# Laboratory Evaluation of Asphalts from Shale Oil

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The objective of this study was to determine the suitability of shele-oil asphalts for paving purposes. Selected shale-oil asphalt coments were characterized both by tests commonly used to specify paving asphalt and by certain special tests. Asphalt-aggregate mixtures were made by using these asphalts, and they too were subjected to tests that are used in specifying paving mixtures. The test results were compared with similar characteristics of petroleum asphalt cements and petroleum asphalt-aggregate mixtures. Based on the laboratory test results, these shale-oil asphalts exhibit somewhat higher temperature susceptibility and lower water susceptibility than the petroleum asphalt, and the properties of the mixtures are shown to be satisfactory when compared with standard specifications.

This research was undertaken to determine the suitability of shale-oil asphalt for paving purposes. Tests of selected shale-oil asphalt cements were made and the results compared with similar characteristics of petroleum asphalt cements and petroleum asphalt-aggregate mixtures.

#### ASPHALT CEMENT PROPERTIES

Crude shale oil was produced from oil shale from the Green River formation in Colorado by the gas combustion process. A sample of the resulting shale-oil residue (LERC #SOA-71-98) was used by selected vendors to produce three grades of asphalt cement. A soft asphalt cement labeled SO AC-5 was produced by vacuum distillation, and а solvent-extracted asphalt cement labeled SO SC-10 was prepared by Kerr-McGee Company through a high-pressure process that uses an aliphatic solvent. The first attempt to produce the third asphalt, an AC-20, by vacuum distillation resulted in a material that was much too hard. There was only enough original residuum for one trial. Since the unfractionated distillate from the residuum had been retained, a predetermined portion was reblended with the hard asphalt to produce a material with the appropriate viscosity at 60°C (140°F); it was labeled SO AC-20. It should be emphasized that the process or processes by which shale oil might be produced commercially have not been determined. The properties of a shale-oil asphalt will undoubtedly depend on the type of process. Therefore, the properties of the shale-oil asphalts reported in this paper should be considered as tentative. For a more detailed discussion, see Button, Epps, and Gallaway (1). The material selected as the control asphalt (2) was a viscosity-graded AC-10 petroleum asphalt cement produced by vacuum reduction by the American Petrofina Company at their Mt. Pleasant, Texas, refinery.

## Laboratory Tests and Results

American Society for Testing and Materials (ASTM), American Association of State Highway and Transportation Officials (AASHTO), and other (3) standard laboratory tests were performed on each asphalt to determine the basic physical and chemical characteristics, including consistency, durability, purity, and safety.

Two nonstandard tests were also conducted: the thermal neutron activation analysis, used to determine the vanadium content of the asphalt, and the actinic-light hardening test, used to determine the asphalt-hardening effects of chemically active (ultraviolet) light (4). The hardening index was computed by dividing viscosity at 25°C (77°F) of the asphalt after exposure to actinic light by its initial viscosity.

The types of tests performed and the results are presented in Table 1. The appropriate properties of each asphalt are displayed on bitumen test data charts (Figures 1-4). The arrows indicate ASTM and AASHTO specification limits for the particular viscosity-graded asphalts.

## Discussion of Test Results

It should be pointed out that the SO AC-20 should not be considered a "normal" asphalt primarily because of the aforementioned method of production. The addition of the unfractionated distillate to the hard asphalt introduced material of higher volatility than would otherwise have been present in a normal vacuum-distillation product. The calculated penetration index (-0.5) and penetration ratio (44 percent) indicate that the material is typical of a normal asphalt that has a relatively low temperature susceptibility. The asphalt is, however, quite susceptible to heat damage, as evidenced by its properties after the thin-film oven test (TFOT) (Table 1). A 2 percent loss on heating indicates the presence of volatile materials; after they were evaporated, the viscosity at 60°C (140°F) became too high to be measured by means of conventional test equipment, and the penetration and ductility fell below specified limits for an AC-20. Also, the flash point and fire point were even lower than those of SO AC-5. In view of the previous discussion, it is not recommended that the results from tests on SO AC-20 be generally applied to evaluate the performance of hard shale-oil asphalts.

Another relatively hard shale-oil asphalt (SO AC-10), prepared by using conventional techniques, was resistant to heat damage, as evidenced by the properties after the TFOT (Table 1). The loss on heating was negligible, and the ductility remained greater than 150 cm (59 in). After the TFOT, changes in viscosity and penetration are what might be expected and are of the order of the corresponding changes in the laboratory standard asphalt. Overall, the properties of the SO AC-10 actually fell nearer to ASTM and AASHTO AC-20 specifications; however, it was termed SO AC-10 primarily because of the viscosity at 60°C. With a penetration index of -1.9 and a penetration ratio of 19 percent, SO AC-10 may be described as a normal asphalt with a high temperature susceptibility.

