OXIDATION (DURABILITY) TESTS ON ASPHALTS USED BY THE TEXAS HIGHWAY DEPARTMENT

by

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ABSTRACT

REPORT: Progress Report No. 7 Research Project No. 15 (2-8-59-9)

TITLE: Oxidation (Durability) Tests on Asphalts Used by Texas Highway Department

PERIOD: June 1, 1962 to June 1, 1963

OBJECTIVE: To establish specifications to assure use of superior asphalts by the Texas Highway Department.

EXPERIMENTAL: Sixty asphalts were tested for susceptibility to hardening by oxidation. The test used was similar to that tentatively proposed by Sub-Committee B-19 of ASTM D-4 on Road and Paving Materials. Fifteen-micron films of each asphalt were oxidized in an air oven in the dark at 225° F for 2 hours. The hardened material and the original asphalt were tested in the Hallikainen viscometer at 77° F and the viscosities calculated at 5×10^{-2} sec^{-1} rate of shear. The quotient obtained by dividing the viscosity of an original asphalt into that of the hardened film was called the relative viscosity and used as an aging index.

CONCLUSIONS: The following conclusion is drawn from the data presented in this report.

1. About 60 percent of the asphalts supplied to the Texas Highway Department during 1959 and 1962 passed the proposed minimum relative viscosities (hardening indices) recommended below.

RECOMMENTATIONS: It is recommended that the following minimum relative viscosities be established as tentative quality specifications for the purchase of asphalt paving cements:

Grade	Relative Viscosity (1)
AC-20	5.0-
AC-10	4.5-
AC-5	4.0-

(1) Fifteen-micron film is oxidized in the dark at 225° F for 2 hours and the viscosity of the hardened film measured at 77°F. Viscosity of the hardened film divided by that of original asphalt gives the relative viscosity of the oxidized material.

FUTURE WORK:

1. The Highway Department Laboratories will test numerous samples received during the 1963 and subsequent seasons for hardening by oxidation (obtain relative viscosities). Thus, no further work on this subject will be done under Research Project No. 15. Some special situations may require attention.

2. An extensive program is underway to determine the rate of hardening of asphalt cements during preparation and handling of the hot mixture, laying on the road and during a year of pavement service.

3. A program is underway on the use of chemical additives to improve the properties of asphalt, especially resistance to hardening by oxidation.

CONTENTS

TABLES

OXIDATION (DURABILITY) TESTS ON ASPHALTS USED BY THE TEXAS HIGHWAY DEPARTMENT

I. OBJECTIVES OF RP-15

The objectives of the project are:

(1) Investigate the paving asphalts used by the Texas Highway Department,

(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 No. 15 "Modifications of Properties of Asphalt" was started February 1, 1959. This report is concerned with objectives (1) and (2) given previously.

During the paving season of 1959 twenty-five drum samples of paving asphalts were obtained from the field by Texas Highway Department personnel and shipped to the Texas Transportation Institute at College Station. Eleven of the samples were 85-100 penetration grade; five were 120-150 penetration grade and nine were 150-200 penetration grade asphalts. The 25 samples represented asphalts from 12 different producers. Progress Reports 1 through 5 of Research Project No. 15 give extensive data on these asphalts. Report No. 4 "Susceptibility of Paving Asphalts to Hardening by Heat, Oxygen and Sunlight" presented data on the hardening by oxidation of the 85-100 and 120-150 penetration asphalts.

In 1962, Texas Highway Department personnel collected small samples from 51 paving projects and shipped them to College Station. This report gives hardening data (relative viscosities) on these 51 asphalts and on the nine 150-200 penetration grade materials collected in 1959.

III. CONCLUSIONS

The following conclusion is drawn from the data presented in this report.

1. About 60 percent of the asphalts supplied to the Texas Highway Department during 1959 and 1962 passed the proposed minimum relative viscosities (hardening indices) recommended below.

IV. RECOMMENDATIONS

It is recommended that the following minimum relative viscosities be established as tentative quality specifications for the purchase of asphalt paving cements:

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(1) Fifteen-micron film is oxidized in the dark at 225° F for 2 hours and the viscosity of the hardened film measured at 77° F. Viscosity of the hardened film divided by that of original asphalt gives the relative viscosity of the oxidized material.

