Evaluation of Physical and Fractional Properties of Asphalt and Their Interrelationship

G. THENOUX, C. A. BELL, AND J. E. WILSON

Presented in this paper are the results of a research study in which physical and fractional properties of original and recovered asphalts were obtained. Asphalts from eight projects in Oregon were evaluated. The relationships between the fractional components (obtained with the Corbett-Swarbrick procedure) and physical properties were examined in detail. The relationships between fractional components and temperature susceptibility parameters were also examined. Also presented are evaluations of four different asphalt extraction-recovery procedures and a pressure oxygen bomb device used for laboratory aging of asphalt.

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Also presented are evaluations of four different asphalt extraction-recovery procedures and of a pressure oxygen bomb device used for laboratory aging of asphalt.

The objectives of this paper are to

1. Present laboratory testing results for asphalt pavement materials used in the research project. These include results from tests on physical properties and chemical composition, as well as results from mathematical calculation of various property indices.
2. Evaluate possible relationships between physical properties and chemical composition and between property indices and chemical composition.
3. Evaluate the results obtained on recovered asphalt samples from four different extraction procedures.
4. Compare the aging of asphalt using Fraass samples in a pressure oxygen bomb (POB) with aging in the rolling thin film oven (RTFO).

RESEARCH PROGRAM

The research program was organized in five different parts. Each part is described briefly.

Project Selection

Eight different highway projects in Oregon were selected to represent a range of performance and highway environments. Figure 1 shows the approximate location of the projects, and Table 1 gives a general description of the present condition of the highway segments under study.

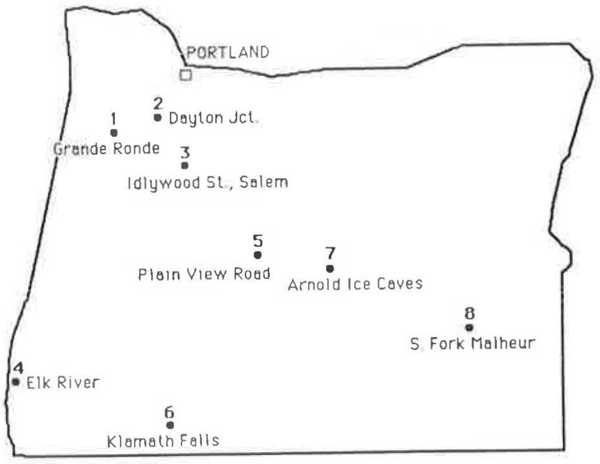

FIGURE 1 Locations of projects.

Core Sampling

Cores were taken from the travel lanes of each project and from shoulders in Projects 5 and 7 (Locations 5s and 7s, respectively, in Table 2). For Project 3, a city street (no shoulders), cores were also taken from a location away from the traffic path (Location 3a in Table 2).

The cores were cut in half: a top layer of approximately 1.5 to 2 in. and a bottom layer ranging from 2 to 4 in. Separate testing was performed on each of the two layers to differentiate environmental effects on the exposed and the unexposed part of the pavement. Table 2 gives detailed information on the top layers of the cores.

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TABLE 1 PROJECT DESCRIPTION

Proj. #	Name and Location	Year of Construc.	ADT for 1985 (1)	Trucks % (1)	Rating Cond. for 1985 (2)	General Observations
1	Grande Ronde - Wallace Bridge, St.Hwy-18	1980	9500	12.5	very good	No significant cracking ravelling or shoving
2	Dayton - Lafayette Jct., St.Hwy-18	1980	4050	12.3	very good	No significant cracking ravelling or shoving
3	Idlywood Street, City of Salem	1974	n/a	n/a	good	No significant cracking ravelling or shoving
4	Elk River - North Port Orford, U.S.Hwy-101	1976	5100	13.9	fair	n/a
5	Plain View Rd. - Deschutes River, U.S.Hwy- 20	1980	3550	13.3	fair	5% raveled 5% cracked
6	Klamath Falls-Green Spring Jct., U.S.Hwy-97	1981	9600	36.8	good/fair	5% raveled 5% cracked
7	Arnold Ice Caves - Horse Ridge, U.S.Hwy- 20	1973	1350	14.8	fair	25% shoved 10% cracking
8	S. Fork Malheur - New Princeton, St.Hwy- 78	1974	190	5.0	poor	95% cracks, 5% spalling 5% ravelling

(1) Kim et al. (*3*).

(2) Unclassified publication of the Pavement Management Unit, OSHD, February 1985.

TABLE 2 CORES AND MIX PROPERTIES

Proj. (#)	Thickness (in)	Max. Sp. Grav.	Air Voids	A/C %	Asphalt Suplier	Mix Type	Mr (ksi)	Nf (1)
1	1.72	2.476	11.1	5.0	Chevron AR4000w	B-mix	862.00	80350
2	2.44	2.580	8.5	5.7	Chevron AR4000w	B-mix	1103.19	10005
3	1.91	2.459	11.8	5.9	Chevron AR4000	B-mix	771.87	276292
3a	-	-	-	-		-	-	-
4	1.44	2.421	5.0	7.0	Douglas AR4000	B-mix	281.94	-
5	1.41	2.497	8.3	5.8	Chevron AR4000	B-mix	568.97	42480
5s	1.83	2.484	9.0	5.6			703.78	129064
6	2.49	2.535	6.1	5.2	Witco AR2000	B-mix	1031.63	4112
7	1.55	2.444	4.3	6.7	Douglas 120/150p	B-mix	243.30	295241
7s	1.92	2.434	4.3	6.9			186.30	1876282
8	1.44	2.158	8.7	7.6	Shell AR2000	C-mix	621.94	87662

(1) Nf, calculated for 100 microstrains

Implementation of the Corbett-Swarbrick Procedure

This part of the project was reported extensively elsewhere (*1*). The Corbett-Swarbrick procedure (current ASTM D 4124) was submitted for revision by ASTM Committee D 04.47, and a new small-scale test was proposed. Although the proposed procedure is not yet an official standard, it was used in this study for the evaluation of asphalt composition. An early description was given by Corbett (*2*).

Laboratory Testing Program

Figure 2 shows a summary of the laboratory testing program. Four groups of tests were performed for all eight projects:

1. Physical properties of original samples and after the RTFO: Original properties of the asphalt were available from the date of construction, but, because the data were incomplete, these properties were measured again using original asphalt that had been stored in sealed cans at room temperature. The repetition of these tests also served to determine whether the stored asphalt underwent changes during the storage period.

Asphalt properties after RTFO were also available from the date of construction, but these data were also incomplete. The stored asphalt was artificially aged in the RTFO and tested for physical properties.

Table 3 gives the results obtained for original asphalts and after the RTFO along with the results already available from the date of construction.

2. Physical properties of core-recovered asphalt: Asphalt was extracted and recovered from the top and bottom layers of each core. The current procedure of the Oregon State Highway Division (OSHD) was used to obtain samples for all eight projects (Method A) (*4*). Three other methods were also used to extract and recover asphalt from Projects 3, 5, and 7. Figure 3 shows the general scheme of the extraction and recovery procedures used.

3. Asphalt chemical fractionation test results: The Corbett-Swarbrick procedure yields four distinct fractions: asphaltenes, saturates, naphthene aromatics, and polar aromatics. All of the results presented in the next section represent the average of two independent tests. Table 4 gives the standard deviation and range for each fraction obtained and the proposed criteria given by ASTM D 4124.

4. Fraass test results before and after POB aging test: This part of the research involved the use of Fraass samples, which were aged in a POB device for 2 and 5 days and subsequently tested for Fraass breaking point and fractional composition. The purpose of this part of the research was to assess the changes in fractional components of the asphalt after the POB aging test. The changes in composition were compared with

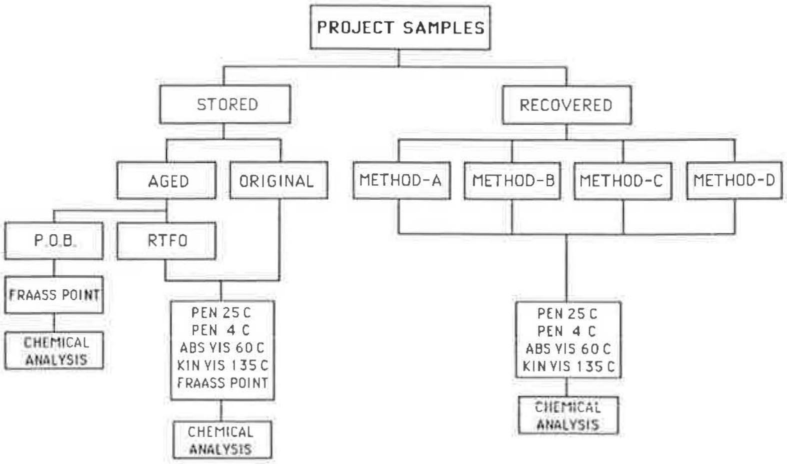

FIGURE 2 Laboratory research program.

