SUSCEPTIBILITY OF PAVING ASPHALTS TO HARDENING BY HEAT, OXYGEN AND SUNLIGHT

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Abstract

Report: Progress Report No. 4 - Research Project No. 15.

Title: Susceptibility of Paving Asphalts to Hardening by Heat, Oxygen, and Sunlight.

Period: February 1, 1961 to October 1, 1962.

Objective: To investigate paving asphalts used by THD to determine how they can be evaluated for quality.

Experimental: Increases in hardness of asphalt films caused by oxidation, photooxidation, volatilization, photochemical action, and polymerization were determined by the microfilm technique. Relative viscosities were evaluated on asphalts extracted after two years of service. Hardening caused by hot mixing, transportation, and laying on the pavements was determined.

Conclusions:

(1) Oxidation in the dark is a very important factor contributing to the hardening of films in an asphalt pavement.

(2) Photooxidation probably is of secondary importance followed by volatilization.

(3) Photochemical action and polymerization are of minor importance.

(4) All effects are accentuated by increases in time and temperature.

(5) Asphalts recovered from pavements after two years usually are quite hard because of the effect of asphalt composition, nature of the aggregate, and design and constructional factors.

(6) Asphalts increase in viscosity considerably during hot mixing, transport to paving site, and laying of the pavement.

(7) Laboratory oxidation tests give valuable information concerning the potential hardening of asphalt during construction and service life of a pavement.

Recommendations: It is recommended that the Texas Highway Laboratories be equipped with an Hallikainen Viscometer and auxiliary equipment in order that oxidation tests on 15-micron films of asphalt can be made using essentially the technique being tentatively considered by A.S.T.M.

Future Work: A considerable number of asphalt cements have been obtained by Highway Department personnel at paving sites during the 1962 season. These asphalts will be tested for susceptibility to oxidation and compared with the serviceabilities of the pavements in which they were used.

SUSCEPTIBILITY OF PAVING CEMENTS TO HARDENING BY HEAT, OXYGEN, AND SUNLIGHT

I. OBJECTIVES FOR RP-15

The objectives of this project are:

(1) Investigate the paving asphalts used by the Texas Highway Department to determine how they can be evaluated for quality.

(2) Establish specifications to assure use of superior asphalts by the Department, and

(3) Determine how the durability of paving asphalts can be improved.

II. HISTORY

Research Project 15 "Modifications of Properties of Asphalt" was initiated on February 1, 1959 and Progress Report No. 1 was issued February 1, 1961. The current report is concerned chiefly with the studies made under objective (1) since the issuance of Progress Report No. 1. Work done during the past 18 months and discussed herein has been concerned with the hardening of asphalt films by (a) oxidation (b) photooxidation (c) volatilization (d) photochemical action and (e) polymerization. Some of the data are elaborations of those discussed in Progress Report No. 1.

III. CONCLUSIONS

The following conclusions are drawn from the data given below:

1. Oxidation in the dark is a very important effect contributing to the hardening of asphalt films in bituminous pavements.

2. Photooxidation (combined action of air and sunlight) of asphalt films results in excessive hardening. However, in a pavement the effect may not be of great importance because the action occurs only with films exposed at the surface of the pavement. The main deteriorating effects of photooxidation may be the acceleration of surface erosion.

3. Volatilization is of secondary importance to oxidation and photooxidation in the hardening of asphalt films.

4. The three above effects are accentuated by increases in time and temperature.

5. Photochemical action (in absence of oxygen) and polymerization are of minor importance although they can cause measurable increases in viscosity.

6. Asphalts recovered from pavements after two years of service show varying increases in hardening depending on (a) composition of the asphalt, (b) nature of the aggregate and (c) design and constructional factors. In certain cases effects (b) and (c) may be more important than effect (a).

7. Asphalt cements increase in viscosity during (a) the short period of residence in the hot mixing process, (b) transportation to the paving site, (c) laying and compaction of the pavement and (d) the service life of the pavement.

8. Laboratory oxidation tests made on 15-micron films of asphalt cement by heating in an air oven at 225°F for 2 hours appear to give valuable information concerning the potential hardening of an asphalt in the preparation and handling of a hot mix and during the service of the pavement.

IV. RECOMMENDATIONS

It is recommended that the Texas Highway Laboratories be equipped with an Hallikainen viscometer and auxilliary equipment, and personnel trained to run oxidation tests on 15-micron films using essentially the technique being tentatively considered by the American Society for Test-Materials (see Appendix).

V. FUTURE WORK

A considerable number of asphalt cements (85-100, 120-150 and 150-200 penetration grade) have been obtained for the Texas Transportation Institute by Texas Highway Department personnel from various construction sites during the summer and fall of 1962. Details of the sites were supplied. These asphalts will be evaluated and compared with the serviceabilities of the pavements in which they were used.

VI. EXPERIMENTAL

1. Materials Investigated

The asphalts used were selected from those used by the Texas Highway Department in the summer of 1959 and described in Table 1, A-1, A-2, A-3, A-6 and A-7 of Progress Report No. 1. Most of the data reported below were obtained on Asphalts 1, 3, 6 and 11 of 85-100 penetration, and 1A, 3A, 6A and 11A of 120-150 penetration.

A few asphalts obtained from other sources are used to illustrate particular effects.

2. Methods for Measuring Viscosity and Quantitative Evaluation of Hardening

The microfilm (sliding plate) viscometer was used to measure the consistencies of both the original and hardened asphalts. This apparatus is illustrated and its operation briefly described in Progress Report No. 1. Also, the use of Relative Viscosity (the quotient obtained by dividing viscosity of the hardened asphalt by that of the original asphalt) as a quantitative measure of hardening is described therein. For the sake of brevity, these discussions are not repeated here.

TABLE 1

Susceptibilities of Representative 85-100 and 120-150 Penetration Asphalts to Oxidation in the Dark

Asphalt	Relative Viscosity
Heated at 225°F for 2 hours	
Viscosities measured at 77°F and calculated at 5×10^{-2} sec^{-1}	
85-100 Penetration	
6	2.1
11	3.3
8	4.0
3	4.3
1	6.0
120-150 Penetration	
6A	2.5
11A	2.6
3A	4.0
1A	4.2
Heated at 190°F for 16 hours	
Viscosities measured at 95°F and calculated at 5×10^{-2} sec^{-1}	
85-100 Penetration	
6	2.9
11	4.7
8	5.2
3	5.8
1	7.7
120-150 Penetration	
11A	3.3
6A	3.8
3A	4.2
1A	5.5

3. Oxidation

Oxidation in the absence of sunlight probably is the effect responsible for the greatest hardening of an asphalt during the manufacture and service life of a bituminous pavement.

Much of the information given in Progress Report No. 1 concerned the oxidation of asphalt films at given temperatures for various periods of time. The temperatures used varied from 225° to 350°F; times ranged from 15 minutes to 164 hours. Recent work has dealt with the testing of selected asphalts at additional times and temperatures. An effort has been made to cover ranges of temperature and time of practical interest to the paving technologist. Some of the older data have been combined with the newer in order to give a complete picture of the effect of oxidation.

