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Final Report

COMPOSITION AND PHYSICAL TEST CHARACTERISTICS OF ORIGINAL AND RECOVERED ASPHALTS AS INFLUENCED BY VARIOUS TEST EXPOSURES

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ABSTRACT

This report summarizes the test data pertaining to a limited number of asphalts and indicates the change in their composition and physical characteristics resulting from laboratory and outdoor weathering exposures. Tentative conclusions and suggestions for additional study have been included.

OBJECTIVE

The purpose of this investigation was to determine the effect of different aggregates and exposures on the composition and physical properties of asphalt.

INTRODUCTION

In 1954 and 1955, a rather comprehensive investigation of asphalt pavement was made in the states of Wyoming, Colorado, Nebraska, Iowa, Minnesota, and Michigan. Attention was primarily directed to pavements containing asphalt from Wyoming crudes, although others were included for comparison. Detailed inspections were made of pavements of ages up to 26 years. Large-size samples were taken from a large number of them and the asphalts were recovered and tested. As much information as was available was gathered concerning the history of each project.

A report of the field and laboratory investigation was submitted in August, 1955, under the title "Field Inspections and Laboratory Tests on Asphalts and Asphalt Pavements" (ERI Report No. 2249-1-P). It was concluded that subgrade soil conditions, base course characteristics, mixture composition and control, construction procedures, and condition of use were more significant in the behavior of the pavement than was the source and characteristics of the asphalt cement.

In the analysis of the data from this phase of the investigation, questions arose concerning the composition of asphalts: whether those of so-called heterogeneous compositions, under the conditions of the Oliensis spot test, may differ greatly from homogeneous asphalts, and under what conditions the heterogeneous may develop from the homogeneous, and to what degree an initially positive spot asphalt may be changed. Further questions concerned the effect upon asphalts of the characteristics of the aggregates with which they were used, the possibility that some aggregate may tenaciously retain resins or oils, thus affecting the test characteristics of the laboratory-recovered asphalt.

To throw some light on these problems, an exploratory investigation was started in the fall of 1955. It was planned to determine the composition of certain asphalts as produced, in terms of asphaltenes, oily constituents, and resins, and also after combining them with different aggregates and subjecting the mixture to various conditions of exposure. This phase of the report summarizes the data which have been accumulated and the conclusions which have been reached. Detailed data, descriptions of procedures, and incidental material are appended as follows:

Appendix I.—Table I—The composition of both original and recovered asphalt. Table II—Standard and special tests of the original and recovered asphalt, and the characteristics of the materials. Table III—Percent change in the asphalt, as related to various exposures, obtained from data in Tables I and II. Graph I—A bar graph showing percent change in the asphalt as related to various exposures as tabulated in Table III.

Appendix II.—Color coded bar graph in which each asphalt is identified by distinctive color and the test data collectively plotted for each different aggregate and exposure.

Appendix III.—Detailed description of equipment and test methods as employed to determine the composition of the asphalts.

MATERIALS USED IN THE INVESTIGATION

Three asphalts of the 85-100 penetration grade were used in the main portion of this investigation. Two were processed from Wyoming crude by Leonard Refineries, Inc., Alma, Michigan, and by Trumbull Asphalt Company, Detroit, Michigan. The third was from Arkansas crude and processed by the Lion Oil Company, El Dorado, Arkansas. A limited study was made of a fourth asphalt prepared from Garland Wyoming crude, known to have positive spot characteristics. It was used in a series of tests in combination with a high resin material known as Duct-spot to determine whether or not the positive spot Garland might be made permanently negative under the conditions of the exposure tests. These materials and the tests in which they were used are described later in the report. The characteristics and compositions of the Leonard, Lion, and Trumbull asphalts are given in Table A.

TABLE A

Asphalt	Penet	Duct	Sp Gr	L on H	Pen Res	S Pt	S Pt/Pen	Spot
Leonard	83	150+	1.024	0.00	75	115.3	1.390	Neg
Lion	95	150+	1.018	0.00	88	114.3	1.203	Neg
Trumbull	86	150+	1.027	0.03	75	113.2	1.316	Neg

Asphalt	% Asphtns	% Oils	% Resins	O/A Ratio	R/A Ratio	R/O Ratio
Leonard	18.57	34.27	47.16	1.846	2.540	1.376
Lion	18.82	36.79	44.39	1.955	2.359	1.207
Trumbull	20.12	34.34	45.54	1.707	2.263	1.326

The composition of the asphalts was determined by methods developed by the United States Bureau of Mines.[1] It was necessary to have most of the equipment specially made, and, once assembled, it was found that the published procedures were not sufficiently detailed to make reproducible separations possible. Con-

[1] H. K. Stanfield, *et al.*, Bureau of Mines, Bulletin 717.

siderable preliminary work was required to establish a workable procedure, but this was finally accomplished and reasonably reproducible results were obtained on successive determinations. Carefully controlled laboratory conditions and extreme accuracy in manipulation are essential. Appendix III contains a detailed description of the apparatus and procedures used in the determination of asphalt composition.

Four aggregates were used in the test mixtures:

1. Ottawa sand from Ottawa, Illinois, composed of clean siliceous particles.

2. Local glacial sand from the Ann Arbor area. This was separated into sizes and recombined to conform to the gradation of the Ottawa sand.

3. Michigan aggregate from a glacial deposit which was used in a Michigan State Highway test pavement between Flint and Pontiac. Limestone dust was added to this aggregate as a filler.