V. FUTURE WORK

1. The Highway Department Laboratories will test numerous samples received during the 1963 and subsequent seasons for hardening by oxidation (obtain relative viscosities). Thus, no further work on this subject will be done under Research Project No. 15. Some special situations may require attention.
2. An extensive program is underway to determine the rate of hardening of asphalt cements during preparation and handling of the hot mixture, laying on the road and during a year of pavement service.
3. A program is underway on the use of chemical additives to improve the properties of asphalt, especially resistance to hardening by oxidation.

VI. EXPERIMENTAL

1. Materials Investigated

(a) Nine 150-200 penetration grade asphalts obtained during the 1959 season (see Progress Report No. 1 dated February 1961).

(b) Fifty-one asphalts from as many projects collected during the 1962 season. Twenty-four were 85-100 penetration; 18 were 120-150 penetration and 9 were 150-200 penetration grade asphalts.

2. Method of Test Used for Evaluating Hardening by Oxidation

The test used is similar to the "Method of Test for Aging Index of Bituminous Materials" tentatively proposed by Sub-Committee B-19 of ASTM D-4 on Road and Paving Materials. The procedure is given in detail in the Appendix of Progress Report No. 4 of Research Project No. 15 dated October 1962. The ASTM proposed method was modified to the extent that 15-micron instead of 5-micron films were used. Fifteen-micron films of the asphalts were oxidized at 225°F for 2 hours in the dark. Viscosities of the original asphalts and oxidized films were determined in the Hallikainen sliding plate viscometer at 77°F. Viscosities were calculated at 5×10^{-2} sec^{-1} rate of shear. Viscosity of a hardened film was divided by that of the original asphalt to obtain the relative viscosity.

TABLE 1

Hardening by Oxidation of 150-200 Penetration Grade Asphalts Obtained by Texas Highway Department During 1959 (1)

Asphalt	Pen. at 77°F	Stokes at 77°F(2) Original Asphalt	Stokes at 77°F(2) Hardened Film	Relative Viscosity(3)
6B	170	0.24×10^6	0.60×10^6	2.5
5B	150	0.32×10^6	0.90×10^6	2.8
12B	168	0.24×10^6	0.72×10^6	3.0
10B	160	0.29×10^6	0.90×10^6	3.1
8B	188	0.22×10^6	0.78×10^6	3.5
11B	177	0.26×10^6	0.96×10^6	3.7
2B	185	0.24×10^6	1.04×10^6	4.4
1B	163	0.32×10^6	1.45×10^6	4.6
7B	172	0.32×10^6	1.72×10^6	5.4
Proposed Limit for AC-5				4.0-

(1) Oxidation of 15-micron film at 225°F for 2 hours.

(2) Measured in thin film (sliding plate) viscometer at rate of shear of 5×10^{-2} sec^{-1}.

(3) Quotient obtained by dividing viscosity of hardened film by viscosity of the original asphalt.

3. Relative Viscosities at 77°F

(a) 150-200 penetration asphalts (1959)

Table 1, facing, shows the relative viscosities obtained by oxidizing the 175 penetration grade asphalts obtained from the field by Texas Highway Department personnel during 1959. Six of the nine asphalts passed the proposed minimum relative viscosity of 4.0 for AC-5 grade.

TABLE 2

Average Relative Viscosities of 85-100 Penetration Grade Asphalts Used by Texas Highway Department During 1962[1]

Asphalt	No. of Samples	Pen. at 77° F	Stokes at 77°F[2] Original Asphalt	Stokes at 77°F[2] Hardened Film	Relative Viscosity[3]
14	1	98	0.85×10^6	2.0×10^6	2.4
2	1	99	0.65×10^6	2.0×10^6	3.1
6	3	85	0.815×10^6	2.6×10^6	3.2
5	1	87	0.80×10^6	2.6×10^6	3.3
11	3	88	1.2×10^6	4.2×10^6	3.5
9	2	83	1.0×10^6	3.7×10^6	3.7
1	3	88	1.35×10^6	5.5×10^6	4.1
3	5	89	1.1×10^6	5.6×10^6	5.1
7	4	87	0.90×10^6	6.9×10^6	7.7
13	1	86	1.2×10^6	12×10^6	10.0
Proposed Limit for AC-20					5.0-

(1) Fifteen-micron films oxidized at 225°F for 2 hours.

