### CHANGES IN ASPHALT CEMENTS DURING PREPARATION LAYING AND SERVICE OF BITUMINOUS PAVEMENTS

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Asphalt paving technologists have been concerned, for several decades, about the changes that take place in asphalt used in a bituminous pavement both during the preparation of the hot mixture and exposure to the environmental conditions encountered in service over a period of months or years.

Within the past few years this problem has been approached by an increasing number of investigators and organizations. Heithaus and Johnson ( l) obtained extensive data on a series of asphaltic concrete test sections which contained a wide variety of asphalt types and grades in hot mixes of two .. 이 글로맨 (2008) - 1950 - 대체 뉴욕 (2008) - 1960 - 1970 - 1980 - 1980 - 1980 - 1980 - 1980 - 1980 - 1980 - 1980 different aggregate gradations and several binder contents. After mix'ing and laying, viscosities of recovered asphalts were determined over a three-year !-~. period of service and their values compared with the viscosities of the original asphalts. The sliding plate viscometer was used to measure viscosities at  $77^{\circ}$  F. Hardening in service was found to increase as void content of the mix increased. A laboratory microfilm hardening test was shown to be valuable for predicting the hardness of road asphalts resulting from preparation and service.

Pauls and Halstead {2) reported a study of the change in the properties of sheet asphalt pavements over a  $19$ -year period of service. They found that, in general, a large proportion of the hardening of the asphalt occurred ir the mixing

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process and rolling of the pavement. It was also concluded that the overall age-hardening in the pavement did not appear to be directly related to the type of asphalt used. The point was made, in the discussion of these data, that the more a pavement is used the less it cracks. It should be remembered that the bituminous pavement at the end of a dead-end street is a notable example of the deterioration (cracking) of a pavement by age-hardening of the asphalt binder used in its construction. Shear by traffic destroys a portion of the hardening that develops with time.

The well-known Zaca-Wigmore Asphalt Test Project ( 3} was installed by the California Division of Highways during 1954-55. The asphalts used were of 200 to 3 00 penetration grade and were found to weather at different rates, but all showed a tendency toward a  $\phi$  decrease in the rate of hardening after 16 to 20 months of service. The data also showed that asphalts produced from different crude sources and methods of production can have various degrees of durability under equivalent conditions of traffic and climate.

In connection with this project viscosities of the original and recovered asphalts were determined using the thin film, sliding plate viscometer  $(4)$  and increases in the ratio of the viscosity of the aged asphalt to that of the original asphalt were used for evaluation of bituminous hardening.

More recently  $(5)$  data have been given for the change in asphalt viscosity during mixing with hot aggregates. Information was obtained from both laboratory and plant runs.

The Texas Highway Department and the Texas Transportation Institute in 1963 entered into a cooperative research endeavor on the changes that occur in

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## LOCATION OF EXPERIMENTAL SITES FIGURE I

asphalt cements from different sources and methods of processing from the time they enter a hot-mix plant until samples are recovered from the pavement over a considerable period of time.

This paper describes the field and laboratory programs and gives data showing the increase in viscosity and other changes in the bitumen during the preparation and laying of the pavement and at intervals through two years of service.

#### FIELD PROGRAM

Hot-mix surfacing studies using  $85-100$  penetration grade asphalt cemerts were located in 13 widely separated districts of the Texas Highway Department (Experimental sites are shown in Figure 1, facing). Asphalts were supplied by 9 different producers. At each site a  $1 \frac{1}{4}$  to  $1 \frac{1}{2}$  inch thick surfacing (about  $125$  lbs./sq/yd.) was being laid by the Highway Department as a part of their annual maintenance program. A spot in the particular highway was selected by District personnel for the experiment and before the paving machine arrived. heavy aluminum foil was tacked to the base by roofing nails. This was done to facilitate removing unbroken slabs of surfacing material and to prevent contamination of the asphalt cement by primer applied to the base. Samples taken from the test sections by District personnel were promptly shipped to the Texa: Transportation Institute in special wooden boxes designed to prevent breakage during transport,

Pertinent information obtained at each site included highway designation, asphalt refinery (Code No.), date pavement surfacing was laid, and temperature

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of the freshly prepared mixture. This information is given in Table 1 page 22. Aggregate samples were taken from the bins at each experimental site and a mineralogical examination made of the materials.

