

Effect of Mixing Temperature on Hardening of Asphaltic Binder in Hot Bituminous Concrete

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Plant mixes of hot bituminous concrete were made in the mixing temperature range of 250 to 400 F, corresponding to viscosity limits of approximately 400 to 15 Saybolt Furol seconds. The asphalt from samples of these mixes was recovered by the modified Abson method, and changes in the penetration, ductility, softening point, and absolute viscosity were measured.

Asphalts from Venezuelan and East Texas crudes were used. The mixes used were base courses, binder courses, wearing surfaces, and certain city street mixes. The aggregates were varied and were either all granite or mixtures of granite, sand, and mineral filler, mixtures of sandy limestone, sand and mineral filler, or mixtures of siliceous gravels, sand, and mineral filler.

Some of the samples were cooled slowly, whereas others were cooled rapidly by quenching in water. The samples cooled slowly showed 65 to 75 percent penetration retained at 250 F with an additional 10 percent drop in percent penetration retained for each 50 F increase in mixing temperature. The samples quenched in water showed essentially no difference in hardening at 250 F from slowly cooled.

No definite difference in hardening was found for different asphalts, different mixes, different aggregates, or different type pugmills.

Samples of paving mixture were found to harden appreciably on storage during the first 15 days; but thereafter the rate of hardening was very slow.

• THE HARDENING of asphaltic binder in hot bituminous concrete has been a matter of concern to asphalt technologists for more than half a century. Hardening involves a loss in penetration, a loss in ductility, an increase in softening point, and an increase in viscosity. When these changes occur, the asphaltic binder becomes brittle, loses flexibility, cracks, and reduces the service life of the pavement. As a rough approximation, it has been found that when the penetration of asphalt recovered from the pavement reaches values of 20 to 30 (7, 14), the pavement will show indications of cracking and will have approached the end of its useful life.

It is a generally accepted fact that a considerable amount of hardening occurs in the mixing operation during the short time that the asphalt is being heated by the hot aggregate. Such hardening may be 20 to 50 percent of the original penetration. Furthermore, it is generally accepted that high mixing temperatures bring about excessive hardening and should be avoided.

In 1943, Nevitt and Donaldson (10) suggested that mixing temperatures be determined by the viscosity of the asphaltic binder rather than mixing at some fixed temperature. They suggested viscosity limits of 75 to 150 Saybolt Furol seconds. Although such limits are arbitrary and have not been checked experimentally, this range of viscosities or some modification of this range has been widely used to specify proper mixing temperatures.

While mixing temperature is an important factor affecting hardening in the mixing

process, there are other mixing variables of considerable importance. Asphalts are known to vary widely in their susceptibility to hardening. It has been suggested that the size of the aggregate, the absorptive and catalytic effect of the aggregate surfaces, the mixing time, and the design of the mixer may all play a part in the hardening that occurs. More large-scale information is needed to evaluate the magnitude of these effects properly and to establish correlations between the results obtained in the field and the laboratory tests that have been developed to predict the hardening behavior of the asphalt during the mixing period and after it has been placed on the road.

SCOPE OF INVESTIGATION

It should be emphasized that the hardening that occurs during the mixing operation is only one of several factors that determine proper mixing temperatures. The moisture content of the aggregate, the mixing time, the completeness of mixing, the mixing power requirements, the proper lay-down and compaction temperatures and, above all, the final performance of the material in the road, all play their parts in selecting the proper mixing temperature. This investigation helps to solve only a part of the overall problem.

This project was concerned primarily with the study of hardening of asphaltic binders in the mix boxes of commercial asphalt plants producing hot bituminous concrete. The tests made were designed so as to obtain the following information regarding hardening:

1. The effect of mixing temperature in the range of 250 to 400 F.
2. The effect of the type of mix.
3. The effect of aggregate composition.
4. The effect of asphalt.
5. The effect of plants.

During the course of the study, certain sidelines of investigation developed: (a) the effect of the rate of cooling on the hardening of paving samples, and (b) the effect of storage on the hardening of loose paving samples.

Samples of six different types of mixes were taken from eight different plants within North Carolina during the 1960 construction season. The asphalts used by these plants were from three different producers, and the aggregates used in each of the mixes were of several different types.

The project was carried out during the period, July 1, 1959, to June 30, 1961, with the financial assistance of the North Carolina State Highway Commission, the U.S. Bureau of Public Roads, and the North Carolina Asphalt Association.

The investigation (Highway Research Project ERD-110-G) was transacted within the Highway Research Program conducted by the Department of Civil Engineering at North Carolina State College.

EQUIPMENT AND PROCEDURES

Sampling

Samples of paving mixtures were taken from trucks immediately after their leaving the mixer and just before they left the plant site. From 2 to 5 min usually elapsed between the time the material left the mixer and the time it was sampled.