TABLE 3 PHYSICAL PROPERTIES OF ORIGINAL AND RTFO SAMPLES

	Data Available from Date of Construction				Data Measured During 1985			
Sample	Pen-4	Pen-25	Vis-60 (poises)	KVis-135 (cStokes)	Pen-4	Pen-25	Vis-60 (poises)	KVis-135 (cStokes)
1-Orig.	18	73	1552	352	23	72	1783	364
2-Orig.	18	73	1552	352	26	77	1613	352
3-Orig.	50	139	-	-	47	128	1169	353
4-Orig.	49	134	1110	340	48	128	1124	335
5-Orig.	20	80	1504	368	22	74	1577	345
6-Orig.	17	85	1052	201	17	88	1059	190
7-Orig.	46	140	762	236	31	128	768	244
8-Orig.	25	100	-	-	15	84	992	190
1-RTFO	-	39	4191	572	16	43	4216	545
2-RTFO	-	39	4191	572	16	44	3960	526
3-RTFO	-	66	4306	608	30	60	4592	665
4-RTFO	-	65	4344	633	32	65	4193	619
5-RTFO	-	46	3858	494	20	52	3858	513
6-RTFO	-	66	1876	255	15	66	1678	247
7-RTFO	-	-	2164	-	30	66	2524	393
8-RTFO	-	60	2051	260	14	54	2068	267

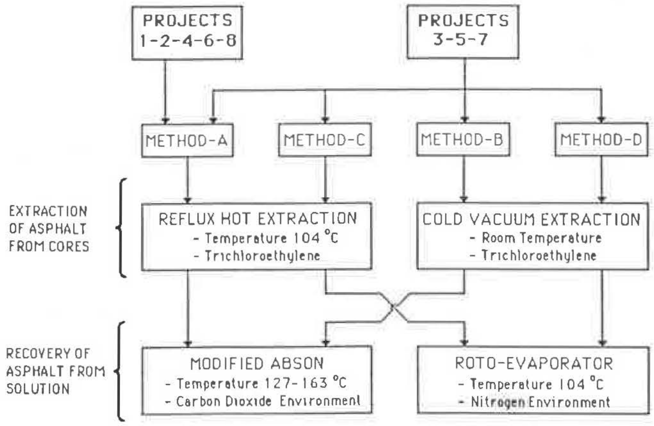

FIGURE 3 Asphalt extraction and recovery procedures.

changes in Fraass temperature and with changes after the RTFO. Only Projects 3, 5, and 7 were used for these tests. The use of the Fraass test sample for aging studies and its advantages are reported elsewhere (*5, 6*). The characteristics of the POB device were reported by Kim et al. (*3*). The conditions for the test were as follows: 100 psi oxygen pressure, 60°C (140°F), and 2 and 5 days of aging.

Because the amount of materials obtained from the aged Fraass sample is relatively small, only one physical property was measured (Fraass brittle point), and the fractional analysis was run only once.

Asphalt Property Indices

To correlate chemical composition analysis with temperature susceptibility, the following indices were calculated:

- Penetration index (*PI*) (*7, 8*):

$$PI = (20 - 50A)/(1 + 50A) \qquad (1)$$

where

A = $[\log P(T1) - \log P(T2)]/(T1 - T2)$,
$T1$ = 25°C,
$T2$ = 60°C,
$P(T1)$ = penetration measured at 25°C, and
$P(T2)$ = penetration calculated at 60°C using the following relationship:

$$P(T2) = \{[-5.42 \log (V60/13{,}000)]/[8.5 + \log (V60/13{,}000)]\} - \log 800 \qquad (2)$$

TABLE 4 REPEATABILITY OF RESULTS FOR INDIVIDUAL CHEMICAL FRACTIONS

Fractions	Actual Testing		ASTM Criteria			
	Single Operator		Single Operator		Multi Laboratory Precision	
	Standard Deviation	Range	Standard Deviation	Range	Standard Deviation	Range
Asphaltenes	0.40	1.1	0.32	0.9	0.95	2.7
Saturates	0.31	1.1	0.44	1.2	0.70	1.9
N-Aromatics	0.88	2.1	1.03	2.9	2.26	6.4
P-Aromatics	0.53	1.6	0.78	2.2	2.37	6.7

where $V60$ = absolute viscosity.

Large negative values of *PI* indicate greater temperature susceptibility. Typical asphalts have values between +2 and −2.

- Viscosity temperature susceptibility (*VTS*) (*9*):

$$VTS = [\log \log V(T2) - \log \log V(T1)]/(\log T1 - \log T2) \quad (3)$$

where

$T1$ = 333 K (60°C),
$T2$ = 408 K (135°F),
$V(T1)$ = absolute viscosity at 60°C in poises, and
$V(T2)$ = kinematic viscosity at 135°C in poises.

where 1 cSt $*$ (0.95/100) $\approx$ 1 poise. Greater VTS indicates greater temperature susceptibility.

- Penetration viscosity number (*PVN*) (*10*):

$$PVN = [(4.258 - 0.7967 \log P25 - \log KVis)/(0.7951 - 0.1858 \log P25)] * (-1.5) \quad (4)$$

where $P25$ is penetration at 25°C, and $KVis$ is kinematic viscosity at 135°C in centistokes. Lower *PVN* indicates greater temperature susceptibility.

- Penetration ratio (*PR*):

$$PR = (\text{Pen @ 4°C, 200 g, 60 sec})/(\text{Pen @ 25°C, 100 g, 5 sec}) \quad (5)$$

Lower *PR* indicates greater temperature susceptibility.

DISCUSSION OF RESULTS

Data from Date of Construction Versus Data from Stored Asphalt

Table 3 gives physical properties of original samples before and after the storage period. Some minor variations in physical properties were noticed. These are attributed to variation in testing over a period of 5 to 11 years, not to any aging that may have occurred during the storage period. By looking at changes in penetration at 25°C, and absolute and kinematic viscosity, it can be observed that there is no general trend in the changes undergone by each asphalt sample (i.e., some tests indicate hardening and others softening or no changes at all). The average variation was around 5 percent in both directions (hardening or softening).

Relationship Among Chemical Fractions

Six possible relationships among the four fractions obtained with the Corbett-Swarbrick analysis were studied. Figures 4–9 show these relationships and include results from original samples, RTFO, and recovered asphalt using Method A.

Initially, the relationships studied were to be based only on the data for original samples. Some trends were noticed in these data, but they were insufficient to use to extrapolate beyond the range shown by the type of asphalt used. To increase the range variation, the data from the RTFO aging test were included, and more clear trends were observed. Plotting the results of original samples together with the RTFO results caused the range of variation of chemical fractions to increase, and the trends found when plotting original results alone were improved.

Results from recovered asphalt, using Method A, cover an even larger range of asphalt fraction proportions. These results were also added to the analysis, but, as seen from Figures 4–9, a different trend is observed for the recovered asphalt than for the original plus RTFO samples.

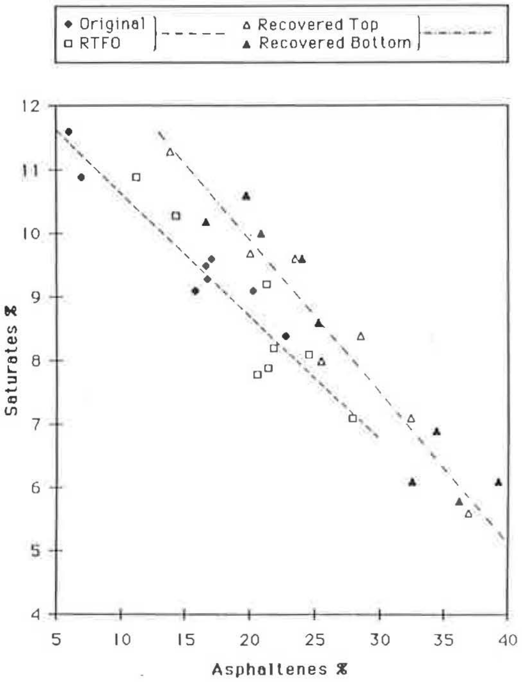

FIGURE 4 Saturates versus asphaltenes.

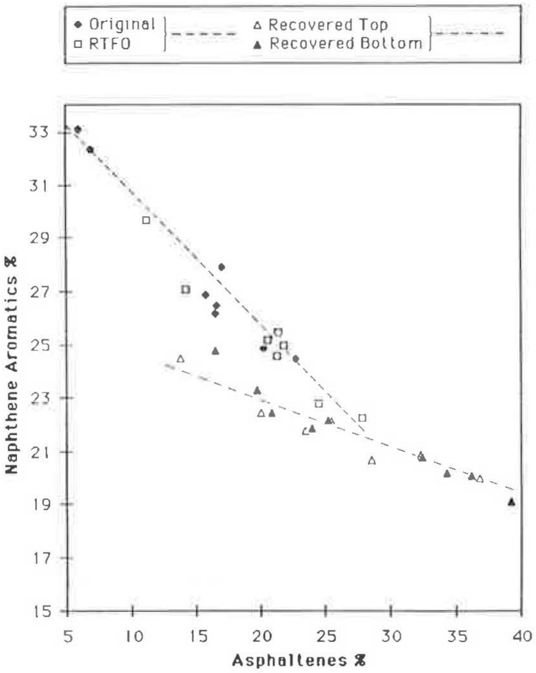

FIGURE 5 Naphthene aromatics versus asphaltenes.

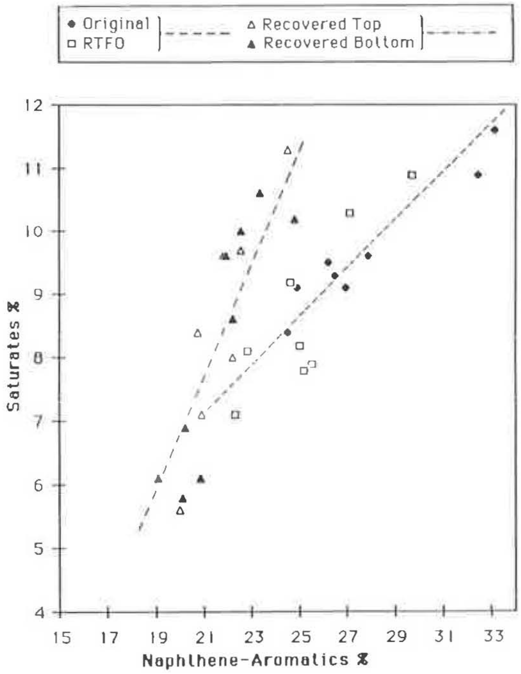

FIGURE 7 Saturates versus naphthene aromatics.