At each field site the following samples were collected:

- ( 1) Original asphalt as supplied to the hot-mix plant.
- (2) Asphalt-aggregate mixture as it issued from the plant.
- ( 3) Asphalt-aggregate mixture when it was placed in the paving machine.
- $(4)$  A 2- x 2-foot sample of the surfacing material taken 1 day after the pavement was laid and compacted.
- $(5)$  A 2- x 2-foot sample of the surfacing taken 2 weeks after laying.
- (6) A 2- x 2-foot sample of the surfacing taken 4 months after laying.
- (7) A  $2 x 2$ -foot sample of the surfacing taken 1 year after laying,
- $(8)$  A 2- x 2-foot sample of the surfacing taken 2 years after laying.

#### lABORATORY PROGRAM

#### Tests on Asphalt-Aggregate Mixtures

Hveem stability and cohesiometer values on the mixture used were supplied by the District in which the experiment was conducted. Densities were determined on each slab after delivery to the Institute and air permeability measurements were made using the Air Permeometer developed by the California Research Corporation and sold by Soil Test, Inc. These data on the various surfacings are shown in Table 2 pages 23 through 25.

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#### Extraction of Asphalt Cements from Paving Mixture

About 25 pounds of asphalt-aggregate mixture or surfacing removed from the road at each site were placed in large Colorado type extractors for separation of the asphalt from the stone. A mixture of 6 parts benzene and l part ethyl alcohol was used to extract the asphalt from the bituminous mixtures. Alcohol was used to assure complete removal of all asphaltic components from the various aggregate surfaces.

The benzene-alcohol solution of asphalt was centrifuged to remove mineral particles that might have passed through the filter paper in the extraction apparatus. The essentially mineral-free solution of asphalt was distilled by the standard Abson procedure until a large portion of the benzene-alcohol mixture was removed and the concentrated solution was then transferred to a thin film evaporator and the remaining solvent removed at 125<sup>0</sup> F and 15 mm. of mercury pressure. With 13 experiments and 8 samples from each, data should have been obtained on 104 original and extracted asphalts. Actually data were obtained on only 94 samples. Ten samples of mixture or pavement slabs were lost, or damaged in one way or another. A common source of contamination on the highway was spillage of lubricating oil or diesel fuel.

#### Flow (Rheological) Data on the Original and Extracted Asphalts

Viscosities at  $77^\circ$ ,  $95^\circ$ ,  $140^\circ$ , and  $275^\circ$  F and ASTM penetration at  $77^\circ$  F, 100 grams, 5 sees were determined on each original and recovered asphalt. Measurement of viscosities at 77<sup>0</sup> and  $95^{\circ}$  F were made in the thin film (sliding plate) Hallikainen viscometer. Values given in this paper were calculated

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## RELATIVE VISCOSITIES OF THE 13 ASPHALTS HARDENED UNDER VARIOUS CONDITIONS AND TIMES

Temperature 77°F



at 5 x  $10^{-2}$  sec  $^{-1}$  rate of shear. Kinematic viscosities at  $140^{\circ}$  and 275<sup>o</sup> F were determined in the Cannon-Manning vacuum capillary tube apparatus,

Viscosities of the thirteen original asphalts varied from  $0.57$  to  $1.18$ megapoises at 77<sup>0</sup> F and from 2.25 to 9.95 stokes at 275<sup>0</sup> F. In order to simplify the presentation of the progress of hardening during the preparation and service of the various bituminous surfacings studied, the measured viscosity of each sample was not used directly. Instead a relative viscosity for each recovered asphalt was calculated by dividing the viscosity of the hardened asphalt at  $77^{\circ}$  F by the viscosity of the original asphalt at the same temperature and rate of shear. The resulting quotient indicates how many fold the asphalt cement increased in hardness because of the treatment or service it encountered. The use of this ratio eliminated the variability caused by differences in the viscosities of the original asphalts and gives a clearer picture of the hardening rates, The viscosities and Relative Viscosities are given in Table 3, pages 26 to 32.

A laboratory hardening test was also made on each original asphalt. Films 15-microns thick were placed on 4cm x 4cm glass plates and exposed in a dark oven at 225<sup>0</sup> for 2 hours. The cool, hardened films were scraped from the glass by a razor blade, the asphalt placed between the plates used in the sliding plate viscometer and viscosities determined at  $77^{\circ}$  F. Viscosity of the hardened asphalt was divided by the viscosity of the original asphalt measured at the same temperature and rate of shear to give the relative viscosity mentioned above, The simple test gives a good indication of the susceptibility of an asphalt to hardening by time, heat and oxidation. Table 4, facing, shows first the relative viscosity

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![](_page_8_Figure_0.jpeg)

obtained for each of the thirteen asphalts by this laboratory test. Next, the relative viscosities are shown for the asphalt recovered from (a) the mixture issuing from the plant, (b) a sample taken from the paver,  $(c)$  a sample removed one day after laying and compaction, (d) after 2 weeks service, (e) after 4 months service, (f) after 1 year of service, and  $(q)$  after 2 years of service. The table also shows the range of relative viscosity values for a particular type of sample and finally the average R. V. for each situation to the nearest 0. 05.