The soft shale-oil asphalt (SO AC-5) possessed a temperature susceptibility in the higher temperature range almost identical to that of the SO AC-10 and SO AC-20, which is to be expected since they have a common origin. The penetration index ( $\pm$ 0.25) and the penetration ratio (26 percent) indicate a normal asphalt. Results from the TFOT indicate a fairly durable asphalt that will resist excessive hardening during mixing and compaction.

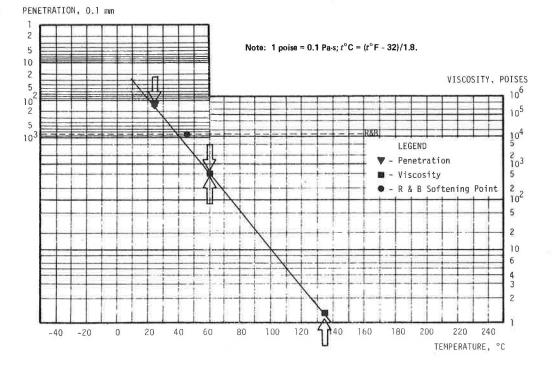
In comparison with the results of tests conducted by Traxler and others  $(\underline{4})$ , the shale-oil asphalts and the laboratory standard asphalt both have very low vanadium contents. Since damage by ultraviolet light in the sun's rays apparently increases with vanadium content, these asphalts may be expected to resist surface hardening that results from exposure to sunlight; very low hardening indexes were determined from the actinic-light hardening tests.

## Table 1. Original asphalt cement properties.

Characteristic Measured	Laboratory Standard AC-10	SO AC-5	SO AC-10	SO AC-20
Viscosity (Pa-s)				
25°C	$5.8 \times 10^{4}$	$4.8 \times 10^{4}$	$2.6 \times 10^{5}$	$2.5 \times 10^{5}$
60°C	158	49	130	199
135°C	0.38	0.13	0.23	0.22
Penetration (mm)				
25°C	11.8	12.3	4.3	7.0
25°C 4°C	2.6	3.2	0.8	3.1
Softening point, ring and ball (°C)	42	46	48	49
Penetration index	-1.4	+0.25	-1.9	-0.5
Specific gravity at 25°C	1.02	1.01	1.03	1.03
Ductility at 25°C (cm)	150+	127	150+	93
Solubility (CH Cl:CCl <sub>2</sub> ) (%)	99.99	100	99.97	100
Flash point (°C)	324	306	294	271
Fire point (°C)	370	355	334	308
Spot test	Negative	Negative	Negative	Negative
Thin-film oven test				
Penetration of residue at 25°C	68	48	24	22
Ductility of residue at 25°C	150+	148	150+	9
Viscosity of residue at 60°C	3050	2070	3650	Too high
Loss of heating (%)	Negative	Negative	Negative	2
Hardening index (actinic light)	1.9	2.5	2.2	1.7
Vanadium content (ppm)	3.4	2.6	-	3.2

Note: 1 Pa·s = 10 poises;  $t^{\circ}C = (t^{\circ}F - 32)/1.8$ ; 1 mm = 0.04 in.

Figure 1. Bitumen test data chart showing properties of SO AC-5.



#### AGGREGATE PROPERTIES

Before discussion of the mixture properties contributed by asphalt cements, the basic characteristics of the aggregates should be presented. The two types of aggregates selected for use in this reseach study are laboratory standard aggregates used at the Texas A&M University materials laboratory  $(\underline{2})$ .

The subrounded siliceous gravel was obtained from a Gifford-Hill plant near the Brazos River at College Station, Texas. A very hard crushed limestone was obtained from White's Mines at a quarry near Brownwood, Texas. Standard sieves (ASTM E-11) were used to separate the aggregates into fractions sized from 19 mm (0.75 in) to less than 75- $\mu$ m (no. 200) mesh. Before the various aggregate sizes were mixed with asphalt, they were recombined according to the ASTM D3515-77 5A grading specification. Standard tests were conducted to determine various physical properties of these aggregates, such as bulk specific gravity, saturated surface-dry (SSD) bulk specific gravity, apparent specific gravity, absorption capacity, abrasion resistance, and unit weight. One additional test (5) was conducted to estimate the optimum asphalt content.

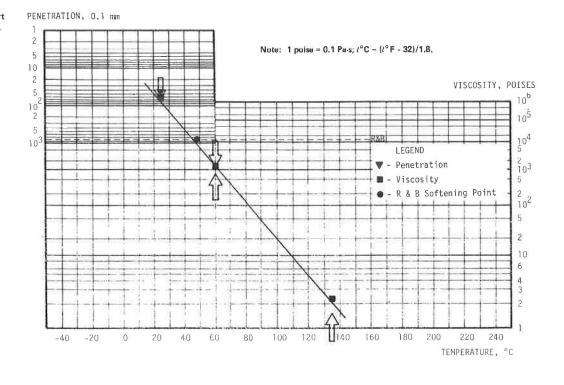
The types of tests and results are presented in Table 2.