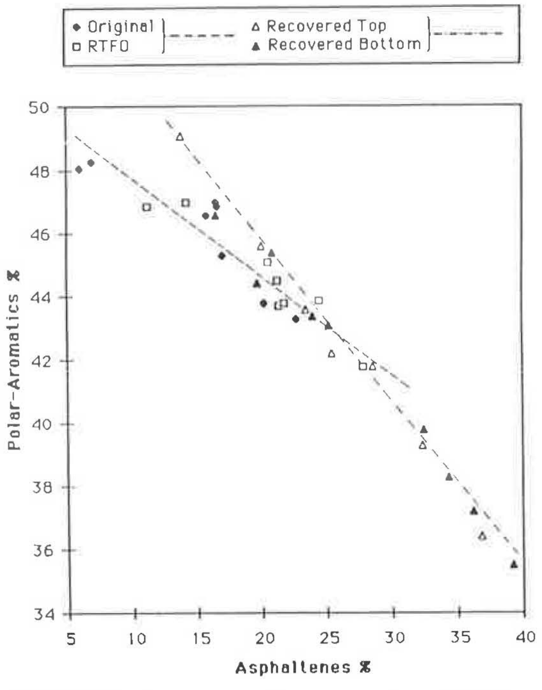

FIGURE 6 Polar aromatics versus asphaltenes.

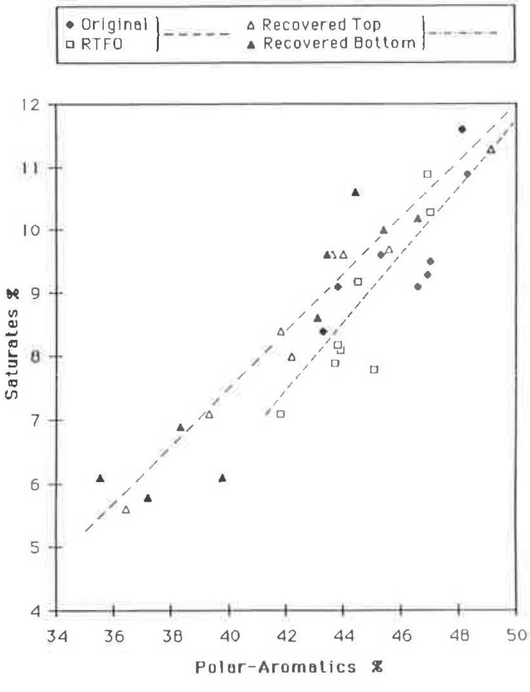

FIGURE 8 Saturates versus polar aromatics.

That recovered asphalt does not show the same relationships that were observed for original plus RTFO samples may be explained as follows:

- Recovered asphalt, after going through the extraction and recovery procedure, may be chemically altered and no longer represent the in-place asphalt.
- The RTFO–accelerated aging procedure does not duplicate the chemical changes undergone by asphalt under natural weathering. The RTFO was designed to simulate aging during construction, not field aging.

On the basis of just the results of this research study, it is difficult to determine how these two factors contribute to the differences in relationships observed in Figures 4–9.

For either the original plus RTFO or the recovered asphalt, relatively good linear relationships were observed between any two chemical fractions. Linear regression equations in which

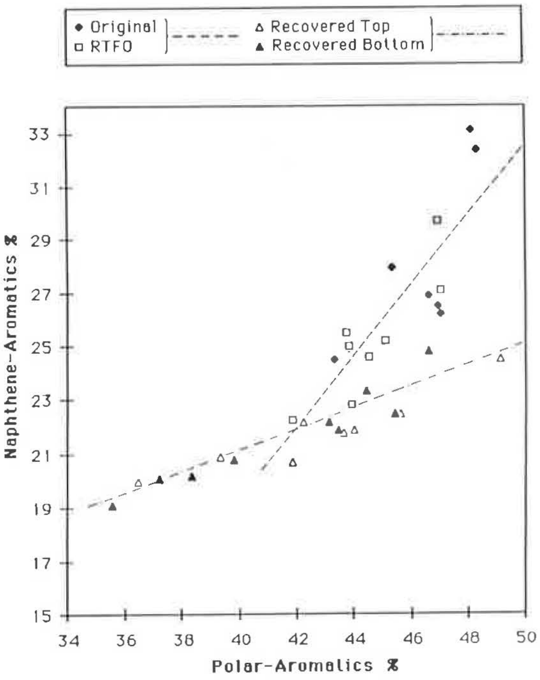

FIGURE 9 Naphthene aromatics versus polar aromatics.

the proportion of one component is expressed as a function of the other are given in Table 5. It can be observed that the relatively good relationships (R^2 greater than 0.8) are those in which the asphaltenes are the independent variable and the saturates, naphthene aromatics, and polar aromatics are the dependent variables, respectively. Because the asphaltenes are the first components obtained during fractionation (before chromatographic analysis), if the percentage of asphaltenes is known the proportion of the other three fractions may be estimated.

The relationships given in Table 5 were obtained from a relatively small population (i.e., the original asphalt samples obtained from the eight projects were from five different suppliers, and three of the projects constructed in 1980 used asphalt from the same supplier). The relationships obtained for recovered asphalt may be considered representative of a larger sample because the recovered asphalt represents samples aged under 22 different environmental and traffic conditions (i.e., samples from eight different environments, from the road surface and bottom layer, from travel lanes, from shoulders, and so forth).

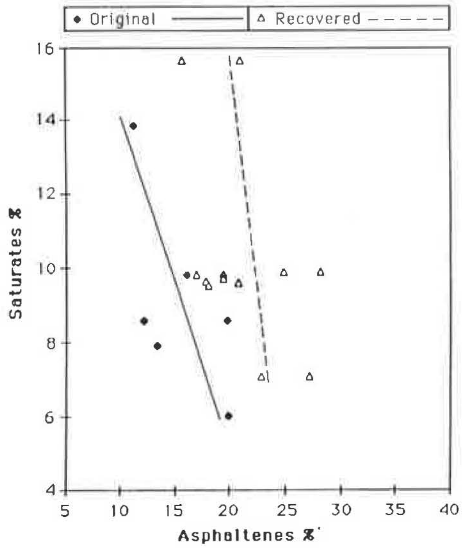

FIGURE 10 Saturates versus asphaltenes in the Michigan Road Test.

Comparison with Other Studies—Relationship Among Chemical Fractions

To determine whether the equations obtained (Table 4) can be extrapolated to other asphalts, results from the Michigan Road Test (*11*) were analyzed. This was the first large-scale program in which the Corbett-Swarbrick analysis was used to characterize asphalt. Figures 10–12 show selected relationships and that, although results from the Michigan Road Test deviate from the relationships developed in this study (Table 4), the general trends are similar. One reason for the deviation may be that the multilaboratory precision range is quite high (ASTM D 4124); these values are given in Table 3. A second reason for some of the large deviations could be that some asphalts may exhibit inexplicable anomalies during repeated trials of the test. This was true when original samples from Project 7 were used, and similar cases have been reported in the literature (*12*).

Recovered asphalt data (*11*) are closer to the trends given by the regression equations than are the original asphalt data. Nevertheless, some deviations were expected because the Abson asphalt recovery procedure used in the present research is a modified version of the original ASTM procedure used in the Michigan Road Test.

One of the objectives of the Michigan Road Test was to relate compositional changes to pavement durability (wear and

TABLE 5 RELATIONSHIP EQUATIONS FOR CHEMICAL FRACTIONS

	Relationship	Original + RTFO			Recover by Method-A	
		Linear Relation	R	Range (+ or -)	Linear Relation	R
1	Saturates vs Asphaltenes	%SA = 12.69-0.197%Asp	0.87	0.93	%SA = 14.49-0.231%Asp	0.89
2	N-Aromatics vs Asphaltenes	%NA = 35.24-0.489%Asp	0.95	2.64	%NA = 27.46-0.206%Asp	0.81
3	P-Aromatics vs Asphaltenes	%PA = 50.65-0.297%Asp	0.87	1.59	%PA = 55.46-0.506%Asp	0.97
4	Saturates vs N-Aromatics	%SA = - 0.78+0.376%NA	0.81	-	%SA = - 9.42+0.808%NA	0.58
5	Saturates vs P-Aromatics	%SA = - 16.63+0.569%PA	0.75	-	%SA = -10.39+0.446%PA	0.87
6	N-Aromatics vs P-Aromatics	%NA = - 34.35+1.341%PA	0.72	-	%NA = - 6.05+0.379%PA	0.72

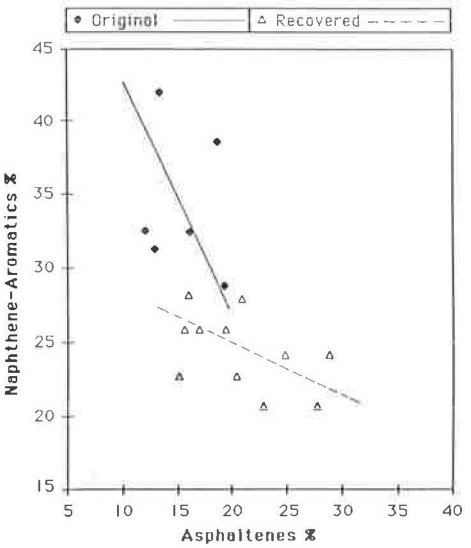

FIGURE 11 Naphthene aromatics versus asphaltenes in the Michigan Road Test.

