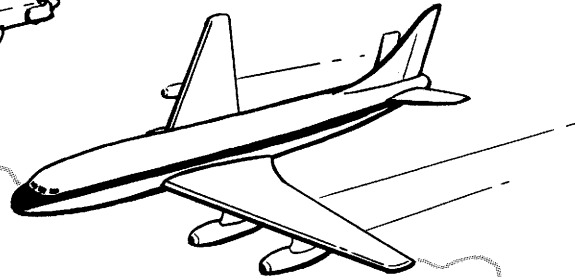
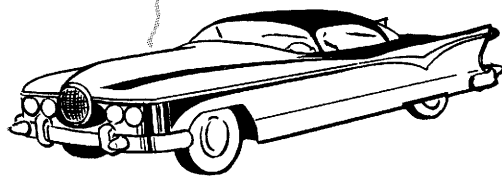


PROPERTIES OF ASPHALT CEMENTS MANUFACTURED IN TEXAS



BY R. N. TRAXLER

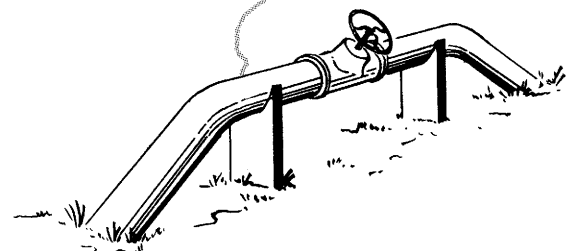
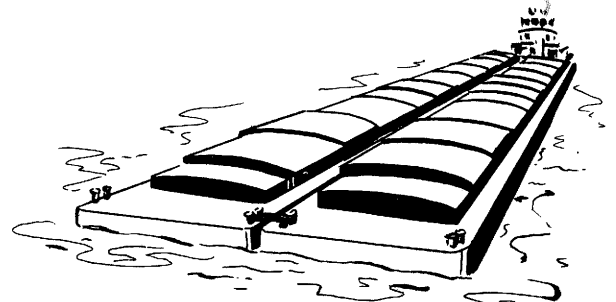
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Properties of Asphalt Cements Manufactured in Texas*

**Dr. R. N. Traxler, Research Engineer
Texas Transportation Institute**

Investigations are in progress on representative asphalts manufactured in Texas and used by the Texas Highway Department. The purposes of these studies are to:

- (1) Determine how binders can best be classified as to quality under handling and service conditions.
- (2) Establish specifications to assure use of superior asphalts by the Department, and
- (3) Determine how the durability of paving asphalts can be improved.

The comments to follow concern some of the results so far obtained under item one, namely, classification as to quality under service conditions.

During mixing, laying, and use of asphaltic pavements, the asphaltic binder is present in the form of thin films. Heat applied to these films, even in the absence of air or light, results in considerable hardening due to the loss of volatile components. Loss of these volatile compounds changes the chemical composition of the asphalt and may be detrimental

to its serviceability if high temperatures are maintained for an appreciable length of time. Early in the history of asphalt technology, volatilization was considered to be the main cause for poor serviceability, but it is now known that this is only a part of the problem, because heating in the presence of oxygen is damaging to an asphalt. Volatilization and oxida-

tion increase the viscosity of all asphalts, and for some the hardening may be excessive. Experience in the field indicates that an asphalt which is highly susceptible to hardening by heat and oxidation may possess limited serviceability as a road binder.

Viscosity Measurements

The data, discussed below, will describe three—and in some cases six—representative 85-100 penetration asphalts, selected from 25 different materials. The asphalts discussed were selected to show a wide range of resistance to hardening and a considerable difference in composition.

Viscosity in absolute units was used to measure the consistencies of the asphalts before and after heating. This method of measurement is more sensitive and informative than the penetration or softening-point pro-

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In Figure III, time of heating the 15-micron film in air at 225°F is plotted versus the relative viscosity "D" of each sample. Results are in the same order as were obtained by the Bureau of Public Roads test and shown in Figure II. Asphalt 1 has high susceptibility to hardening and Asphalt 6 shows a low rate of hardening. Asphalt 3 again is intermediate in its reaction to heat and oxygen.