(2) Measured in thin film (sliding plate) viscometer at rate of shear of 5×10^{-2} sec^{-1}.

(3) Quotient obtained by dividing viscosity of hardened film by viscosity of the original asphalt.

(b) 85-100 Penetration Asphalts (1962)

Data on the twenty-four 85-100 penetration (AC-20) asphalts are shown in Table A-1 of the Appendix. Average values for the materials supplied by each of the 10 producers are given in Table 2. facing (only one sample was obtained from producers 2, 5, 13 and 14). Seven of the 10 producers supplied asphalts that passed the proposed minimum relative viscosity of 5.0 for AC-20.

TABLE 3

Average Relative Viscosities of 120-150 Penetration Grade Asphalts Used by Texas Highway Department During 1962(1)

Asphalt	No. of Samples	Pen. at 77°F	Stokes at 77°F(2) Original Asphalt	Stokes at 77°F(2) Hardened Film	Relative Viscosity (3)
2A	1	123	0.46×10^6	1.2×10^6	2.6
6A	5	138	0.39×10^6	1.17×10^6	3.0
1A	2	130	0.64×10^6	2.2×10^6	3.4
11A	3	139	0.41×10^6	1.6×10^6	3.9
7A	2	125	0.50×10^6	2.0×10^6	4.0
3A	4	139	0.43×10^6	2.7×10^6	6.3
13A	1	118	0.72×10^6	6.0×10^6	8.4
Proposed Limit for AC-10					4.5-

(1) Fifteen-micron film oxidized at 225°F for 2 hours.

(2) Measured in thin film (sliding plate) viscometer at rate of shear of 5×10^{-2} sec^{-1}.

(3) Quotient obtained by dividing viscosity of hardened film by viscosity of the original asphalt.

(c) 120-150 Penetration Asphalts (1962)

Data on the hardening of the eighteen 120-150 penetration (AC-10) asphalts are shown in Table A-2 of the Appendix. Average values for the materials obtained from each of the seven producers are shown in Table 3, facing. Only one sample each of asphalts 2A and 13A were obtained. Asphalts from five of the seven producers passed the proposed minimum relative viscosity of 4.5 for AC-10.

(d) 150-200 Penetration Asphalts (1962)

Detailed data on nine samples of 175 penetration grade asphalt from four producers are shown in Table A-3 of the Appendix. These asphalts were used by the Texas Highway Department in 1962. Average values for the materials obtained from each of the four producers are given in Table 4 below. Only one sample was obtained from the producers of Asphalts 1B and 11B. Asphalts from two of the producers passed the proposed minimum relative viscosity of 4.0 for AC-5.

TABLE 4

Average Relative Viscosities of 150-200 Penetration Grade Asphalts Obtained by Texas Highway Department During 1962[1]

Asphalt	No. of Samples	Pen. at 77°F	Stokes at 77°F[2] Original Asphalt	Stokes at 77°F[2] Hardened Film	Relative Viscosity[3]
6B	3	164	0.24×10^6	0.65×10^6	2.7
11B	1	185	0.26×10^6	0.80×10^6	3.1
7B	4	175	0.26×10^6	1.36×10^6	5.1
1B	1	172	0.48×10^6	2.6×10^6	5.5
Limits for AC-5					4.0-

(1) Fifteen-micron film oxidized at 225°F for 2 hours.

(2) Measured in thin film (sliding plate) viscometer at rate of shear of 5×10^{-2} sec^{-1}.

(3) Quotient obtained by dividing viscosity of hardened film by viscosity of the original asphalt.