An examination of the data shown in Tables 3 and 4 indicates that approximately a two- to threefold increase in hardening occurs during the combined mixing cycle (at temperatures of  $250^{\circ}$  to  $325^{\circ}$  F), hauling to the paving site, laying and compacting the hot mixture and the first day on the road. Some time between two weeks and four months of service the rate of hardening increases rapidly, It also is evident that differences in hardening may occur with the same producer's asphalt at sites where different conditions existed, Such a situation is illustrated in Figure II, facing, for one producer's asphalt. The most obvious differences in these two experimental sites are the times of year the pavements were laid, and the kind of aggregate used. When pavements are made at two sites, with asphalt from a producer of uniform asphalt, differences in rate of hardening, after a particular time, are most likely caused by chemical changes in the films of asphalt induced by heat, oxygen, or reactions within the asphaltic components at the aggregate-bitumen interface.

### Micro-ductility Tests on the Original and Recovered Asphalt Cements at  $77^{\circ}$  F

The microductility machine developed by the California Division of Highways

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### ORIGINAL AND RECOVERED ASPHALTS FROM FIELD TEST SITES 1 THROUGH 14

### Viscosity at 770F for Sample from Each Site Possessing Highest Microductility Value

![](_page_10_Picture_170.jpeg)

 $(6)$  was used to evaluate the asphalts obtained at the thirteen sites discussed above, The apparatus is constructed to pull the asphalt at a rate of 0, 5 em. per minute. A small sample ( 0. 05 gm. ) is required for a test. The practice followed was to obtain at least three determinations that check quite closely and use their average as the accepted value.

An analysis was made of the relationship between ductility and viscosity at 77° F for 94 samples. For a particular site the sample possessing the highest ductility was selected and compared with its viscosity at  $77^{\circ}$  F. The viscosities of the high ductility samples ranged from 1 to 3. million poises. Data are shown in Table 5, page 11. A log-log plot of viscosity versus ductility for the original, plant, 1 day and 4 months samples from Site No. 1 is given in Figure III, page 21.

In 1940 ( 7) measurements, on series of progressively harder asphalts processed from given sources, showed that the maximum ductility by the standard ASTM test at  $77^{\circ}$  F, 5 cms/min. was associated with asphalts possessing viscosities between about 1 and 3 megapoises at  $77^{\circ}$  F.

The greatest advantage of the new micro apparatus is the small sample required and the fact that definite values are obtained for the most ductile material, whereas with the standard ASTM test, high ductilities are recorded as 100+, 150+, or 200+ ems.

- 12 -

![](_page_12_Picture_69.jpeg)

## ASPHALTENE CONTENTS, PERCENT

- 13 -

### CHANGE IN COMPOSITION OF ASPHALT CEMENTS DURING SERVICE IN PAVEMENTS

Extensive investigations of the compositions of asphalts have been conducted during the past 3 decades but will not be reviewed here because that type of information can be readily obtained elsewhere ( 8).

The original asphalts and satisfactory samples obtained after one and two years of service were analyzed for asphaltene and petrolene contents. Petrolenes were extracted from the original asphalts by means of n-pentane. Alundum extraction thimbles (RA 84 Dense) were filled with glass wool and about 2. 5 grams of molten asphalt poured over the fibrous mass. The alundum thimbles were placed in slightly larger paper thimbles which fitted into a Soxhlet Extractor. One hundred and fifty ml. of n-pentane was refluxed through the asphalt coated glass wool for 22 to 23 hours. The resulting solution of petrolenes was stripped of solvent, the residue cooled and weighed to determine the amount of extracted material ( petrolenes). This subtracted from the weight of the original sample gave the amount of n-pentane insoluble material (asphaltenes). This procedure avoided the weighing of asphaltenes in air thereby precluding errors caused by the rapid oxidation of unprotected asphaltenes. Table 6, facing, gives the asphaltene data. In the eleven experimental sites, for which the necessary samples were available, the asphaltene contents after one year in service were appreciably higher than in the original asphalt cements. The analyses on the bitumens extracted after 2 years of service showed an increase in asphaltenes for seven of the eleven samples over that obtained after

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# VISCOSITIES (POISES) OF PETROLENES AT 77º F

![](_page_14_Picture_10.jpeg)

one year of service. A definite decrease was noted in the four other two-year old samples (values underscored in the table). There is no good explanation for this behavior because in three cases the decreased asphaltene content occurred in one of a pair of samples from a particular refinery. There does not appear to be anything wrong with the analyses - in fact excellent checks were obtained on the four samples which. might be considered out-of-line. This situation may be another example that we know very little about what happens to an asphalt in service.