## DETERMINATION OF OPTIMUM ASPHALT CONTENT

The optimum asphalt content for each of the two laboratory standard aggregates was determined by using the laboratory standard asphalt. Then the identical asphalt content was used when each of the



Figure 2. Bitumen test data chart showing properties of SO AC-10.



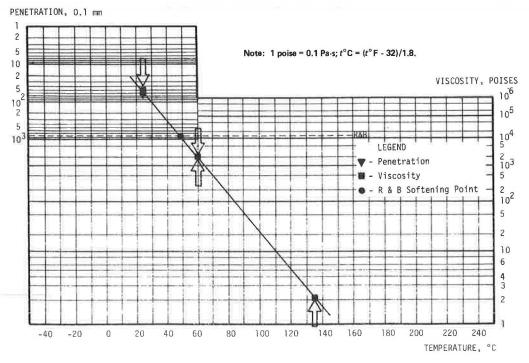
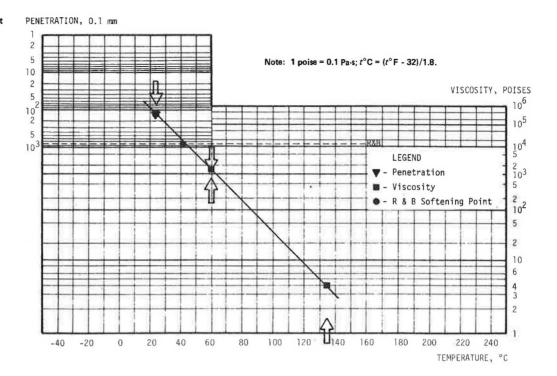


Figure 3. Bitumen test data chart showing properties of SO AC-20.

shale-oil asphalts was mixed with these aggregates, although some design procedures would indicate a somewhat different optimum for different viscosities of binder. Determination of optimum asphalt content was accomplished in accordance with the test program shown by the flowchart in Figure 5.

The selection of the optimum was based primarily on the results of the test series conducted on the Marshall specimens by using the mixture design selection procedures described by the Asphalt Institute ( $\underline{6}$ ). However, both the results of the test series conducted on the Hveem specimens and engineering judgment also entered into the final selection. The optimum contents were 3.8 percent for the gravel and 4.5 percent for the limestone.

It should be noted that some of the properties of the compacted mixtures at optimum asphalt content did not meet the criteria established by the Asphalt Institute ( $\underline{6}$ ). Undoubtedly, the quality of these mixtures could have been improved by adjusting the aggregate gradation and/or the asphalt content. However, since these mixtures were to be used as laboratory standards for test comparisons and not for highway paving, no attempt was made to further adjust the mixture design. Figure 4. Bitumen test data chart showing properties of laboratory standard asphalt.



### Table 2. Physical properties of aggregates.

			Test Results		
Aggregate Grading	Test Designation	Physical Property	Gravel	Limestone	
Coarse material <sup>a</sup>	ASTM C127, AASHTO T85	Bulk specific gravity	2.261	2.663	
		SSD bulk specific gravity	2.640	2.678	
		Apparent specific gravity	2.672	2.700	
		Absorption (%)	0.72	0.7	
fine material <sup>b</sup>	ASTM C218, AASHTO T84	Bulk specific gravity	2.551	2.537	
		SSD bulk specific gravity	2.597	2.597	
		Apparent specific gravity	2.675	2.702	
		Absorption (%)	1.8	2.2	
	Centrifuge kerosene equivalent	Surface capacity (% by weight of dry ag- gregate)	3.0	4.1	
Project design gradation	ASTM C127 and C128, AASHTO	Bulk specific gravity	2.580	2.589	
	T84 and T85	Apparent specific gravity	2.671	2.701	
		Absorption (%)	1.3	1.56	
	ASTM C29, AASHTO T19	Compacted unit weight (kg/m <sup>3</sup> )	2066	1954	
	Centrifuge kerosene equivalent and oil equivalent	Estimated optimum asphalt content (% by weight of dry aggregate)	4.7	5.5	
Grading C	ASTM C131, AASHTO T96	Abrasion resistance (% loss)	19	23	
<9.5 mm to >4.75 mm	Oil equivalent	Surface capacity (% oil retained by weight of dry aggregate)	1.8	2.3	

Note:  $1 \text{ kg/m}^3 = 0.06 \text{ lb/ft}^3$ .

