

CORRELATION OF COMPOSITION WITH RHEOLOGY
AND DURABILITY OF ASPHALTS

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Progress Report No. 5
Research Project No. 15

E 83-62

Submitted to
Research Committee of the
Texas Highway Department

December 1, 1962

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ABSTRACT

REPORT: Progress Report No. 5 Research Project No. 15

TITLE: Correlation of Composition With Rheology and Durability of Asphalts

PERIOD: February 1, 1959 to December 1, 1962

OBJECTIVES: Investigate paving asphalts used by THD to determine how they can be evaluated for quality and their durabilities improved.

EXPERIMENTAL: A number of representative paving asphalts were analyzed by (1) a modified Traxler and Schweyer Component Analysis procedure using pentane, butanol and acetone, (2) a modified Pitchford and Axe method using pentane, isopropanol and acetone, and (3) the Rostler analytical procedure using pentane, 85%, 98%, and fuming sulfuric acid. The data obtained were correlated with oxidation of 15-micron films of each asphalt for 2 hours in a dark oven at 225°F, and in some cases with photooxidation of 15-micron films under radiation from a 175 Watt Mazda Sunlamp for 2 hours at 190°F. Changes in composition caused by oxidation were determined. A correlation was established between Saturates content by the Traxler and Schweyer method and viscosity of the asphalt at 275°F.

CONCLUSIONS:

1. Dispersion Coefficients calculated from a modified Traxler and Schweyer Component Analyses correlate well with susceptibilities to oxidation as measured by Relative Viscosities. Asphalt No. 8 is the one exception among the asphalts investigated.
2. Dispersion Coefficients do not correlate very well with susceptibilities of the asphalts to photooxidation.
3. Data obtained by a modified Pitchford and Axe procedure correlate well with susceptibilities to oxidation.
4. The coefficient established by Rostler does not correlate well with durability as measured by the Relative Viscosities of oxidized films. A modified Rostler coefficient gave an improved correlation but was not equal to that calculated from a modified Traxler and Schweyer Component Analysis.
5. Oxidation of 1/8 inch films for 15 hours at 325°F results in a definite increase in Asphaltene content and a marked decrease in percent Resins. There is a tendency for Cyclics (unsaturated or aromatic oils) to increase during oxidation. Saturates (paraffinic oils) may increase or decrease depending on the source of the asphalt.

6. Percent Saturates correlate fairly well with the high temperature viscosities of the asphalts.

RECOMMENDATIONS: None.

FUTURE WORK: No further work will be done on asphalts studied under RP-15 until improved methods of analysis have been perfected. Such work is progressing at A. & M. College under a grant from the Petroleum Research Fund of the American Chemical Society. At present a more logical and precise method for determining the paraffine content of an asphalt is under development. If it offers promise the procedure will be applied to the asphalts discussed in this report.

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CORRELATION OF COMPOSITION WITH RHEOLOGY
AND DURABILITY OF ASPHALTS

I. OBJECTIVES OF RP-15

The objectives of this project are to:

- (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 February 1, 1959.

The investigations reported below are concerned with objectives (1) and (3). During the past fifteen years considerable progress has been made in understanding the composition of asphalt. However, the information now available is inadequate to explain fully the causes for the variety of properties encountered in commercial asphalts. The correlations discussed below, although helpful, are not as satisfactory as we believe will be possible when new techniques and procedures are developed. We are certain that within a few years the data given below will be completely revised and will result in sound explanations for the differences in durability and viscosity encountered in paving asphalts.

III. CONCLUSIONS

- (1) Dispersion Coefficients calculated from a modified Traxler and Schweyer Component Analysis correlate well with susceptibilities to oxidation as measured by Relative Viscosities. Asphalt No. 8 is the one exception among the asphalts investigated.
- (2) Dispersion Coefficients do not correlate very well with susceptibilities of the asphalts to photooxidation.
- (3) Data obtained by a modified Pitchford and Axe procedure correlate well with susceptibilities to oxidation.
- (4) The coefficient established by Rostler does not correlate well with durability as measured by the Relative Viscosities of oxidized films. A modified Rostler coefficient gave an improved correlation but was not equal to that calculated from a modified Component Analysis.
- (5) Oxidation of 1/8 inch films for 15 hours at 325° F results in a definite increase in Asphaltene content and a marked decrease in percent Resins. There is a tendency for Cyclics (unsaturated or aromatic oils) to increase during the test. Saturates (paraffinic oils) may increase or decrease depending on the source of the asphalt.
- (6) Percent Saturates correlate fairly well with the high temperature viscosities of the asphalts.

IV. RECOMMENDATIONS

No recommendations are made.