4. Minnesota aggregate from near Waterville, Minnesota, which was the source of aggregate for asphalt pavement laid on Minnesota Route T.H. 13 north of that town. This aggregate was soft, absorptive, and included silt and clay. No filler was added.

Both the Michigan and Minnesota aggregates were regraded to conform to the specifications of the respective states. These gradings are not the same. Most of the exposure tests were made upon mixtures containing the Michigan and Minnesota aggregates.

Aggregate gradations are shown in Table B.

TABLE B

Sieve Size	% Retained on Sieves			
	Sand		Aggregate	
	Ottawa	Local	Michigan	Minnesota
1/2 in.	0.0	0.0	2.40	9.9
No. 10	0.0	0.0	56.3	44.4
No. 20	0.0	0.0		
No. 30	1.0	1.0		
No. 40	40.0	40.0	8.9	26.0
No. 50	32.0	32.0		
No. 80	23.0	23.0	17.6	9.8
No. 100	2.0	2.0		
No. 200	2.0	2.0	8.8	3.1
P-200	0.0	0.0	6.0	6.8
	100.0	100.0	100.0	100.0

CONDITIONS OF TEST EXPOSURES

Mixtures were made of the various asphalts and subjected to several exposure conditions. Batches of 2000 grams were prepared, all containing 5.5% of asphalt by weight of the batch. All were prepared under carefully controlled conditions of time and temperature, and all were exposed to heated air in a force-draft oven. Some were simply cooled for a specified period of time before extraction and recovery of the asphalt. Others were subjected to further exposure conditions, and included two series, (k) and (l), which were exposed to the outdoor weather for 60 days. Series (l) was compacted before exposure. Series (k) was loosely spread in a shallow pan to a depth of approximately 1/2 inch. The modified Abson method was used to recover the asphalt from the mixtures.

All exposures were not performed on all asphalts or all aggregates, since this investigation was intended to be exploratory rather than exhaustive. There is no definite correlation between these conditions of test and actual paving exposure. However, a comparison of the results obtained from the most severe (n) exposure with observations and tests from the earlier survey of pavements of various ages and conditions indicates that this exposure may represent conditions fully as severe as those to which a pavement would be subjected over long years of normal service. The conditions of mixing and exposure are outlined in Table C.

TABLE C

Test Exposure	Mat'l Heating		Mix Time	Mixture Heating			Test Conditions
	Agg °F	Asphalt °F	Hobart, min	Time, min	Temp, °F	Type of Oven	
a	400	300	1	30	350	F. Draft	Cool 45 min, extract and recover. Ottawa and Local sand only.
a_1	350	300	1	30	325	F. Draft	Cool 45 min, extract and recover. Michigan and Minnesota aggregates.
b	400	300	1	30	350	F. Draft	72-hr exposure in air in laboratory.
c	400	300	1	30	350	F. Draft	24-hr exposure in air in laboratory.
d	400	300	1	30	350	F. Draft	48-hr exposure in air in laboratory.
e	400	300	1	30	350	F. Draft	72 hr in force-draft oven at 140°F.
f	400	300	1	30	350	F. Draft	24 hr in force-draft oven at 140°F.
g	400	300	1	30	350	F. Draft	48 hr in force-draft oven at 140°F.
h	350	300	1	30			90 min in force-draft oven at 325°F.
i	Not run during this program.						
j	350	300	1	30			90 min in force-draft oven at 325°F.
k	350	300	1	30	325	F. Draft	Mix loose spread and expose 60 days outdoors.
l	350	300	1	30	325	F. Draft	Compacted to 4" diameter x 4-1/2" cylender and exposed to 60 days outdoors.
m	350	300	1	30	325	F. Draft	Cool 45 min, extract and recover.
n	350	300	1	30	325	F. Draft	180 min in force-draft oven at 325°F.

SUMMARY OF RESULTS OF EXPOSURES

Table D is a summary of the detailed data of Appendix I. Relationships between constituents of the asphalts, original and as recovered from the mixtures, are expressed by the resin/asphaltene ratio. Exposures (b) to (g), although somewhat more severe than (a) and (a_1), were conducted only on a few mixtures and are not included in Table D. It should be noted that two distinct series of the (n) exposure were conducted.

CHANGE IN PHYSICAL PROPERTIES DUE TO CONDITIONS OF EXPOSURE

As will be noted from Table A, the composition of the Leonard, Lion, and Trumbull asphalts are quite similar for the original materials.

All exposures produced changes in all three of the asphalts, as is indicated by the change in the relative percentages of their constituents. Asphaltenes always increased and resins always decreased with the exception of one very slight apparent increase. These changes resulted in decreases in the resin-asphaltene ratio which became greater as the severity of the exposure increased. Oils increased in some cases and decreased in others, with the increases greatly outnumbering the decreases. It appears that the tendency is for asphaltenes and oils to gain at the expense of the resins.

Changes occurred in the physical test characteristics, reflecting those in composition and were of course greater as the conditions of exposure became more severe. Penetration decreased, softening point increased, in isolated instances ductility was measurably lower, and tendencies developed for the asphalts to produce positive spot in the Oliensis test.

When the loose mixtures were exposed to forced-draft air at 325°F for 30 minutes in Exposure (a_1), penetration decreased considerably, but the ductility was not measurably affected. A longer exposure of 90 minutes further reduced penetrations and measurably reduced ductilities of Leonard and Lion in the (h) exposure with the Minnesota aggregate mixture. All ductilities were above 150 cm on asphalt recovered from the Michigan aggregate mixtures, and the same was true of Trumbull with the Minnesota aggregate mixture subjected to Exposure (h). The 3-hour forced-draft air exposure of the (n) exposure resulted in great reduction in both penetration and ductility.