<sup>a</sup> Material retained on 4.75-mm (no. 4) sieve from project design gradation. <sup>b</sup>Material passing 4,75-mm (no. 4) sieve from project design gradation.

# PERFORMANCE OF SHALE-OIL ASPHALTS IN PAVING MIXTURES

## Test Results on Gyratory-Compacted Specimens

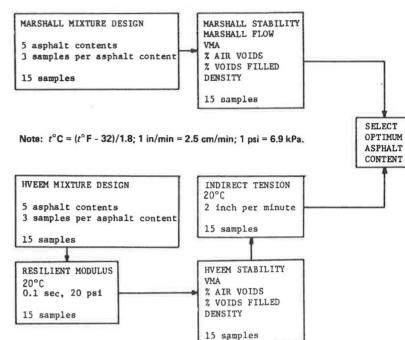
Table 3 presents the basic physical properties of the gyratory-compacted specimens. The test sequence performed on the gyratory-compacted specimens is presented in the flowchart in Figure 6 and is discussed below.

1. Resilient modulus--By using the optimum asphalt contents previously determined for each of the aggregates, 30 specimens of each of the eight asphalt-aggregate mixtures (four asphalts with two aggregates) were compacted in accordance with test method TEX-206-F. The resilient modulus of each of these specimens was measured at 20°C (68°F) by using the Schmidt device ( $\underline{7}$ ) (see Table 4).

2. Tensile strength--Twenty-seven of the 30 specimens were selected and divided into three groups of 9 each and conditioned at temperatures of -25, 1, and 20°C (-13, 33, and 68°F), respectively. Then they were subdivided into groups of 3 each, and the splitting tensile test (8) was conducted at loading-head displacement rates of 5.1, 0.51, and 0.051 cm/min (2, 0.2, and 0.02 in/min). A computer program with a plotting subroutine was used to reduce the data. A summary of the test results is presented in Table 5; each value represents an average of three specimen values, unless otherwise indicated.

3. Recovered asphalt properties--After the splitting tensile test, certain specimens were se-

Figure 5. Test program for determination of optimum asphalt content.

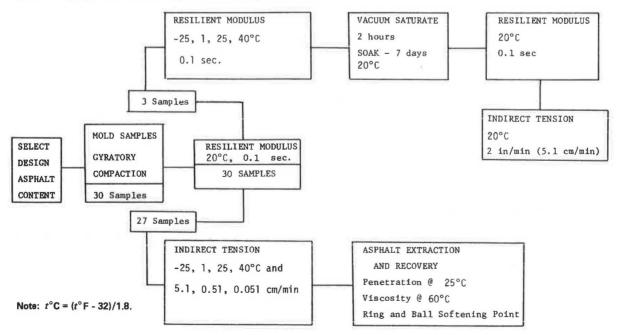


### Table 3. Basic physical properties of gyratory-compacted specimens.

	Rounded Gravel Aggregate				Crushed Limestone Aggregate				
Physical Property	Laboratory Standard	SO AC-5	SO AC-10	SO AC-20	Laboratory Standard	SO AC-5	SO AC-10	SO AC-20	
Bulk specific gravity of compacted mix	2.43	2.40	2.42	2.42	2.42	2.45	2.45	2.46	
Maximum specific gravity of mixture	2.50	2.51	2.50	2.50	2.51	2.50	2.52	2.51	
Asphalt absorption (% by weight of aggregate)	1.0	1.2	0.91	0.91	1.6	1.3	1.6	1.3	
Effective asphalt content (% of total mix) Voids in mineral aggregate (VMA) (% of bulk	2.7	2.5	2.8	2.8	2.8	3.1	2,8	3.0	
volume)	9.3	10.4	9.6	9.6	10.6	9.5	9.5	9.1	
Air void content (% of total volume)	2.8	4.4	3.2	3.2	3.6	2.0	2.8	2.0	
VMA filled with asphalt (% of VMA)	76	67	73	73	74	84	79	84	

Note: Each value represents an average of 30 specimens.

## Figure 6. Test program to determine strength and water susceptibility of mixes.



lected for extraction and recovery of each of the asphalt cements. Extraction was conducted in accordance with ASTM D2172-75 (method B). Penetration at 25°C (77°F), viscosity at 25°C and 60°C (140°F), and ring-and-ball softening point were measured to

Table 4. Simple statistics of resilient modulus of gyratory-compacted specimens at  $20^\circ$  C.

