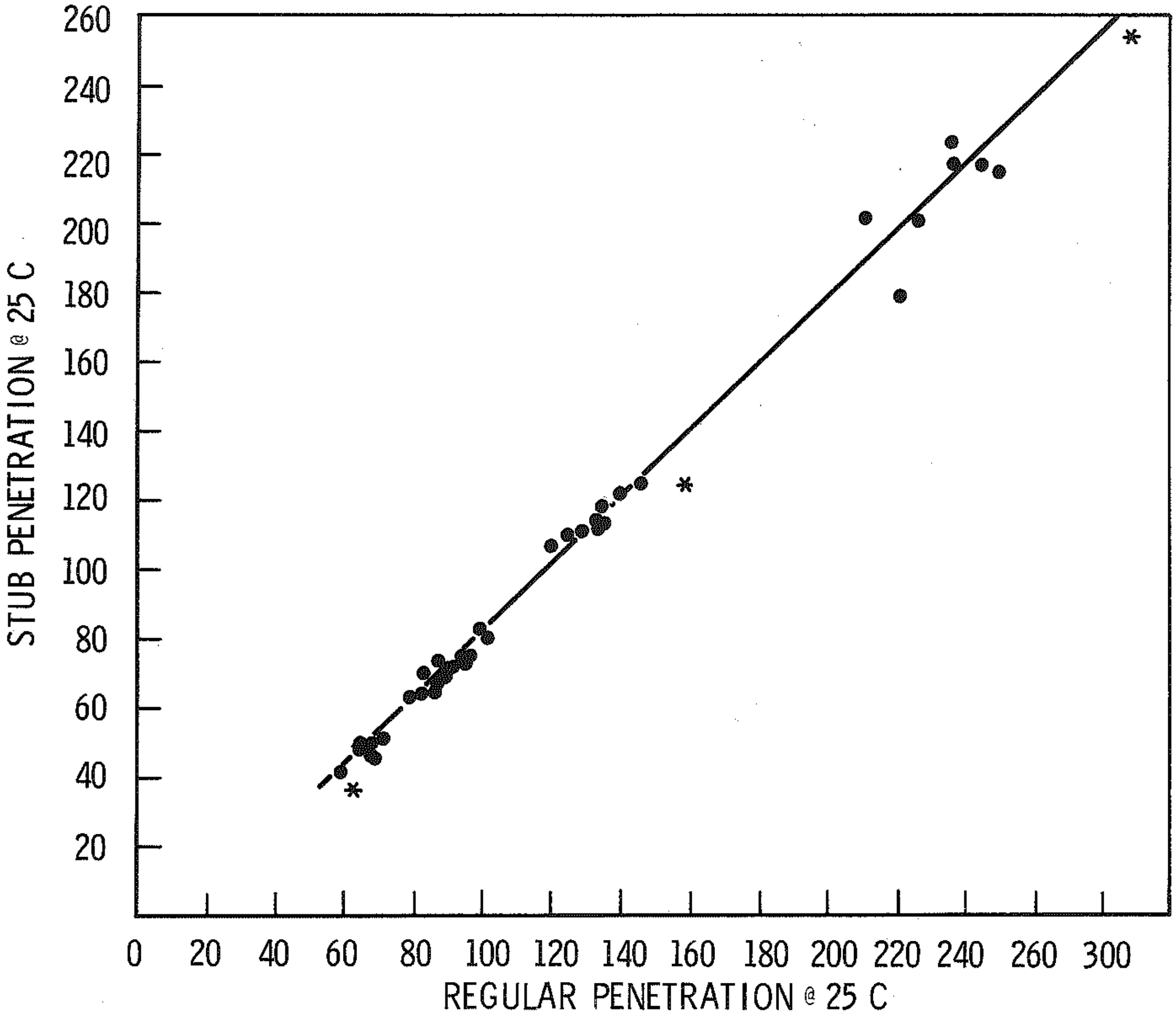

Figure 27. Regular penetration versus stub penetration for 43 Michigan asphalts.

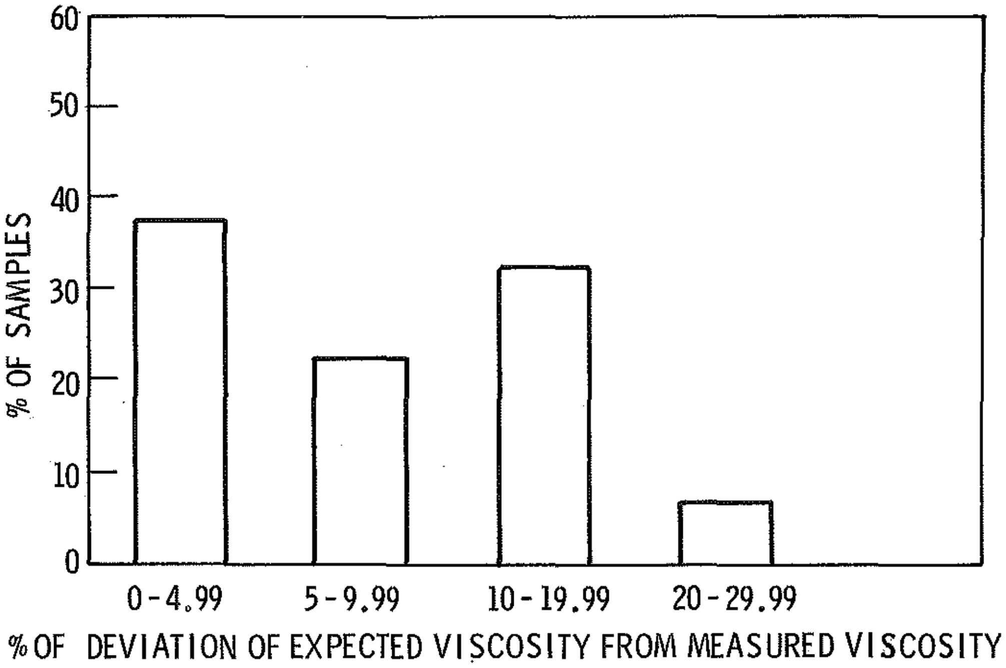

Figure 28. Deviations in predicting viscosity from standard penetration.

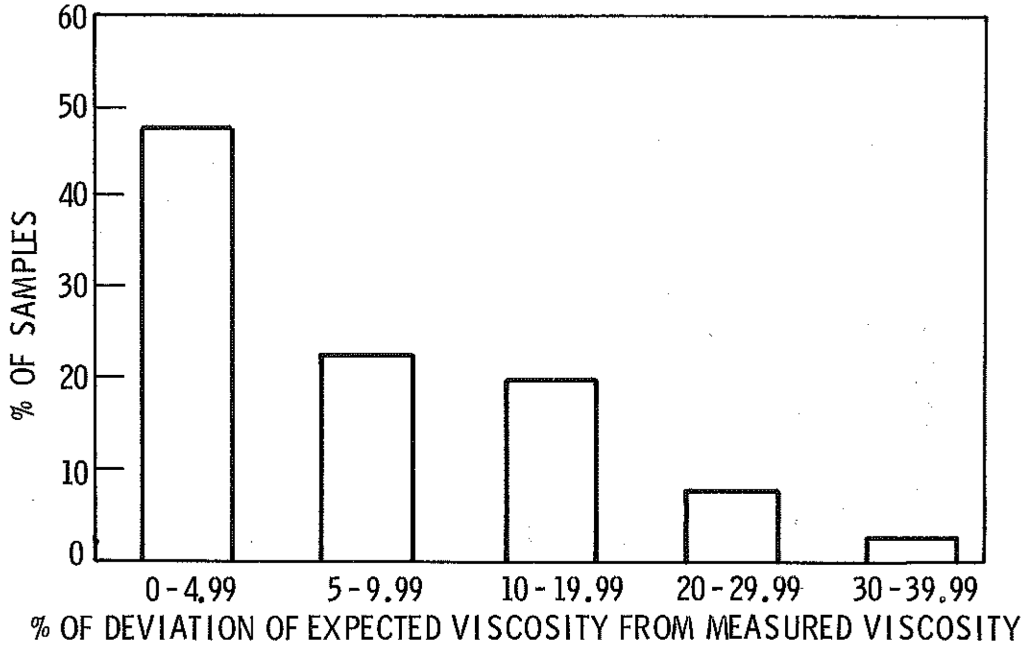

Figure 29. Deviation in predicting viscosity from stub penetration.