The outdoor Exposures (k) and (l), made only on mixtures of asphalt and Michigan and Minnesota aggregates, were exposed to 60 days of outdoor weathering on the roof of a building and then were dried to remove moisture before the

TABLE D

SUMMARY OF RESULTS OF EXPOSURES (a), (a_1), (h), (k), (l), and (n)

Test Data	Leonard			Lion			Trumbull		
	Orig	Rcvd	% Change	Orig	Rcvd	% Change	Orig	Rcvd	% Change
Exposure (a). Mix data: Heat aggregate to 400°F, asphalt to 300°F. Mix 1 minute. Exposure: In force-draft oven at 350°F for 30 min; cool 45 min, extract, and recover asphalt.									
Ottawa Sand Aggregate									
No. of Tests		3			3			3	
Res/Asphtn	2.54	1.88	- 25.9	2.36	1.84	- 22.2	2.26	1.58	- 30.1
Penet	83	47	- 43.4	95	59	- 37.9	86	45	- 47.7
Duct	150+	150+	--	150+	150+	--	150+	150+	--
S Pt, °F	115.3	123.1	+ 6.70	114.3	122.0	+ 6.80	113.2	126.7	+ 11.9
S Pt/Pen	1.39	2.62	+ 88.4	1.20	2.07	+ 71.9	1.32	2.82	+113.9
Skelly "S" Spot	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
Local Sand Aggregate									
No. of Tests		3			3			3	
Res/Asphtn	2.54	1.69	- 33.5	2.36	1.71	- 27.7	2.26	Not determined	
Penet	83	53	- 36.2	95	76	- 20.0	86	46	- 46.5
Duct	150+	150+	--	150+	150+	--	150+	150+	--
S Pt, °F	115.3	124.9	+ 8.3	114.3	118.9	+ 4.1	113.2	127.2	+ 12.4
S Pt/Pen	1.39	2.36	+ 69.5	1.20	1.57	+ 30.1	1.32	2.77	+110.2
Skelly "S" Spot	Neg	Neg	--	Neg	Neg	--	Neg	Sl Pos	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
Exposure (a_1). Mix data: Heat aggregate to 350°F, asphalt to 300°F. Mix 1 minute. Exposure: In force-draft oven at 325°F, 30 min; cool 45 min, extract, and recover asphalt.									
Michigan Aggregate									
No. of Tests		3			3			3	
Res/Asphtn	2.54	1.95	- 23.3	2.36	1.86	- 21.2	2.26	1.82	- 19.6
Penet	83	58	- 30.1	95	76	- 20.0	86	55	- 36.1
Duct	150+	150+	--	150+	150+	--	150+	150+	--
S Pt, °F	115.3	119.7	+ 3.8	114.3	114.1	- 0.2	113.2	118.2	+ 4.5
S Pt/Pen	1.39	2.06	+ 48.4	1.20	1.50	+ 24.1	1.32	2.15	+ 63.3
Skelly "S" Spot	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
Minnesota Aggregate									
No. of Tests		3			3			3	
Res/Asphtn	2.54	1.97	- 22.6	2.36	1.85	- 21.7	2.26	1.89	- 16.4
Penet	83	46	- 44.6	95	73	- 23.2	86	53	- 38.4
Duct	150+	150+	--	150+	150+	--	150+	150+	--
S Pt, °F	115.3	126.1	+ 9.4	114.3	127.6	+ 11.7	113.2	122.0	+ 7.8
S Pt/Pen	1.39	2.74	+ 97.3	1.20	1.75	+ 45.3	1.32	2.30	+ 74.9
Skelly "S" Spot	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
Exposure (h). Mix data: Heat aggregate to 350°F, asphalt to 300°F. Mix 1 minute. Exposure: In force-draft oven at 325°F, 90 min; cool, extract, and recover asphalt.									
Michigan Aggregate									
No. of Tests		1			1			1	
Res/Asphtns	2.54	1.75	- 31.0	2.36	2.07	- 12.2	2.26	1.56	- 30.9
Penet	83	38	- 54.2	95	56	- 41.1	86	40	- 53.5
Duct	150+	150+	--	150+	150+	--	150+	150+	--
S Pt, °F	115.3	129.6	+ 12.3	114.3	122.0	+ 6.8	113.2	127.4	+ 12.6
S Pt/Pen	1.39	3.41	+145.3	1.20	2.18	+ 81.1	1.32	3.19	+142.0
Skelly "S" Spot	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
Minnesota Aggregate									
No. of Tests		1			1			1	
Res/Asphtns	2.54	1.62	- 36.3	2.36	1.52	- 35.6	2.26	1.62	- 28.5
Penet	83	30	-63.9	95	41	- 56.8	86	32	- 62.8
Duct	150+	93	- 38.0	150+	123	- 18.0	150+	150+	--
S Pt, °F	115.3	132.1	- 14.5	114.3	129.2	- 13.1	113.2	131.5	+ 16.2
S Pt/Pen	1.39	4.40	+216.8	1.20	3.15	+162.2	1.32	4.11	+212.4
Skelly "S" Spot	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--

TABLE D (Continued)

Exposure (k). Mix data: Heat aggregate to 350°F, asphalt to 300°F. Mix 1 minute.
Exposure: Loosely spread in shallow pan approximately 1/2 in. deep, stand outdoors for 60 days. Before extraction dry at 150°F for 24 hr, extract, and recover the asphalt.