Table A-1, of the Appendix, gives extensive oxidation data on five 85-100 penetration asphalts for times ranging from 15 minutes to 32 hours and temperatures from 190 to 350° F. Table A-2, in the Appendix, gives oxidation data on four representative 120-150 penetration asphalts for times ranging from one to 32 hours and temperatures from 190 to 300°F.

Table 1, facing, gives Relative Viscosities on the five 85-100 and four 120-150 penetration asphalts obtained by heating 15-micron films at 225° F for 2 hours and at 190°F for 16 hours. In some cases the 85-100 penetration asphalt from a particular producer is more susceptible to oxidation than is the softer asphalt. For other asphalt the reverse is true. These effects probably result from variations in composition of the asphalts because of differences in source and processing.

4. Photooxidation

Oxidation in the presence of sunlight (photooxidation) is much more severe than that which occurs in the dark. However, the short wave lengths of sunlight penetrate into asphalt only to a depth of about 3 microns. Although the effect is very severe on the films of asphalt exposed at the surface of a pavement it probably does not cause much hardening and deterioration of the pavement mass. Photooxidation is mainly responsible for hardening and subsequent erosion of the exposed surface films.

Experiments first were conducted at 190°F for 16 hours. Viscosity measurements of the hardened asphalts were made at 77°F. The treated asphalts were very hard and possessed so much non-Newtonian flow that the data obtained in the microfilm viscometer were not very accurate.

It was decided to conduct a second series of experiments at 190°F but for shorter periods of time. Also, in this series the viscosities were measured at 95°F to eliminate some of the rheological problems encountered in the hardened samples. Table A-3 in the Appendix gives data on eleven 85-100 penetration asphalts heated at 190°F for 1, 2, and 3 hours. Viscosities were measured at 95°F and calculated at 5×10^{-2} sec^{-1} and at a power input of 1000 $ergs/sec/cm^3$. Table A-4 gives similar data on five 120-150 penetration asphalts photooxidized at 190°F for 2 hours.

TABLE 2

Photooxidation of 15-Micron Films
at 190°F for 2 hours

Viscosities measured at 95°F and calculated at 5×10^{-2} sec^{-1}

Asphalt No.	Relative Viscosity
85-100 Penetration Asphalts	
6	3.1
5	3.7
3	6.3
11	6.9
8	7.2
9	7.4
4	8.0
10	8.0
2	10.0
7	11.7
1	12.3
120-150 Penetration Asphalts	
6A	3.9
11A	5.3
3A	6.0
1A	11.2

Table 2, facing, compares eleven 85-100 penetration asphalts subjected to photooxidation at 190°F for 2 hours and the same information on four 120-150 asphalts. The reversal in relative susceptibility of the two grades of asphalt from sources 3 and 11 results from differences in composition caused by source and processing.

TABLE 3

Volatilization

15-Micron Films of Various Grades of
Asphalt Heated in Nitrogen at 225°F for 4 Hours

Viscosities measured at 77°F and calculated at 5×10^{-2} sec.$^{-1}$

Asphalt No.	Relative Viscosity
85-100 Penetration Grade	
6	1.3
11	2.8
1	3.8
120-150 Penetration Grade	
6A	2.3
11A	3.0
1A	3.9
150-200 Penetration Grade	
6B	2.85
11B	3.9
1B	5.3

5. Volatilization

Several decades ago the assumption was made that paving asphalts hardened chiefly by evaporation of the lighter oils, but this concept has been refuted by experiments conducted during the past 30 years. In the current investigation numerous experiments were conducted to determine the importance of volatilization in the hardening of thin films of paving asphalts. The procedure was essentially that used for evaluating the effect of oxidation in the dark except that nitrogen was substituted for air as the atmosphere in contact with the asphalt.

Table A-6 of Progress Report No. 1 gives data on the relative viscosities of eleven 85-100 penetration, five 120-150 penetration and nine 150-200 penetration asphalt heated in nitrogen at 225°F for 4 hours. As would be expected volatilization becomes a more important factor with the softer asphalts. This is further illustrated by the data shown in Table 3, facing.

More recently, the effect of volatilization has been evaluated at higher temperatures (275 and 300° F) for shorter periods of time (15, 30, and 60 minutes). The data are somewhat erratic probably because of the difficulties encountered in quickly attaining temperature equilibrium in the nitrogen system. Data are given in Table A-5 of the Appendix. The hardening developed at 275°F is shown in Table 4, following. Some of the hardening is caused by polymerization, mentioned in section 7 below.

Finally, hardening caused by heating 15 micron films in nitrogen for 16 hours at 190°F was determined on five representative 85-100 penetration and four 120-150 penetration asphalts. Data are shown in Table 5, following. Discrepancy in the relative viscosities of hard and soft asphalts from the same source may be due to experimental error in the test runs or by differences in source or processing.

TABLE 4

Volatilization

15-Micron Films of 85-100 Penetration
Asphalts Heated in Nitrogen at 275°F for 15, 30, & 60 minutes

Viscosities measured at 77°F and calculated at 5×10^{-2} sec^{-1}

Time Heated (Minutes)	Relative Viscosity
Asphalt No. 1	
15	1.5
30	2.0
60	2.7
Asphalt No. 3	
15	1.7
30	1.75
60	1.85
Asphalt No. 6	
15	1.4
30	1.6
60	1.7

TABLE 5

Volatilization

15-Micron Films Heated in Nitrogen at 190°F for 16 Hours

Viscosities measured at 95°F and calculated at 1000 ergs/sec/cm^3

Asphalt	Relative Viscosity
85-100 Penetration Asphalts	
11	1.4
3	1.6
6	1.75
8	2.1
1	2.6
120-150 Penetration Asphalts	
6A	1.6
11A	1.9
3A	2.5
1A	3.0

TABLE 6

Photochemical Action

Effect of Film Thickness on Hardening of 85-100 Penetration Asphalts Exposed Between Glass Plates at 190°F for 18 Hours

Viscosities measured at 77°F and calculated at 5×10^{-2} sec^{-1}

Asphalt No.	Film Thickness, Microns		
	26	13.5	6.5
	Relative Viscosity		
1	1.4	1.9	2.9
3	1.5	2.2	4.6
6	1.2	1.2	1.9

TABLE 7

Photochemical Action

3 Micron Films of 85-100 and 120-150 Penetration Asphalts Exposed Between Glass Plates at 190°F for 16 Hours

Viscosities measured at 95°F and calculated at 1000 $ergs/sec/cm^3$

Asphalt No.	Relative Viscosity
85-100 Pen. Asphalts	
6	3.2
1	3.5
11	3.6
8	4.0
3	5.2
120-150 Pen. Asphalts	
3A	2.8
6A	3.8
11A	6.2
1A	8.0

6. Photochemical Action

Because of the rapid oxidation of asphalt which occurs in the presence of sunlight it was thought advisable to determine the effect of light in the absence of oxygen. The experiments were conducted by placing films of the asphalt between 4 x 4 cm plates of Pyrex 7740 glass which transmits 90 percent of the short wavelengths of light. The assembly was exposed to the radiation from a 175-watt Mazda Sunlamp. Distance between lamp and asphalt film was eleven inches.