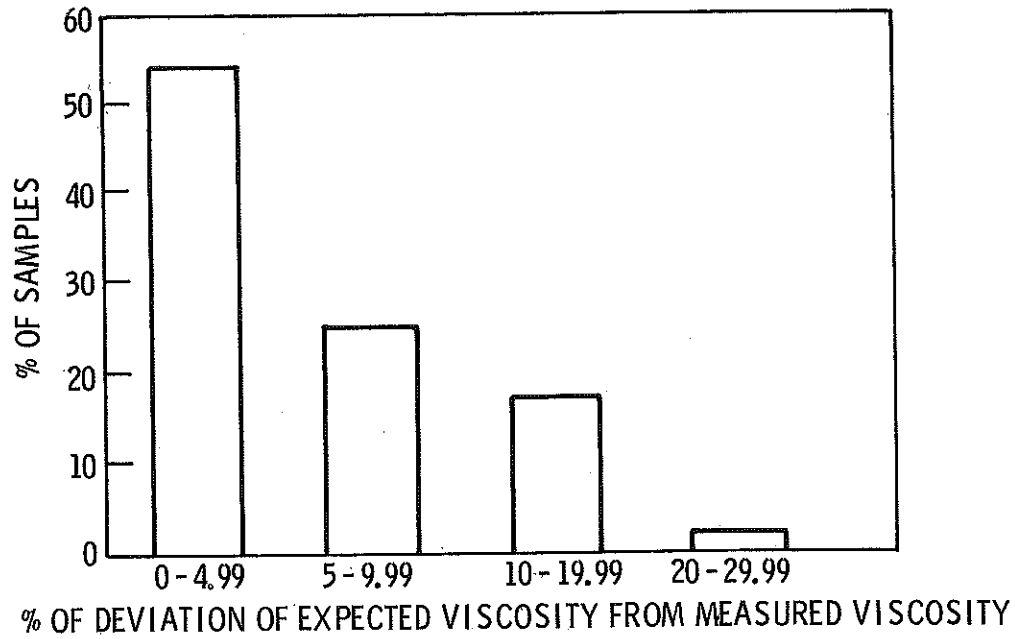

Figure 30. Deviations in predicting viscosity from submerged penetration.

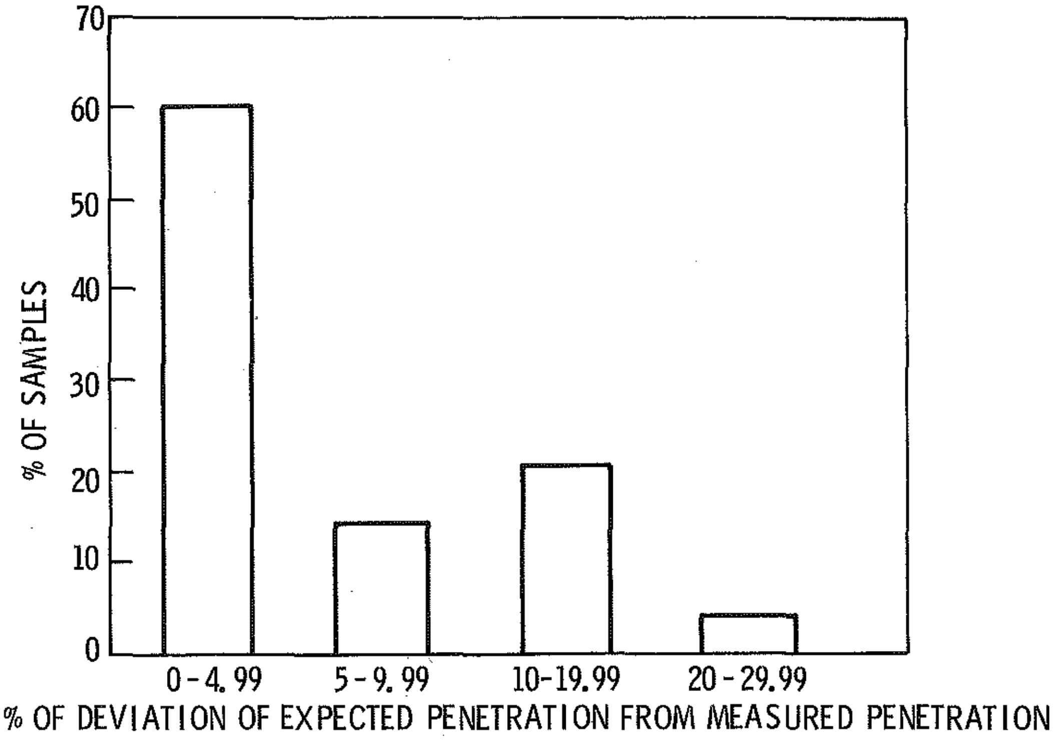

Figure 31. Deviations in predicting standard penetration from submerged penetration.

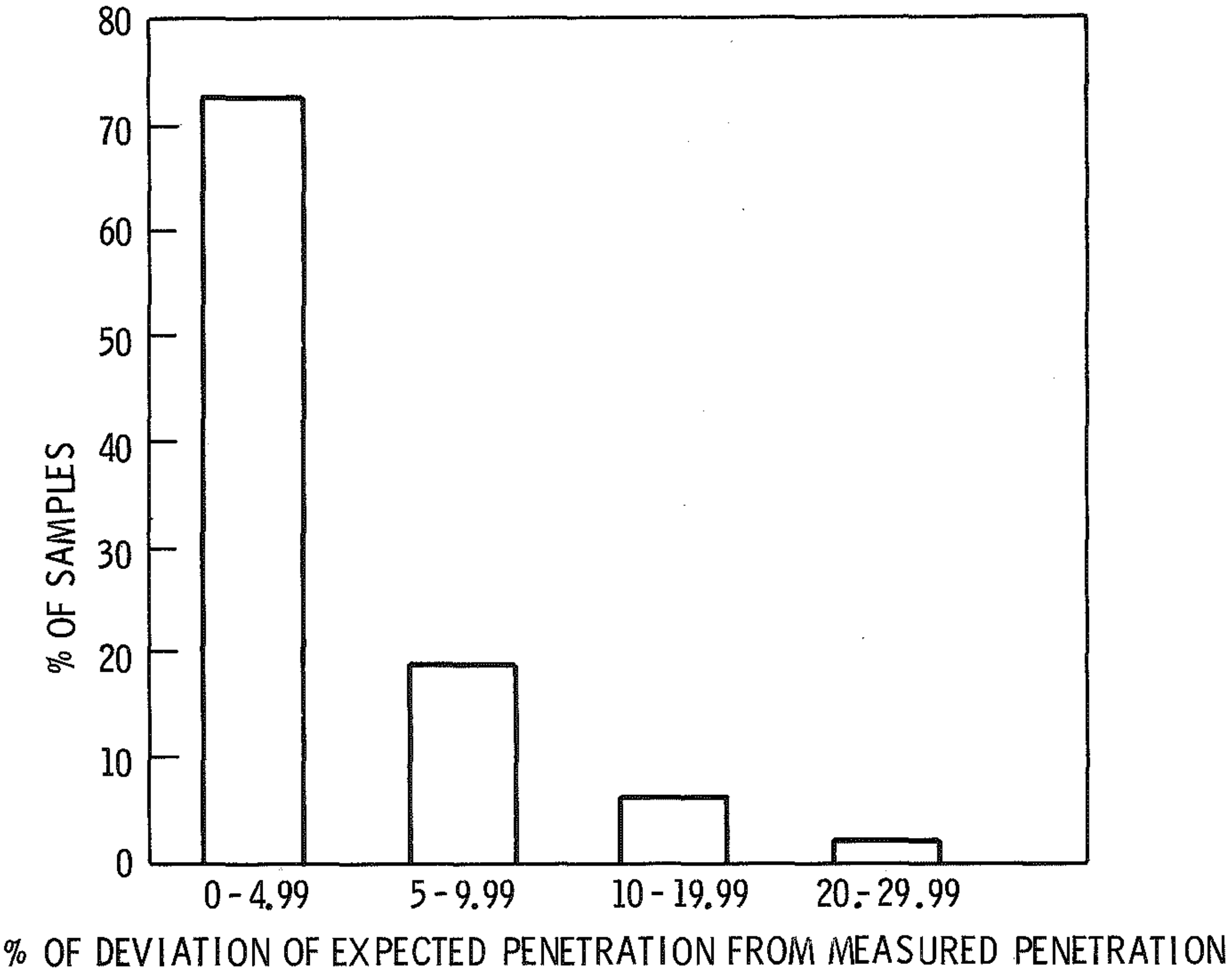

Figure 32. Deviations in predicting standard penetration from stub penetration.