Test Data	Leonard			Lion			Trumbull		
	Orig	Rcvd	% Change	Orig	Rcvd	% Change	Orig	Rcvd	% Change
Michigan Aggregate									
No. of Tests		3			3			3	
Res/Asphtns	2.54	1.57	- 38.3	2.36	1.54	- 34.7	2.26	1.25	- 45.0
Penet	83	32	- 61.5	95	38	- 59.6	86	30	- 64.7
Duct	150+	150+	--	150+	150+	--	150+	150+	--
S Pt, °F	115.3	135.7	+ 17.7	114.3	133.2	+ 16.6	113.2	136.6	+ 20.7
S Pt/Pen	1.39	4.24	+ 205.2	1.20	3.51	+192.1	1.32	4.51	+243.0
Skelly "S" Spot	Neg	1 Trace 2 Neg	--	Neg	Neg	--	Neg	Pos	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
Minnesota Aggregate									
No. of Tests		1			3			3	
Res/Asphtns	2.54	1.72	- 32.2	2.36	1.28	- 45.76	2.26	1.32	- 41.6
Penet	83	33	- 60.2	95	39	- 58.5	86	30	- 63.8
Duct	150+	150+	--	150+	(?)*	--	150+	(?)*	--
S Pt, °F	115.3	132.8	+ 15.1	114.3	134.8	+ 17.9	113.2	133.7	+ 18.1
S Pt/Pen	1.39	4.02	+ 189.5	1.20	3.34	+178.3	1.32	4.31	+235.4
Skelly "S" Spot	Neg	Neg	--	Neg	1 Trace 2 Neg	--	Neg	Neg	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--

*Ductility test values erratic. Not reliable. Not included.

Variation. Same as above (Minnesota Aggregate) except that two mixes with Leonard asphalt, which contained moisture accumulated from outdoor exposure, were dried at 250°F in force-draft oven for 24 hr. These were the first outdoor samples to be recovered. As the 250°F temperature appeared to affect the asphalt extremely adversely, subsequent samples were dried at 150°F.

Test Data	Leonard Orig	Leonard Rcvd	% Change
No. of Tests		2	
Res/Asphtns	2.54	0.80	- 68.5
Penet	83	7	- 91.6
Duct	150+	3	+ 98.0
S Pt, °F	115.3	207.0	+ 80.0
S Pt/Pen	1.39	32.42	+2210.79
Skelly "S" Spot	Neg	1 Pos 1 Neg	--
100% Xylene	Neg	Neg	--

Note: The application of 250°F temperature for removing moisture from the mixture was not accidentally applied, but was initially considered a safe temperature, in view of the temperature of 300°F to which the asphalt was heated in preparing the mixture.

Exposure (l). Mix data: Heat aggregate to 350°F, asphalt to 300°F. Mix 1 minute.
Exposure: Compact mix into cylinders, 4 in. diameter x 4-1/2 in. high and subject to outdoor weathering for 60 days. Before extraction dry at 150°F in force-draft oven for 24 hr, extract, and recover the asphalt.

Test Data	Leonard			Lion			Trumbull		
	Orig	Rcvd	% Change	Orig	Rcvd	% Change	Orig	Rcvd	% Change
Michigan Aggregate									
No. of Tests		3			3			3	
Res/Asphtns	2.54	1.80	- 29.2	2.36	1.87	- 20.7	2.26	1.47	- 35.1
Penet	83	52	- 37.4	95	63	- 33.3	86	50	- 42.3
Duct	150+	150+	--	150+	150+	--	150+	150+	--
S Pt/°F	115.3	125.5	+ 8.8	114.3	122.4	+ 7.1	113.2	126.2	+ 11.5
S Pt/Pen	1.39	2.45	+ 76.4	1.20	1.94	+ 60.3	1.32	2.54	+ 93.3
Skelly "S" Spot	Neg	1 Trace 2 Neg	--	Neg	--	--	Neg	1 Sl Pos 2 Neg	--
100% Xylene	Neg	Neg	--	Neg	--	--	Neg	Neg	--

TABLE D (Continued)

Test Data	Leonard			Lion			Trumbull		
	Orig	Rcvd	% Change	Orig	Rcvd	% Change	Orig	Rcvd	% Change
				Exposure (l) (Concluded)					
Minnesota Aggregate									
No. of Tests		1			3			3	
Res/Asphtns	2.54	1.91	- 24.7	2.36	1.68	- 28.8	2.26	1.57	- 30.7
Penet	83	44	- 47.0	95	61	- 36.1	86	46	- 46.1
Duct	150+	150+	--	150+	150+	--	150+	150+	--
S Pt, °F	115.3	128.1	+ 11.1	114.3	122.3	+ 7.1	113.2	126.4	+ 11.7
S Pt/Pen	1.39	2.91	+109.5	1.20	2.02	+ 68.3	1.32	2.73	+107.5
Skelly "S" Spot	Neg	Trace	--	Neg	Neg	--	Neg	Neg	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--

Variation. Same as above (Minnesota Aggregate) except that two mixes with Leonard asphalt, which contained moisture accumulated from outdoor exposure were dried at 250°F in force-draft oven for 24 hr. These were the first outdoor samples to be recovered. As the 250°F temperature appeared to affect the asphalt extremely adversely, subsequent samples were dried at 150°F.