Actinic light (short wavelengths) penetrates a very short distance (probably about 3 microns) into a film of asphalt. Thus, it was necessary to determine the effect of film thickness on the change that would occur in the asphalt. Films 26, 13, and 6.5 micron thick were exposed to radiation at 190°F for 18 hours. Viscosities were measured at 77°F and calculated at 5×10^{-2} sec^{-1}. Table 6, facing, indicates that the relatively thick films did not harden appreciably under this environment.

Later it was decided to evaluate five 85-100 penetration asphalts and four 120-150 penetration cements by conducting tests on 3 micron films at 190°F for 16 hours (a convenient time interval). To reduce the amount of non-Newtonian flow, viscosities were measured at 95°F. Calculations were made at a power input of 1000 $ergs/sec/cm^3$. Data are shown in Table 7, facing. The relative order of hardening of the two grades is different probably because of differences in source and processing of the hard and soft asphalts.

7. Polymerization

Slight hardening occurs when asphalts are held at elevated temperatures under conditions where oxidation, photooxidation, volatilization or photochemical action cannot occur. This has been called condensation polymerization. Some data are given in Table 3 of Progress Report No. 1. Three representative 85-100 penetration asphalt were used in those experiments, which were conducted for 4 hours at temperatures ranging from 225 to 375°F. It was found that all three asphalts hardened to some extent. But between 325°F and 350°F asphalt No. 1 showed depolymerization or cracking which resulted in a decrease in viscosity and relative viscosity. Depolymerization occurred in Asphalt No. 3 between 350 and 375°F. Polymerization was moderate in Asphalt No. 6 and no polymerization occurred at temperatures up to 375°F.

Since the publication of Progress Report No. 1, a series of runs have been made using eight 85-100 penetration asphalts. Temperature was maintained at 190°F and the test was continued for 18 hours. Viscosities were measured at 77°F and calculated at 1000 ergs/sec/cm^3. At 190°F the relative viscosities of the hardened film were quite low in spite of the long period of time. Data are shown in Table A-6, of the Appendix.

Finally, runs were made on five 85-100 penetration asphalts and on four 120-150 penetration asphalts at 190°F for 16 hours. In this case viscosities were measured at 95°F and calculated at 1000ergs/sec/cm^3.

TABLE 8

Polymerization of 85-100 and 120-150 Penetration Asphalts - 15 Micron Films Heated in the Dark Between Glass Plates at 190°F for 16 Hours

Viscosities measured at 95°F and Calculated at 1000 ergs/sec/cm^3

Asphalt No.	Relative Viscosity
85-100 Pen. Asphalts	
6	1.4
11	1.5
3	1.5
8	1.6
1	1.7
120-150 Pen. Asphalts	
6A	1.1
11A	1.2
1A	1.9
3A	1.95

Table 8, facing, gives the values obtained. Polymerization is a rather insignificant factor in hardening unless the asphalt is subjected to moderately high temperatures for a considerable period of time. Some asphalts will depolymerize if the temperature is excessive.

TABLE 9

Effect of Environment on Relative Viscosities - 15 Micron Films Treated at 190°F for 16 Hours

Viscosities measured at 95°F and calculated at 1000 ergs/sec/cm^3

Asphalt No.	Photooxidation(1)	Oxidation	Photochemical(2)	Volatilization	Polymerization
			85-100 Penetration Asphalt		
6	3.4	3.2	3.2	1.75	1.4
11	8.3	5.8	3.6	1.4	1.5
8	8.4	6.6	4.0	2.1	1.6
3	11.6	7.0	5.2	1.6	1.5
1	13.8	9.5	3.5	2.6	1.7
			120-150 Penetration Asphalt		
6A	4.0	3.9	3.8	1.6	1.1
11A	6.2	3.5	6.2	1.9	1.2
3A	7.7	5.0	2.8	2.5	1.95
1A	14.5	7.0	8.0	3.0	1.9

(1) Two hours instead of 16 hours.

(2) 3 Microns instead of 15 microns.

8. Comparison of Five Environmental Effects on the Hardening of Asphalt Cements

It is difficult to compare the effects of different environments on asphalt because of the great difference in hardening resulting from experiments conducted at a given time and temperature. The use of long times and high temperatures is required to obtain substantial values for polymerization and photochemical action, but such conditions in photooxidation tests result in asphalts so hard that measurement of the viscosity is difficult and not very accurate.

Data have been collected from the tables shown in the preceding pages where the temperature of test was 190°F and the time was 16 hours except for photooxidation where the time was reduced to 2 hours. In all cases the film used was 15 microns thick except for photochemical action where it was reduced to 3 microns. The asphalts in Table 9 are arranged in order of increasing susceptibility to photooxidation. The same order is not always maintained for the other environments. For some of the tests the effects become quite involved. For example, it must be realized that the hardening by photooxidation includes also the effects due to polymerization, volatilization and photochemical action. Further, the oxidation test data include hardening caused by polymerization and volatilization. Finally, volatilization and photochemical test data include hardening caused by polymerization.

It can be concluded that, for most of the asphalts studied, the environments causing hardening can be arranged in the following order of decreasing effectiveness: (1) photooxidation, (2) oxidation, (3) photochemical, (4) volatilization, and (5) polymerization.

9. Relative Viscosities after 24 to 27 Months in Service

The asphalts discussed in Progress Report No. 1 and in the present report were collected in the field by Texas Highway Department personnel at the site of pavements laid in 1959. About two years later a number of the pavements were inspected. Samples were removed and brought to the Texas Transportation Institute Laboratories where the asphalt was extracted from the pavement sample using the Colorado Highway Department apparatus. The asphalt was recovered by the Abson procedure. Viscosities of the asphalts were measured in the microfilm viscometer at 77°F and calculated at 5×10^{-2} sec^{-1}. Relative Viscosities of the extracted asphalts were calculated.

Although at first glance the data obtained seem to be confusing and not particularly enlightening, careful examination of the available information reveals conditions that may account for some of the unexpected Relative Viscosities obtained.

Since the correlation between laboratory data and field serviceability is important, further work is being initiated. Samples were collected (summer of 1962) from the field and sent to TTI for laboratory testing. When failure occurs in the pavement made from a particular asphalt, the field information will be compared with tests made on the retained asphalt sample.