APPENDIX

TABLE A

STANDARD PEN-VISCOSITY (at 77 F and 2 x 10^{-2} sec^{-1})
REGRESSION ANALYSIS ON 43 SAMPLES

```
POLYNOMIAL COEF. A0,A1,...,A(ND)
 0.2257E 02 -0.1926E 01
LINEAR LOG FUNCTION, Y=C*X**A1
 C=     0.6322E 10

MEAN=     0.6424E 06
SAMPLE STANDARD DEVIATION OF Y=     0.9668E 06
STANDARD ERROR OF ESTIMATE=  0.121733E 06
CORRELATION COEFFICIENT=  0.98198

INDEPENDANT & DEPENDANT VARIABLES  ESTI. Y        %ERROR
     0.5900E 02       0.2450E 07      0.2458E 07       0.3
     0.6300E 02       0.3250E 07      0.2167E 07     -33.3
     0.6300E 02       0.2600E 07      0.2167E 07     -16.7
     0.6300E 02       0.2440E 07      0.2167E 07     -11.2
     0.6400E 02       0.1940E 07      0.2102E 07       8.4
     0.6800E 02       0.2420E 07      0.1870E 07     -22.7
     0.6900E 02       0.2330E 07      0.1819E 07     -22.0
     0.7100E 02       0.1910E 07      0.1721E 07      -9.9
     0.7900E 02       0.1230E 07      0.1401E 07      13.9
     0.8200E 02       0.1330E 07      0.1304E 07      -1.9
     0.8300E 02       0.1120E 07      0.1274E 07      13.8
     0.8600E 02       0.9320E 06      0.1190E 07      27.7
     0.8700E 02       0.1300E 07      0.1164E 07     -10.5
     0.8700E 02       0.1040E 07      0.1164E 07      11.9
     0.8900E 02       0.9830E 06      0.1114E 07      13.3
     0.9000E 02       0.1070E 07      0.1090E 07       1.9
     0.9100E 02       0.9160E 06      0.1067E 07      16.5
     0.9500E 02       0.1030E 07      0.9824E 06      -4.6
     0.9600E 02       0.8040E 06      0.9628E 06      19.7
     0.9900E 02       0.8120E 06      0.9074E 06      11.7
     0.1010E 03       0.8720E 06      0.8731E 06       0.1
     0.1240E 03       0.5600E 06      0.5881E 06       5.0
     0.1250E 03       0.5540E 06      0.5791E 06       4.5
     0.1280E 03       0.4880E 06      0.5533E 06      13.4
     0.1280E 03       0.4650E 06      0.5533E 06      19.0
     0.1320E 03       0.4630E 06      0.5214E 06      12.6
     0.1330E 03       0.4880E 06      0.5139E 06       5.3
     0.1330E 03       0.4840E 06      0.5139E 06       6.2
     0.1340E 03       0.4740E 06      0.5065E 06       6.9
     0.1340E 03       0.4720E 06      0.5065E 06       7.3
     0.1370E 03       0.4560E 06      0.4854E 06       6.4
     0.1450E 03       0.3570E 06      0.4351E 06      21.9
     0.1590E 03       0.5500E 06      0.3644E 06     -33.8
     0.2100E 03       0.1950E 06      0.2132E 06       9.4
     0.2200E 03       0.1880E 06      0.1950E 06       3.7
     0.2260E 03       0.1940E 06      0.1851E 06      -4.6
     0.2350E 03       0.1690E 06      0.1717E 06       1.6
     0.2360E 03       0.1560E 06      0.1703E 06       9.2
     0.2370E 03       0.1740E 06      0.1689E 06      -2.9
     0.2440E 03       0.1630E 06      0.1597E 06      -2.0
     0.2450E 03       0.1440E 06      0.1585E 06      10.0
     0.2490E 03       0.1610E 06      0.1536E 06      -4.6
     0.2890E 03       0.2080E 06      0.1153E 06     -44.6

% OF ESTIMATED Y WITHIN RANGE OF TESTED VALUE
RANGE 0-4.99% 5-9.99% 10-19.99% 20-29.99% 30-39.99% 40-49.99% >50%
          27.9    23.3        32.6         9.3         4.7         2.3    0.0
```

TABLE B

STANDARD PEN-VISCOSITY (at 77 F and 2 x 10^{-2} sec^{-1})
REGRESSION ANALYSIS ON 40 SAMPLES

```
POLYNOMIAL COEF. A0,A1,...,A(ND)
 0.2278E 02 -0.1978E 01
LINEAR LOG FUNCTION, Y=C*X**A1
 C=     0.7828E 10

MEAN=     0.6370E 06
SAMPLE STANDARD DEVIATION OF Y=     0.9275E 06
STANDARD ERROR OF ESTIMATE=  0.758442E 05
CORRELATION COEFFICIENT=  0.99213

INDEPENDANT & DEPENDANT VARIABLES  ESTI. Y        %ERROR
     0.5900E 02      0.2450E 07      0.2460E 07        0.4
     0.6300E 02      0.2600E 07      0.2160E 07      -16.9
     0.6300E 02      0.2440E 07      0.2160E 07      -11.5
     0.6400E 02      0.1940E 07      0.2094E 07        7.9
     0.6800E 02      0.2420E 07      0.1858E 07      -23.2
     0.6900E 02      0.2330E 07      0.1805E 07      -22.5
     0.7100E 02      0.1910E 07      0.1706E 07      -10.7
     0.7900E 02      0.1230E 07      0.1381E 07       12.3
     0.8200E 02      0.1330E 07      0.1283E 07       -3.6
     0.8300E 02      0.1120E 07      0.1252E 07       11.8
     0.8600E 02      0.9320E 06      0.1167E 07       25.3
     0.8700E 02      0.1300E 07      0.1141E 07      -12.2
     0.8700E 02      0.1040E 07      0.1141E 07        9.7
     0.8900E 02      0.9830E 06      0.1091E 07       11.0
     0.9000E 02      0.1070E 07      0.1067E 07       -0.3
     0.9100E 02      0.9160E 06      0.1044E 07       14.0
     0.9500E 02      0.1030E 07      0.9588E 06       -6.9
     0.9600E 02      0.8040E 06      0.9391E 06       16.8
     0.9900E 02      0.8120E 06      0.8836E 06        8.8
     0.1010E 03      0.8720E 06      0.8494E 06       -2.6
     0.1240E 03      0.5600E 06      0.5661E 06        1.1
     0.1250E 03      0.5540E 06      0.5571E 06        0.6
     0.1280E 03      0.4880E 06      0.5316E 06        8.9
     0.1280E 03      0.4650E 06      0.5316E 06       14.3
     0.1320E 03      0.4630E 06      0.5002E 06        8.0
     0.1330E 03      0.4880E 06      0.4928E 06        1.0
     0.1330E 03      0.4840E 06      0.4928E 06        1.8
     0.1340E 03      0.4740E 06      0.4855E 06        2.4
     0.1340E 03      0.4720E 06      0.4855E 06        2.9
     0.1370E 03      0.4560E 06      0.4647E 06        1.9
     0.1450E 03      0.3570E 06      0.4154E 06       16.4
     0.2100E 03      0.1950E 06      0.1997E 06        2.4
     0.2200E 03      0.1880E 06      0.1821E 06       -3.1
     0.2260E 03      0.1940E 06      0.1727E 06      -11.0
     0.2350E 03      0.1690E 06      0.1598E 06       -5.4
     0.2360E 03      0.1560E 06      0.1585E 06        1.6
     0.2370E 03      0.1740E 06      0.1572E 06       -9.7
     0.2440E 03      0.1630E 06      0.1484E 06       -9.0
     0.2450E 03      0.1440E 06      0.1472E 06        2.2
     0.2490E 03      0.1610E 06      0.1425E 06      -11.5

% OF ESTIMATED Y WITHIN RANGE OF TESTED VALUE
RANGE 0-4.99% 5-9.99% 10-19.99% 20-29.99% 30-39.99% 40-49.99% >50%
          37.5    22.5        32.5         7.5         0.0         0.0     0.0
```