Aggregate	Asphalt	Mean Resilient Modulus (kPa × 10 <sup>6</sup> )	SD (kPa × 10 <sup>6</sup> )	Coefficient of Variation (%)
Gravel	Laboratory			
	Standard	3.55	0.414	12
	SO AC-5	6.55	1.13	17
	SO AC-10	13.0	1.07	8
	SO AC-20	8.47	1.31	16
Limestone	Laboratory			
	Standard	4.98	0.69	14
	SO AC-5	7.35	0.73	10
	SO AC-10	13.4	1.47	11
	<b>SO AC-20</b>	9.79	1.04	11

Note: 1 kPa = 0.145 psi.

## Table 5. Summary of splitting tensile test data.

quantify any asphalt hardening that might have taken place during the mixing and compacting procedures. The properties of the asphalts recovered from gravel and limestone are given in Table 6. Although hardening occurred, it was not excessive.

4. Resilient modulus and water susceptibility--The remaining 3 specimens of the original 30 were tested to determine whether or not the asphalts were susceptible to damage by water. The resilient modulus of the specimens was measured at -25, 1, 20, 25, and 40°C (-13, 33, 68, 77, and 104°F) by using a load of approximately 320 N (72 lbf) for a duration of 0.1 s. Figure 7 shows resilient moduli as a function of temperature for the gravel specimens. The curve shapes are similar for the limestone specimens, but the values are a little higher at the higher temperatures. Note the higher temperature susceptibility exhibited by SO AC-5 and SO AC-10 between 10 and 40°C (50-104°F), which corresponds with viscosity data in this temperature range. Then the specimens were submerged in water and vacuum saturated at approximately 25 mm (1 in) of mercury (absolute pressure) for 2 h and allowed to soak at atmospheric pressure for seven days. After soaking, while the specimens were still in the saturated

		Labora	tory Standa	rd	SO AC-	-5		SO AC-	-10		SO AC-	-20	
Displacement Rate Temperat (cm/min) (°C)	Temperature (°C)	Stress (Pa)	Strain (cm/cm)	Modulus (kPa)	Stress (Pa)	Strain (cm/cm)	Modulus (kPa)	Stress (Pa)	Strain (cm/cm)	Modulus (kPa)	Stress (Pa)	Strain (cm/cm)	Modulu (kPa)
Gravel													
5.1	20	110	0.0029	38	140	0.0026	58	310	0.0038	82	160	0.0025	75
	1	390	0.0027	170	410	0.0013	354	450	0.0007	984	400	0.0009	470
	-25	490	0.0012	418	360	0.0006	625	340	0.0004	1042	370	0.0006	668
Soak	20	100	0.0050	21	200	0.0026	76	200	0.0038	55	230	0.0020	114
0.51	20	50	0.0043	12	80	0.0032	25	230	0.0032	87	100	0.0023	46
	1	250	0.0020	130	380	0.0018	212	400	0.0016	257	300	0.0014	232
	-25	380	0.0009	498	460	0.0008	578	370	0.0009	457	430	0.0009	519
0.051	20	20	0.0041	5	30	0.0037	9	80	0.0048	18	60	0.0022	30
	1	110	0.0018	59	110	0.0021	61	250 <sup>a</sup>	0.0024 <sup>a</sup>	102 <sup>a</sup>	340	0.0011	348
	-25	340	0.0012 <sup>a</sup>	331 <sup>a</sup>	270	0.0011	246	390 <sup>b</sup>	0.0014 <sup>b</sup>	271 <sup>b</sup>	410	0.0011	385
Limestone			×										
5.1	20	150	0.0025	60	130	0.0023	69	250	0.0029	89	150	0.0017	94
	1	520	0.0018	290	480 <sup>a</sup>	0.0011 <sup>a</sup>	462 <sup>a</sup>	590	0.0006	1089	500	0.0011	479
	-25	630 <sup>a</sup>	$0.0012^{a}$	553 <sup>a</sup>	500	0.0011	553	470	0.0005	955	590	0.0010	598
Soak	20	90	0.0059	16	120	0.0038	32	190	0.0031	63	240	0.0022	109
0.51	20	90	0.0041	23	70	0.0034	19	270	0.0030	97	120	0.0017 <sup>a</sup>	70 <sup>a</sup>
	1	310	0.0022	150	420	0.0013	337	490	0.0014	361	400	0.0014	280
	-25	630	0.0030 <sup>a</sup>	226 <sup>a</sup>	540	0.0012	479	470	0.0011	456	600	0.0012	500
0.051	20	40	0.0040	11	40	0.0028	12	90	0.0042	21	70	0.0023	32
	1	140	0.0021'	70	470	0.0014	340	380 <sup>a</sup>	$0.0020^{a}$	$200^{a}$	200	0.0017	120
	-25	410	0.0030	156	500	0.0011	481	480 <sup>a</sup>	0.0024 <sup>a</sup>	205 <sup>a</sup>	570	0.0013	462

Notes: 1 cm = 0.4 in; t° C = (t° F -32)/1.8; 1 kPa = 0.145 psi.