Test Data	Leonard Orig	Leonard Rcvd	Leonard % Change
No. of Tests		2	
Res/Asphtns	2.54	1.71	- 32.86
Penet	83	36.5	- 56.0
Duct	150+	150+	--
S Pt, °F	115.3	132.0	+ 15.3
S Pt/Pen	1.39	3.65	+162.4
Skelly "S" Spot	Neg	1 Sl Pos 1 Neg	--
100% Xylene	Neg	Neg	--

Note: The application of 250°F temperature for removing moisture from the mixture was not accidentally applied, but was initially considered a safe temperature, in view of the initial temperature of 300°F, to which the asphalt was heated in preparing the mixture. Although this temperature affected the asphalt in the compacted mixture much less than the loosely spread mixture in exposure (k), it is demonstrated that high drying temperatures applied to mixtures prior to extraction and recovery may materially affect test results.

Exposure (n). Mix data: Heat aggregate to 350°F, asphalt to 300°F. Mix 1 minute.
Exposure: In force-draft oven for 3 hr, extract, and recover the asphalt.

FIRST SERIES

Test Data	Leonard Orig	Leonard Rcvd	Leonard % Change	Lion Orig	Lion Rcvd	Lion % Change	Trumbull Orig	Trumbull Rcvd	Trumbull % Change
Michigan Aggregate									
No. of Tests		1			0			1	
Res/Asphtns	2.54	1.54	- 39.5				2.26	1.31	- 42.2
Penet	83	24	- 71.1				86	19	- 77.9
Duct	150+	19	- 87.3				150+	7	- 95.8
S Pt, °F	115.3	142.0	+ 23.1		No test performed		113.2	158.2	+ 39.8
S Pt/Pen	1.39	5.9	+325.6				1.32	8.3	+532.6
Skelly "S" Spot	Neg	Sl Pos	--				Neg	Pos	--
100% Xylene	Neg	Neg	--				Neg	Neg	--
Minnesota Aggregate									
No. of Tests		1			1			1	
Res/Asphtns	2.54	1.16	- 54.2	2.36	Not determined		2.26	Not determined	
Penet	83	19	- 77.1	95	27	- 71.6	86	27	- 68.6
Duct	150+	6	- 96.0	150+	9	- 94.0	150+	52	- 65.3
S Pt, °F	115.3	162.9	+ 41.2	114.3	151.5	+ 32.6	113.2	139.8	+ 23.5
S Pt/Pen	1.39	8.6	+516.7	1.20	5.6	+366.5	1.32	5.2	+293.5
Skelly "S" Spot	Neg	Pos	--	Neg	Neg	--	Neg	Sl Pos	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--

TABLE D (Continued)

Test Data	Leonard			Lion			Trumbull		
	Orig	Rcvd	% Change	Orig	Rcvd	% Change	Orig	Rcvd	% Change
		Exposure (n) (continued). Mix data: Heat aggregate to 350°F, asphalt to 300°F. Mix 1 minute. Exposure: In force-draft oven for 3 hr, extract, and recover the asphalt.							
SECOND SERIES (See Appendix II)									
<u>Michigan Aggregate</u> (Clean, unused material)									
No. of Tests		6			3			3	
Res/Asphtns	Composition tests not performed in this test series.								
Penet	83	25	- 69.9	95	31	- 67.4	86	22	- 74.4
Duct	150+	No ductility tests performed in this series.							
S Pt, °F	115.3	144.7	+ 25.5	114.3	145.0	+ 26.9	113.2	150.9	+ 33.3
S Pt/Pen	1.39	6.06	+336.0	1.20	4.78	+298.33	1.32	6.99	+429.5
Skelly "S" Spot	Neg	5 Neg 1 Ring	--	Neg	Neg	--	Neg Ring	--	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
<u>Heptane-Xylene Solvent</u>									
10% Xylene	--	6 Pos	--	--	Pos	--	--	Pos	--
15% Xylene	--	5 Pos 1 Ring	--	--	Pos	--	--	Pos	--
20% Xylene	--	6 Pos	--	--	1 Pos 2 Ring	--	--	Pos	--
25% Xylene	--	2 Pos 4 Ring	--	--	2 Neg 1 Ring	--	--	Pos	--
30% Xylene	--	5 Ring 1 Neg	--	--	Neg	--	--	1 Pos 1 Ring	--
35% Xylene	--	5 Neg 1 Ring	--	--	Neg	--	--	1 Neg 2 Ring	--
<u>Michigan Aggregate</u> (Reused aggregate, recovered from original mix)									
No. of Tests		6			3			3	
Res/Asphtns	2.54	Composition tests not performed in this test series.							
Penet	83	25	-69.9	95	32	- 66.3	86	27	- 68.6
Duct	150+	Ductility tests not performed in this test series.							
S Pt, °F	115.3	146.0	+ 26.6	114.3	142.3	+ 24.5	113.2	143.1	+ 26.4
S Pt/Pen	1.39	6.06	+336.0	1.20	4.54	+278.3	1.32	5.56	+321.2
Skelly "S" Spot	Neg	4 Neg 2 Ring	--	Neg	Neg	--	Neg	Ring	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
<u>Heptane-Xylene Solvent</u>									
10% Xylene	--	Pos	--	--	Pos	--	--	Pos	--
15% Xylene	--	5 Pos 1 Ring	--	--	Pos	--	--	Pos	--
20% Xylene	--	Pos	--	--	Ring	--	--	Pos	--
25% Xylene	--	3 Pos 3 Ring	--	--	Neg	--	--	Pos	--
30% Xylene	--	Ring	--	--	Neg	--	--	1 Pos 2 Ring	--
35% Xylene	--	Neg	--	--	Neg	--	--	2 Neg 1 Ring	--