TABLE C

STUB PEN-VISCOSITY (at 77 F and 2 x 10^{-2} sec^{-1})
REGRESSION ANALYSIS ON 43 SAMPLES

```
POLYNOMIAL COEF. A0,A1,....,A(ND)
 0.2093E 02 -0.1653E 01
LINEAR LOG FUNCTION, Y=C*X**A1
 C=    0.1229E 10

MEAN=    0.6424E 06
SAMPLE STANDARD DEVIATION OF Y=    0.9668E 06
STANDARD ERROR OF ESTIMATE=  0.939999E 05
CORRELATION COEFFICIENT=  0.98888

INDEPENDANT & DEPENDANT VARIABLES  ESTI. Y        %ERROR
    0.3700E 02      0.3250E 07     0.3148E 07      -3.1
    0.4200E 02      0.2450E 07     0.2553E 07       4.2
    0.4500E 02      0.2330E 07     0.2278E 07      -2.2
    0.4600E 02      0.2420E 07     0.2197E 07      -9.2
    0.4800E 02      0.2600E 07     0.2048E 07     -21.2
    0.4800E 02      0.2440E 07     0.2048E 07     -16.1
    0.5000E 02      0.1940E 07     0.1914E 07      -1.3
    0.5100E 02      0.1910E 07     0.1852E 07      -3.0
    0.6300E 02      0.1230E 07     0.1306E 07       6.2
    0.6400E 02      0.1330E 07     0.1273E 07      -4.3
    0.6500E 02      0.9320E 06     0.1241E 07      33.1
    0.6700E 02      0.1040E 07     0.1180E 07      13.5
    0.6900E 02      0.9830E 06     0.1124E 07      14.3
    0.7000E 02      0.1120E 07     0.1098E 07      -2.0
    0.7200E 02      0.9160E 06     0.1048E 07      14.4
    0.7300E 02      0.1070E 07     0.1024E 07      -4.3
    0.7300E 02      0.1030E 07     0.1024E 07      -0.6
    0.7400E 02      0.1300E 07     0.1001E 07     -23.0
    0.7500E 02      0.8040E 06     0.9793E 06      21.8
    0.8000E 02      0.8720E 06     0.8803E 06       0.9
    0.8300E 02      0.8120E 06     0.8283E 06       2.0
    0.1090E 03      0.5540E 06     0.5280E 06      -4.7
    0.1100E 03      0.5600E 06     0.5201E 06      -7.1
    0.1110E 03      0.4880E 06     0.5124E 06       5.0
    0.1110E 03      0.4650E 06     0.5124E 06      10.2
    0.1120E 03      0.4840E 06     0.5048E 06       4.3
    0.1130E 03      0.4740E 06     0.4975E 06       4.9
    0.1140E 03      0.4880E 06     0.4903E 06       0.5
    0.1150E 03      0.4630E 06     0.4832E 06       4.4
    0.1180E 03      0.4720E 06     0.4631E 06      -1.9
    0.1180E 03      0.4560E 06     0.4631E 06       1.6
    0.1250E 03      0.5500E 06     0.4210E 06     -23.4
    0.1250E 03      0.3570E 06     0.4210E 06      17.9
    0.1800E 03      0.1880E 06     0.2305E 06      22.6
    0.2010E 03      0.1940E 06     0.1921E 06      -1.0
    0.2020E 03      0.1950E 06     0.1905E 06      -2.3
    0.2150E 03      0.1610E 06     0.1718E 06       6.7
    0.2170E 03      0.1630E 06     0.1692E 06       3.8
    0.2170E 03      0.1560E 06     0.1692E 06       8.5
    0.2210E 03      0.1740E 06     0.1642E 06      -5.6
    0.2210E 03      0.1440E 06     0.1642E 06      14.0
    0.2240E 03      0.1690E 06     0.1606E 06      -5.0
    0.2530E 03      0.2080E 06     0.1313E 06     -36.9

% OF ESTIMATED Y WITHIN RANGE OF TESTED VALUE
RANGE 0-4.99% 5-9.99% 10-19.99% 20-29.99% 30-39.99% 40-49.99% >50%
         53.5    14.0       16.3       11.6        4.7        0.0     0.0
```

TABLE D

STUB PEN-VISCOSITY (at 77 F and 2 x 10^{-2} sec^{-1})
REGRESSION ANALYSIS ON 40 SAMPLES

```
POLYNOMIAL COEF. A0,A1,...,A(ND)
 0.2112E 02 -0.1698E 01
LINEAR LOG FUNCTION, Y=C*X**A1
 C=     0.1482E 10

MEAN=     0.6370E 06
SAMPLE STANDARD DEVIATION OF Y=     0.9275E 06
STANDARD ERROR OF ESTIMATE=  0.726746E 05
CORRELATION COEFFICIENT=  0.99275

INDEPENDANT & DEPENDANT VARIABLES  ESTI. Y         %ERROR
     0.4200E 02       0.2450E 07      0.2601E 07       6.1
     0.4500E 02       0.2330E 07      0.2313E 07      -0.7
     0.4600E 02       0.2420E 07      0.2228E 07      -7.9
     0.4800E 02       0.2600E 07      0.2073E 07     -20.3
     0.4800E 02       0.2440E 07      0.2073E 07     -15.0
     0.5000E 02       0.1940E 07      0.1934E 07      -0.3
     0.5100E 02       0.1910E 07      0.1870E 07      -2.1
     0.6300E 02       0.1230E 07      0.1307E 07       6.2
     0.6400E 02       0.1330E 07      0.1272E 07      -4.4
     0.6500E 02       0.9320E 06      0.1239E 07      32.9
     0.6700E 02       0.1040E 07      0.1177E 07      13.2
     0.6900E 02       0.9830E 06      0.1120E 07      13.9
     0.7000E 02       0.1120E 07      0.1093E 07      -2.4
     0.7200E 02       0.9160E 06      0.1042E 07      13.7
     0.7300E 02       0.1070E 07      0.1017E 07      -4.9
     0.7300E 02       0.1030E 07      0.1017E 07      -1.2
     0.7400E 02       0.1300E 07      0.9942E 06     -23.5
     0.7500E 02       0.8040E 06      0.9718E 06      20.9
     0.8000E 02       0.8720E 06      0.8710E 06      -0.1
     0.8300E 02       0.8120E 06      0.8182E 06       0.8
     0.1090E 03       0.5540E 06      0.5151E 06      -7.0
     0.1100E 03       0.5600E 06      0.5072E 06      -9.4
     0.1110E 03       0.4880E 06      0.4995E 06       2.4
     0.1110E 03       0.4650E 06      0.4995E 06       7.4
     0.1120E 03       0.4840E 06      0.4919E 06       1.6
     0.1130E 03       0.4740E 06      0.4846E 06       2.2
     0.1140E 03       0.4880E 06      0.4774E 06      -2.2
     0.1150E 03       0.4630E 06      0.4704E 06       1.6
     0.1180E 03       0.4720E 06      0.4502E 06      -4.6
     0.1180E 03       0.4560E 06      0.4502E 06      -1.3
     0.1250E 03       0.3570E 06      0.4083E 06      14.4
     0.1800E 03       0.1880E 06      0.2198E 06      16.9
     0.2010E 03       0.1940E 06      0.1823E 06      -6.0
     0.2020E 03       0.1950E 06      0.1807E 06      -7.3
     0.2150E 03       0.1610E 06      0.1626E 06       1.0
     0.2170E 03       0.1630E 06      0.1601E 06      -1.8
     0.2170E 03       0.1560E 06      0.1601E 06       2.6
     0.2210E 03       0.1740E 06      0.1552E 06     -10.8
     0.2210E 03       0.1440E 06      0.1552E 06       7.8
     0.2240E 03       0.1690E 06      0.1517E 06     -10.3

% OF ESTIMATED Y WITHIN RANGE OF TESTED VALUE
RANGE 0-4.99% 5-9.99% 10-19.99% 20-29.99% 30-39.99% 40-49.99% >50%
          47.5    22.5       20.0         7.5        2.5        0.0     0.0
```