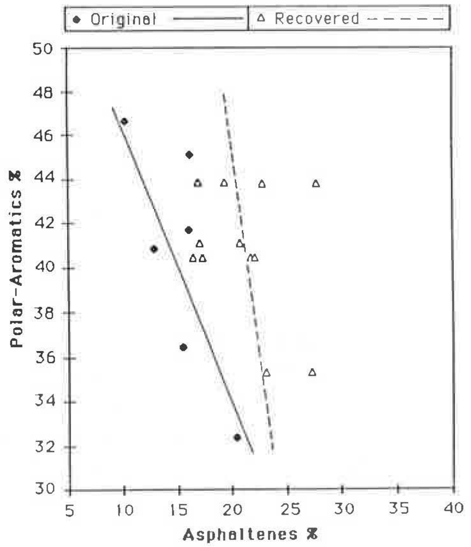

FIGURE 12 Polar aromatics versus asphaltenes in the Michigan Road Test.

weathering qualities). The test was conducted on a 6-mi test section where meticulous care was exercised in controlling mix and construction variables (e.g., aggregate gradation, binder content, temperatures, placing and compaction, and so forth). Although considered a well-controlled experiment, the Michigan test did not result in any definition of a "desirable" asphalt in terms of fractional composition.

Relationship Between Chemical Composition and Physical Properties

Four physical properties of all samples were measured: penetration at 40°C (ASTM D 5, AASHTO T49), penetration at

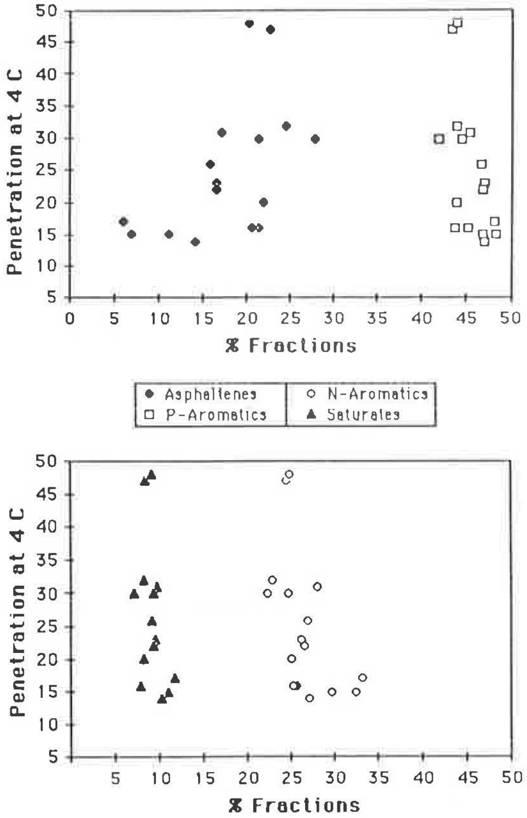

FIGURE 13 Penetration at 4°C versus chemical fractions: original and RTFO samples.

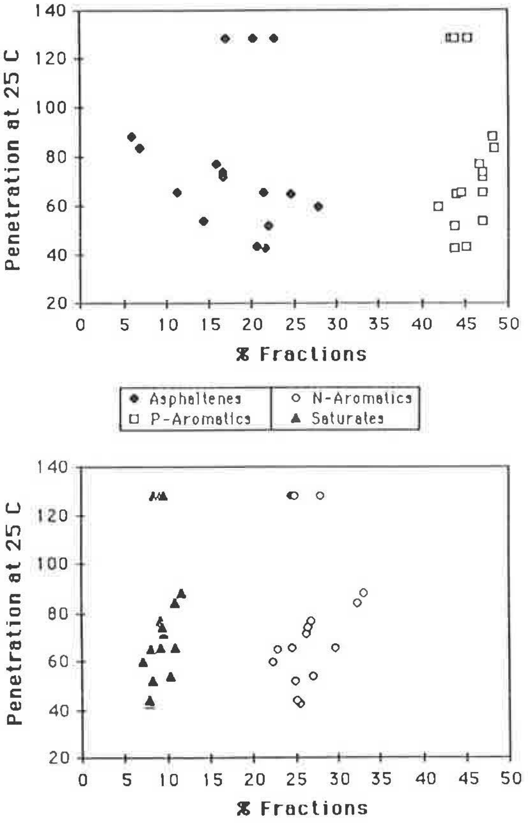

FIGURE 14 Penetration at 25°C versus chemical fractions: original and RTFO samples.

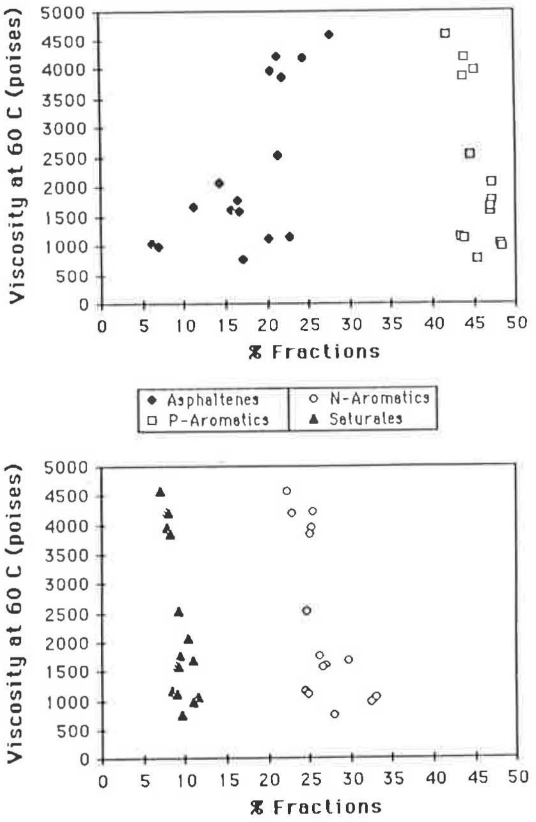

FIGURE 15 Viscosity at 60°C versus chemical fractions: original and RTFO samples.

25°C (ASTM D 5, AASHTO T49), absolute viscosity at 60°C (ASTM D 2171, AASHTO T202), and kinematic viscosity at 135°C (ASTM D 2170, AASHTO T201).

With the amount of data gathered during the research, it was possible to look for relationships between individual chemical fractions and each of the physical properties measured. The study of these relationships was done with two different groups: original samples combined with RTFO samples (Figures 13–16) and recovered asphalt using Method A (Figures 17–20).

Analysis indicates that the compositional profile of recovered asphalt differs from that of original or RTFO samples, or both (Figures 4–9). For this reason, the study of relationships between physical properties and chemical fractions should be treated separately.

Penetration at 4°C Versus Chemical Composition

Figure 13 shows the relationships for original and RTFO samples. Figure 17 shows the relationships for recovered asphalt. The approximate vertical distribution of the data points for saturates, naphthene aromatics, and polar aromatics indicates that penetration at 4°C is independent of the percentage of these three fractions or quite sensitive to changes in the percentage of any of these fractions. The variable distribution of the asphaltene fraction may indicate the following two effects:

1. The asphaltene fraction has an impact on penetration at 4°C, but the significant scatter in the data suggests that some other physicochemical property of the asphaltenes may be significant.
2. The test for penetration, in general, may not be sensitive enough, or, because of its empirical nature, the test may not measure the effect of other variables such as shear rate, shear stresses, and changes in volume (*13*). It is possible that both of these are true.

Penetration at 25°C Versus Chemical Composition

Figure 14 shows the relationships for original and RTFO samples. Figure 18 shows the relationships for recovered asphalt. The relationships between chemical fractions and penetration at 25°C were found to be similar to those for penetration at 4°C.

Although the relationships for penetration (at both 4°C and 25°C) versus chemical fractions look similar for both groups of samples (original plus RTFO and recovered asphalt), it was observed that the naphthene aromatics and the polar aromatics showed "opposite" behavior in both groups of samples (i.e., for original plus RTFO samples the naphthene aromatics show a larger variability than do the polar aromatics). For recovered asphalt larger variability was found for the polar aromatics than for the naphthene aromatics.

The observed phenomena indicate again that the recovered asphalt may not necessarily represent the material that was in

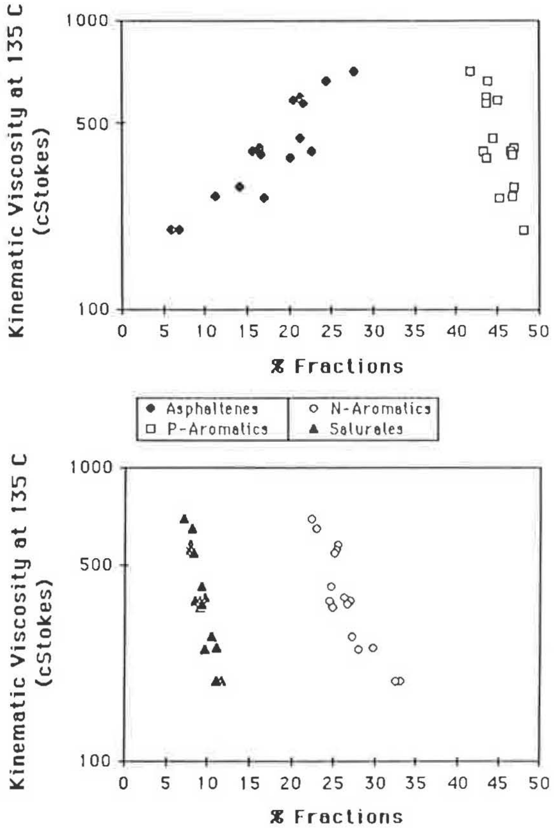

FIGURE 16 Kinematic viscosity versus chemical fractions: original and RTFO samples.