Petrolenes from the original asphalts and the extracted bitumens after two years of service in the pavement were measured for viscosity at  $77^{\circ}$  F in the sliding plate viscometer. In most cases films 6 to 12 microns thick were used in this measurement. Table 7, facing, gives the viscosity values obtained and shows that the ratio of viscosities between 2 years old and original samples ranged from 1.05 to 20.1. The high value of twentyfold increase in viscosity after 2 years of service occurred in a pavement made with highly absorbent limestone" A portion of the low viscosity oil absorbed by the larger pieces of aggregate is not removed during extraction process. Thus, the loss of these low viscosity materials from the extracted material may be a partial answer to the exceptionally high viscosity petrolenes found in the sample extracted from this particular pavement after two years of service. It is evident that much more work must be done to learn what changes can take place in the films of asphalt used to hold together the aggregate in a pavement surfacing during service. The studies made to date certainly indicate that the problem is one of considerable complexity.

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#### SUMMARY AND CONCLUSIONS

This paper presents data on the hardening of 85-100 penetration asphalt cements (based on viscosity measurements) from the time they entered the hot mix plant through the laying of the pavement surfacing and two years of service, Thirteen asphalts (made at nine different refineries), and employed by the Texas Highway Department in their maintenance program at 13 locations in the State, were used in the study.

Data obtained show that relatively slow hardening occurs during the preparation of a paving mixture at temperatures of  $250-325^{\circ}$  F, laying the surfacing and during the first 2 weeks of service. However, from then on up to 2 years the hardening of the asphalts proceeds at a much more rapid rate which varies among the different asphalts combined with different aggregates under various service conditions .

The microductility apparatus devised by the California Division of Highways was used to test the original and recovered asphalts discussed in this paper. For each asphalt (starting with the original material and testing the progressively harder samples) it was found that the highest ductility value at 77<sup>0</sup> F was always associated with a viscosity of 1 to 3 megapoises at 77<sup>0</sup> F. One advantage of this apparatus is that a number is always obtained for high ductility values and the resort of listing data as  $100+, 150+,$  or  $200+$  is not necessary.

Analyses have shown that asphaltene content increases during preparation and laying of the pavement and up to one year of service. Between one and two years of service in the pavement the a sphaltene content usually increases, but

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in a few situations an unexplained decrease is noted. Viscosities of the petrolenes, separated from the original asphalts and from the materials extracted after 2 years of service in the pavement, were determined at  $77^{\circ}$  F. Over the period of 2 years the petrolenes increased in viscosity from 1. 05 to 20. 1 fold. The increase in asphaltene content and petrolene viscosities at 77<sup>0</sup> F with time do not in every case explain the hardening of the films of asphalt cement during service.

#### Literature Cited

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- ( 8) Highway Research Board Bibliographies "Characteristics of Bituminous Materials," Bibliography No. 35 (1935), Bibliography No. 40 (Supplement<br>to No. 35) (1966) to No.  $35)$  (1966).

## Appendix

![](_page_19_Picture_44.jpeg)

![](_page_20_Figure_0.jpeg)

FIGURE III

GENERAL INFORMATION ON VARIOUS EXPERIMENTAL SITES IN TEXAS  $^\star$ 

![](_page_21_Picture_105.jpeg)

\*All asphalts used were 85-100 Penetration Grade.

![](_page_22_Picture_145.jpeg)

### TESTS ON ORIGINAL MIX AND SAMPLES OF SURFACINGS REMOVED FROM THE HIGHWAYS

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![](_page_23_Picture_148.jpeg)

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![](_page_24_Picture_65.jpeg)

### FLOW PROPERTIES OF ORIGINAL 90 PENETRATIONS ASPHALTS AND THOSE RECOVERED FROM MIXTURES WITH VARIOUS AGGREGATES

![](_page_25_Picture_16.jpeg)

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TABLE 3 CONTINUED - l

![](_page_26_Picture_262.jpeg)

TABLE 3 CONTINUED - 2

![](_page_27_Picture_255.jpeg)

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![](_page_28_Picture_248.jpeg)