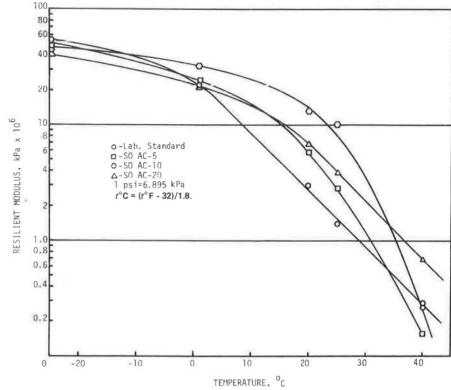
All values measured at the point of failure.

<sup>a</sup>Average of two specimen values. <sup>b</sup>Single specimen value.

## Table 6. Recovered asphalt properties.

Aggregate	Test	Laboratory Standard	SO AC-5	SO AC-10	SO AC-20
Extracted from gravel	Penetration at 25°C (mm) Viscosity (Pa·s)	5.5	4,6	1.9	3.0
	25°C	$3.9 \times 10^{5}$	$2.8 \times 10^{5}$	$2.3 \times 10^{6}$	$2.0 \times 10^{6}$
	60°C	463	143	881	3300
	Ring and ball softening point (°C)	54	57	57	67
Extracted from limestone	Penetration at 25°C (mm) Viscosity (Pa·s)	5.3	5.0	1.7	3.5
	25°C	$3.8 \times 10^{5}$	$3.2 \times 10^{5}$	$2.4 \times 10^{6}$	$1.5 \times 10^{6}$
	60°C	432	152	801	1310
	Ring and ball softening point (°C)	54	49	58	61

Note: t° C = (t° F<sub>1</sub>-32)/1.8; 1 Pa·s = 10 poises.



condition, the resilient modulus of each specimen was again measured at 20°C; then the splitting tensile test was conducted at 20°C and 5.08 cm/min (2 in/min). Figures 8 and 9 show comparisons of mixture characteristics before and after soaking in water.

## Test Results on Marshall-Compacted Specimens

Marshall tests were performed to determine the compactibility and stability of mixtures containing shale-oil asphalt and to afford a direct comparison of Marshall specimens containing shale-oil asphalt with Marshall specimens containing the laboratory standard asphalt.

After the three shale-oil asphalts had been mixed at the optimum asphalt contents, each was combined with two laboratory standard aggregates to prepare Marshall specimens by the application of 50 blows to each face of the specimens. After the dimensions and density of each specimen had been determined, the resilient modulus was determined at  $20^{\circ}C$  (68°F) by using a load of approximately 320 N (72 lbf) for a duration of 0.1 s.

The Marshall stability test was then conducted in accordance with ASTM D1559. The test results for the Marshall-compacted specimens is presented in Table 7.

#### Discussion of Laboratory Test Results

### Gyratory-Compacted Specimens

The resilient modulus (Table 4) indicates that the order of stiffness of the asphalt mixtures is the same for mixtures containing gravel or. limestone. The order from low to high follows: laboratory standard, SO AC-5, SO AC-20, and SO AC-10.

Simple statistics for the resilient modulus tests are given in Table 4. For a laboratory test such as this, coefficients of variation of 10 percent or less are considered excellent; therefore, coefficients of variation up to 17 percent should be considered reasonable.

The results of the splitting tensile test would normally be expected to yield the highest tensile strength and highest elastic moduli at the highest loading rate and the lowest temperature, and the converse should be true regarding tensile strain. Generally, this trend is fairly consistent with the data presented herein (Table 5); however, there are specific instances in which this is not true. Because of the lack of precision inherent in data of this type, the heterogeneity of individual asphalt specimens, and the fact that only three specimens were tested at each condition, it is reasonable to expect some inconsistencies.

The mode of failure of the splitting tensile test specimens ranged from physically unnoticeable at  $20^{\circ}C$  ( $68^{\circ}F$ ) and 0.051 cm/min (0.02 in/min) to catastrophic at  $-25^{\circ}C$  ( $-13^{\circ}F$ ) and 5.1 cm/min (2 in/min). At  $-25^{\circ}C$  the failure plane was well defined in such a way that the larger aggregates within the failure plane were severed, which indicated that the tensile strength of the matrix equaled or exceeded that of the aggregates.