TABLE D (Concluded)

Test Data	Leonard			Lion			Trumbull		
	Orig	Rcvd	% Change	Orig	Rcvd	% Change	Orig	Rcvd	% Change
				Exposure (n) (Concluded)					
Minnesota Aggregate (Clean, unused material)									
No. of Tests		3			3			3	
Res/Asphtns	2.54	Composition tests not performed in this test series.							
Penet	83	18	- 78.3	95	26	- 72.6	86	16	- 81.4
Duct	150+	Ductility tests not performed in this test series.							
S Pt, °F	115.3	162.0	+ 40.5	114.3	156.4	+ 36.8	113.2	162.3	+ 43.4
S Pt/Pen	1.39	9.18	+560.4	1.20	6.14	+411.7	1.32	10.5	+695.5
Skelly "S" Spot	Neg	1 Neg 2 Ring	--	Neg	Neg	--	Neg	Ring	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
Heptane-Xylene Solvent									
10% Xylene	--	Pos	--	--	Pos	--	--	Pos	--
15% Xylene	--	Pos	--	--	Pos	--	--	Pos	--
20% Xylene	--	Pos	--	--	1 Pos 2 Ring	--	--	Pos	--
25% Xylene	--	2 Pos 1 Ring	--	--	Neg	--	--	Pos	--
30% Xylene	--	1 Pos 2 Ring	--	--	Neg	--	--	2 Pos 1 Ring	--
35% Xylene	--	2 Neg 1 Ring	--	--	Neg	--	--	1 Neg 2 Ring	--
Minnesota Aggregate (Reused aggregate, recovered from original mix)									
No. of Tests		3			3			3	
Res/Asphtns	2.54	Composition tests not performed in this test series.							
Penet	83	19	- 77.1	95	26	- 72.6	86	14	- 83.7
Duct	150+	Ductility tests not performed in this test series.							
S Pt, °F	115.3	158.4	+ 37.4	114.3	157.0	+ 37.4	113.2	167.1	+ 47.6
S Pt/Pen	1.39	8.2	+490.0	1.20	6.1	+408.3	1.32	11.73	+788.6
Skelly "S" Spot	Neg	Ring	--	Neg	Neg	--	Neg	Ring	--
100% Xylene	Neg	Neg	--	Neg	Neg	--	Neg	Neg	--
Heptane-Xylene Solvent									
10% Xylene	--	Pos	--	--	Pos	--	--	Pos	--
15% Xylene	--	2 Pos 1 Ring	--	--	Pos	--	--	Pos	--
20% Xylene	--	Pos	--	--	Pos	--	--	Pos	--
25% Xylene	--	2 Pos 1 Ring	--	--	2 Neg 1 Ring	--	--	Pos	--
30% Xylene	--	1 Pos 2 Ring	--	--	Neg	--	--	Pos	--
35% Xylene	--	1 Neg 2 Ring	--	--	Neg	--	--	Ring	--

asphalt was extracted. Drying was accomplished in a forced-draft oven at 150°F for 24 hours for all samples except four of the Leonard asphalt with Minnesota aggregate, which were dried at 250°F. Two of these were from the (k) exposure, which were loosely spread in a shallow pan and the other two were from the (l) exposure and were compacted specimens.

The loosely spread mixtures subjected to the (k) exposure were more greatly affected than the compacted specimens used in the (l) exposure, as is indicated by the comparative changes in the recovered asphalt. Ductilities of the asphalts for both (k) and (l) exposures all remained above 150 cm in the Michigan aggregate mixtures. The asphalts recovered from both the (k) and (l) exposures on Minnesota aggregate mixtures, which were dried at 150°F before extraction, remained above 150 cm in ductility with the exception of one (k) exposure Lion and one (k) exposure Trumbull asphalt. Companion test values do not substantiate the low ductility values obtained, and for that reason these two tests have been eliminated for comparative purposes. The two Leonard (k) and the two (l) exposure mixtures with Minnesota aggregate which were heated to 250°F to remove moisture prior to extraction showed marked changes in both penetration and ductility. They have been reported in Table D as special exposure conditions to illustrate the susceptibility of asphalts to change when exposed in thin films.

Throughout the series, softening points increased as did the possibly significant softening point/penetration ratio, which received comment in the correlation of field and laboratory studies reported in August, 1955. It was noted that pavements containing asphalts in which the softening point/penetration ratio had increased to a value approximately 5.50 times that of the original material are likely to become dry and brittle. Above such value map cracking becomes prevalent, attended with raveling and crack spalling as the value exceeds 6.00 times the original. During the current series of laboratory tests more severe exposure, such as that of the (n) test, produced softening point/penetration values greater than the critical maximum. None approached it in the outdoor (l) exposure series, where mixtures were compacted. It was rarely approached when the mixtures were exposed in a loose condition as employed in the (k) test. In the preceding field study, an Iowa pavement from which samples were taken from the traveled wheel paths and also from between them, reflected the effect of traffic densification in the values of the softening point/penetration ratios. The asphalt from the wheel-path samples had a ratio of 2.60, and that from between wheel paths, a ratio value of 4.40. The desirability of providing thorough compaction to a paving mixture is demonstrated by both laboratory and field observations.

SPOT CHARACTERISTICS AS AFFECTED BY EXPOSURE CONDITIONS

Whereas all three asphalts were negative in spot (Skelly "S" solvent) and continued so through most of the test exposures, there appears to be a tendency, with increasing severity in test conditions, to develop positive spots. In this investigation, all the recovered asphalts remained negative after the (h) exposure, even through, under that exposure with Minnesota aggregates, all suffered over 50% losses in penetration, and two were measurably lower than the original in ductility.