V. FUTURE WORK

No further work will be done on asphalts being studied under RP-15 until improved methods of analysis have been perfected. Such work is progressing at A. & M. College under a grant from the Petroleum Research Fund of the American Chemical Society. At present a more logical and precise method for determining the paraffine content of an asphalt is under development. If it offers promise the procedure will be applied to the asphalts discussed in this report.

VI. EXPERIMENTAL

1. Materials

(a) Representative asphalts of various consistencies were selected from those discussed in Progress Reports 1, 2, 3, and 4 of Research Project No. 15.

(b) Solvents used for fractionation of asphalts were n-pentane, 1-butanol, acetone, and isopropanol.

(c) Sulfuric acids of various concentrations were used for fractionation of asphalts.

2. Procedures

(a) Modified Traxler and Schweyer Component Analysis, using n-pentane, 1-butanol, and acetone, was used to fractionate a considerable number of asphalts. Briefly, the procedure is:

A weighed sample of the asphalt is digested for 30 minutes with 20 parts of boiling 1-butanol. The solution is then maintained at 122°F (50°C) for 16 to 18 hours, filtered and washed repeatedly with hot 1-butanol. The insoluble material is called asphaltics. The asphaltene content (determined on a separate sample using n-pentane as the solvent) is subtracted from the amount of asphaltics to give the quantity of resin present. The solution of oil in the 1-butanol is carefully distilled to remove the solvent. The recovered oil is refluxed with 35 parts of acetone for 15 minutes, allowed to cool in the room and then placed in a deep freeze at -10°F. After 16 to 18 hours the solution is decanted from the precipitated saturates (paraffinic type oils). Finally the solution is stripped of acetone leaving the cyclic or aromatic oils. Four fractions are obtained:

Asphaltenes, A
Resins, R
Saturates (paraffinics), S
Cyclics (aromatics), C

(b) Pitchford and Axe Component Analysis which employs n-pentane and isopropanol was used to fractionate a few of the same asphalts analyzed by method (a). Pitchford and Axe's procedure is briefly as follows:

Asphaltenes are determined by treatment with n-pentane. The solution is stripped of pentane and the recovered malthenes (petrolenes) refluxed for 2 hours with about 50 parts of isopropanol, filtered and washed with hot isopropanol. The recovered materials are called resins. The isopropanol is distilled from the solution and the residue is the oil from the asphalt. By this procedure three fractions are obtained:

Asphaltenes, A
Resins, R
Oils, O

(c) Rostler and Sternberg Chemical (Sulfuric acid) Fractionation.

Briefly, the procedure depends on the successive use of 85% 98% and fuming sulfuric acid. However, the first step is the separation of asphaltenes. The pentane soluble material is then digested with 85% H_2SO_4 and the material removed is called "nitrogen bases". Next the unreacted material is treated with 98% H_2SO_4 to remove "1-Acidaffins". The compounds not removed by this strong acid are then treated with fuming H_2SO_4 to extract "2-Acidaffins". The materials not attacked by the fuming H_2SO_4 are called paraffins. This procedure gives five fractions:

Asphaltenes, A
Nitrogen Bases, N
1-Acidaffins, A_1
2-Acidaffins, A_2
Paraffins, P

(d) Viscosity Determinations were made using the microfilm (Hallikainen) and the Cannon-Manning vacuum type capillary viscometers. The operations of these instruments have been described in Progress Reports 1 and 2 of Research Project No. 15.

(e) Durability Tests included oxidation of 15-micron films in a dark oven for 2 hours at 225°F and photooxidation of 15-micron films for 2 hours at 190°F using the radiation from a 175 Watt Mazda Sunlamp. The viscosity of a hardened film was determined in the microfilm viscometer at 77° or 95°F and this value divided by the viscosity of the original asphalt at the same temperature. The quotient (called the Relative Viscosity of the hardened material) serves as a quantitative measure of the susceptibility of the asphalt to oxidation or photooxidation. This method of evaluation has been used and explained in Progress Reports 1, 3, and 4 of Research Project No. 15.

TABLE 1

Correlation of Dispersion Coefficients With Durabilities

Asphalt No.	Coefficient of Dispersion ⁽¹⁾ $X = \frac{R + C}{A + S}$	Susceptibility to Oxidation Relative Vis. ⁽²⁾	Susceptibility to Photooxidation Relative Vis. ⁽³⁾
<u>85-100 Penetration Asphalts</u>			
6	1.22	2.1	3.4
4	1.03	2.5	10.1
5	0.96	2.8	4.5
10	0.94	3.3	10.1
11	0.67	3.3	8.3
9	0.49	3.5	7.5
3	0.55	4.3	8.5
8	3.61	4.4	8.4
2	0.49	4.5	12.4
1	0.50	6.0	13.9
7	0.59	6.2	13.7
<u>120-150 Penetration Asphalts</u>			
6A	1.78	2.5	3.9
11A	0.79	2.6	5.3
3A	0.65	4.0	6.0
1A	0.61	4.2	11.2

(1) R = Resins, C = Cyclics, A = Asphaltenes, S = Saturates.