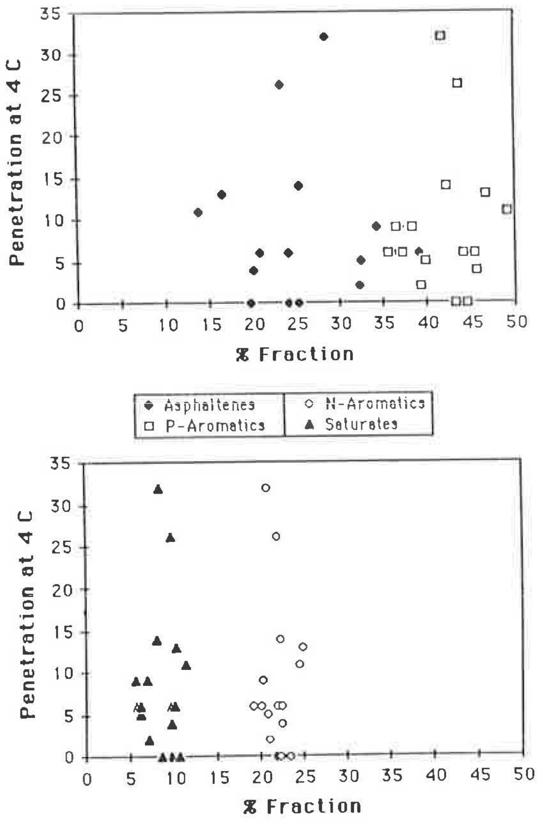

FIGURE 17 Penetration at 4°C versus chemical fractions: Recovery Method A.

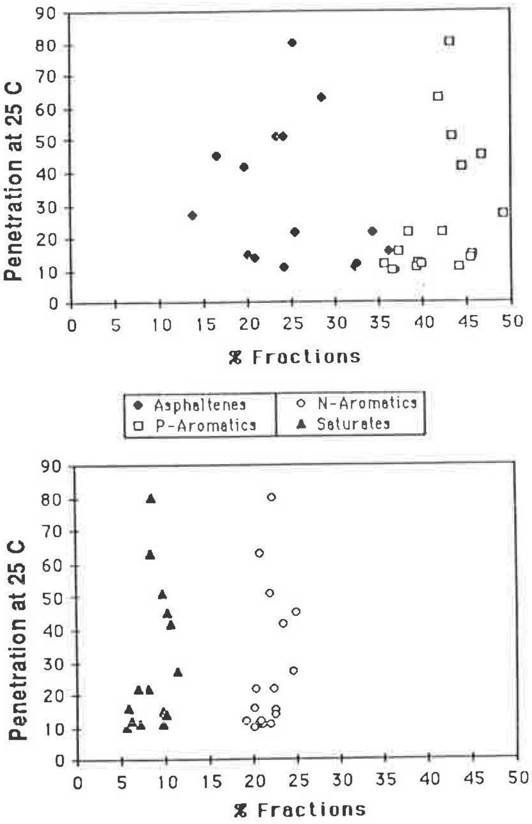

FIGURE 18 Penetration at 25°C versus chemical fractions: Recovery Method A.

place in the road. The same reasoning given when discussing relations among fractions may be applied here to partly explain these differences in behavior between recovered asphalt and original asphalt.

Absolute Viscosity at 60°C Versus Chemical Composition

Figure 15 shows the relationships for original and RTFO samples. Figure 19 shows the relationships for recovered asphalt. The relationships for viscosity at 60°C look quite similar to those observed for penetration, but there are more noticeable trends.

The relationship for asphaltenes is more pronounced than it is for the penetration tests. Viscosity at 60°C shows some type of dependency on the percentage concentration of the asphaltene fraction, but with large variability in the lower viscosity range. This variability may be attributed to the capillary viscometer because recording lower viscosity values manually may be subject to more imprecision than is present in the higher range of viscosity.

The general trend for the relationship between viscosity at 60°C and asphaltenes indicates that the higher the asphaltene content the higher the viscosity. For the other three fractions the relationship is opposite.

Comparison of results for original plus RTFO samples with those for recovered asphalt indicates that the relationships are similar. The results are also similar to those found with the penetration data (i.e., the naphthene aromatic fraction shows a larger variability for original samples than for recovered asphalt and vice versa for the polar-aromatics fraction.

Kinematic Viscosity Versus Chemical Composition

Figure 16 shows the relationships for original plus RTFO samples. Figure 20 shows the relationships for recovered asphalt. Both figures show the kinematic viscosity axis in logarithmic scale. The logarithmic viscosity at 135°C exhibits a good relationship with all four fractions for both original asphalt and recovered asphalt. The greater the percentage content of asphaltenes and the lower the percentage content of the other three fractions, the higher the viscosity at 135°C.

The reason for the better relationship between a physical flow property measured in the higher temperature range (viscosity at 135°C) and chemical fractions may be explained by the following extract from a paper by Petersen (*14*):

> At higher temperatures (Newtonian flow region) the polar interactions between molecules dominate in influencing the flow behavior and the effects of molecular shape or geometry are minimized. At lower temperatures, the kinetic energy of the molecules is lowered and the molecules tend to associate or agglomerate into immobilized entities with a more or less ordered spatial arrangement which is influenced by the geometry of the molecule and its polar functionality.

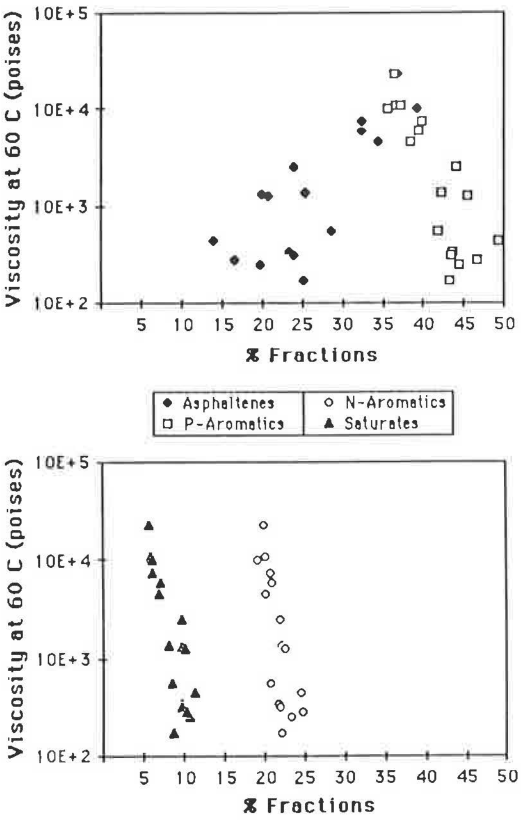

FIGURE 19 Viscosity at 60°C versus chemical fractions: Recovery Method A.

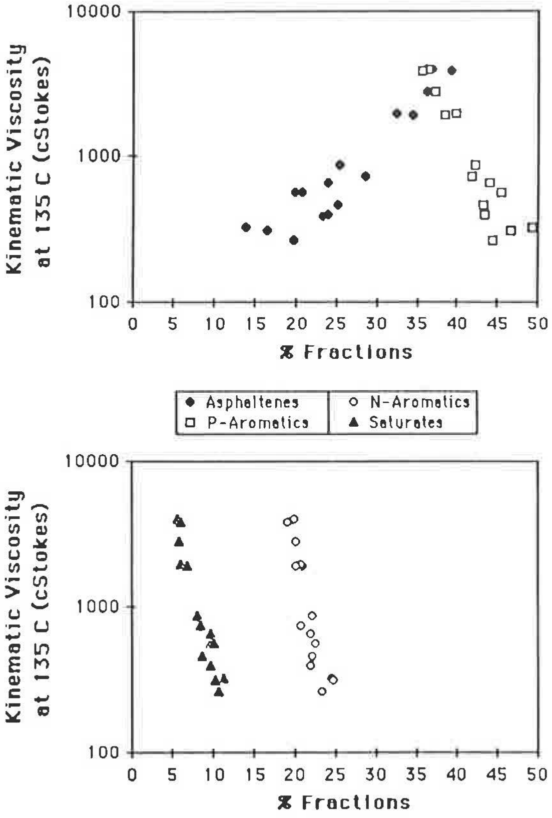

FIGURE 20 Kinematic viscosity versus chemical fractions: Recovery Method A.

Thus, at lower temperatures, the flow property of asphalt may be influenced not only by the percentage concentration of certain types of molecules but also by their polar functionality, spatial arrangement, and geometry.

Relationship Between Chemical Composition and Temperature Susceptibility

Temperature susceptibility can be defined as the rate of change of viscosity (or another measure of asphalt consistency) with temperature and is dependent on the temperature range considered. The influence of temperature susceptibility on pavement construction and performance was addressed in a significant study conducted by Button et al. (*12*).

As was the evaluation of composition versus physical properties, that for composition versus temperature susceptibility was done with two separate groups, original samples combined with RTFO samples (Figures 21–24) and recovered asphalt using Method A (Figures 25–28).