Change in the spot characteristics in two of the asphalts was evident after (k) exposure of the loosely spread Michigan aggregate mixtures, but (l) exposure of the compacted mixtures produced very minor change. With the Minnesota aggregate after (k) exposure, one of the mixtures was dried at 250°F to remove moisture prior to extraction. The remaining mixtures were dried at 150°F. The higher temperature apparently affected the properties of the recovered asphalt, thus precluding a valid comparison. Eliminating the results of over heating, the effects of (k) and (l) exposure on Minnesota aggregate mixtures produced less change in spot than Michigan aggregate mixtures, although reduction in penetration was somewhat greater. Asphalts recovered from both Michigan and Minnesota aggregate mixtures, after the relative severe exposure (n), showed appreciable change. In the laboratory study the asphalts tended to become positive as the exposures became more severe. It may be that this also is a tendency of asphalts in service, which has been overlooked. There is evidence that change from negative to positive has occured in some instances in pavements which have continued to give good service, as have others which probably were laid with positive spot asphalts. Examples are the pavements of Colorado Springs, Colorado, and McCook, Nebraska, the latter some 28 years of age, which were noted in the field survey. Perhaps an investigation might uncover many such instances and assist in the evaluation of the spot test as a criterion of acceptability.

A second test series involving (n) exposure was conducted for the dual purpose of providing more comprehensive information on the reaction of the asphalt to the most severe (n) exposure, on spot-test characteristics, to investigate possible influence of different aggregates,and to determine the effect of asphalt components which might be absorbed or adsorbed by the aggregate particles. In this second (n) series, percentages of asphalt components were not determined, the test equipment having been dismantled. In the investigation relative to the effect of asphalt fractions retained on the surface of the aggregate, aggregate recovered from an initial mixture was re-mixed with the same type of asphalt. A comparison of the properties of the asphalts recovered from the fresh and reused aggregates may be made from the results shown in Table D. The results do not appear to be conclusive. It would seem that an aggregate might retain certain components of an asphalt which had been recovered from it, and that, when such aggregate was reused, the second recovered asphalt might show less change from its original properties. As will be noted from Table D, there is no consistent evidence that this is the case with the aggregates used in this study.

The (n) exposure caused reduction in the penetration of the recovered asphalts in the order of 66 to 83%. Softening point temperatures increased between 23.0 to 40.0%. With the Michigan aggregate mixtures, for all asphalts, only one softening point/penetration ratio approached the cricial maximum. With Minnesota aggregates, both new and reused, the critical softening point/ penetration ratio was exceeded in numerous instances.

If a series of exposure tests of increasing intensity, with composition and physical tests on the recovered asphalt, could be initiated and related to parallel tests on the same asphalt from a pavement of various ages, it may be that ultimately a procedure could be developed which would make it possible to estimate the potential service life of that asphalt under defined local conditions.

DEGREE OF HETEROGENEITY

In the special (n) series, attention was directed to the degree of heterogeneity developed by asphalts in the exposure test as related to the type of aggregate and its condition. Each original asphalt was negative to Skelly "S" and in 100% xylene, but was positive in 100% heptane. Increased percentages of xylene were used in successive steps to determine at what point the spot changed from positive to negative. Near the transition point from positive to negative, generally a narrow ring of dark material enclosed a circle only slightly darker or even the same shade as the stain surrounding the ring. These cases are designated as "Ring" in the table. On the basis of these determinations, it appears that the Leonard and Trumbull are somewhat more heterogeneous than is the Lion. This confirms indications in exposures (l) and (k).

It is generally believed that changes in asphalts may be somewhat attributable to characteristics of the aggregates with which they are used and that these changes may be variously developed under conditions of prolonged or severe exposures. Michigan and Minnesota aggregates were employed in an effort to demonstrate such differences. The Michigan aggregate was a natural glacial gravel of a quality acceptable for local state highway use. The Minnesota aggregate was admittedly inferior by State Highway Department standards and was used solely to reduce haulage costs. It was quite soft, absorptive, and contained a high dust content. The asphalt recovered from the paving in which it was used had hardened excessively and had a softening point/penetration ratio of from 5.46 to 6.80 at the time of sampling.

Considering Exposures (a_1), (h), (k), and (l), comparison of the recovered asphalts do not show consistent differences which may be attributed to the aggregate. On the average, however, the asphalts from the Minnesota aggregate mixtures did have a somewhat lower resin/asphaltene ratio, a greater loss in penetration, and a higher softening point. After (n) exposure conditions, however, the decreases in penetration and increases in softening point were always higher after the asphalt had been recovered from the Minnesota aggregate mixtures. It would appear, therefore, that the characteristics of the aggregate may affect the properties of the asphalt recovered from it.

MODIFICATION OF NATURALLY POSITIVE SPOT ASPHALT

Garland asphalt is by nature a positive spot material. It was desired to determine whether or not it could be made and would remain negative under exposure by adding a high resin product, Duct-spot, produced by Century Refining Company, Garden City, Kansas.

The Duct-spot was added in increments of 3% up to 12%, at the producer's recommended temperature of 400°F, to the Garland heated to the same temperature. At 6% the combination still remained positive but at 9% and 12% produced negative spots. The 9% and 12% combinations were mixed with Michigan aggregate and subjected to three exposures of increasing severity. Composition and physical test results before and after recovery are given in Table E.