TABLE E

SUBMERGED PEN-VISCOSITY (at 77 F and 2 x 10^{-2} sec^{-1})
REGRESSION ANALYSIS ON 43 SAMPLES

```
POLYNOMIAL COEF. A0,A1,...,A(NO)
 0.1836E 02 -0.1220E 01
LINEAR LOG FUNCTION, Y=C*X**A1
 C=     0.9440E 08

MEAN=     0.6424E 06
SAMPLE STANDARD DEVIATION OF Y=     0.9668E 06
STANDARD ERROR OF ESTIMATE=  0.791998E 05
CORRELATION COEFFICIENT=  0.99195

INDEPENDANT & DEPENDANT VARIABLES  ESTI. Y        %ERROR
    0.1500E 02      0.3250E 07      0.3468E 07       6.7
    0.2000E 02      0.2600E 07      0.2441E 07      -6.1
    0.2000E 02      0.2450E 07      0.2441E 07      -0.4
    0.2100E 02      0.2440E 07      0.2300E 07      -5.7
    0.2100E 02      0.2330E 07      0.2300E 07      -1.3
    0.2100E 02      0.1940E 07      0.2300E 07      18.6
    0.2200E 02      0.2420E 07      0.2173E 07     -10.2
    0.2400E 02      0.1910E 07      0.1954E 07       2.3
    0.3500E 02      0.1330E 07      0.1233E 07      -7.3
    0.3500E 02      0.1230E 07      0.1233E 07       0.3
    0.3800E 02      0.9320E 06      0.1116E 07      19.7
    0.3900E 02      0.1300E 07      0.1081E 07     -16.9
    0.3900E 02      0.1040E 07      0.1081E 07       3.9
    0.4000E 02      0.1120E 07      0.1048E 07      -6.4
    0.4000E 02      0.1070E 07      0.1048E 07      -2.1
    0.4000E 02      0.9830E 06      0.1048E 07       6.6
    0.4100E 02      0.1030E 07      0.1017E 07      -1.3
    0.4400E 02      0.9160E 06      0.9330E 06       1.9
    0.4500E 02      0.8720E 06      0.9077E 06       4.1
    0.4800E 02      0.8040E 06      0.8390E 06       4.4
    0.5000E 02      0.8120E 06      0.7982E 06      -1.7
    0.7000E 02      0.5600E 06      0.5295E 06      -5.5
    0.7200E 02      0.4880E 06      0.5116E 06       4.8
    0.7200E 02      0.4880E 06      0.5116E 06       4.8
    0.7300E 02      0.4650E 06      0.5030E 06       8.2
    0.7600E 02      0.4840E 06      0.4789E 06      -1.0
    0.7700E 02      0.5540E 06      0.4713E 06     -14.9
    0.7700E 02      0.4740E 06      0.4713E 06      -0.6
    0.7800E 02      0.4630E 06      0.4640E 06       0.2
    0.8000E 02      0.4720E 06      0.4499E 06      -4.7
    0.8100E 02      0.4560E 06      0.4431E 06      -2.8
    0.8500E 02      0.5500E 06      0.4178E 06     -24.0
    0.8700E 02      0.3570E 06      0.4061E 06      13.8
    0.1440E 03      0.1950E 06      0.2196E 06      12.6
    0.1520E 03      0.1880E 06      0.2056E 06       9.4
    0.1550E 03      0.1940E 06      0.2007E 06       3.5
    0.1610E 03      0.1630E 06      0.1917E 06      17.6
    0.1640E 03      0.1610E 06      0.1874E 06      16.4
    0.1660E 03      0.1740E 06      0.1846E 06       6.1
    0.1830E 03      0.1440E 06      0.1639E 06      13.8
    0.1880E 03      0.1560E 06      0.1586E 06       1.7
    0.1910E 03      0.1690E 06      0.1556E 06      -7.9
    0.2130E 03      0.2080E 06      0.1362E 06     -34.5

% OF ESTIMATED Y WITHIN RANGE OF TESTED VALUE
RANGE 0-4.99% 5-9.99% 10-19.99% 20-29.99% 30-39.99% 40-49.99% >50%
         46.5    25.6       23.3        2.3         2.3         0.0    0.0
```

TABLE F

SUBMERGED PEN-VISCOSITY (at 77 F and 2 x 10^{-2} sec^{-1})
REGRESSION ANALYSIS ON 40 SAMPLES

```
POLYNOMIAL COEF. A0,A1,...,A(ND)
 0.1850E 02 -0.1257E 01
LINEAR LOG FUNCTION, Y=C*X**A1
 C=    0.1081E 09

MEAN=     0.6370E 06
SAMPLE STANDARD DEVIATION OF Y=     0.9275E 06
STANDARD ERROR OF ESTIMATE=  0.546690E 05
CORRELATION COEFFICIENT=  0.99579

INDEPENDANT & DEPENDANT VARIABLES  ESTI. Y         %ERROR
     0.2000E 02       0.2600E 07       0.2502E 07       -3.8
     0.2000E 02       0.2450E 07       0.2502E 07        2.1
     0.2100E 02       0.2440E 07       0.2353E 07       -3.6
     0.2100E 02       0.2330E 07       0.2353E 07        1.0
     0.2100E 02       0.1940E 07       0.2353E 07       21.3
     0.2200E 02       0.2420E 07       0.2219E 07       -8.3
     0.2400E 02       0.1910E 07       0.1989E 07        4.2
     0.3500E 02       0.1330E 07       0.1238E 07       -6.9
     0.3500E 02       0.1230E 07       0.1238E 07        0.7
     0.3800E 02       0.9320E 06       0.1116E 07       19.8
     0.3900E 02       0.1300E 07       0.1081E 07      -16.9
     0.3900E 02       0.1040E 07       0.1081E 07        3.9
     0.4000E 02       0.1120E 07       0.1047E 07       -6.5
     0.4000E 02       0.1070E 07       0.1047E 07       -2.2
     0.4000E 02       0.9830E 06       0.1047E 07        6.5
     0.4100E 02       0.1030E 07       0.1015E 07       -1.5
     0.4400E 02       0.9160E 06       0.9285E 06        1.4
     0.4500E 02       0.8720E 06       0.9027E 06        3.5
     0.4800E 02       0.8040E 06       0.8323E 06        3.5
     0.5000E 02       0.8120E 06       0.7907E 06       -2.6
     0.7000E 02       0.5600E 06       0.5180E 06       -7.5
     0.7200E 02       0.4880E 06       0.5000E 06        2.5
     0.7200E 02       0.4880E 06       0.5000E 06        2.5
     0.7300E 02       0.4650E 06       0.4914E 06        5.7
     0.7600E 02       0.4840E 06       0.4671E 06       -3.5
     0.7700E 02       0.5540E 06       0.4595E 06      -17.1
     0.7700E 02       0.4740E 06       0.4595E 06       -3.1
     0.7800E 02       0.4630E 06       0.4521E 06       -2.3
     0.8000E 02       0.4720E 06       0.4380E 06       -7.2
     0.8100E 02       0.4560E 06       0.4312E 06       -5.4
     0.8700E 02       0.3570E 06       0.3941E 06       10.4
     0.1440E 03       0.1950E 06       0.2092E 06        7.3
     0.1520E 03       0.1880E 06       0.1954E 06        4.0
     0.1550E 03       0.1940E 06       0.1907E 06       -1.7
     0.1610E 03       0.1630E 06       0.1818E 06       11.5
     0.1640E 03       0.1610E 06       0.1776E 06       10.3
     0.1660E 03       0.1740E 06       0.1750E 06        0.6
     0.1830E 03       0.1440E 06       0.1548E 06        7.5
     0.1880E 03       0.1560E 06       0.1496E 06       -4.1
     0.1910E 03       0.1690E 06       0.1467E 06      -13.2

% OF ESTIMATED Y WITHIN RANGE OF TESTED VALUE
RANGE 0-4.99% 5-9.99% 10-19.99% 20-29.99% 30-39.99% 40-49.99% >50%
           55.0    25.0        17.5        2.5         0.0         0.0    0.0
```