(2) 15-micron films oxidized in dark air oven for 2 hours at 225°F.

(3) 15-micron films oxidized under radiation from 175 Watt Mazda Sunlamp for 2 hours at 190°F.

3. Correlation of Composition with Durability

After a number of years experience it has been concluded that although 3 components reveal considerable information about an asphalt a much clearer picture is obtained by using a Dispersion Coefficient, X, calculated from four components. This was the reason for modifying the original Traxler-Schweyer Component Analysis. The Dispersion Coefficient is obtained by dividing the sum of the Resins and Cyclics by the sum of the Asphaltenes and Saturates. High Resin + Cyclics content, which results in a high value for X, means that the asphalt is well dispersed. When the sum of the Asphaltenes plus Saturates is large, and X is small, a poorly dispersed asphalt is indicated.

Some correlations given in Progress Report No. 1 are included in this report to show how the results obtained by the Modified T. & S. Component Analysis correlate with the hardening of the asphalt by oxidation. Table 1 compares the Relative Viscosities of eleven 85-100 and four 120-150 penetration asphalts with the Dispersion Coefficient, X. A good correlation is obtained between this coefficient and the Relative Viscosity of asphalts oxidized in the dark for 2 hours at 225°F. Asphalt 8 is the only one which does not yield a reasonably good correlation. This is a manifestation of the unusual composition of Asphalt 8.

When the Relative Viscosities obtained by photooxidation are compared with Coefficient X the correlation is not so satisfactory. For example, asphalts 4 and 10 are well dispersed materials (high values for X) but are

quite susceptible to hardening by photooxidation (RV = 10.1 for each). Again Asphalt 8 does not correlate because of its high X value. Nevertheless, there is enough correlation to indicate that we are approaching an understanding of the effects of composition on the durability of paving asphalts under different environments. Data obtained on 150-200 penetration asphalts similar to those shown in Table 1 did not correlate at all well. This difficulty was probably caused by the increased effect of volatilization experienced when conducting oxidation tests on the soft asphalts. Data are shown in Table A-1 of the Appendix.

TABLE 2

Comparison of Analyses by 1-Butanol (T & S) and
Isopropanol (P & A) Methods of Fractionation

Asphalt No.	Percent					
	Asphaltenes by		Resin by		Oil by	
	T & S	P & A	T & S	P & A	T & S	P & A
1A	11.0	11.0	21.0	30.2	68.0	58.8
3A	17.5	17.5	25.5	41.0	57.0	41.5
6A	1.0	1.0	55.0	90.0	44.0	9.0

TABLE 3

Component Analysis and Dispersion Coefficient by 1 - Butanol and Isopropanol
Procedures and Their Correlation With Relative Viscosities of Asphalt
Films Oxidized for 16 Hours at 190°F

Asphalt No.	Procedure	Asphal- tenes	Resin	Saturates	Cy- clics	$X = \frac{R+C}{A+S}$	R.V. (1)
1A	1-butanol	11.0	21.0	51.0	17.0	0.61	5.5
	isopropanol	11.0	30.2	36.2	22.6	1.15	5.5
3A	1-butanol	17.5	25.5	43.0	14.0	0.65	4.2
	isopropanol	17.5	41.0	25.0	16.5	1.35	4.2
6A	1-butanol	1.0	55.0	35.0	9.0	1.78	3.8
	isopropanol	1.0	90.0	6.0	3.0	13.0	3.8

(1) Relative Viscosity of 15-micron film oxidized in the dark for 16 hours at 190°F. Viscosities were measured at 95°F and calculated at $5 \times 10^{-2} \text{ sec}^{-1}$.

4. Comparison of 1-Butanol-Acetone and Isopropanol Separations

A number of asphalts were analyzed by the Pitchford and Axe isopropanol method described in Section 2-b above. Data obtained on three representative 120-150 penetration asphalts are given in Table 2 facing. Values on the same asphalts by the Traxler and Schweyer 1-butanol procedure are shown in order that the two methods of analysis may be compared. The isopropanol treatment indicates a higher resin and a lower oil content than does the 1-butanol separation.