The two procedures used for evaluating the rate of hardening of asphalts give the same general classification for all of the asphalts that have been investigated. However, data obtained with the 15-micron films were most consistent. More uniform data would be expected from the very thin films, for the following reason. When a 3175-micron (one-eighth-inch) layer is heated, a hardened film develops on the surface which frequently can be noted visually, especially after heating for ten to fifteen hours. Prior to testing in the viscometer it is necessary to thoroughly blend this hard surface film with the softer asphalt under it. Even with most careful at-

tention to this operation, blending may not be perfect. The sample is in reality a mass of original asphalt contaminated with some hardened material. The same problem is presented by the hardened 15-micron film, but to a much lesser degree.

Effect of Temperature and Time on Hardening of Asphalt Films Heated in Air

Attention was next turned to an investigation of the hardening of representative asphalt films at temperatures in the range 225 to 350°F and times from 15 to 120 minutes. These temperatures encompass those to which a hot asphalt mixture may be exposed. High temperatures in the uncompacted asphaltic mixtures may be retained up to 30 minutes or more. Figure IV shows the temperature-relative viscosity data obtained by heating 15-micron films of Asphalts 1, 3, and 6 in air for fifteen minutes.

As an example of how the data in Figure IV can be used, let us assume that the temperature of the asphalt in a hot-mix plant reaches 350°F and that the mass of loose asphaltic mix-

ture remains near this temperature for fifteen minutes and then drops to 325°F, and fifteen minutes later to 300°F. This is an extreme but not impossible situation since 45 minutes may elapse before a hot mix is laid and properly compacted. From Figure IV it will be noted that the above conditions would result in a 26-fold hardening of Asphalt 1 over a 45-minute period. For the same conditions, Asphalt 3 would harden 19.5 fold and Asphalt 6 would increase 8.5 fold in viscosity. Data of this type makes possible a comparison of the effect of different time and temperature conditions on the various asphalts manufactured and used in Texas.

Heating Asphalts in Nitrogen Atmosphere

The increase in viscosity discussed here was caused chiefly by (a) volatilization of oils, and (b) oxidation. To obtain information concerning the relative importance of these two factors, additional experiments were made using the 25 asphalts. In these tests, 15-micron films were heated at

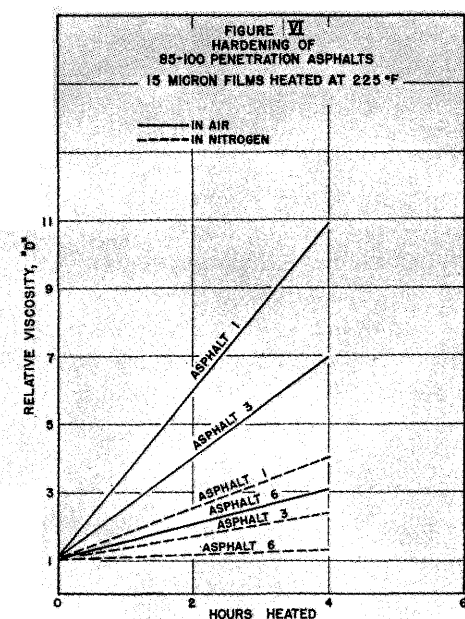


TABLE 1

Analyses on 85-100 Penetration Asphalts

Asphalt	Asphaltenes				Coefficient of Disperson "X" = $\frac{R+C}{A+S}$
	Fraction IV A	Fraction IV R	Fraction III S	Fraction II C	
1	17.8	17.2	49.0	16.0	0.50
2	19.0	13.0	48.0	20.0	0.49
3	24.6	20.4	40.0	15.0	0.55
4	9.3	41.7	40.0	9.0	1.03
5	8.1	33.9	43.0	15.0	0.96
6	1.0	41.0	44.0	14.0	1.22

225°F under one atmosphere of nitrogen. With no oxygen present, any increase in viscosity may be assumed to be caused chiefly by evaporation of oils from the asphalt films. At the test temperature of 225°F, some polymerization may occur but certainly most of the increase in consistency can be ascribed to volatilization. Asphalts 1 and 3 show greater hardening (higher relative viscosities) when heated in nitrogen than does Asphalt 6. Figure V is a plot of hours heated in nitrogen versus relative viscosity, "D." Materials were the same as shown in Figure III.