TABLE G

STANDARD PEN-SUBMERGED PEN (at 77 F)
REGRESSION ANALYSIS ON 48 SAMPLES

```
POLYNOMIAL COEF. A0,A1,...,A(ND)
-0.3638E 02   0.8636E 00

MEAN=     0.7467E 02
SAMPLE STANDARD DEVIATION OF Y=     0.5524E 02
STANDARD ERROR OF ESTIMATE=  0.662689E 01
CORRELATION COEFFICIENT=  0.99278

INDEPENDANT & DEPENDANT VARIABLES  ESTI. Y          %ERROR
     0.5900E 02      0.2000E 02      0.1457E 02      -27.1
     0.6300E 02      0.2100E 02      0.1803E 02      -14.2
     0.6300E 02      0.1500E 02      0.1803E 02       20.2
     0.6300E 02      0.2000E 02      0.1803E 02       -9.9
     0.6400E 02      0.2000E 02      0.1889E 02       -5.5
     0.6400E 02      0.2100E 02      0.1889E 02      -10.0
     0.6800E 02      0.2600E 02      0.2235E 02      -14.1
     0.6800E 02      0.2200E 02      0.2235E 02        1.6
     0.6900E 02      0.2100E 02      0.2321E 02       10.5
     0.7100E 02      0.2400E 02      0.2494E 02        3.9
     0.7900E 02      0.3500E 02      0.3185E 02       -9.0
     0.8200E 02      0.3500E 02      0.3444E 02       -1.6
     0.8300E 02      0.4000E 02      0.3530E 02      -11.8
     0.8600E 02      0.3800E 02      0.3789E 02       -0.3
     0.8700E 02      0.3900E 02      0.3875E 02       -0.6
     0.8700E 02      0.3900E 02      0.3875E 02       -0.6
     0.8900E 02      0.4000E 02      0.4048E 02        1.2
     0.9000E 02      0.4000E 02      0.4135E 02        3.4
     0.9100E 02      0.4400E 02      0.4221E 02       -4.1
     0.9400E 02      0.4600E 02      0.4480E 02       -2.6
     0.9500E 02      0.4100E 02      0.4566E 02       11.4
     0.9600E 02      0.4800E 02      0.4653E 02       -3.1
     0.9900E 02      0.5000E 02      0.4912E 02       -1.8
     0.1010E 03      0.4500E 02      0.5084E 02       13.0
     0.1190E 03      0.6600E 02      0.6639E 02        0.6
     0.1240E 03      0.7000E 02      0.7071E 02        1.0
     0.1250E 03      0.7700E 02      0.7157E 02       -7.0
     0.1280E 03      0.7300E 02      0.7416E 02        1.6
     0.1280E 03      0.7200E 02      0.7416E 02        3.0
     0.1320E 03      0.7800E 02      0.7762E 02       -0.5
     0.1330E 03      0.7600E 02      0.7848E 02        3.3
     0.1330E 03      0.7200E 02      0.7848E 02        9.0
     0.1340E 03      0.8000E 02      0.7934E 02       -0.8
     0.1340E 03      0.7700E 02      0.7934E 02        3.0
     0.1370E 03      0.8100E 02      0.8194E 02        1.2
     0.1390E 03      0.8300E 02      0.8366E 02        0.8
     0.1450E 03      0.8700E 02      0.8884E 02        2.1
     0.1590E 03      0.8500E 02      0.1009E 03       18.7
     0.2100E 03      0.1440E 03      0.1450E 03        0.7
     0.2200E 03      0.1520E 03      0.1536E 03        1.1
     0.2260E 03      0.1550E 03      0.1588E 03        2.5
     0.2350E 03      0.1910E 03      0.1666E 03      -12.8
     0.2360E 03      0.1880E 03      0.1674E 03      -10.9
     0.2370E 03      0.1660E 03      0.1683E 03        1.4
     0.2440E 03      0.1610E 03      0.1743E 03        8.3
     0.2450E 03      0.1830E 03      0.1752E 03       -4.3
     0.2490E 03      0.1640E 03      0.1787E 03        8.9
     0.2890E 03      0.2130E 03      0.2132E 03        0.1
```

TABLE G (continued)

```
% OF ESTIMATED Y WITHIN RANGE OF TESTED VALUE
RANGE 0-4.99% 5-9.99% 10-19.99% 20-29.99% 30-39.99% 40-49.99% >50%
        60.4    14.6       20.8        4.2         0.0         0.0    0.0
```

TABLE H

STANDARD PEN-STUB PEN (at 77 F)
REGRESSION ANALYSIS ON 48 SAMPLES

```
POLYNOMIAL COEF. A0,A1,...,A(ND)
-0.1438E 02  0.9590E 00

MEAN=    0.1089E 03
SAMPLE STANDARD DEVIATION OF Y=    0.6118E 02
STANDARD ERROR OF ESTIMATE=  0.580666E 01
CORRELATION COEFFICIENT=  0.99549

INDEPENDANT & DEPENDANT VARIABLES  ESTI. Y          %ERROR
     0.5900E 02       0.4200E 02       0.4220E 02         0.5
     0.6300E 02       0.4800E 02       0.4604E 02        -4.1
     0.6300E 02       0.3700E 02       0.4604E 02        24.4
     0.6300E 02       0.4800E 02       0.4604E 02        -4.1
     0.6400E 02       0.4800E 02       0.4700E 02        -2.1
     0.6400E 02       0.5000E 02       0.4700E 02        -6.0
     0.6800E 02       0.5000E 02       0.5084E 02         1.7
     0.6800E 02       0.4600E 02       0.5084E 02        10.5
     0.6900E 02       0.4500E 02       0.5179E 02        15.1
     0.7100E 02       0.5100E 02       0.5371E 02         5.3
     0.7900E 02       0.6300E 02       0.6138E 02        -2.6
     0.8200E 02       0.6400E 02       0.6426E 02         0.4
     0.8300E 02       0.7000E 02       0.6522E 02        -6.8
     0.8600E 02       0.6500E 02       0.6810E 02         4.8
     0.8700E 02       0.6700E 02       0.6906E 02         3.1
     0.8700E 02       0.7400E 02       0.6906E 02        -6.7
     0.8900E 02       0.6900E 02       0.7098E 02         2.9
     0.9000E 02       0.7300E 02       0.7193E 02        -1.5
     0.9100E 02       0.7200E 02       0.7289E 02         1.2
     0.9400E 02       0.7500E 02       0.7577E 02         1.0
     0.9500E 02       0.7300E 02       0.7673E 02         5.1
     0.9600E 02       0.7500E 02       0.7769E 02         3.6
     0.9900E 02       0.8300E 02       0.8057E 02        -2.9
     0.1010E 03       0.8000E 02       0.8248E 02         3.1
     0.1190E 03       0.1070E 03       0.9975E 02        -6.8
     0.1240E 03       0.1100E 03       0.1045E 03        -5.0
     0.1250E 03       0.1090E 03       0.1055E 03        -3.2
     0.1280E 03       0.1110E 03       0.1084E 03        -2.4
     0.1280E 03       0.1110E 03       0.1084E 03        -2.4
     0.1320E 03       0.1150E 03       0.1122E 03        -2.4
     0.1330E 03       0.1120E 03       0.1132E 03         1.0
     0.1330E 03       0.1140E 03       0.1132E 03        -0.7
     0.1340E 03       0.1180E 03       0.1141E 03        -3.3
     0.1340E 03       0.1130E 03       0.1141E 03         1.0
     0.1370E 03       0.1180E 03       0.1170E 03        -0.8
     0.1390E 03       0.1220E 03       0.1189E 03        -2.5
     0.1450E 03       0.1250E 03       0.1247E 03        -0.3
     0.1590E 03       0.1250E 03       0.1381E 03        10.5
     0.2100E 03       0.2020E 03       0.1870E 03        -7.4
     0.2200E 03       0.1800E 03       0.1966E 03         9.2
     0.2260E 03       0.2010E 03       0.2024E 03         0.7
     0.2350E 03       0.2240E 03       0.2110E 03        -5.8
     0.2360E 03       0.2170E 03       0.2120E 03        -2.3
     0.2370E 03       0.2210E 03       0.2129E 03        -3.7
     0.2440E 03       0.2170E 03       0.2196E 03         1.2
     0.2450E 03       0.2210E 03       0.2206E 03        -0.2
     0.2490E 03       0.2150E 03       0.2244E 03         4.4
     0.2890E 03       0.2530E 03       0.2623E 03         3.9
```

TABLE H (continued)

```
% OF ESTIMATED Y WITHIN RANGE OF TESTED VALUE
RANGE 0-4.99% 5-9.99% 10-19.99% 20-29.99% 30-39.99% 40-49.99% >50%
       72.9    18.8        6.3        2.1         0.0        0.0    0.0
```

MODEL CCP1

CANNON CONE-PLATE VISCOMETER

Instructions

Instructions for measuring asphalt viscosity with the Cannon Cone-Plate Viscometer are contained in the accompanying "Proposed Method of Test for Viscosity of Asphalt using a Cone-Plate Viscometer". The Cannon Cone-Plate viscometer (which is a modification of the American Oil Company design developed by Dr. A. W. Sisko) has three cones, each with a cone angle about 0.5 degrees. Photographs of the basic cone-plate assembly and also the complete system with constant temperature bath are contained in Bulletin 51.

The following paragraphs contain information on the cone-plate viscometer which is not covered in the proposed test method, but which can be important in maintaining the equipment in good operating condition and obtaining good results from this instrument.

1. The cone and shaft is an integral unit which slips through the lower bearing, the drum, and the upper bearing in this order. A pin is provided to position the drum on the shaft and allow for easy removal of the cone. Take special care in inserting the shaft through the bearings to avoid damage to the bearings.