Temperatures were measured by inserting dial-type asphalt thermometers with 6-in. stems in the top of the load. Usually, two thermometers were used by the project personnel and checked with the inspector's thermometer. Each of the thermometers used by the project personnel was calibrated against mercury thermometers by placing the two together in a heated oil bath.

The temperature of the aggregate was raised or lowered until the material leaving the mix box and falling into the truck had reached the desired temperature. The aggregate and asphalt temperatures were taken from plant thermometers or pyrometers. When the truck to be sampled had pulled out from under the mix box and the temperature had been checked, the top 5 or 6 in. of the batch to be sampled was scraped away and the underlying material scooped up from several spots and placed in a cardboard box

or a peck-bucket, depending on whether the sample was to be quenched or unquenched. Unquenched samples were placed in a cardboard box approximately 18 by 12 by 12 in. and allowed to cool at a natural rate.

Quenched samples were placed in a peck-bucket that had been filled with tap-water

ASPHALT

Group	Sample	Type	Mix			Aggregate					
			Quantity (ton batch)	Mixing Time (sec)	Specified Temp (°F)	Composition	Type	Percent	Source	2-In Sieve	1/4-Sieve
I	17-20A	Bituminous concrete base course—H-B (BCBC)	3	35	285	Gresham No 1 stone	Granite	35 0	Wake Co	100 0	97
						Erwin No 11 gravel	Assorted gravel	38 0	Harnett Co	-	-
						Local sand	Silica	28 4	Cumberland Co	-	-
						Mineral filler	Limestone	0 6	Buchanan, Va	-	-
II	21-24	Bituminous concrete surface course—1-2 (1-2 WS)	3	45	285	Gresham No 1 stone	Granite	15 0	Wake Co	-	-
						Erwin No 11 gravel	Assorted gravel	14 0	Harnett Co	-	-
						Local sand	Silica	34 0	Cumberland Co	-	-
						Gresham screenings	Granite	34 0	Wake Co	-	-
						Mineral filler	Limestone	3 0	Buchanan, Va	-	-
III	25-25C	Bituminous concrete surface course—1-2 (1-2 WS)	3	45	285	Gresham No 1 stone	Granite	15 0	Wake Co	-	-
						Erwin No 11 gravel	Assorted gravel	14 0	Harnett Co	-	-
						Local sand	Silica	34 0	Cumberland Co	-	-
						Gresham screenings	Granite	34 0	Wake Co	-	-
						Mineral filler	Limestone	3 0	Buchanan, Va	-	-
IV	26-28	Bituminous concrete surface course—mod F-2 (Mod F-2 WS)	2	30	-	Rolesville stone	Granite	-	Wake Co	-	-
						Rolesville screenings	Granite	-	Wake Co	-	-
V	29-32A	Bituminous concrete surface course—mod F-2 (Mod F-2 WS)	2	45	-	Rocky Mount No 11 stone	Granite	-	Nash Co	-	-
						Rocky Mount No 13 stone	Granite	-	Nash Co	-	-
						Local sand	Silica	-	Nash Co	-	-
						Rocky Mount screenings	Granite	-	Nash Co	-	-
VI	33-37	Sand asphalt surface course—F1 (SAWS)	2	45	285	Local sand	Silica	66 7	Nash Co	-	-
						Rocky Mount screenings	Granite	33 3	Nash Co	-	-
VII	38-41	Bituminous concrete surface course—1-3 (1-2 WS)	2	45	285	Rocky Mount No 13 stone	Granite	30 0	Nash Co	-	-
						Local sand	Silica	35 0	Nash Co	-	-
						Rocky Mount screenings	Granite	35 0	Nash Co	-	-
VIII	42-45	Bituminous concrete surface course—mod F-2 (Mod F-2 WS)	1	45	-	No 11 stone	Granite	50 0	Burke Co	-	-
						Local sand	Silica	25 0	Burke Co	-	-
						Screenings	Granite	25 0	Burke Co	-	-
IX	46-50	Bituminous concrete binder course—H (H binder)	3	45	285	Hendrick No 5 gravel	Assorted gravel	37 0	Anson Co	-	-
						Hendrick No 13 gravel	Assorted gravel	23 0	Anson Co	-	-
						Hendrick fine sand	Silica	37 5	Anson Co	-	-
						Mineral filler	Limestone	2 5	Buchanan, Va	-	-
X	51-54	Bituminous concrete surface course—1-2 (1-2 WS)	3	45	285	Hendrick No 13 gravel	Assorted gravel	28 0	Anson Co	-	-
						Hendrick blended sand	Silica	67 5	Anson Co	-	-
						Mineral filler	Limestone	4 5	Buchanan, Va	-	-
XI	55-55E	Bituminous concrete surface course—mod F-2 (Mod F-2 WS)	2	30	-	Rolesville stone	Granite	-	Wake Co	-	-
						Rolesville screenings	Granite	-	Wake Co	-	-
XII	56-60	Bituminous concrete binder course—H (H binder)	2	55	285	New Bern 7/8-in stone	Sandy shell limestone	31 0	Craven Co	-	-
						New Bern No 11 stone	Sandy shell limestone	28 0	Craven Co	-	-
						Local sand	Silica	24 0	Craven Co	-	-
						New Bern screenings	Sandy shell limestone	15 0	Craven Co	-	-
						Mineral filler	Limestone	2 0	Buchanan, Va	-	-
XIII	61-64	Bituminous concrete surface course—1-2 (1-2 WS)	2	55	285	New Bern No 13 stone	Sandy shell limestone	28 0	Craven Co	-	-
						Local sand	Silica	15 0	Craven Co	-	-
						New Bern screenings	Sandy shell limestone	53 0	Craven Co	-	-
						Mineral filler	Limestone	4 0	Buchanan, Va	-	-
						McKenzie sand	Silica	98 0	Scotland Co	-	-
Mineral filler	Limestone	2 0	Buchanan, Va	-	-						
XIV	65-68	Sand asphalt base course—F-1 (SABC)	3	45	285	Teer No 11 stone	Traprock	40 0	Durham Co	-	-
						Duncan sand	Silica	60 0	Harnett Co	-	-
XV	70-74	Bituminous concrete surface course—mod F-2 (Mod F-2 WS)	2	52	-	Local coarse sand	Silica	65 0	Cumberland Co	-	-
						Local fine sand	Silica	30 0	Sampson Co	-	-
						Mineral filler	Limestone	5 0	Buchanan, Va	-	-
XVI	75-78	Sand asphalt surface course—F-1 (SAWS)	3	50	285	Local coarse sand	Silica	65 0	Cumberland Co	-	-
						Local fine sand	Silica	30 0	Sampson Co	-	-
						Mineral filler	Limestone	5 0	Buchanan, Va	-	-
XVII	79-82A	Sand asphalt surface course—F-1 (SAWS)	2	-	-	-	-	-	-	-	-
						-	-	-	-	-	-
						-	-	-	-	-	-
XVIII	83-85	Sand asphalt surface course—F-1 (SAWS)	2	-	285	Local sand	Silica	14 0	Craven Co	-	-
						New Bern screenings	Sandy shell limestone	32 0	Craven Co	-	-
						Mineral filler	Limestone	4 0	Buchanan, Va	-	-
XIX	90-92	Bituminous concrete surface course—1-2 (1-2 WS)	1	45	285	Gold Hill No 13 stone	Argillite	32 0	Rowan Co	-	-
						Harrison sand	Silica	23 0	Montgomery Co	-	-
						Woodleaf screenings	Granite	45 0	Rowan Co	-	-
						-	-	-	-	-	-