TABLE 10

Relative Viscosities of 85-100 Penetration
Asphalts Recovered After 24.25 to 27.5 Months in Pavement

THD Dist.	Project & Location	Months in Service	Condition	Asphalt No.	Viscosity @ 77°F (Megapoises) Orig.	Recov'd	Relative Viscosity
8	DF 581 (15), U. S. 83 Taylor County, 1 mile S of Jones County Line to Freeway at Kirby Lake.	27.0	Hot-mix, excellent condition, heavy city traffic 15% trucks.	7	0.8	6	7.5
2	351-2-7, U. S. 81, Wise County, 1 mile N. of Alvard past Dry Creek.	24.25	1-1/2" hot-mix. Surface contained many large patches. Failure caused by stripping within 10 days after construction. Dirty aggregate, limited compaction, cool rainy weather.	10	1.0	13	13
12	C-111-4-9, 288, Brazoria County.	27.5	Hot-mix over concrete. Wire mesh reinforcement in the bituminous layer. Thickness 3-1/2 to 5". Surface in good condition.	6	0.7	10	14
12	C-389-5, 6, 16 & 23, State 146. Galveston County 100' east of intersection with State 85.	27.5	Shell gravel mix in good condition.	6	0.7	16	23
12	C-367-2-22, C-367-3-14, & C-307-5-8. U.S. 87, Galveston County, Bolivar Island ferry N.E. of landing.	27.25	Surface in fairly good condition. Highly absorptive aggregate & high surface area.	11	1.3	32	25
					Average		16.5

(a) 85-100 Penetration Grade Asphalts

Table 10, facing, shows the locations, time in service, condition of pavement when inspected, source of the asphalt, viscosities and Relative Viscosities of asphalts extracted from five pavements prepared with four different 90 penetration asphalts.

The following comments throw some light on the data presented.

Asphalt 7, which by laboratory tests is highly susceptible to oxidation, shows a Relative Viscosity of only 7.5 on the extracted asphalt. This low value probably resulted because the mixture was dense, and air could not reach the asphalt films.

Asphalt 10, which is moderately susceptible to hardening by laboratory tests, shows a Relative Viscosity of 13. Poor compaction, permitting air to reach the asphalt films in the pavement probably is the cause for this moderately high R.V.

Asphalt 6, which by laboratory tests is highly resistant to oxidation, shows R.V. values of 14 and 23 for the extracted asphalt. Causes for these high values are not evident.

Asphalt 11, which by laboratory tests is moderately resistant to oxidation, has a Relative Viscosity of 25 for the extracted binder. Highly absorbtive aggregate with a large surface area may be responsible for the excessive hardening during service. Large amounts of oil may have been removed from the asphalt by the stone.

Thus, some explanation may be offered for the results obtained except for those on Asphalt No. 6.

Hardening in the laboratory by oxidizing 15-micron films at 225°F for 2 hours in the dark as follows:

Asphalt No.	Relative Viscosity
7	6.2
10	3.3
6	2.1
11	3.3

TABLE 11

Relative Viscosities of 120-150 Penetration
Asphalts Recovered After 26.5 to 27.5 Months in Pavement

THD Dist.	Project & Location	Months in Service	Condition	Asphalt No.	Viscosity @ 77°F (Megapoises) Orig.	Recov'd	Relative Viscosity
5	294-2-8, U.S. 180 Gaines County, from FM 1312 to Dawson County Line.	27.25	Hot-mix, base was preserved with RC-2 prior to hot-mix overlay. Pavement flushed in numerous areas due to RC-2.	1A	0.59	3.5	6
11	C-694-1-6, FM 83, Sabine County. Pineland to Hamphill. 2.7 miles east of Highway 6.	26.5	Surface good. Small amount of patches at edges. Films thin and difficult to recover.	11A	0.49	13	26.5
11	694-2-6, FM 83(944) Sabine County. S.E. of Hemphill 7 miles E. of Highway 6.	26.5	Surface fairly good. Subsurface moisture weakened base.	11A	0.49	14	28.5
17	675-6-1, 675-7-1, IH 45, Walker County, loop around Huntsville.	27.5	Sandstone base single surface treatment. Poor bond. Embedment 39%. No traffic. Grass growing in surface near edge.	6A	0.36	11	30.5
9	R-2306-2-1, FM 2412 Coryell County.	26.75	Crushed limestone base. Seal coated, surface in good condition.	6A	0.36	19	53
8	2372-1-1, FM 2412, Fisher County, 10 miles N.E. of Roby.	27.0	A single with pea gravel with wide size distribution. Embedment 85-90% fairly heavy prime. Medium heavy density base of caliche. Surface good.	7A	0.40	29	72.5
						Average	36

(b) 120-150 Penetration Grade Asphalts

Table 11, facing, gives information available on this softer grade.

Asphalt 1A, which is highly susceptible to hardening by laboratory tests, shows a Relative Viscosity of only 6 for the extracted asphalt. This surely results from fluxing of the binder by the RC-2 used as primer. Flushing of the asphalt occurred in the pavement.

Asphalt 11A, which is resistant to hardening by laboratory oxidation tests, shows Relative Viscosities of 26.5 and 28.5 for the extracted cement. There appears to be no particular aspect of the pavement that could account for the excessive hardening of the asphalt in the pavements.

Asphalt 6A is resistant to hardening in the laboratory tests but shows Relative Viscosities of 30.5 and 53 for the extracted material. Probably low traffic resulted in poor compaction which accounted for the value of 30.5. Crushed limestone could have absorbed enough oil from the asphalt to materially harden the films yielding the R.V. of 53.

Asphalt 7A is very susceptible to hardening by the laboratory tests. The caliche may also have absorbed oil from the films resulting in the very high value of 72 for the extracted asphalt.

Hardening in the laboratory by oxidizing 15-micron films at 225°F for 2 hours in the dark are as follows:

Asphalt No.	Relative Viscosity
1A	4.2
11A	2.6
6A	2.5
7A	4.8

TABLE 12

Relative Viscosities of 150-200 Penetration
Asphalts Recovered After 24.25 to 27.25 Months in Pavement

THD Dist.	Project & Location	Mos. in Service	Condition	Asphalt No.	Viscosity @ 77° F (Megapoises) Orig.	Recov'd	Relative Viscosity
4	C-791-5-6, FM 278 Hanford Co., 5 miles N. of Hutchinson Co. Line.	24.25	Essentially free of distress. Medium traffic, wheel path embedment - 80% to -50%. Silicious gravel.	8B	0.21	2.1	10.0
4	R-2335-1-4, FM 2387 Hanford Co., 1.5 miles on FM 2387 from point where FM 1261 goes North.	24.25	Low traffic road. Embedment 50%. Some edge repairs, Occasional patch on outside 2' of road. Narrow shoulder.	8B	0.21	2.9	13.8
3	903-18-1, FM 171 Wichita Co., on line C (not on State Sys.) Co. Road.	26.25	Surface good - low traffic. About 60% embedment.	10B	0.27	3.7	13.5
3	681-1-5, FM 171 Wichita Co., 6 miles N.E. of Wichita Falls.	26.25	Gravel base, limestone cover aggregate. Embedment 90 - 95%. Surface good-no patches.	10B	0.27	14	52
3	903-17-1, FM 171 Wichita Co. on Line B (not on State Sys.) Co. Road.	26.25	Surface good - low traffic. 60-70% embedment.	10B	0.27	6.4	24
23	DF 194 (10) U.S. 84, Coleman Co., 4.5 miles E. of Santa Anna.	27.0	Shoulders only. Limestone caliche base. Single treatment. Embedment 40%. Asphalt dead & surface dry.	7B	0.3	4.6	15.5
23	DF 303 (7) U.S. 67, Coleman Co., 2 miles W. of Santa Anna.	27.0	Shoulders only. Crushed limestone base. Embedment 30%. Lt. prime. Condition fair.	7B	0.3	10	33
5	380-1-16, U.S. 62 Lubbock Co., 1/2 milesSW City of Lubbock.	27.25	A triple surface treatment with appl. of asph. Embedment 90%. Surface good.	1B	0.3	14	47
						Average	26

(c) 150-200 Penetration Grade Asphalts

Table 12, facing, shows available data on these vary soft asphalts.