TABLE 5

Average Relative Viscosities of Asphalts Received by Texas Highway Department from Four Major Producers During 1962(1)

85-100 Penetration Grade Asphalts (AC-20)

Asphalt	Viscosity-Stokes at 77°F		Relative Viscosity of Hardened Film
	Original Asphalt	Hardened Film	
6	0.815×10^6	2.6×10^6	3.2
11	1.2×10^6	4.2×10^6	3.5
1	1.35×10^6	5.5×10^6	4.1
3	1.1×10^6	5.6×10^6	5.1
Proposed Limit for AC-20			5.0-
	120-150 Penetration Grade Asphalts (AC-10)		
6A	0.39×10^6	1.17×10^6	3.0
1A	0.64×10^6	2.2×10^6	3.4
11A	0.41×10^6	1.6×10^6	3.9
3A	0.43×10^6	2.7×10^6	6.3
Proposed Limit for AC-10			4.5-
	150-200 Penetration Grade Asphalt (AC-5)		
6B	0.24×10^6	0.65×10^6	2.7
11B	0.26×10^6	0.80×10^6	3.1
1B	0.48×10^6	2.60×10^6	5.5
3B	----	----	---
Proposed Limit for AC-5			4.0-

(1) Fifteen-micron film oxidized at 225°F for 2 hours. Viscosities on original asphalt and hardened film determined in Hallikainen viscometer at 77°F and rate of shear of 5×10^{-2} sec^{-1}.

4. Average Relative Viscosities of Asphalts Received from Four Major Producers During 1962

Table 5, facing, is a compilation of the average viscosities at 77°F and relative viscosities of the three penetration grade materials produced from four major sources and collected during 1962.

Considering the 85-100 penetration (AC-20) grade materials: three of the producers met the proposed minimum relative viscosity of 5.0. Producer of Asphalt No. 3 missed by a small margin.

Referring to the 120-150 penetration (AC-10) grade asphalts: three of the producers met the proposed minimum relative viscosity of 4.5.

Samples of 150-200 penetration (AC-5) grade asphalts were obtained from only three of the four major producers. Of these two passed the proposed minimum relative viscosity of 4.0.

VII. APPENDIX

Index

TABLE A-1

Hardening by Oxidation of 85-100 Penetration Grade Asphalt Obtained by Texas Highway Department During 1962[1]

Asphalt	TTI No.	Pen. at 77°F	Stokes at 77°F[2] Original Asphalt	Stokes at 77°F[2] Hardened Film	Relative Viscosity[3]
14	138	98	0.85×10^6	2.0×10^6	2.4
2	39	99	0.65×10^6	2.0×10^6	3.1
6	12	79	0.80×10^6	2.8×10^6	3.5
	9	86	0.85×10^6	3.0×10^6	3.6
	150	89	0.80×10^6	2.0×10^6	2.5
Average		85	0.815×10^6	2.6×10^6	3.2
5	129	87	0.80×10^6	2.6×10^6	3.3
11	96	88	1.1×10^6	3.2×10^6	2.9
	27	81	1.5×10^6	6.0×10^6	4.0
	32	96	1.0×10^6	3.6×10^6	3.6
Average		88	1.2×10^6	4.2×10^6	3.5
9	75	85	1.0×10^6	3.4×10^6	3.4
	144	90	1.0×10^6	4.0×10^6	4.0
Average		87	1.0×10^6	3.7×10^6	3.7
1	69	78	1.75×10^6	6.0×10^6	3.4
	113	88	1.3×10^6	5.6×10^6	4.3
	6	99	1.0×10^6	4.8×10^6	4.8
Average		88	1.35×10^6	5.5×10^6	4.1

TABLE A-1 (Cont.)

Asphalt	TTI No.	Pen. at 77°F	Stokes at 77°F(2) Original Asphalt	Stokes at 77°F(2) Hardened Film	Relative Viscosity (3)
3	108	82	1.4×10^6	5.4×10^6	3.9
	3	83	1.1×10^6	5.2×10^6	4.7
	93	88	1.0×10^6	6.0×10^6	6.0
	57	94	1.1×10^6	5.8×10^6	5.3
	87	98	0.9×10^6	5.4×10^6	6.0
Average		89	1.1×10^6	5.6×10^6	5.1
7	72	85	1.0×10^6	7.6×10^6	7.6
	101	86	0.9×10^6	6.0×10^6	6.6
	124	87	0.85×10^6	6.0×10^6	7.1
	112	92	0.85×10^6	8.0×10^6	9.5
Average		87	0.90×10^6	6.9×10^6	7.7
13	54	86	1.2×10^6	12×10^6	10.0
Limit for AC-20					5.0-

(1) Oxidation of 15-micron film at 225°F for 2 hours.