Figure VI shows the combined data from Figures II and V for comparison.

Chemical Composition and Hardening

It will now be interesting to determine whether there is any connection between the susceptibility of an asphalt to hardening and its content of dispersing or flocculating materials. Each asphalt was subjected to component analysis. To extend the study, the asphaltene (n-pentane insoluble) content was subtracted from Fraction I, and the difference, Fraction IV, has been called asphaltic resins. Table 1 gives the analytical results for six asphalts. The asphaltenes are high-molecular-weight compounds which, to some extent, must be dispersed in the asphalt. The oily Fractions III have a flocculating effect on the asphaltenes. However, the Fractions IV are dispersing or protective agents for the hard asphaltenes and Fractions II also have a marked dispersing effect.

It is logical that a coefficient of dispersion expressed by the ratio between asphaltenes (A) + Fraction III (S) and Fraction IV (R) + Fraction II (C) should indicate the colloidal characteristics of an asphalt. This ratio, $\frac{R+C}{A+S}$, is shown as Coefficient of Dispersion, "X," in Table 1 and is the expression used below. Again, reviewing the compositions, if the amounts of asphaltenes (A) and Frac-

TABLE 2
Relationship Between Composition and Resistance to Hardening for 85-100 Penetration Asphalts

Asphalt	Relative Viscosity, D		Coefficient of Dispersion
	15 micron film heated for 4 hours @ 225°F in Nitrogen		$\frac{R+C}{A+S}$ (Table 1)
	Air		
Poor Resistance to Heat and Oxygen			
1	3.8	10.9	0.50
2	3.7	9.1	0.49
3	2.2	6.9	0.55
Average	2.2	9.0	0.51
Good Resistance to Heat and Oxygen			
4	2.0	4.8	1.03
5	2.0	4.0	0.96
6	1.25	3.0	1.22
Average	1.75	3.9	1.07

tion III (S) are excessive, as in Asphalts 1, 2, and 3, the value of the coefficient, "X," is low and the asphalt is a poorly dispersed or gel-type system. When Fraction IV (R) and Fraction II (C) predominate, as in Asphalts 4, 5, and 6, the value of "X" is high and the asphalt is a well-dispersed sol-type asphalt possessing little internal structure.

Is there any relationship between the Relative Viscosity "D" of a heated asphalt and the composition of the asphalt as expressed by the Coefficient of Dispersion "X"? The data shown in Table 2 indicate that a correlation does exist. For the six asphalts there is a range in the values for "D," representing differences in hardening resulting from heating in the absence and presence of air, and in the values for "X" indicating considerable variation in degree of dispersion. The six asphalts in Table 2 are listed in two groups which may be classed as poor or good in reference to degree of dispersion and rate of hardening. Three, with a high average value of

"D," possess a low average value for "X." The other three asphalts with a low average value for relative viscosity "D" after heating for four hours at 225°F show a high average value for "X." Thus, we may draw the general conclusion that the susceptibility of an asphalt to hardening by heat and oxidation is related to its colloidal nature (degree of dispersion), which in turn is governed by the chemical composition.

The data reported above do not represent all of the work that has been done on the hardening of asphalt by heat, oxygen, and sunlight. We are rapidly learning the "how" of the hardening process. So far, we have only an inkling concerning the "why" of the reactions, but a fuller understanding of the chemical composition of asphalt will clarify the reactions responsible for the hardening which occurs during hot mixing and in service. This increased knowledge will lead to the eventual establishment of quality specifications more discriminatory than are available at present.

cedures. Several years ago, a viscometer was devised for evaluating the viscosity of thin (5 to 50 microns*) films of asphalt. This apparatus (shown in Figure 1) has made possible precise measurements of the hardening of asphalts by heat, oxidation, or the action of actinic light. The microviscometer used in the experiments described below is the apparatus sold by Hallikainen Instruments, of Berkeley, California.