If the recovered asphalt properties (Table 6) are compared with the original asphalt properties (Table 1), it is seen that, as a result of heating during mixing and compacting, the penetration at  $25^{\circ}$ C (77°F) of each asphalt cement decreased slightly more than 50 percent and the viscosity at 25°C increased by slightly less than one order of The viscosity at 60°C (140°F) of the magnitude. "soft" asphalts (laboratory standard and SO AC-5) increased by a factor of three, whereas that of the "hard" asphalts (SO AC-10 and SO AC-20) increased considerably more. Hardening of all the shale-oil asphalts was quite comparable to that of the petroleum asphalt. Interestingly, the penetration of the recovered asphalt indicates the same order of stiffness of the asphalt cements as mentioned before

Figure 7. Resilient modulus of gravel specimens as a function of temperature.

Figure 8. Resilient modulus at 20° C of gravel specimens before and after soaking.

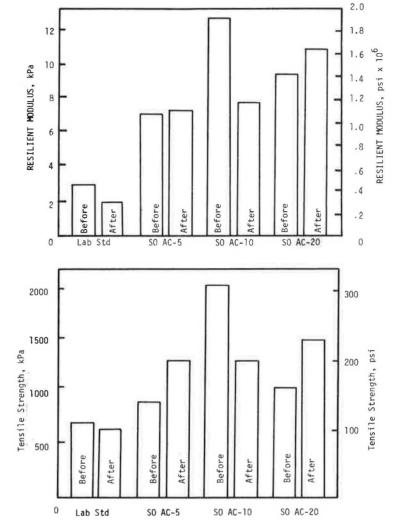


Figure 9. Splitting tensile strength of gravel specimens before and after soaking.

#### Table 7. Test results for Marshall specimens.

	Rounded Gravel Aggregate				Crushed Limestone Aggregate				
Physical Property	Laboratory Standard	SO AC-5	SO AC-10	SO AC-20	Laboratory Standard	SO AC-5	SO AC-10	SO AC-20	
Bulk specific gravity of compacted mix	2.44	2,42	2.43	2.39	2.45	2.42	2.46	2.42	
Maximum specific gravity of compacted mix	2.49	2.51	2.50	2.50	2.53	2.50	2.52	2.51	
Asphalt absorption (% by weight of aggregate)	0.75	1.2	0.91	0.91	1.7	1.3	1.6	1.3	
Effective asphalt content (% of total mix) Voids in mineral aggregate (VMA) (% of bulk	2.9	2.5	2.8	2.8	2.6	3.1	2.7	3.0	
volume)	9.1	9.8	9.3	10.8	10.5	10.7	9.1	10.6	
Air void content (% of total volume)	2.1	3.7	2.8	4.2	3.0	3.5	2.3	3.6	
VMA filled with asphalt (% of VMA)	80	70	76	67	78	75	81	74	
Marshall stability (N)	5650	6140	6850	10 990	12 190	10 270	11 390	15 260	
Marshall flow (mm)	1.8	1.5	1.5	1.8	2.8	2	3	2.5	
Resilient modulus at 20°C (kPa)	3930	7860		11 170	4070	8000		11 580	

Note: 1 N = 0.225 lbf; 1 mm = 0.4 in; t° C = (t° F -32)/1.8; 1 kPa = 0.145 psi.

in discussion of resilient modulus and, generally, the splitting tensile test.

The most apparent result of the water susceptibility study was that the resilient moduli of the mixtures that used laboratory standard asphalt and SO AC-10 with both aggregates were adversely affected by soaking in water, whereas the mixtures that used SO AC-5 and SO AC-20 were not appreciably affected (Figure 8). This same trend was generally prevalent in the postsoaking results of the splitting tensile tests at  $20^{\circ}C$  (68°F) and 5.1 cm/min (2 in/min) (Figure 9). With one exception, that of SO AC-5 plus limestone, mixtures that contained SO AC-5 and SO AC-20 actually displayed an increase in tensile strength after water soaking. Consider a theory to explain these phenomena: Shale oil contains larger amounts of basic nitrogen than does petroleum. Large amounts of basic nitrogen in the shale-oil asphalts act as antistripping agents [as Kommes and Stanfield (<u>9</u>) and J. Claine Petersen (U.S. Department of Energy, Laramie, Wyoming) have noted] unless these compounds are removed by some procedure such as the solvent de-asphalting process. The laboratory standard and the SO AC-10 asphalts might therefore be expected to exhibit higher water susceptibility than the SO AC-5 and SO AC-20 asphalts. Further, if it is assumed that the water had little effect on the mixtures that contain SO AC-5 and SO AC-20, the increase in strength and stiffness may have been due to thixotrophy since the specimens had aged at least one week more and since the before-soaking tests were normally conducted on the day after specimen fabrication. Tests have shown that the resilient modulus of freshly made laboratory specimens will increase significantly during the first four days of curing under room conditions, as D.N. Little (Texas Transportation Institute) noted in July 1978.