TABLE E

Material	% Components			Penet	Soft Point	S Pt/Pen Ratio	Duct	Skelly "S" Spot
	Asphtns	Oils	Resin					
Summary of composition and physical properties of Garland and Duct-spot								
Garland orig	21.91	41.27	36.43	91	114.1	1.25	150+	Pos
Garland after 400°F	22.16	41.80	36.04	--	-----	----	----	---
Duct-spot orig	16.17	35.82	48.02	160	102.2	0.64	148	Neg
Duct-spot after 400°F	15.87	34.64	49.49	--	-----	----	----	---
Garland + 6% Duct	21.37	40.23	38.39	87	112.64	1.29	150+	Pos
Garland + 9% Duct	21.10	39.09	39.81	91	109.80	1.21	150+	Neg
Garland *12% Duct	Not tabulated			93	109.00	1.17	150+	Neg
Recovered Garland + 9% Duct-spot after mixing with Michigan aggregate								
Exposure (m)	22.89	34.16	42.95	63	116.80	1.85	150+	Pos
Exposure (j)	26.28	44.69	29.03	35	129.00	3.69	150+	Pos
Exposure (n)	28.53	37.46	34.01	28	140.00	5.00	18	Pos
Recovered Garland + 12% Duct-spot after mixing with Michigan aggregate								
Exposure (m)	23.67	41.37	34.96	54	126.10	2.34	150+	Pos
Exposure (j)	26.97	40.39	32.64	34	136.60	4.02	150+	Pos
Exposure (n)	31.10	36.84	32.06	23	160.20	6.96	8	Pos

NOTE: Exposures (m), (j), and (n), same as Exposures (a), (h), and (n), respectively; see Table C, this report.

*A single determination of composiiion gave results which appear inconsistent with those in the Garland and the 6% and 9% Duct-spot combinations. Because of time limitations only one determination was made on each material.

Table E presents the properties of Garland, Duct-spot, and of combinations of both materials prior to and after exposure. Exposure (m) was the same as (a_1). In the initial test series the (a_1) exposure produced relatively little change in the Wyoming asphalts and their spot remained negative. The recovered Garland Duct-spot blends from the (m) exposure were positive. Exposure (j) was identical with (h). Being more severe, the change in Wyoming asphalts increased, but their spots remained negative. The recovered Garland Duct-spot blends from (j) exposure were positive. Exposure (n) was the same in all tests. It produced greatest change in all asphalts and changed spots to slightly positive and positive.

CONCLUSIONS

From the results of these exploratory investigations, a number of observations can be made. Some confirm those made in the earlier field study. Others, based only on the few materials and the number of duplicate tests which could be run under the limitations of this program, should not be regarded as conclusive.

1. The Bureau of Mines test for the determination of asphalt composition was rather exhaustively studied. Details of procedure were developed which permitted reproducible results. Further refinement dealing with laboratory temperature and humidity conditions, temperature of solvents, and preparation and handling of the anhydrous alumina would doubtless result in still greater accuracy.

2. A comprehensive study of the concurrent changes in composition and physical properties of asphalts should be interesting and informative. The scope of this study was too limited for conclusions.

3. The original compositions of the Leonard, Lion, and Trumbull asphalts are quite similar in terms of the percentages of asphaltenes, oils, and resins.

4. All exposures, from the mildest oven exposure (a_1) to the most severe (n), resulted in changes in the percentages of components of the asphalts and in changes in physical properties. No accurate correlation is possible between the laboratory test conditions and conditions of pavement service, but the characteristics of the recovered asphalts from the (n) exposure, compared with field samples, indicate that it is probably equivalent to several, perhaps many, years of service in a pavement.

5. Hardening increased in all three asphalts under the increasingly severe exposures, but generally the Wyoming asphalts hardened somewhat more rapidly than did the Arkansas. However, under the most severe exposure (n), a Wyoming had the same penetration as the Arkansas with higher ductility and

lower softening point/penetration ratio.

6. Rate of hardening and its practical significance deserves serious study based upon predictive laboratory test sequences and parallel field studies. The earlier pavement survey found asphalts of original penetration of 130 to 260, which, after two or three years, were down to penetration of 50 to 80. Perhaps most of this was due to mixing temperatures, but after a few years penetration losses in well-compacted pavements appear to be slow. Softening points may have behaved similarly, with ultimate critical hardening being approached slowly and depending on the natural characteristics of the asphalt and the conditions of exposure.

7. Both the field survey and the laboratory series emphasize the greater resistance to change of asphalts in dense, highly compacted mixtures.

8. The asphalts were all originally negative in spot and remained so until the most severe oven test and outdoor exposures were reached. Apparently all three tended toward heterogeneity, but the Wyoming reached that condition somewhat earlier. This is further shown by the xylene equivalent tests made on asphalts recovered from the (n) exposure.

9. The xylene equivalent technique offers a simple method of studying the tendency of asphalts to develop heterogeneity.

10. Many satisfactory pavements containing positive spot asphalt were found in the earlier field investigation. Some were doubtless laid with positive spot asphalt. Other evidence shows that negative asphalts may change to positive in service, as occurred in some of the outdoor exposures of this study. An extended study of the significance of positive spot asphalts in existing pavements would be very useful.

11. Asphalt properties appear to be somewhat affected by the nature and composition of the aggregates with which an asphalt is combined. The changes in asphalt were generally greater with the Minnesota than with the Michigan aggregates used in this test.

12. The special series in which Duct-spot additive was used with positive Garland asphalt did not produce combinations which were negative under any exposure test.