(2) Measured in thin film (sliding plate) viscometer at rate of shear of 5×10^{-2} sec^{-1}.

(3) Quotient obtained by dividing viscosity of hardened film by viscosity of the original asphalt.

TABLE A-2

Hardening by Oxidation of 120-150 Penetration Grade Asphalt Obtained by Texas Highway Department During 1962[1]

Asphalt	TTI No.	Pen. at 77°F	Stokes at 77°F[2] Original Asphalt	Stokes at 77°F[2] Hardened Film	Relative Viscosity[3]
2A	146	123	0.46×10^6	1.2×10^6	2.6
6A	40	134	0.375×10^6	1.2×10^6	3.2
	46	145	0.37×10^6	1.0×10^6	2.7
	58	134	0.41×10^6	1.55×10^6	3.8
	61	123	0.44×10^6	1.15×10^6	2.6
	103	139	0.36×10^6	0.92×10^6	2.5
Average		138	0.39×10^6	1.17×10^6	3.0
1A	121	133	0.66×10^6	2.2×10^6	3.3
	64	128	0.62×10^6	2.2×10^6	3.5
Average		130	0.64×10^6	2.2×10^6	3.4
7A	13	118	0.50×10^6	2.2×10^6	4.4
	118	132	0.50×10^6	1.8×10^6	3.6
Average		125	0.50×10^6	2.0×10^6	4.0
11A	34	124	0.54×10^6	1.7×10^6	3.1
	82	151	0.32×10^6	1.7×10^6	5.4
	130	141	0.36×10^6	1.4×10^6	3.9
Average		139	0.41×10^6	1.6×10^6	4.1

TABLE A-2 (Cont.)

Asphalt	TTI No.	Pen. at 77°F	Stokes at 77°F(2) Original Asphalt	Stokes at 77°F(2) Hardened Film	Relative Viscosity(3)
3A	22	119	0.54×10^6	3.4×10^6	6.3
	16	148	0.38×10^6	3.0×10^6	7.9
	115	134	0.46×10^6	2.2×10^6	4.8
	79	155	0.34×10^6	2.2×10^6	6.5
Average		139	0.43×10^6	2.7×10^6	6.3
13A	145	118	0.72×10^6	6.0×10^6	8.4
Limit for AC-10					4.5-

(1) Oxidation of 15-micron film at 225°F for 2 hours.

(2) Measured in thin film (sliding plate) viscometer at rate of shear of 5×10^{-2} sec^{-1}.

(3) Quotient obtained by dividing viscosity of hardened film by viscosity of the original asphalt.

TABLE A-3

Hardening by Oxidation of 150-200 Penetration Grade Asphalt Obtained by Texas Highway Department During 1962[1]

Asphalt	TTI No.	Pen. at 77°F	Stokes at 77°F[2] Original Asphalt	Stokes at 77°F[2] Hardened Films	Relative Viscosity[3]
6B	28	157	0.26×10^6	0.72×10^6	2.8
	19	165	0.23×10^6	0.62×10^6	2.7
	49	169	0.23×10^6	0.60×10^6	2.6
Average		164	0.24×10^6	0.65×10^6	2.7
11B	43	185	0.26×10^6	0.80×10^6	3.1
7B	151	161	0.28×10^6	1.2×10^6	4.2
	133	163	0.30×10^6	2.0×10^6	6.7
	88	186	0.25×10^6	1.2×10^6	4.8
	109	191	0.22×10^6	1.05×10^6	4.8
Average		175	0.26×10^6	1.36×10^6	5.1
1B	76	172	0.48×10^6	2.6×10^6	5.5
Limits for AC-5					4.0-

(1) Oxidation of 15-micron film at 225°F for 2 hours.

(2) Measured in thin film (sliding plate) viscometer at rate of shear of 5×10^{-2} sec^{-1}.

(3) Quotient obtained by dividing viscosity of hardened film by viscosity of original asphalt.