Asphalt 8B, is fairly resistant to oxidation in the laboratory tests. The Relative Viscosities of 10 and 13.8 on the extracted asphalt seem justified.

Asphalt 10B shows, in the laboratory, about the same susceptibility to hardening as Asphalt 8B so the R.V. value of 13.5 for the extracted material appears to be reasonable. But the values of 52 and 24 cannot be explained except that the limestone cover may have absorbed oil from the asphalt to give the R.V. value of 52.

Asphalt 7B, is one of the asphalts most susceptible to oxidation as measured in the laboratory. No plausible explanation can be offered for the difference in the 15.5 and 33 R.V. values obtained on the extracted binders.

Asphalt 1B, shows the same behavior in the laboratory tests as does Asphalt 7B. No explanation can be offered for the 52 Relative Viscosity obtained on the extracted asphalt.

Hardening in the laboratory by oxidizing 15-micron films at 225°F for 2 hours in the dark are as follows:

Asphalt No.	Relative Viscosity
8B	3.7
10B	3.4
7B	4.7
1B	4.7

The general conclusion can be drawn that the 135 and 175 penetration asphalts show greater relative increases in viscosity during service than do the 90 penetration cements.

TABLE 13

Progressive Increase in Relative Viscosity of Asphalt No. 9

Sample	Pen. @ 77/100/5	Viscosity (Stokes) @ 275°F	Viscosity (Poises)(2) @ 77°F	Relative Viscosity
Original Asphalt(1) Temp. 300°F.	86	2.3	1.0×10^6	----
Recovered from mix leaving hot-mix plant. Temp. 285°F.	72	2.4	1.4×10^6	1.4
Recovered at laydown machine. 3 1/2 hrs. after leaving plant. Temp. 265°F.	68	2.6	1.9×10^6	1.9
Recovered from sample taken from road 15 days after pavement was laid. Very hot weather.	48	3.0	3.8×10^6	3.8
Original asphalt. 15-micron film subjected to oxidation for 2 hrs. at 225°F.	--	---	4.0×10^6	4.0

(1) Taken immediately prior to entering hot-mix plant.
(2) Calculated at 1,000 $ergs/sec/cm^3$.

TABLE 14

Increase in Viscosity of Asphalt No. 13 from Entrance into Hot Mix Plant to Lay Down Machine

Sample	Pen. @ 77/100/5	Viscosity (Stokes) @275°F	Viscosity (Poises)(1) @ 77°F	Relative Viscosity
Original Asphalt	81	5.7	1.6×10^6	---
Recovered from mix at Laydown machine Temp. 290°F.	44	9.0	8.85×10^6	5.5

(1) Calculated at 1000 $ergs/sec/cm^3$.

10. Increase of Relative Viscosity During Handling

When a molten asphalt flows to a hot mix machine and is spread as thin films over the surface of hot mineral (aggregate), in the presence of an oxidizing atmosphere (air), oxidation and volatilization occur. Further, during transport of the hot mixture to the paving site, additional hardening can and often does take place. In the process of transferring the hot mixture to the lay-down machine and during the spreading and compaction operation there is still more opportunity for oxidation and volatilization. Finally, after the pavement is opened to traffic, unless it is well compacted, additional oxidation of the asphalt films occurs at the surface and down into the mass.

This progressive hardening is illustrated in Table 13, facing. These data were obtained during construction on U.S. 77 from Rockdale, Texas to the Lee County Line. Asphalt used was 85-100 penetration grade from producer No. 9. The work was started on August 7, 1962 during a period of hot weather.

Table 14 shows the increase in hardness which occurred in an 85-100 grade asphalt from the time it passed into an hot mix plant until the mixture was laid on North U.S. 281 in Hidalgo County. The aggregate used was gravel and caliche. Asphalt was supplied by Producer No. 13. Date of laying was July 25, 1962 during extremely warm weather. During this constructional operation the viscosity of the asphalt increased 5.5 fold.

11. Evaluation of Durability

Straight oxidation (in absence of sunlight) at a relatively high temperature (225° F) for a short period of time (2 hours) serves as a quality test for asphalts.

Heating of a 5 micron film at 225° F for 2 hours has been selected by ASTM as the basis for their "Tentative Test for Aging Index of Bituminous Materials" (65th Annual Meeting, New York, June 24-29, 1962). The procedure is given in the Appendix. We believe that a 15 micron film more nearly approaches the geometry of the films in a pavement than does 5 microns. Also, the use of the thicker film reduces the problems encountered in preparing and testing the asphalt samples.

VII. APPENDIX

A - Tables

INDEX

TABLE A-1

Hardening of 15-Micron Films by Oxidation in the Dark

Asphalt No. 1 (85-100 Pen.)

Original Viscosities of Asphalt No. 1

1.1×10^6 poises at 77°F
1.3×10^5 poises at 95°F

Calculated at 5×10^{-2} sec.$^{-1}$

Temperature °F	Viscosity at 77°F (Megapoises)	Relative Viscosity
	Heated for 15 Minutes	
250	2.3	2.1
275	3.5	3.2
300	5.5	5.0
325	9.0	8.2
350	14.0	12.7
	Heated for 30 Minutes	
250	4.0	3.6
275	6.6	6.0
300	12.0	10.9
325	22.0	20.0
350	50.0	45.0
	Heated for 1 Hour	
200	0.26*	2.0
225	0.34*	2.6
250	7.2	6.5
275	12.5	11.4
300	21.0	19.1
325	43.0	39.0
	Heated for 2 Hours	
225	6.6	6.0
250	12.0	10.9
275	22.0	20.0
300	44.0	40.0

*Viscosities were measured at 95°F.

Temperature °F	Viscosity at 77° F (Megapoises)	Relative Viscosity
	Heated for 4 Hours	
225	12.0	10.9
	Heated for 16 Hours	
190	1.0*	7.7
225	30.0	27.0
	Heated for 32 Hours	
225	42.0	37.0

*Viscosities were measured at 95° F.

TABLE A-1 (Cont. 1)

Hardening of 15-Micron Films by Oxidation in the Dark

Asphalt No. 3 (85-100 Pen.)

Original Viscosities of Asphalt No. 3

1.2×10^6 poises at 77°F
1.9×10^5 poises at 95°F

Calculated at 5×10^{-2} sec.$^{-1}$

Temperature °F	Viscosity at 77° F (Megapoises)	Relative Viscosity
	Heated for 15 Minutes	
275	3.0	2.5
300	4.8	4.0
325	8.0	6.6
350	11.0	9.2
	Heated for 30 Minutes	
275	4.4	3.7
300	9.0	7.5
325	15.0	12.5
350	37.0	31.0
	Heated for 1 Hour	
275	8.6	7.2
300	16.0	13.3
325	30.0	25.0
	Heated for 2 Hours	
225	5.2	4.3
250	9.0	7.5
275	15.5	12.9
300	35.0	29.0
	Heated for 4 Hours	
225	9.0	7.5
	Heated for 16 Hours	
190	1.1*	5.8

*Viscosities were measured at 95° F.