Penetration Index Versus Chemical Composition

Penetration index (PI) values were calculated using penetration at 25°C (Pen 25) and viscosity at 60°C (Vis 60) (*15*). Figure 22 shows the relation for original and RTFO samples and Figure 26 shows the relation for recovered asphalt. It appears that combining Pen 25 and Vis 60 into one index improves the relationships for each of the four fractions. Pen 25 did not show

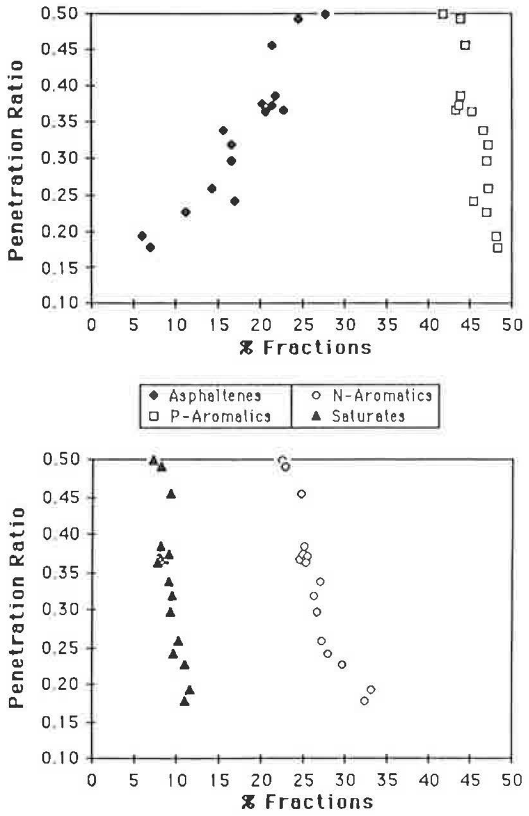

FIGURE 21 Penetration ratio versus chemical fractions: original and RTFO samples.

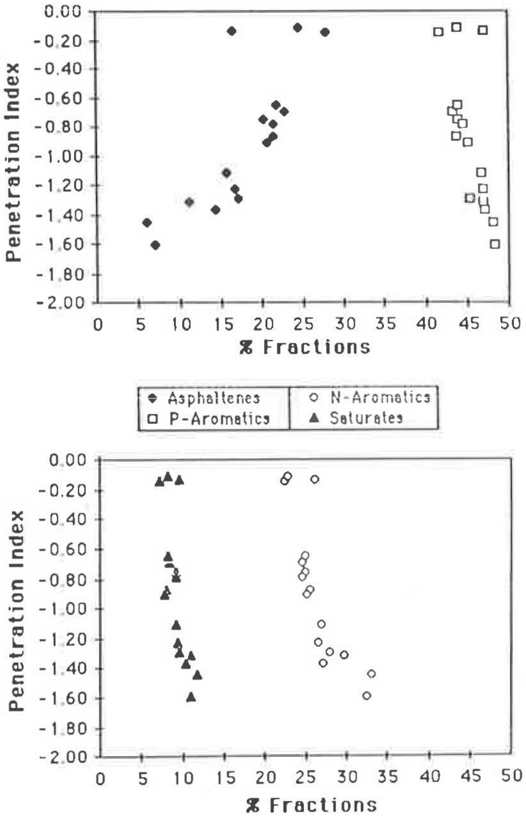

FIGURE 22 PI versus chemical fractions: original and RTFO samples.

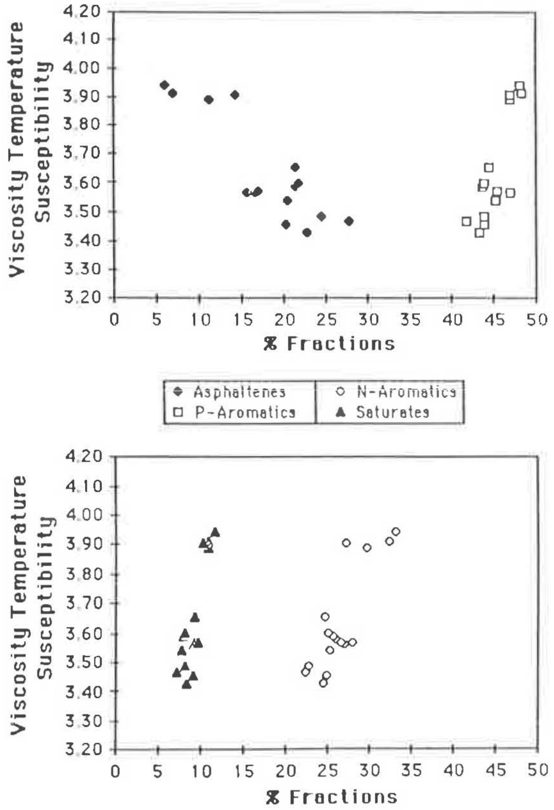

FIGURE 23 Viscosity temperature susceptibility versus chemical fractions: original and RTFO samples.

a good relationship with asphaltene content, but the PI shows a clear dependency on the percentage content of asphaltenes.

Viscosity Temperature Susceptibility Versus Chemical Fractions

The viscosity temperature susceptibility (VTS) parameter was obtained by using the viscosity values measured at 60°C and 135°C. Figure 23 shows the relation for original and RTFO samples, and Figure 27 shows the relation for recovered asphalt.

For both original and RTFO samples there is a correlation between VTS and asphaltene content, although the data are scattered. The percentage content of the other three fractions shows little deviation with changes in VTS-values for both original and recovered asphalt. Although there was some correlation between viscosity at 60°C and 135°C and each of the four generic fractions, it appears that the VTS within this temperature range is not clearly dependent on fractional composition.

Penetration Ratio Versus Chemical Fractions

Penetration ratio (PR) relationships are shown in Figure 21 for original asphalt and Figure 25 for recovered asphalt. The PR parameter used here measures temperature susceptibility of asphalt at 4°C and 25°C.

There is a clear correlation between all four chemical fractions and PR for original and RTFO samples, but poor

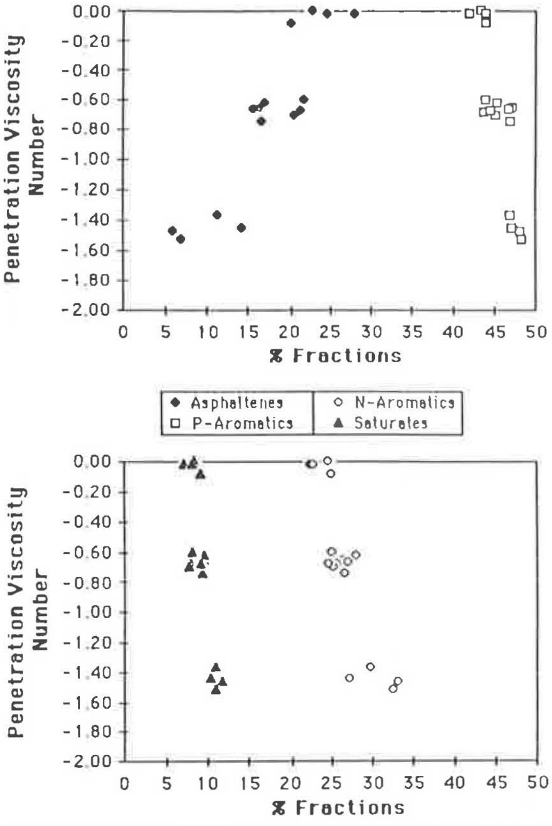

FIGURE 24 Penetration viscosity number versus chemical fractions: original and RTFO samples.

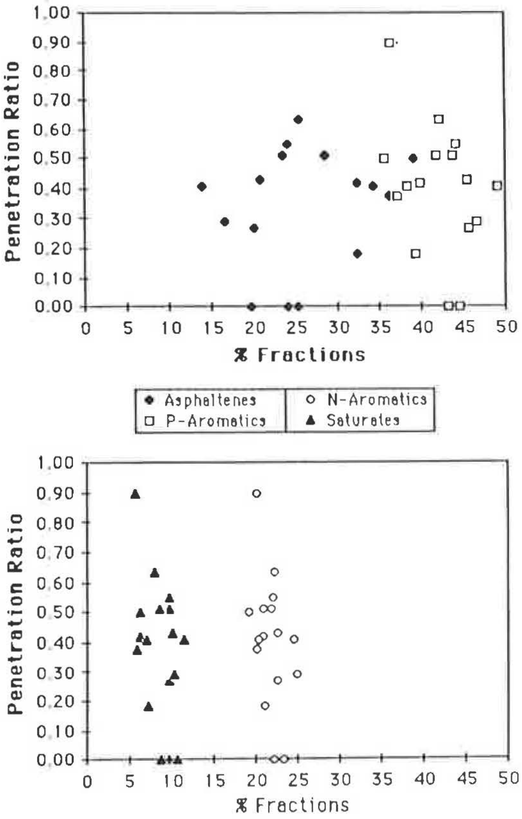

FIGURE 25 Penetration ratio versus chemical fractions: Recovery Method A.

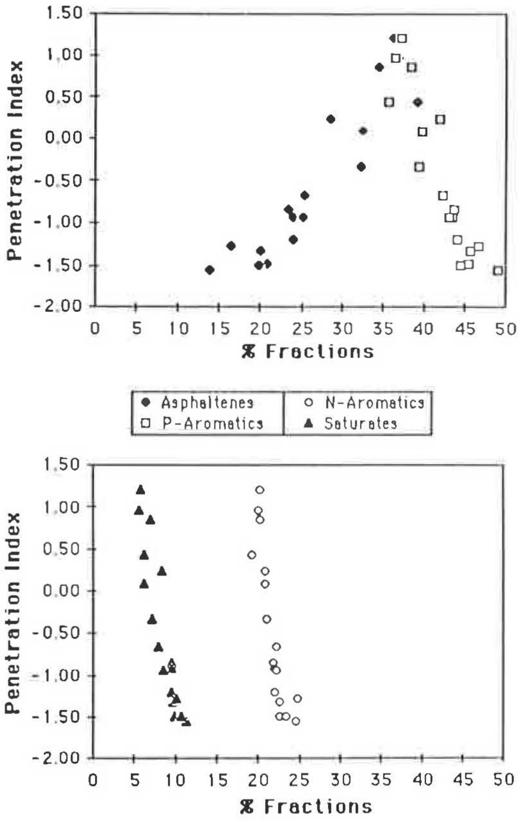

FIGURE 26 PI versus chemical fractions: Recovery Method A.

correlation was observed for recovered asphalt. This big difference among both groups of samples may be explained partly by the arguments given earlier for relationships between fractions. However, the differences may also be due to the recovery procedure during which some kinds of preferential molecular arrangements are destroyed, which would reduce any chance of common behavior within the range of temperature within which penetration values were measured.