It was decided to fractionate the oils from the isopropanol treatment by acetone as in the Traxler and Schweyer method in order that the Dispersion Coefficients could be calculated from the data supplied by both the 1-butanol and isopropanol procedures. Data are shown in Table 3, facing, together with the Relative Viscosities of the three asphalts after oxidation in the dark for 16 hours at 190°F. Both methods of fractionation give Dispersion Coefficients that correlate with resistance to oxidation as measured by Relative Viscosity. The well dispersed asphalts (with high values of X) are most resistant to oxidation (low R.V. values).

TABLE 4

Correlation of Rostler and Sternberg Data
With Susceptibility to Oxidation

Asphalt No.	R.V. (1)	Coefficient X (2)	Rostler Coefficient (3)	Modified Rostler Coefficient (4)
85-100 Penetration Asphalts				
1	6.0	0.50	1.19	1.50
3	4.3	0.55	0.90	1.45
11	3.3	0.67	0.75	1.76
6	2.1	1.22	1.33	5.65
120-150 Penetration Asphalts				
1A	4.2	0.61	1.05	2.68
3A	4.0	0.65	1.03	2.46
11A	2.6	0.79	0.97	2.68
6A	2.5	1.78	1.49	8.70

(1) Relative Viscosity of 15-micron films oxidized in dark oven for 2 hours at 225°F.

(2) Resins + Cyclics .
Asphaltenes + Saturates

(3) Nitrogen Bases + 1-Acidaffins .
Paraffins + 2-Acidaffins

(4) Nitrogen Bases + 1-Acidaffins + 2-Acidaffins .
Asphaltenes + Paraffins

5. Correlation of Composition by Rostler-Sternberg Sulfuric Acid Method with Relative Viscosities of Oxidized Asphalts

Data obtained on four 85-100 and four 120-150 penetration asphalts by the Rostler procedure are given in Tables A-2 and A-3 of the Appendix. The Rostler coefficient $\frac{N + A_1}{P + A_2}$ was calculated and compared with the hardening caused by oxidation. Poor correlation was obtained as shown in Table 4, facing.

Additional calculations were made using other ratios of the experimental values obtained by the Rostler procedure. For example, the Asphaltene content was used with the other components to obtain a modified Rostler Coefficient. These values did not correlate with the Relative Viscosities (see Table 4). It must be concluded from the available data that the Rostler method of analysis is not reliable for predicting the susceptibility of an asphalt to hardening by oxidation.

TABLE 5

Change in Composition Resulting From Oxidation
of 1/8 inch Films of 85-100 Penetration
Asphalts for 15 Hours at 325°F

Asphalt No.	Asphaltenes	Resins	Saturates	Cyclics
1	+ 9.2	-12.2	+1.0	+2.0
2	+ 6.0	- 3.0	-6.0	+3.0
3	+ 5.1	- 5.1	-2.5	+2.5
4	+12.2	-15.9	+3.3	+0.4
5	+ 9.3	-10.3	+1.0	0.0
6	+ 9.0	- 9.0	0.0	0.0

6. Change in Composition Caused by Oxidation

Six 85-100 penetration grade asphalts were oxidized in the form of 1/8 inch (3175 micron) films for 15 hours at 325°F. This is essentially the Bureau of Public Roads Thin Film Test except the time of oxidation was increased from 5 to 15 hours. Each oxidized sample was thoroughly mixed while hot and a portion analyzed by the modified Traxler and Schweyer component analysis. Table 5 shows that for each asphalt the oxidized samples showed a marked increase in Asphaltenes over the original. Likewise each showed a decrease in Resin content indicating that much of the increased Asphaltene content was derived from the Resins. For two of the samples the Saturate content decreased slightly, for three it increased and for Asphalt 6 the Saturate content did not change with oxidation. Four of the asphalts showed a small increase of Cyclics in the oxidized sample. Asphalt 5 and 6 showed no change. This confirms a long established belief that oxidation (hardening) of an asphalt results chiefly in a change of Resins into Asphaltenes.

TABLE 6

Correlation of Composition With Viscosity

Asphalt No.	% Saturates ⁽¹⁾	Viscosity, Stokes @ 275°F
85-100 Penetration Asphalts		
8	23	7.0
3	40	5.4
10	40	4.8
4	40	4.3
6	44	3.5
11	49	3.4
5	43	3.3
9	50	3.1
2	48	3.1
1	49	3.0
7	48	2.6
120-150 Penetration Asphalts		
6A	35	4.2
3A	43	3.8
11A	45	3.1
1A	51	2.3
7A	47	1.9

(1) 1-butanol soluble-acetone insoluble fraction of the asphalt.