TABLE A-1 (Cont. 2)

Hardening of 15-Micron Films by Oxidation in the Dark

Asphalt No. 6 (85-100 Pen.)

Original Viscosities of Asphalt No. 6

0.7×10^6 poises at 77°F
8.4×10^4 poises at 95°F

Calculated at 5×10^{-2} sec.$^{-1}$

Temperature °F	Viscosity at 77° F (Megapoises)	Relative Viscosity
	Heated for 15 Minutes	
275	1.3	1.85
300	1.3	1.85
325	1.9	2.7
350	2.7	3.9
	Heated for 30 Minutes	
275	1.5	2.2
300	2.3	3.3
325	4.3	6.1
350	12.0	17.2
	Heated for 1 Hour	
200	0.12*	1.4
225	0.15*	1.8
250	0.16*	1.9
275	2.3	3.3
300	3.5	5.0
325	10.1	14.4
	Heated for 2 Hours	
225	1.5	2.1
250	2.2	3.1
275	4.2	6.0
300	6.6	9.5
	Heated for 4 Hours	
225	2.1	3.0
	Heated for 16 Hours	
190	0.24*	2.9
225	4.4	6.3
	Heated for 32 Hours	
225	7.2	10.3

*Viscosities were measured at 95° F.

TABLE A-1 (Cont. 3)

Hardening of 15-Micron Films by Oxidation in the Dark

Asphalt No. 8 (85-100 Pen.)

Original Viscosity of Asphalt No. 8

0.9×10^6 poises at 77°F
1.0×10^5 poises at 95°F

Calculated at 5×10^{-2} sec.$^{-1}$

Temperature °F	Viscosity at 77°F (Megapoises)	Relative Viscosity
	Heated for 30 Minutes	
275	2.45	2.7
300	3.6	4.0
	Heated for 2 Hours	
225	3.6	4.0
	Heated for 4 Hours	
225	6.6	7.3
	Heated for 16 Hours	
190	0.52*	5.2

*Viscosities were measured at 95°F.

TABLE A-1 (Cont. 4)

Hardening of 15-Micron Films by Oxidation in the Dark

Asphalt No. 11 (85-100 Pen.)

Original Viscosity of Asphalt No. 11

1.3×10^6 poises at 77°F
1.4×10^5 poises at 95°F

Calculated at 5×10^{-2} sec.$^{-1}$

Temperature °F	Viscosity at 77°F (Megapoises)	Relative Viscosity
	Heated for 1 Hour	
200	0.24*	1.7
225	0.36*	2.6
250	0.42*	3.0
275	0.70*	5.0
300	1.50*	10.7
	Heated for 2 Hours	
225	4.3	3.3
	Heated for 4 Hours	
225	6.8	5.5
	Heated for 16 Hours	
190	0.66*	4.7

*Viscosities were measured at 95°F.

TABLE A-2

Hardening of 15-Micron Films by Oxidation in the Dark

Asphalt 1A (120-150 Pen.)

Original Viscosity of Asphalt No. 1A

0.59×10^6 poises at 77°F
6.6×10^4 poises at 95°F

Calculated a 5×10^{-2} sec.$^{-1}$

Temperature °F	Viscosity at 77°F (Megapoises)	Relative Viscosity
	Heated for 1 Hour	
275	4.4	8.8
300	10.0	20.0
	Heated for 2 Hours	
225	2.5	4.2
	Heated for 4 Hours	
225	4.3	7.4
	Heated for 16 Hours	
190	0.36*	5.5
225	13	22
	Heated for 32 Hours	
225	31	52

*Viscosities measured at 95°F.

TABLE A-2 (Cont. 1)

Hardening of 15-Micron Films by Oxidation in the Dark

Asphalt No. 3A (120-150 Pen.)

Original Viscosity of Asphalt No. 3A

0.54×10^6 poises at 77° F
1.0×10^5 poises at 95°F

Calculated at 5×10^{-2} sec.$^{-1}$

Temperature °F	Viscosity at 77° F (Megapoises)	Relative Viscosity
	Heated for 1 Hour	
275	3.5	6.3
300	7.6	13.5
	Heated for 2 Hours	
225	2.2	4.0
	Heated for 4 Hours	
225	3.8	7.0
	Heated for 16 Hours	
190	0.42*	4.2

*Viscosities determined at 95° F.

TABLE A-2 (Cont. 2)

Hardening of 15-Micron Films by Oxidation in the Dark

Asphalt No. 6A (120-150 Pen.)

Original Viscosity of Asphalt No. 6A

0.36×10^6 poises at 77°F
5×10^4 poises at 95°F

Calculated at 5×10^{-2} sec.$^{-1}$

Temperature °F	Viscosity at 77°F (Megapoises)	Relative Viscosity
	Heated for 1 Hour	
275	1.35	3.2
300	2.6	6.2
	Heated for 2 Hours	
225	0.9	2.5
	Heated for 4 Hours	
225	1.3	3.6
	Heated for 16 Hours	
190	0.19*	3.8
225	4.0	11
	Heated for 32 Hours	
225	7.8	22

*Viscosities measured at 95°F.

TABLE A-2 (Cont. 3)

Hardening of 15-Micron Films by Oxidation in the Dark

Asphalt No. 11A (120-150 Pen.)

Original Viscosity of Asphalt No. 11A

0.49×10^6 poises at 77°F
9×10^4 poises at 95°F

Calculated at 5×10^{-2} sec.$^{-1}$

Temperature °F	Viscosity at 77°F (Megapoises)	Relative Viscosity
	Heated for 2 Hours	
225	1.3	2.6
	Heated for 4 Hours	
225	2.1	4.3
	Heated for 16 Hours	
190	0.30*	3.3

*Viscosities measured at 95°F.

TABLE A-3

Hardening of 15-Micron Films of 85-100 Penetration Asphalts by Photooxidation at 190°F

Viscosities (poises) at 95°F
Calculated at 5×10^{-2} sec. $^{-1}$

Asphalt No.	Original	Hardened	Relative Viscosity
	Heated for 1 Hour		
6	8.4×10^4	2.0×10^5	2.4
11	1.4×10^5	4.0×10^5	2.9
1	1.3×10^5	8.2×10^5	6.3
	Heated for 2 Hours		
6	8.4×10^4	2.6×10^5	3.1
5	1.1×10^5	4.0×10^5	3.7
4	1.0×10^5	8.0×10^5	8.0
11	1.4×10^5	9.6×10^5	6.9
10	1.25×10^5	1.0×10^6	8.0
9	1.25×10^5	9.2×10^5	7.4
8	1.0×10^5	7.2×10^5	7.2
3	1.9×10^5	1.2×10^6	6.3
2	1.3×10^5	1.3×10^6	10.0
7	8.4×10^4	9.8×10^5	11.7
1	1.3×10^5	1.6×10^6	12.3
	Heated for 3 Hours		
6	8.4×10^4	4.4×10^5	5.2
11	1.4×10^5	1.5×10^6	10.7
1	1.3×10^5	4.0×10^6	30.8

TABLE A-3 (Cont.)