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TABLE 3 CONTINUED - 4

			Sliding Plate Viscometer. at $5x10^{-2}$ sec <sup>1</sup>   Vacuum Capillary. Stroke Rate of Shear - Megapoises							Pen. at 770F	
Site	Producer	Age of Sample	$77^\circ$ F	R.V.	950F	R.V.	$140$ <sup>O</sup> F	R.V.	$275^{\circ}$ FR.V.		$100$ gm/5sec
$\overline{7}$	15	Original	1.00	$\frac{1}{2}$	0.11		1165	aas	2.75	$\equiv$	92.5
		Plant	1.75	1.75	0.17	1.55	1660	1.4	3.35	1.2	75
		Paver	1.85	1.85	0.24	2.2	2060	1.75	3.55	1.3	66
		1 day	2.85	2.85	0.31	2.8	2430	2.1	3.70	1.35	58
		2 weeks	4.35	4.35	0.33	3.0	2960	2.5	3.75	1.35	54
		4 months	13.20	13.20	0.86	7.8	5370	4.6	4.45	1.60	36.5
		1 year	14.50	14.50	1.28	11.6	8100	6.9 <sup>°</sup>	5.00	1.80	33.5
		2 years	21.40	21.40	2.20	20.0	15100	13.0	5.90	2.15	29
12	$\overline{2}$	Original	0.86		0.088	me.	1190	$\equiv$	2.25		77
		Plant	1.20	1.4	0.116	1.3	1430	1.2	2.80	1.1	68
		Paver	1.40	1.6	0.130	1.5	1780	1.5	2.95	1.15	62
		1 day	2.05	2.4	0.196	2.2	1995	1.7	3.15	1.25	55
		2 weeks	2.20	2.6	0.210	2.4	2175	1.8	3.17	1.25	53.5
		4 months	4.95	5.7	0.400	4.5	3340	2.8	3.70	1.45	39.5
		1 year	13.20	15.3	0.690	7.8	8775	7.4	5.16	2.00	25.5
		2 years	25.00	29.0	2.300	26.0	15600	13.0	6.97	2.75	19

![](_page_30_Picture_40.jpeg)

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- $\sqrt{\omega_{\rm B}^2}$  . Then
- $\label{eq:2} \begin{split} \mathcal{L}_{\text{max}} &= \frac{1}{2} \frac{1}{M} \left( \frac{1}{M} \right) \frac{1}{M} \frac{M}{M}, \end{split}$
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- $\label{eq:zeta} \begin{array}{c} \gamma_{\rm e} = \frac{1}{2} \frac{1}{2} \left( \frac{1}{2} \left( \frac{1}{2} \right) \right) \left( \frac{1}{2} \right) \, , \\ \gamma_{\rm e} = \frac{1}{2} \frac{1}{2} \left( \frac{1}{2} \left( \frac{1}{2} \right) \right) \, . \end{array}$
- $\begin{split} \mathbf{w} &= \mathbf{w} \mathbf{e}^\top \mathbf{e}^\top \mathbf{e}^\top \mathbf{e}^\top \mathbf{e}^\top \mathbf{e}^\top \\ \mathbf{e}^\top_\mathbf{e} &= \mathbf{e}^\top \mathbf{e}^\top \mathbf{e}^\top \mathbf{e}^\top \mathbf{e}^\top \mathbf{e}^\top \mathbf{e}^\top \end{split}$

 $\int_{\mathbb{R}^{n-1}} e^{-\frac{1}{2} \pi i \omega_{\text{max}} \left( \frac{1}{2} \left( \omega_{\text{max}} + \frac{1}{2} \left( \omega_{\text{max}} + \frac{1}{2} \right) \right) \right)} d\omega_{\text{max}}$ 

TABLE 3 CONTINUED - 6

			Sliding Plate Viscometer. at 5x10 <sup>-2</sup> sec <sup>1</sup>				Vacuum Capillary. Stroke				Pen. at
Site	Producer	Age of	Rate of Shear - Megapoises						77°F		
		Sample	770F	R.V.	$95$ <sup>O</sup> F	R.V.	$140$ <sup>O</sup> F		$R.V.$ 275 <sup>o</sup> F R.V.		$100$ qm/5sec
13	5	Original	0.81	$\overline{\phantom{a}}$	0.074	$\overline{\phantom{a}}$	940		2.60	$\equiv$	90
		Plant	1.78	2.2	0.200	2.7	1590	1.7	3.30	1.25	59
		Paver	1.98	2.5	0.200	2.7	1920	2.0	3.40	1.30	57.5
		$1$ day									
		2 weeks	2.26	2.8	0.244	3.3	1990	2.1	$3.5^{\circ}$	1.35	52
		4 months	4.66	5.75	0.250	3.4	2200	2.3	3.65	1.40	48
		1 year	7.96	9.85	0.660	8.9	5690	6.0		$4.35$   1.65	30
		2 years	17.80	22.00	1.160	15.7	7790	8.3	5.20	2.00	26

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