Bureau of Public Roads Film Test

In this test, which is becoming widely used, a one-eighth-inch layer of asphalt is placed in a shallow aluminum pan and heated in an oven at 325°F for five hours. The heated asphalt is tested for penetration, soft-

ening point, and ductility. Retained per cent of original penetration is calculated.

This procedure was followed in our original investigations, but the times of heating were 5, 10, and 15 hours in order to obtain more definite information concerning the rate at which the various asphalts hardened. After removal from the oven, the oxidized asphalt was thoroughly mixed in an attempt to obtain a uniform sample. The viscosities of the original and of each hardened sample were determined at 77°F in the microfilm viscometer using films 25 to 30 microns thick. The viscosity of each sample was determined at three rates of shear. From the resulting experimental data, the viscosity of each asphalt was calculated at a rate of

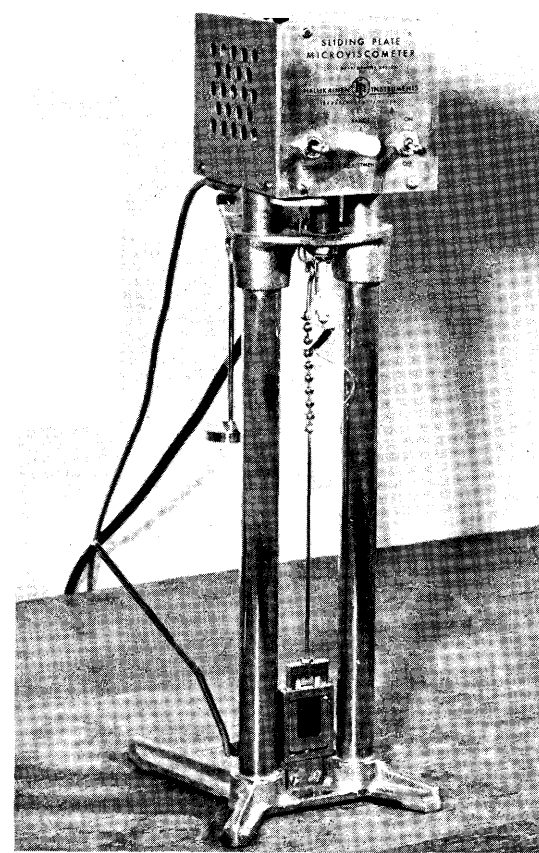
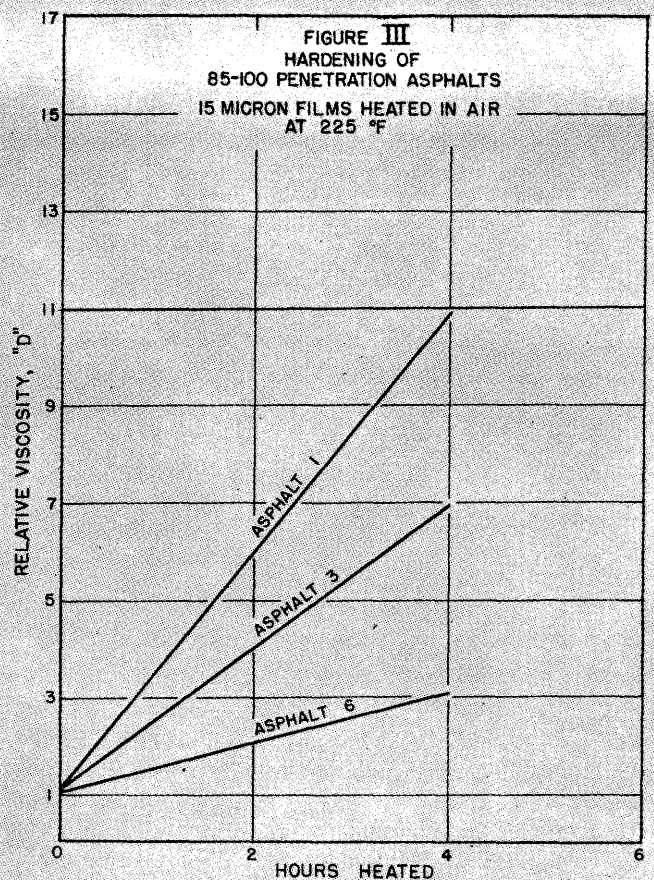
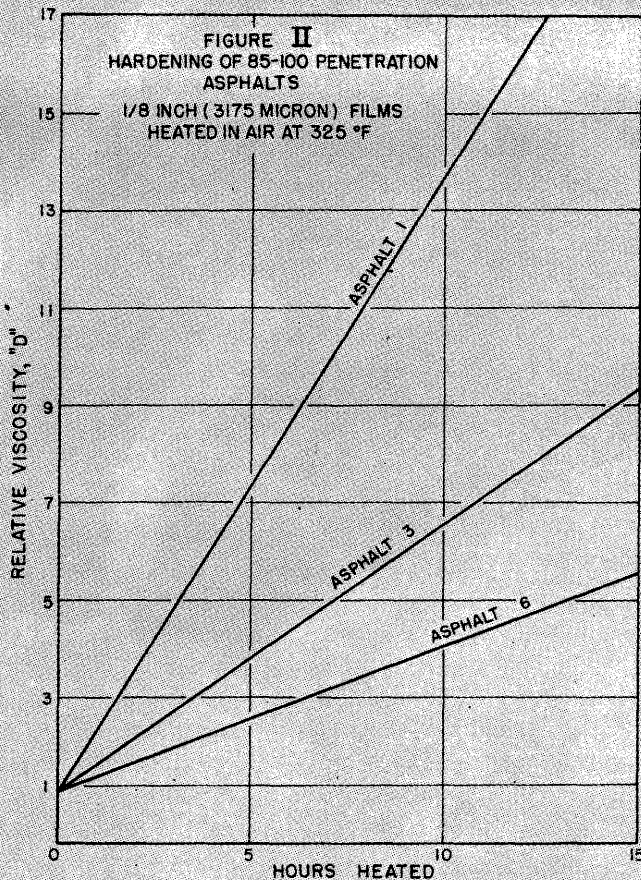
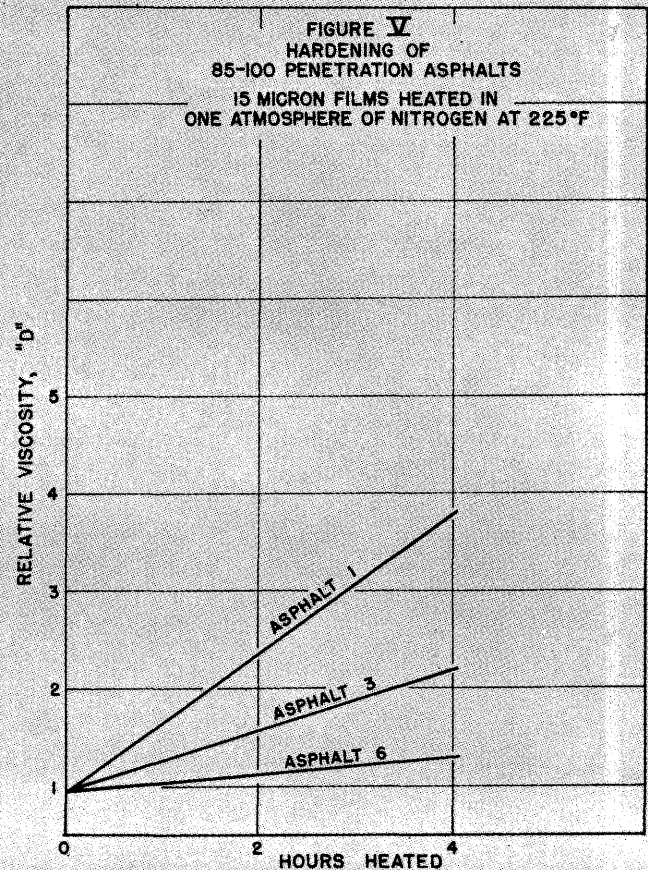
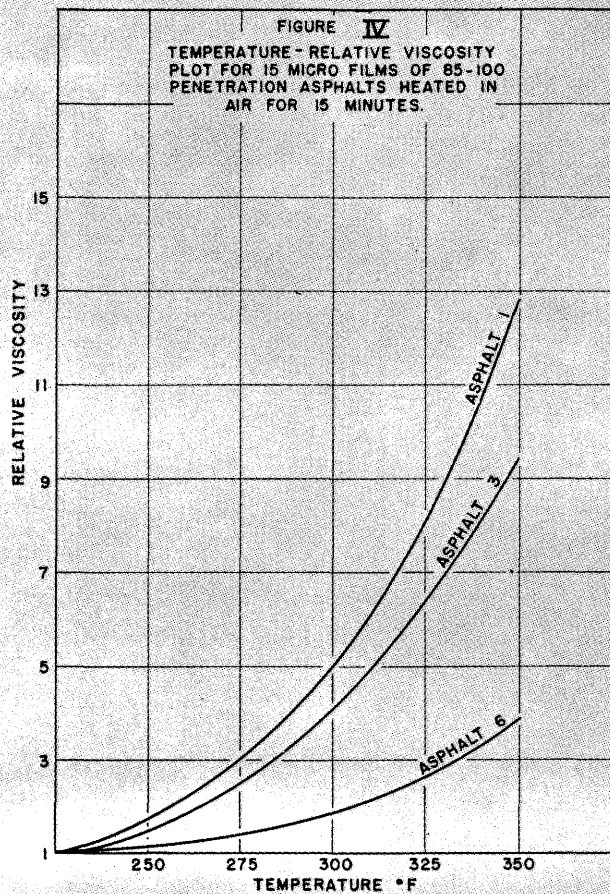