7. Relation Between Viscosity and Composition

The data on composition available at the present time have been studied in relation to the high temperature viscosities of the asphalts. Percent Saturants as determined by the Traxler and Schweyer Component Analyses method appear to give the best correlation. Saturants are the 1-butanol soluble and acetone insoluble portion of the asphalt. Table 6, facing, shows the relationship for both 85-100 and 120-150 penetration asphalts. Asphalt No. 5 is somewhat out of line as is No. 7A. These discrepancies may be removed when we have developed a more accurate method for determination of the saturated hydrocarbons (paraffines) in an asphalt. It is logical to assume that a high paraffin content would result in increased susceptibility to change of temperature and thus a low viscosity at elevated temperatures (e.g. 275°F).

VII. APPENDIX

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TABLE A-1

Correlation of Dispersion Coefficients
with Durabilities

150-200 Penetration Asphalts

Asphalt No.	Coefficient of Dispersion (1) $X = \frac{R + C}{A + S}$	Susceptibility to Oxidation Relative Viscosity (2)
12B	0.90	2.5
5B	0.75	2.9
6B	1.50	3.3
10B	0.68	3.4
2B	0.55	3.6
8B	2.90	3.7
11B	0.73	3.8
1B	0.51	4.7
7B	0.61	4.7

(1) R = resins; C = cyclics; A = asphaltenes; S = saturates.

(2) 15-micron films oxidized in dark oven for 2 hours at 225°F.

TABLE A-2

Analysis of 85-100 Penetration Asphalts by the
Rostler-Sternberg Sulfuric Acid Method

<u>Asphalt No. 1</u>	
Asphaltenes (A)	17.4%
Nitrogen Bases (N)	26.2
1-Acidaffins (A ₁)	18.6
2-Acidaffins (A ₂)	21.2
Paraffins (P)	16.6
Rostler Coefficient (1)	1.19
Relative Viscosity (2)	6.0

<u>Asphalt No. 3</u>	
Asphaltenes (A)	28.5%
Nitrogen Bases (N)	13.1
1-Acidaffins (A ₁)	20.8
2-Acidaffins (A ₂)	25.2
Paraffins (P)	12.4
Rostler Coefficient (1)	0.90
Relative Viscosity (2)	4.3

<u>Asphalt No. 11</u>	
Asphaltenes (A)	18.8%
Nitrogen Bases (N)	20.0
1-Acidaffins (A ₁)	14.9
2-Acidaffins (A ₂)	28.9
Paraffins (P)	17.4
Rostler Coefficient (1)	0.75
Relative Viscosity (2)	3.3

<u>Asphalt No. 6</u>	
Asphaltenes (A)	2.5%
Nitrogen Bases (N)	30.2
1-Acidaffins (A ₁)	25.4
2-Acidaffins (A ₂)	29.3
Paraffins (P)	12.6
Rostler Coefficient (1)	1.33
Relative Viscosity (2)	2.1

(1) $\frac{N + A_1}{P + A_2}$.

(2) 15-micron films oxidized in dark oven for 2 hours at 225°F.

TABLE A-3

Analysis of 120-150 Penetration Asphalts
by the Rostler-Sternberg Sulfuric Acid Method

<u>Asphalt No. 1A</u>	
Asphaltenes (A)	11.0%
Nitrogen Bases (N)	22.0
1-Acidaffins (A ₁)	23.6
2-Acidaffins (A ₂)	27.1
Paraffins (P)	16.3
Rostler Coefficient (1)	1.05
Relative Viscosity (2)	4.2
<u>Asphalt No. 3A</u>	
Asphaltenes (A)	17.5%
Nitrogen Bases (N)	22.4
1-Acidaffins (A ₁)	19.5
2-Acidaffins (A ₂)	29.2
Paraffins (P)	11.4
Rostler Coefficient (1)	1.03
Relative Viscosity (2)	4.0
<u>Asphalt No. 11A</u>	
Asphaltenes (A)	11.0%
Nitrogen Bases (N)	27.2
1-Acidaffins (A ₁)	16.8
2-Acidaffins (A ₂)	28.7
Paraffins (P)	16.3
Rostler Coefficient (1)	0.97
Relative Viscosity (2)	2.6
<u>Asphalt No. 6A</u>	
Asphaltenes (A)	1.0%
Nitrogen Bases (N)	29.2
1-Acidaffins (A ₁)	30.0
2-Acidaffins (A ₂)	30.6
Paraffins (P)	9.2
Rostler Coefficient (1)	1.49
Relative Viscosity (2)	2.5

(1) $\frac{N + A_1}{P + A_2}$

(2) 15-micron films oxidized in dark oven for 2 hours at 225°F.