Penetration Viscosity Number Versus Chemical Composition

The penetration viscosity number (PVN) parameter is calculated from the penetration value measured at 25°C and the kinematic viscosity measured at 135°C. Figure 24 shows the relation for original plus RTFO samples, and Figure 28 shows the relation for recovered asphalt. PVN covers a larger range of temperatures in comparison with the other three parameters analyzed, and within this temperature range asphalt materials exhibit a wide range of consistency. Thus poor relations were expected between PVN-values and fractional components.

Although, as seen in Figures 24 and 28, there was a poor relation for the PVN-values, the relations with the PI-values determined using a narrower temperature range were similarly bad. The authors have no reasonable explanation for this phenomenon other than to remind the reader that both of these temperature susceptibility parameters (PI and PVN) are based on empirical relationships among the different physical properties and are therefore not fundamental descriptions of material behavior.

Comparison with Other Research—Relationship Between Chemical Composition and Temperature Susceptibility

Values of asphaltene content and temperature susceptibility for 70 asphalts were tabulated by Anderson and Dukatz (*16*) and later plotted by Button et al. (*12*). The temperature susceptibility parameters used in the study were penetration index, viscosity temperature susceptibility, and penetration viscosity number. Button et al. did not observe any relation between any temperature susceptibility parameter and asphaltene content. The differences between the present study and that of Button et al. may be explained as follows:

1. The asphaltene fraction reported by Button et al. was obtained using the Rostler analysis (ASTM D 2006) in which the asphaltene has been precipitated in *n*-pentane. The asphaltene fraction measured in this study, using the Corbett-Swarbrick procedure (ASTM D 4124), was precipitated in *n*-heptane. The amounts of asphaltenes precipitated with each of these two procedures are different (*17*).
2. Button et al. (*12*) plotted all 70 asphalts in one figure in which laboratory asphalt and field asphalt are combined as one set of data. As discussed earlier, field asphalt shows a quite different chemical profile than does "original" asphalt and thus should be treated separately.
3. This research study used a limited number of asphalts, and the data range was artificially increased by using laboratory-aged asphalt with original materials. Nevertheless,

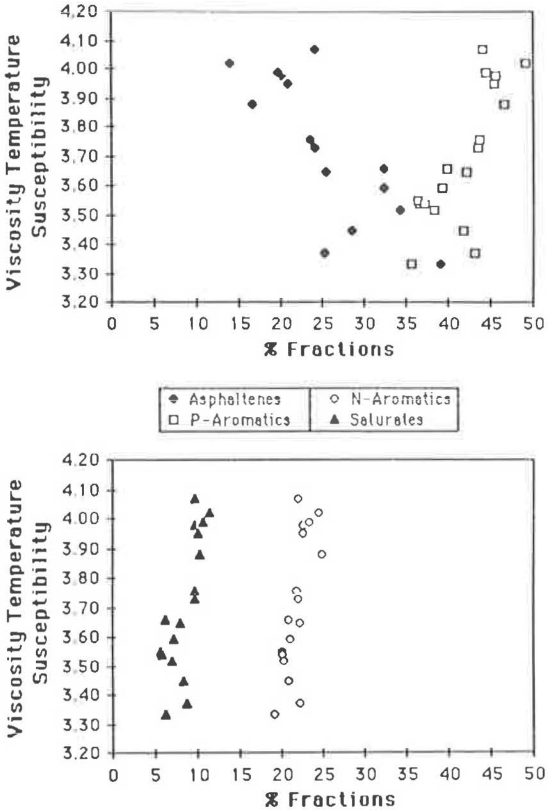

FIGURE 27 Viscosity temperature susceptibility versus chemical fractions: Recovery Method A.

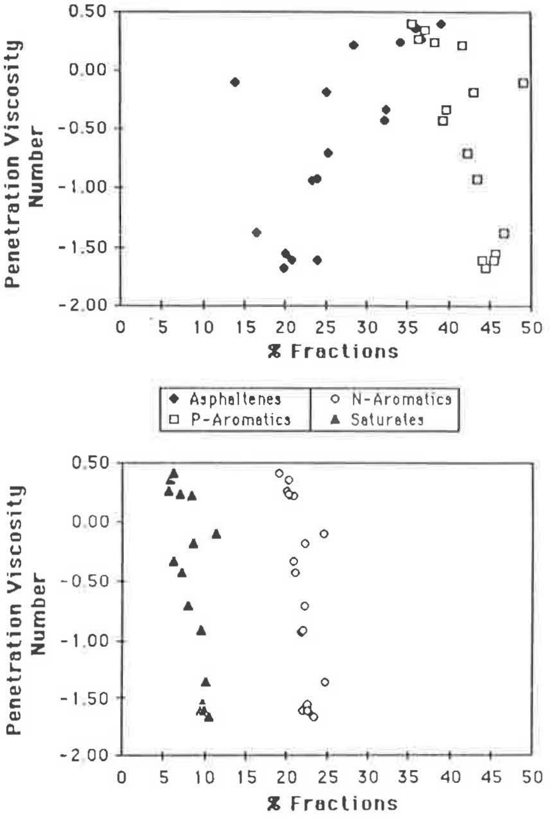

FIGURE 28 Penetration viscosity number versus chemical fractions: Recovery Method A.

this was not done with the field asphalt, and the same kind of correlation was found.

Response of Individual Asphalts

Up to this point, the asphalt analyses of all eight projects have been grouped as one set of data. This permits, to a certain extent, generalization of some of the findings. However, each asphalt used may be studied independently to determine how the aging effect (as measured by increases in hardening) relates to asphalt composition.

For each asphalt sample there are four data points that may be considered: for the original sample, for the sample after RTFO, and for samples recovered from the top and bottom of the core. Unfortunately, the number of points is too small to allow the use of some statistical tools. Thus a descriptive analysis was done to complement the discussion presented previously.

To examine the relationship of each chemical fraction with all of the physical properties and temperature susceptibility parameters used in the present study for all eight projects, 256 plots were created and analyzed. These plots are not included here but will be discussed. The following general observations may be made:

1. All asphalt experienced changes in physical properties and fractional composition with aging. However, asphalts from Projects 2, 4, and 5 experienced relatively small changes in composition, but their physical properties showed significant changes. Asphalt from Project 6 showed the opposite behavior: relatively small changes in physical properties but significant changes in composition. Asphalts from Projects 1, 3, 7, and 8 underwent significant changes in physical properties and composition.
2. For all eight projects, the proportion of asphaltenes increased with consistency as measured by penetration at 4°C and 25°C and viscosity at 60°C and 135°C. The proportions of the other three fractions were reduced.
3. The temperature susceptibility parameters showed quite distinct behavior for all eight asphalts and all four parameters used. Asphalt Samples 1, 3, and 5 showed no variation in VTS and PVN with asphalt composition whereas PR and PI showed significant variations. Asphalt Samples 2, 4, 6, and 8 showed erratic behavior in all four temperature susceptibility parameters. Sample 7 showed some correlation between fractions and all four temperature susceptibility parameters.

From this analysis, the only observation that can be made is that different asphalts behave differently and age differently. The different behavior shown by all of the samples of original and aged material suggests that, to better characterize asphalt properties after aging, more than one aging condition should be studied. For example, asphalt samples could be aged at three or four different RTFO conditions, and, after physical or fractional properties, or both, were measured, the rate of change in measured properties of the different asphalts could be compared. Measuring absolute changes of asphalt properties based on one aging condition may not reflect overall aging behavior.

Comparison of Recovered Asphalt Using Four Different Extraction Procedures

Four methods were used to extract and recover asphalt samples from cores from Projects 3, 5, and 7. The laboratory procedures for these four methods (A, B, C, and D) are summarized in Figure 3. The physical properties and composition analysis of these samples are summarized in Figure 29, which shows that the four methods of extraction and recovery did not give consistent results. The following factors contributed to the differences:

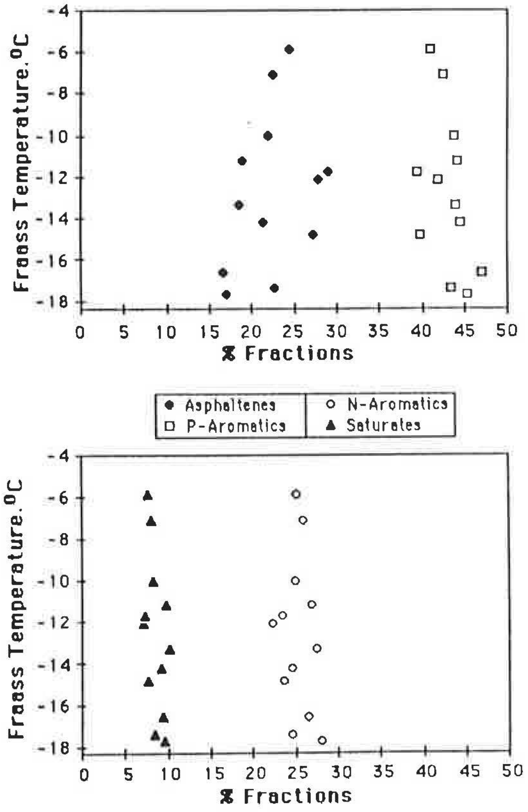

FIGURE 29 Fraass brittle point versus chemical fractions: Projects 3, 5, and 7.