Hardening of 15-Micron Films of 85-100 Penetration Asphalts by Photooxidation at 190°F

Viscosities (poises) at 95°F
Calculated at 1000 ergs/sec/cm^3

Asphalt No.	Original	Hardened	Relative Viscosity
		Heated for 1 Hour	
6	7.1×10^4	1.9×10^5	2.7
11	1.2×10^5	4.0×10^5	3.3
1	1.15×10^5	8.5×10^5	7.4
		Heated for 2 Hours	
6	7.1×10^4	2.4×10^5	3.4
5	8.9×10^4	4.0×10^5	4.5
4	8.2×10^4	8.3×10^5	10.1
11	1.2×10^5	1.0×10^6	8.3
10	1.13×10^5	1.14×10^6	10.1
9	1.3×10^5	9.7×10^5	7.5
8	8.2×10^4	6.85×10^5	8.4
3	1.7×10^5	1.45×10^6	8.5
2	1.13×10^5	1.4×10^6	12.4
7	7.1×10^4	9.7×10^5	13.7
1	1.15×10^5	1.6×10^6	13.9
		Heated for 3 Hours	
6	7.1×10^4	4.4×10^5	6.2
11	1.2×10^5	1.9×10^6	15.8
1	1.15×10^5	3.4×10^6	29.6

TABLE A-4

Hardening of 15-Micron Films of 120-150 Penetration Asphalts by Photooxidation at 190°F

Heated for 2 Hours

Asphalt No.	Original	Hardened	Relative Viscosity
	Viscosities (poises) at 95°F Calculated at 5×10^{-2} sec.$^{-1}$		
6A	5.0×10^4	1.95×10^5	3.9
11A	9.0×10^4	4.8×10^5	5.3
3A	1.0×10^5	6.0×10^5	6.0
1A	6.6×10^4	7.4×10^5	11.2
	Viscosities (poises) at 95°F Calculated at 1000 ergs/sec/cm^3		
6A	4.45×10^4	1.8×10^5	4.0
11A	7.9×10^4	4.9×10^5	6.2
3A	8.1×10^4	6.3×10^5	7.8
1A	5.0×10^4	7.3×10^5	14.6

TABLE A-5

Volatilization of 85-100 Penetration Asphalts
15-Micron Films Heated in Nitrogen

Viscosities Measured at 77°F and Calculated at 5×10^{-2} sec^{-1}

Temp. °F	Time (Minutes)	Relative Viscosity of Asphalts No. 1	No. 3	No. 6
275	15	1.5	1.7	1.4
275	30	2.0	1.75	1.6
275	60	2.7	1.85	1.7*
300	15	1.8	1.5*	1.4
300	30	2.2	1.9	1.4*
300	60	2.7*	2.2	1.4*

*These values appear to be low.

TABLE A-6

Polymerization of 85-100 Penetration Asphalts
15-Micron Films Heated Between Glass Plates
at 190°F for 18 Hours

Viscosities measured at 77°F and Calculated at 1000 ergs/sec/cm^3

Asphalt No.	Viscosity (Megapoises) Original	Viscosity (Megapoises) Heated	Relative Viscosity
6	0.7	0.8	1.15
4	0.8	1.0	1.25
8	1.0	1.4	1.40
11	1.2	1.8	1.50
10	1.0	1.6	1.60
3	1.2	1.9	1.60
1	1.1	1.8	1.80
7	0.9	1.8	2.00

B. PROPOSED METHOD OF TEST FOR AGING INDEX OF BITUMINOUS MATERIALS[1,2]

Scope

1. This method[3] describes a procedure for the measurement of the increase in viscosity which occurs when a semi-solid bituminous material is heated in a thin film under the conditions specified.

Summary of Method

2. A film of bituminous material 5μ in thickness is heated in air for 2 hours at 225°F. The viscosity of the original material and of the sample after heating are measured at 77°F using a sliding-plate microviscometer.

Definition

3. Aging Index of a Bituminous Material is the ratio of its viscosity at 77°F after heating under specified conditions to its original viscosity at 77°F.

Apparatus

4. (a) Microviscometer, sliding-plate type, Shell Development Co. design.[4] By means of quickly operated spring claws, one of a pair of

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[1]This proposed method is under the jurisdiction of the ASTM Committee D-4 on Road and Paving Materials.

[2]Published as information only, June, 19--.

[3]This method is the method actually used in the cooperative program. The test described in this method is still under study by Subcommittee B-19 of ASTM Committee D-4, however, and various changes may be made from time to time.

[4]Manufactured by Hallikainen Instruments, 1341 Seventh St., Berkeley, Calif.

matched glass plates is clamped to the viscometer frame and the other to a device for adding loads from 0.1 g to 10,000 g. The sample is placed between the two glass plates and the desired force is applied through a simple balance beam pivoted on agate bearings and polished steel knife edges. An electronic circuit is used to follow the movement of the glass plate by controlling a simple servo motor. This drives an insulated micrometer and causes it to maintain a high-resistance contact with a flag attached to the glass plate. Movement of the glass plate may be measured by reading the micrometer as a function of time, or can be recorded by use of a suitable millivolt recorder.[5]

(b) Viscosity Plates of polished borosilicate-plate glass, 20± 0.1 by 30± 0.1 mm and 6 ± 0.1 mm thick, in matching pairs.[6]

(c) Viscosity Water Bath, maintained at 77°F constant to ±0.2°F. The bath specified in the Method of Test for Penetration of Bituminous Materials (ASTM Designation: D5)[7] may be used. The depth of water above the perforated shelf must be at least 5 in. and should be approximately 7 in.

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[5] Varian Model G-10 has been found satisfactory for this purpose.

[6] Polished plates are available in matched pairs from Hallikainen Instruments, 1341 Seventh Street, Berkeley, Calif., or from Herron Optical Co., 9117 South Main Street, Los Angeles, Calif.

[7] 1961 Book of ASTM Standards, Part 4.

(d) Viscosity Bath Thermometer. — An ASTM Saybolt Viscosity Thermometer having a range of 66 to 80°F and conforming to the requirements for thermometer 17F as prescribed in ASTM Specifications E 1.[8]

(e) Infrared Lamp, operated from a variable transformer.

(f) Aging Oven, as described in the Method of Test for Loss on Heating of Oil and Asphaltic Compounds (ASTM Designation: D 6).[7] The circular revolving shelf shall be turned over so that the aging plates may be placed on the flat surface. The oven temperature shall be maintained at 225 ± 2°F.

(g) Aging Oven Thermometer. — An ASTM Softening Point (High) Thermometer having a range of 85 to 392°F and conforming to the requirements for thermometer 16F as prescribed in ASTM Specifications E 1, or an ASTM Fuel Rating (Mixture) Thermometer having a range of 200 to 350°F and conforming to the requirements for thermometer 86F as prescribed in Specifications E 1.[8]

(h) Aging Plates of polished borosilicate-plate glass in matching pairs.[6] The plates must be of such size that they can be placed on the revolving shelf of the oven without interfering with the movement of the shelf. The dimensions of the plates, however, must not be less than 40 by 40 by 6 mm thick.