Figure 1—Hallikainen Microfilm Viscometer

*There are 25,400 microns to one inch.





shear of 5×10^{-2} reciprocal seconds. This procedure was used to obtain comparable viscosities for the asphalts possessing various degrees of internal structure or non-Newtonian flow.

The viscosity of each hardened sample is divided by the original viscosity of the asphalt. The quotient, which may be called the relative viscosity, is a measure of the hardening of the asphalt, and is represented by "D" in this article. A high value for the relative viscosity "D" means that the asphalt hardens excessively; a low value indicates good resistance to hardening and, thus, anticipated superior durability under service conditions.

Figure II is a plot of heating time at 325°F versus value of "D" for three asphalts shown. The best straight line was established through

the experimental data. Asphalt 1 shows poor resistance to hardening whereas Asphalt 6 is an example of an asphalt with low susceptibility to hardening by heat and oxygen. Asphalt 3 is intermediate in its reaction to heat and oxygen under the conditions of the test.

Shell Thin Film Test

Griffin, Miles, and Penther of the Shell Oil Company developed a procedure in which very thin films of asphalt were oxidized and the viscosities measured by the microfilm viscometer. They oxidized a 5-micron film of asphalt on a glass plate in an oven for two hours at 225°F. Two modifications of this procedure were made in the work described below. First, 13- to 15-micron films were used and second, oxidation initially

was conducted for two and four hours to obtain information concerning the rate of hardening. The 15-micron films were employed to expedite preparation of the samples and also because 15 microns seems to be in the range of optimum average thickness for asphalt films present in most types of pavement.

Viscosity measurements were again made using 25- to 30-micron films in the microfilm viscometer. Viscosities were determined on each original asphalt and oxidized sample at three different rates of shear. From these experimental data, the viscosities at 5×10^{-2} reciprocal seconds were calculated. The quotient, obtained by dividing the viscosity of the hardened asphalt by the viscosity of the original material, is shown as the relative viscosity, "D."