2. Binding of the threads on the plate in the base of the instrument must be prevented. Using the spanner wrench provided, screw the plate into the base until it is snug; do not apply force such as to make subsequent removal difficult. Plate and base should be about the same temperature when threaded together. After a run, the instrument can be cleaned by heating on a hot plate (or with a heat gun) until asphalt has softened sufficiently for the cone to be easily rotated and then lifted. The instrument is removed from the hot plate, the plate removed, and the asphalt wiped off. The cone can be warmed with a heat gun and excess asphalt wiped off; the cone may be left in place in the instrument. The cone and plate can then be cooled with water, and cleaned with solvents.

3. A one-way clutch is provided on the drum, so that the string may be attached after a sample is in place, and easily wound or rewound on the drum.

4. High loads may fracture the asphalt. A bucket of sand or shredded paper, etc. can be used to catch the falling weights.

5. The cylindrical scale fits over the end of the cone shaft as shown in the photograph in Bulletin 51. For very slow angular velocities, the 20X objective of the telescope is useful along with the pointer to indicate the appropriate reading. The pointer can be used without the telescope at high angular velocities. The needle of the pointer is threaded and thereby can be raised or lowered.

6. Each cone-plate assembly is checked for alignment and a viscosity standard is measured with each cone prior to shipment. Thus, the instrument leaves Cannon Instrument Co. in good operating condition. However, there is a possibility of misalignment being produced by rough handling in shipment; therefore, alignment should be checked before measuring samples.

Place the large cone in the bearing assembly and attach the plate. Observe the gap between the outer edge of the cone and the plate from all angles as the cone is rotated slowly. The gap should appear uniform. If there is a noticeable difference in the height of the gap as one moves around the circumference of the cone, the alignment is unsatisfactory and must be corrected. A set of feeler gages can be useful in checking the uniformity of the gap, and is somewhat better than reliance on observation only.

Misalignment can be corrected by adding shim washers to one or more of the three spacers on the support posts of the bearing assembly to tilt the plate holder relative to the cone such that the plate can be brought into proper alignment with the cone.

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AREA CODE 814, 466-6232
VISCOMETERS OF ALL TYPES VISCOSITY AND LABORATORY BATHS VISCOMETER HOLDERS

Cannon Cone-Plate Viscometer
Model CCP1

Parts List

CCP1-1	Viscometer frame with ball bearings and plate holder
CCP1-2	Plate
CCP1-3	No. 2 Cone (0.94 cm radius)
CCP1-4	No. 4 Cone (1.88 cm radius)
CCP1-5	No. 8 Cone (3.75 cm radius)
CCP1-6	Drum with one-way clutch
CCP1-7	Cylindrical scale
CCP1-8	Bubble level
CCP1-9	Metal index pointer
CCP1-10	Telescopic viewer
CCP1-11	Viewer support
CCP1-12	Pulley
CCP1-13	Pulley Support
CCP1-14	Colorlith support table
CCP1-15	Aluminum table legs (Pair)
CCP1-16	Set of small weights (1, 2, 2)
CCP1-17	Set of large weights and weight holder (Two 2 K gm and Three 5 K gm)
CCP1-19	Spanner wrench
CCP1-20	Heavy Nylon Cord (5 ft length)

Sept. 1, 1972
Fifth Draft

APPENDIX G

FOR COMMITTEE USE ONLY

ASTM SUB-COMMITTEE D04.44

Method of Test for Viscosity of Asphalt Cements Using a Cone-Plate Viscometer

1. Scope

1.1 This method covers the determination of the viscosity of asphalt cements by means of a cone-plate viscometer. It is applicable to materials having viscosities in the range of 10^3 to 10^{10} poises and is therefore suitable for use at temperatures where viscosity is in the range indicated. The shear rate may vary between approximately 10^{-3} to 10^2 reciprocal seconds (sec^{-1}) and the method is suitable for determination on materials having either Newtonian or non-Newtonian flow properties.

2. Summary of Method

2.1 The sample is placed between the cone and plate assembly which is then brought to the test temperature. Weights acting through a pulley apply torque to the cone and the angular velocity of the cone is measured. Viscosity in poises and shear rate in reciprocal seconds (sec^{-1}) are calculated from the angular velocity, torque and calibration constants.

2.2 Some asphalt cements may fracture at shear stresses within the range of this instrument. This fracture stress may be reported.

3. Definitions

3.1 Viscosity - A general term referring to the resistance to deformation or internal friction of a liquid and as determined by this method, is expressed as the ratio of shear stress to shear rate, whether this ratio is constant or not. The unit of viscosity obtained by dividing the shearing stress in dynes per square centimeter by the rate of shear in reciprocal seconds (sec^{-1}) is called the poise. The SI unit of viscosity has the dimensions of Newton x seconds/meter, and is equivalent to 10 poises.

3.2 Newtonian Liquid - A liquid in which the rate of shear is proportional to the shearing stress.

3.3 Non-Newtonian Liquid - A liquid in which the rate of shear is not proportional to the shearing stress.

4. Apparatus

4.1 Cone-Plate Viscometer (1,2) - (Figure 1) with metric weights from 10 to 20,000 g. It is used for measuring the viscosities in the range of 10^3 to 10^{10} poises at shear rates from 10^{-3} to 10^2 sec^{-1}. Important dimensions of each cone and approximate constants are given in Table 1. The approximate data of Table 2 may be helpful in the selection of the proper cone and load.

4.2 Thermometers - Calibrated mercury - in glass thermometers of suitable range and graduated to 0.1F (0.05C) shall be used. They shall

---

(1) Sisko A.W., "Determination and Treatment of Asphalt Viscosity Data" Highway Research Board, Highway Research Record No. 67 (1965)

(2) Manufactured by the Cannon Instrument Company, P.O. Box 16, State College, Pennsylvania 16801

conform to the requirements of ASTM Designation E1. Calibrated ASTM kinematic viscosity thermometers are satisfactory. Other thermometric devices are permissible providing their accuracy, precision and sensitivity are equal or better than ASTM kinematic viscosity thermometers.

4.3 Bath - A water, alcohol or ethylene glycol bath suitable for the immersion of the plate and cone and of such height that the cone is immersed to a depth of at least 6 cm. The efficiency of the stirring and balance between heat losses and heat input must be such that the temperature of the water does not vary by more than ±0.1F(0.05C).

4.4 Timer - A stop watch or other timer graduated in divisions of 0.1 sec. or less and accurate to within 0.01 per cent when tested over intervals of not less than 15 minutes. Electrical timing devices may be used only on electrical circuits in which frequency is controlled to an accuracy of 0.05 percent or better.

4.4.1 Alternating current frequencies which are intermittently and not continuously controlled, as provided by some public power systems, can cause large errors, particularly over short timing intervals, when used to actuate electrical timing devices.

4.5 Ohmmeter - or any electrical device capable of indicating that contact between cone and plate is maintained prior to, and during the test.

5. Calibration

5.1 The shear stress constant K_S, the shear rate constant K_D and the friction correction F, are determined as follows:

5.1.1 To calculate the shear stress constant, K_S, proceed as follows:

Using an accurate micrometer, measure the cone radius, r, (diameter/2) to an accuracy of ± 0.005 cm (± 0.002 in). The effective drum radius is the drum radius plus half the string thickness; measure the effective drum radius, R, to an accuracy of ± 0.005 cm (± 0.002 in).

Calculate K_S by:

$$K_S = \frac{3g\ R}{2\pi r^3} \qquad \frac{\text{dynes/cm}^2}{\text{g}} \qquad (1)$$

where r = radius of cone, cm
R = effective radius of drum, cm, and
g = gravitational constant, 980 dynes/g

5.1.2 Determine the shear rate constant, K_D, for each cone by direct calibration with viscosity standards (see Table 3 for available calibration standards). This is obtained by the following procedure:

Measure the angle of rotation, θ, in degrees, and the time, t, in seconds, at applied loads, L, from 5 to 500 grams (the range of applied loads will depend on the size of the cone being calibrated).

Plot the angular velocity, θ/t, in degrees/sec, as the ordinate versus the applied load, L, in grams, as the abscissa as shown in the example of Figure 2. Determine the slope, m, of the line and calculate K_D by:

$$K_D = \frac{K_S}{\eta^m} \ \mathrm{deg}^{-1} \qquad (2)$$

where K_S has the value determined in Eq (1)
η = viscosity of standard oil, poises and
m = slope of regression line resulting from plotting θ/t versus L.