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[8] 1961 Book of ASTM Standards, Parts 4, 7, and 8.

Preparation of Sample

5. Heat the sample in an oven, maintained at a temperature not over 325°F, until the asphalt reaches a temperature of 250° to 300°F. Avoid prolonged exposure of the bulk sample to temperatures of 250° to 300° F. In no case should the material be heated above a temperature 50°F below the flash point (C.O.C.). After melting, thoroughly stir the sample until it is homogeneous and free from air bubbles.

Procedure

6. (a) Viscosity Specimen Preparation. — Place a drop of the melted sample on one of a pair of viscosity plates which have previously been cleaned with benzene, dried with paper tissue, and weighed to the nearest 0.1 mg. Cover the asphalt with the second plate and use the infrared lamp to melt the asphalt thoroughly. As the asphalt softens, press and work the plates together. Repeat as necessary to form a uniform layer of asphalt having a thickness between 10 and 100μ. Check the uniformity of the film by viewing it in transmitted light. After forming the film allow the plates to cool. Scrape any excess asphalt from the edges of the plates with a razor blade and then wipe the plates with a cloth dipped in benzene. Weigh on an analytical balance to the nearest 0.1 mg to determine the thickness.

(b) Calculate the film thickness as follows:

$$\text{Film thickness, } \mu = \frac{10^4 W}{lwd} = \frac{10^4 W}{6} = 1667W$$

where:

d = density of sample, in grams per cubic centimeter (for most asphalts assume d = 1),

l, w = length and width of the glass plate, in centimeters (for the plates obtained from the recommended sources, l = 2.00 and w = 3.00), and

W = weight of sample, in grams.

(c) Viscosity Measurement. — Allow the sample loaded between the viscometer plates, as described in Paragraph (a), to cool in air for 60 to 70 min. Then place in the viscometer which has been standing in the water bath maintained at 77± 0.2°F. After loading the plates in the viscometer, allow 2 to 5 minutes for the sample to come to the bath temperature.

Clean the contact flag and micrometer point by rubbing lightly with crocus cloth (Note 1). Position the flag beneath the micrometer and move the micrometer, by driving the motor, until it makes contact with the flag. Apply the load by adding a weight to the weight holder on the beam and measure the displacement either by reading the micrometer as a function of time or by recording the displacement graphically on a millivolt recorder (Note 2). By the use of four different loads, determine the viscosity of each sample at four shear rates, which bracket the shear rate of 0.05 sec^{-1}; suggested loads are given in Table I. It is usually possible to make a single viscosity determination with 0.1-mm movement; therefore, a total movement of 0.5 mm should be sufficient for four shear rates. In no case should the total movement exceed 1.50 mm as this changes the effective area of the sample.

Note 1. — In routine operation, cleaning of the contact flag and micrometer is required only about once a day.

Note 2. — More detailed instructions for measuring the viscosity are given in the instruction manual supplied with the viscometer.

TABLE I

SUGGESTED LOADINGS FOR DIFFERENT GRADES OF ASPHALT

Standard Penetration at 77°F	Approximate Viscosity in Poises at 77°F	Range of Loads in Grams
300	7×10^4	20 to 200
100	7×10^5	100 to 1000
50	3×10^6	500 to 5000
20	2.3×10^7	2000 to 10000

Preparation of Specimens for Aging

7. Prepare the specimens for aging by either of the methods described in Paragraph (a) or (b). Method A will be suitable only if the edges of the aging plates are square and polished.

(a) Method A:

(1) Prepare a film of the material to be aged between two glass aging plates by the procedure given in Section 6, except that the film thickness must be between 9 and 11μ.

(2) Calculate the film thickness as follows:

$$\text{Film thickness, } \mu = \frac{10^4 W}{lwd}$$

where:

d = density of sample, in grams per cubic centimeter (for most asphalts, assume d = 1)

l, w = length and width of aging plates, in centimeters, and

W = weight of sample, in grams.

(3) Warm the plates with the infrared lamp to soften the asphalt and then separate them by sliding the upper plate from the lower one.

(b) Method B:

(1) Place 15 to 35 mg of the material to be aged in the middle of a clean aging plate which has previously been weighed to the nearest 0.1 mg. Weigh the plate plus sample to the nearest 0.1 mg. Cover the asphalt with a second clean aging plate and use an infrared lamp to melt the asphalt thoroughly. As the asphalt softens, press the plates together and squeeze out the asphalt to form a uniform circle, which does not reach the edge of the plates, having a diameter (in millimeters) between $10.8\sqrt{W}$ and $11.9\sqrt{W}$, where W = weight of sample, in milligrams. Then the film will be between 9 and 11μ in thickness.

(1) Warm the plates with the infrared lamp to soften the asphalt, Then rotate one plate 30 to 45 degrees with respect to the other, so that the corners of the plates are somewhat offset, and pull the plates apart without sliding one over the other.

Aging

8. (a) Place the separated plates near the outer edges of the revolving shelf of the aging oven, which is maintained at 225°F ± 2°F. Heat the samples in the oven for 2 hours ± 1 minute with the oven shelf revolving at 5 to 6 rpm during the entire aging period. Determine the temperatures by means of the specified thermometer with the bottom of the thermometer bulb 1/4 in. above the revolving shelf and 1/4 in. from the outer edges of the glass plates.

(b) At the conclusion of the prescribed heating period, remove the plates from the oven, scrape the asphalt off the plates with a razor blade which has been cleaned with benzene and dried, and transfer it to a clean, weighed viscosity plate. Prepare a viscosity specimen as described in Section 6(a) and (b). Determine the viscosity as described in Section 6(c).

Calculations

9. (a) Shearing Stress and Shear Rate. — For both aged and original samples, calculate the shearing stress for each load and the shear rate for the resulting plate movement as follows:

Shearing stress, dynes per cm^2

$$= \frac{A \times 980}{B} = 163.3 \times A$$

where:

A = weight applied, in grams, and

B = area of plate, in square centimeters.

$$\text{Shear rate, } sec^{-1} = \frac{A}{B \times C}$$

where:

A = plate movement, in microns,

B = time, in seconds, and

C = film thickness, in microns.

(b) Viscosity. — Calculate the viscosity for each load as follows:

$$\text{Viscosity, poises} = \frac{A}{B} \quad \text{(Note 3)}$$

where:

A = shearing stress, in dynes per square centimeters, and

B = shear rate, in $seconds^{-1}$.

Plot log shear rate versus log viscosity and draw the best straight line through the points. From this line read the viscosity at a shear rate of 0.05 sec^{-1}.

Note 3. — Other methods for calculating the viscosity are given in the operating manual provided with the microviscometer.[9]

(c) Aging Index. — From the viscosities of the aged and original samples as calculated in accordance with Paragraphs (a) and (b), calculate the aging index as follows:

$$\text{Aging index} = \frac{A}{B}$$

where:

A = viscosity of aged sample, and

B = viscosity of original sample.

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[9] A special slide rule for rapid calculations of the viscosity from the recorded data is available from Hallikainen Instruments, 1341 Seventh Street, Berkeley, Calif.