1. The extraction procedure of Methods A and C uses high temperatures (104°C) for a relatively long period of time (as long as 2 hr); for Methods B and D the extraction procedure is done at room temperature. Another difference between the two major extraction procedures is in the filtering devices used. However, the samples are centrifuged in all four methods to decant fine particles that are not filtered properly.
2. The recovery procedure for Methods A and B uses a different gas environment during solvent recovery than is used in Methods C and D. Methods A and B use carbon dioxide at a rate of 2000 mL/min whereas Methods C and D use nitrogen at an unspecified rate. These differences are important because, from OSHD experience, it has been observed that variation of the flow rate of the gas used causes differences in asphalt extracted from the same cores.
3. A third difference to be considered in the present analysis is related to the familiarity of the laboratory technicians with the procedures used. Method A is the only procedure that has been used in routine work for a number of years; the other three methods were used for the first time in this research study.

Analysis of Fraass Test Results and POB Aging Test

This part of the study constitutes an extension of the overall objectives of the research. The POB test was used with the RTFO test to produce accelerated aging of asphalt binders. The POB device was used in conjunction with the Fraass test to evaluate oxidative aging. Samples were prepared on Fraass plates and tested for Fraass breaking point (Institute of Petroleum IP-80/53) before and after aging. Changes in Fraass temperature and in fractional composition were analyzed. It should be noted that the POB test ages asphalt in an oxygen-rich environment in an attempt to simulate long-term aging, whereas the RTFO uses high temperature and simulates short-term construction effects.

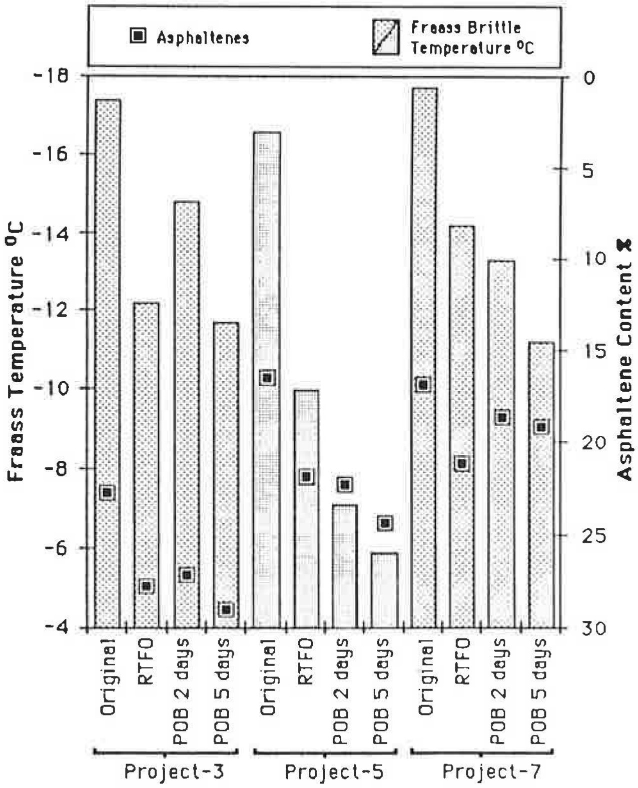

FIGURE 30 Comparison of results of POB test.

Figure 30 shows the relationships between Fraass temperature and all four fractional components for results from three projects studied (Projects 3, 5, and 7) before and after aging. Figure 30 shows that the asphaltenes are the only fraction that has a relationship with Fraass temperature; the other three fractions are more independent. Figure 31 shows similar relationships but is arranged so that each project can be analyzed separately and the effects of each of the aging procedures used can be compared. On the basis of the three samples used (small sample size), the following effects can be observed:

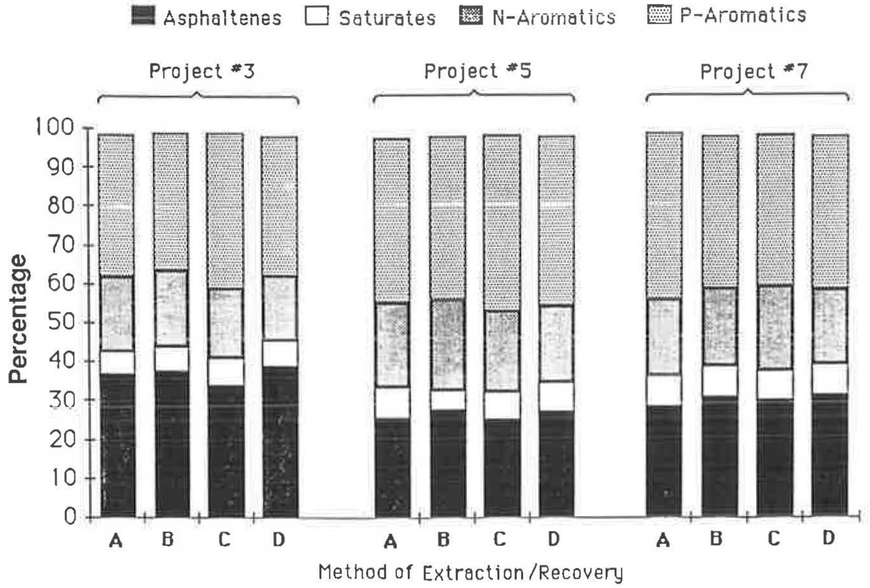

FIGURE 31 Comparison of four extraction and recovery procedures.

- The POB 5-day test was the most severe aging test; it caused much greater changes in composition than did the RTFO test.
- Asphaltene content increases with aging, but the initial asphaltene content of any of the original samples was not related to the total amount of aging after RTFO and POB tests.
- Project 5 was the most susceptible to aging based on laboratory "performance."

CONCLUSIONS

The following conclusions were drawn on the basis of the results of this study:

1. Asphalt samples stored in sealed cans at room temperature for periods of up to 12 years did not show significant variations in their physical properties. Minor variations did not give a clear indication that physical changes were due to aging, and these variations were attributed to the reproducibility of the test results.
2. The analysis of relationships between chemical components showed that recovered asphalt did not have the same profile as original and RTFO samples, which indicates that recovered asphalt, after going through the extraction and recovery procedure, may be chemically altered and no longer represent the in-place asphalt or that the RTFO aging test may not duplicate the changes in asphalt under natural weathering and in contact with mineral aggregates, or both.
3. All physical properties did show some correlation with all four fractions. Better correlations were found at higher temperatures (kinematic viscosity) than at lower temperatures (penetration at 4°C). This may be due to the effects of molecular shape and geometry, which are minimized in the higher temperature range, and to the effect of the testing procedures used at different temperatures.
4. Relatively good relations were found between fractional composition and temperature susceptibility. Better correlations were found for the PR and PI indices in the low temperature range than for VTS and PVN in the high temperature range. Regression analyses showed that the four indices used were distinctly different and that fractional composition had entirely different effects on all four.
5. A certain level of generalization about rheological and chemical behavior of original and aged asphalt was made possible by studying a relatively small group of asphalts. However, analysis of individual asphalts showed that different asphalts do behave differently and age differently. The different types of behavior shown by all of the samples of original and aged materials suggest that more than one aging condition should be studied. For example, asphalt samples should be aged at three or four different RTFO conditions, and the rate of changes in measured properties should be compared for the different asphalts. Measuring absolute changes of asphalt properties under one aging condition may not reflect overall aging behavior.
6. Asphalt extracted and recovered from cores showed a different compositional profile than did the original asphalt. Thus care is advised when using data from recovered and original asphalts together. The four methods used to extract and recover asphalt samples did not give consistent results, and both physical properties and composition measured after recovery were significantly different.
7. Insufficient data were gathered for meaningful conclusions about asphalt low-temperature behavior and its relation to asphalt composition. With the few data available (from Projects 3, 5, and 7), it was observed that, in general, asphalt composition did not show great dependency on the Fraass brittle temperature, which suggests that other molecular properties (e.g., molecular size, molecular structuring, and molecular geometry) may be more important than fractional composition in relation to low-temperature behavior. It was observed that asphaltene content increases with aging in a proportion that is relatively similar to the increase in Fraass temperature. However, the initial proportion of asphaltenes on all three projects was not related to the total amount of laboratory aging.

8. The POB test, in general, did not simulate field aging in terms of the percentage change of asphaltenes. However, the POB showed enough flexibility that it may be adjusted to simulate asphalt aging conditions of zones with different types of environments. The POB did cause greater changes in composition than did the RTFO.

RECOMMENDATIONS

1. Fractional composition of original and laboratory-aged asphalt should be analyzed separately from that of recovered asphalt when studying physical properties of asphalt versus composition.
2. The temperature susceptibility parameters were not comparable because they measured property indices in temperature ranges within which the components of asphalt have different influences. Regression models, based on fractional composition, may be built to predict temperature susceptibility, but a larger set of samples is needed to account for laboratory testing variations.
3. For consistency, the Oregon State Highway Division Laboratory should continue to use the same extraction and recovery procedure (Method A) they have used to date. If interest persists in using the cold vacuum extractor or a Rotoevaporator, or both, for recovery, more research is recommended to produce compatible results or to establish correlations.
4. More testing of the POB device with a larger number of samples is recommended. This will permit a statistical analysis rather than a descriptive discussion of results.

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