5.1.3 Determine the friction correction F by one of the following methods:

(a) using the formula

$$F = L - 1/m\ (\theta/t)\mathrm{grams} \qquad (3)$$

where F = friction correction in grams
L = Applied load in grams
m = slope of the regression line
θ = measured angle of rotation, degrees
t = measured time of rotation, seconds

The value of F is calculated for each load point and the average is determined.

(b) The friction correction F is determined from the plot of 5.1.2 as the intercept with the abscissa.

6. Preparation of Sample

6.1 Heat the sample in an oven at a temperature not over 325°F (163 C) until it has become sufficiently fluid to pour, occasionally stirring the sample to aid heat transfer and to assure uniformity. Transfer a minimum of 200 ml into a suitable container and heat to a temperature of 250 to 300 F (120 to 150C). In no case, should the material be heated above a temperature of 50F (28C) below the flash point (C.OC). After melting, thoroughly, stir the sample until it is homogeneous and free from air bubbles.

7. Preparation of Apparatus

7.1 Maintain the bath at test temperature within ± 0.02F (0.01C). Apply the necessary corrections, if any, to all thermometer readings.

7.2 Select the proper size cone to allow measurement of viscosity over a 100 fold shear rate range, preferably at loads of 100, 300, 1000, 3000 and 10,000 g or up to fracture of the sample. (See Table 1 for approximate recommended viscosity ranges for each cone.

7.3 Place the cone in position in the viscometer, and the plate in place. Tighten the plate firmly, but do not force.

8. Procedure

8.1 Raise the cone and place sufficient hot, prepared sample onto the center of the plate beneath the raised cone. Lower the cone to rest on the sample and place a load of approximately 1000 g on top of the shaft to ensure contact between the cone and plate.

8.2 Place the cone-plate viscometer on a hot plate and allow it to remain there until an ohmmeter, or other electrical device, indicates contact between the cone and plate. Remove the viscometer from the hot plate, allow it to cool until the cone and plate are cool enough to touch. Remove with a non-scratching blade any asphalt on the edge of the cone and on the plate around the cone.

8.3 Place the viscometer in position in the constant temperature bath. Allow at least 30 minutes for it to attain the bath temperature. Level the viscometer.

8.4 Remove the weight from top of the shaft.

8.5 Alternate No. 1 - Measure the angular velocity for increasing loads using at least five different weights starting with the smallest and applying them successively at no more than 10 minute intervals between each load application.

8.6 Alternate No. 2 - Measure angular velocity for decreasing loads using at least five different weights starting with the largest and apply them successively at no more than 10 minute intervals between each application.

8.7 The cone shall be allowed to rotate approximately one degree before recording data for each weight.

8.8 The angle of rotation of the cone shall be sufficient to ensure a minimum time of 20 seconds, measured to the nearest 0.1 sec. While the test is in progress contact between cone and plate shall be verified continually or intermittently at frequent intervals, since cone and plate separation may occur as the angle of rotation increases. If contact is lost the test must be made with a smaller angle of rotation. Select a larger cone and repeat the test starting with section 7.3.

8.9 Upon completion of the test, remove the viscometer from the constant temperature bath. Clean the plate and cone with several rinsings of an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent.

9. Calculation of Viscosity

9.1 Select the calibration factors corresponding to the cone and cord used. For each load and angular velocity calculate the shear stress, S, the shear rate, D, and the viscosity, η, by:

$$S = K_S \ (L-F), \ \text{dynes/cm}^2 \qquad (4)$$

$$D = K_D \ (\theta/t), \ \text{sec}^{-1} \qquad (5)$$

$$\eta = \frac{S}{D}, \ \text{poises} \qquad (6)$$

10. Report

10.1 Report whether alternate procedure No. 1 or No. 2 was used.

10.2 Report test temperature, viscosity, shear rate and, if fracture occurs, the shear stress resulting in fracture.

11. Precision

11.1 Repeatability - Duplicate results obtained by the same operator on the same sample using the same apparatus should not be considered suspect unless they differ by more than __per cent of their mean.

11.2 Reproducibility - Two results obtained by different operators in different laboratories on the same sample should not be considered suspect unless they differ by more than __ per cent of their mean.

TABLE 1

APPROXIMATE INSTRUMENT CONE SIZES AND CONSTANTS

Cone No. (a)	Approx. Cone Radius cm (b)	Approx. Cone Angle Deg (c)	Approximate Cone Constant	
			K_D deg^{-1}	K_S dynes/cm^2/g
8	3.75	0.5	2.0	31
4	1.88	0.5	2.0	250
2	0.94	0.5	2.0	2000

(a) Other cone sizes may be used.

(b) Exact cone and drum radii must be measured to determine K_S by calculation.

(c) Exact cone angle may be calculated from the determination of K_D by viscosity standards and measured cone and drum radii. K_D is the reciprocal of the angle between the cone and plate.

TABLE 2

APPROXIMATE LOADS AND VISCOSITIES AT SHEAR RATES OF 1, 10^{-1}, AND 10^{-2} SEC^{-1}

Cone No	Load g	Approximate Viscosities in Megapoises At Shear Rates of		
		1 sec^{-1}	10^{-1} sec^{-1}	10^{-2} sec^{-1}
8	100	0.003	0.03	0.3
	1000	0.03	0.3	3
	10000	0.3	3	30
4	100	0.025	0.25	2.5
	1000	0.25	2.5	25
	10000	2.5	25	250
2	100	0.2	2	20
	1000	2	20	200
	10000	20	200	2000
Angular velocity in deg/sec		0.5	0.05	0.005

TABLE 3

VISCOSITY STANDARDS

Viscosity Standard	Approximate Viscosity Poises	
	At 68F	At 86F
N 30,000[a]	1500	-
N 190,000[a]	8000	-

(a) Available in 1 pt containers, price $25.00. F.O.B. State College, Pa. Purchase orders should be addressed to Cannon Instrument Company, P.O. Box 16, State College, Pa. 16801

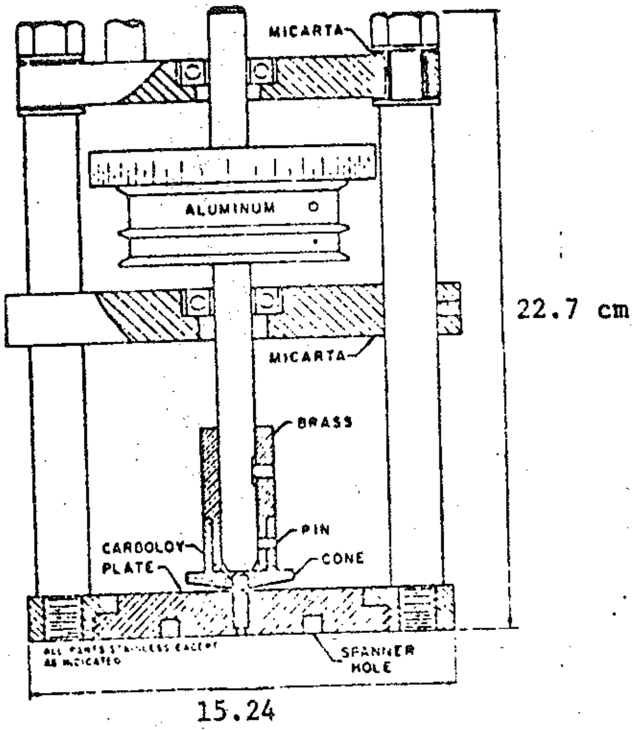

Figure 1. Assembly view of viscometer.

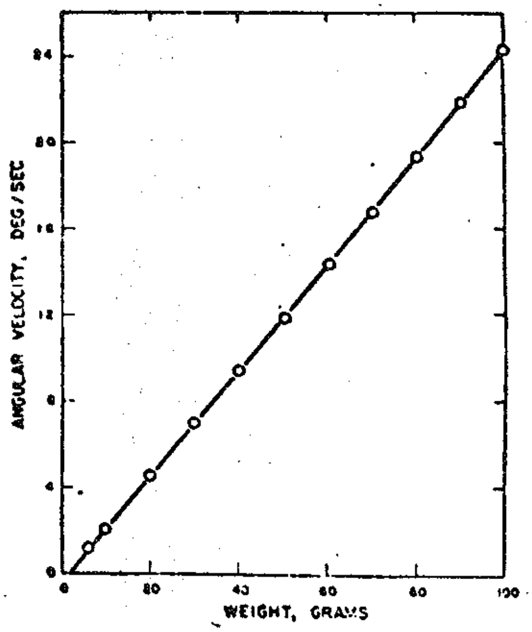

Figure 2. Calibration of instrument.