Resilient modulus (stiffness) as a function of temperature of the mixtures made with shale-oil asphalt was not strikingly different from those made with petroleum asphalt (Figure 7). The slopes of these plots are indicators of asphalt temperature susceptibility. At the lower temperature, SO AC-10 exhibits the lowest temperature susceptibility. At the higher temperatures, laboratory standard and SO AC-20 exhibit significantly lower temperature susceptibilities. This illustrates the fact that asphalt temperature susceptibility depends on the temperature range within which it is defined. Mixture stiffness as a function of temperature showed that shale-oil asphalts have slightly lower temperature susceptibilities at lower service temperatures.

### Marshall-Compacted Specimens

According to the Asphalt Institute (6), the medium traffic category requires 50 blows per face on each specimen and should result in a Marshall stability that exceeds 2224 N (500 lbf). The stability of all the mixtures exceeded this value (Table 7). Based on the stiffness of the SO AC-10 relative to the other asphalts tested, the Marshall stability of mixtures containing this material was surprisingly low. However, the comparatively low stability of the rounded gravel specimens was not surprising, since round, smooth aggregates usually produce mixtures that have low stabilities. The bulk specific gravity of the compacted mixtures that possess similar aggregates indicated that all the mixtures were about equal in compactibility. Since all the mixtures of a given aggregate contained identical quantities of asphalt cement, received equal compactive effort, and were in the same viscosity range during compaction, it can be stated that the air void contents indicated that SO AC-20 was the least compactible and SO AC-10 was the most compactible.

#### CONCLUSIONS

Based on the previous discussions of shale-oil asphalts from the Green River formation, the following conclusions appear warranted.

 Shale-oil asphalt can be produced by conventional methods in acceptable grades for highway paving mixtures.

2. Difficulties encountered in producing the SO AC-20 asphalt from shale oil for this research were due to the vendor's problems in obtaining reliable viscosity data during sample preparation and had nothing to do with the fact that the residuum came from shale oil.

3. The vanadium content of shale-oil asphalt is low compared with that of about 65 petroleum asphalts tested by Traxler and others  $(\underline{4})$ .

4. Adhesive properties of shale-oil asphalt are sufficient to produce adequate paving mixtures and compare favorably with those of petroleum asphalts.

5. Paving mixtures that contain shale-oil asphalts appear to show superior resistance to damage by water; however, mixtures prepared from the solvent-precipitated asphalt showed some water susceptibility and possibly some loss of Marshall stability.

6. Hardening of the shale-oil asphalts as a result of heating during mixing and compacting was slightly higher than that of the petroleum asphalt.

7. The stiffness as a function of temperature of mixtures made with shale-oil asphalt was not strikingly different from the stiffness of those made with petroleum asphalt.

8. The Marshall stability of mixtures made with shale-oil asphalt was more than adequate and compared well with the Marshall stability of those made with petroleum asphalt.

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#### REFERENCES

- J. W. Button, J. A. Epps, and B. M. Gallaway. Laboratory Evaluation of Selected Shale-Oil Asphalts. Laramie Energy Research Center, U.S. Department of Energy, Rept. 3695-1, Jan. 1978.
- J. W. Button, J. A. Epps, and B. M. Gallaway. Test Results of Laboratory Standard Asphalts, Aggregates, and Mixtures. Texas Transportation Institute, Texas A&M Univ., College Station, Materials Laboratory Note 1, 1977.
- 3. Manual of Testing Procedures, Volume 1. Texas State Department of Highways and Public Transportation, Austin, 1974.
- 4. R. N. Traxler, F. H. Scrivner, and W. E. Kuykendall, Jr. Loss of Durability in Bituminous Pavement Surfaces: Importance of Chemically Active Solar Radiation. Texas Transportation Institute, Texas A&M Univ., College Station, Res. Rept. 127-3, Nov. 1971.
- F. N. Hveem. Establishing the Oil Content for Dense-Graded Bituminous Mixtures. California Department of Highways and Public Works, Sacramento, July-Aug. 1942.
- Mix Design Methods for Asphalt Concrete, 4th ed. Asphalt Institute, College Park, MD, Manual Series 2 (MS-2), March 1974, section 3.12.
- R. J. Schmidt. A Practical Method for Measuring the Resilient Modulus of Asphalt-Treated Mixes. HRB, Highway Research Record 404, 1972, pp. 22-32.
- W. O. Hadley, W. R. Hudson, and T. W. Kennedy. Evaluation and Prediction of the Tensile Properties of Asphalt-Treated Materials. Center for Highway Research, Univ. of Texas at Austin, Res. Rept. 98-9, May 1971, pp. 63-76.
- W. C. Kommes and K. E. Stanfield. Properties of Shale-Oil Asphalts from Colorado Oil Shale. Petroleum and Oil-Shale Experiment Station, Laramie, WY, Intra-Bureau Rept. OSRD-45, 1951.

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