^aPercentage of total by weight

^bProduced by atmospheric and vacuum distillation

TABLE 3
THIN FILM OVEN TEST RESULTS ON ORIGINAL ASPHALTS

Group	Sample No.	Plant	Producer	Crude Source	Weight Loss		Penetration		
					(G)	(%)	Original (0.1 mm)	Residue (0.1 mm)	Retained (%)
II	21-24	A	1	Venezuela	0.2832	0.551	93	51	54.8
IV	26-28	B	2	East Texas	0.1980	0.385	96	53	55.2
V	29-32A	C	2	East Texas	0.1455	0.283	91	51	56.0
VII	38-41	C	2	East Texas	0.2000	0.389	94	50	53.2
VIII	42-45	D	2	East Texas	0.1937	0.377	93	50	53.8
IX	46-50	E	1	Venezuela	0.3666	0.712	91	48	52.7
XII	56 60	F	1	Venezuela	0.3983	0.774	94	49	52.1
XIV	65-68	E	1	Venezuela	-	-	92	46	50.0
XV	70-74	G	3	Venezuela (70%) Texas (30%)	0.0770	0.149	90	53	58.9
XVII	79-82A	B	2	East Texas	0.2006	0.389	90	46	51.1
XVIII	83-85	F	1	Venezuela	0.2792	0.543	84	47	56.0
XIX	90-92	H	3	Venezuela (70%) Texas (30%)	0.0920	0.178	90	54	60.0

TABLE 4
TEST RESULTS ON QUENCHED AND UNQUENCHED SAMPLES TAKEN AT PLANTS

Group	Sample No.	Plant	Asphalt Producer	Type of Mix	Mixing Temp. (°F)	Ash (%)	Penetration			Softening Point			Ductility		Viscosity			
							Original (0.1 mm)	Recovered (0.1 mm)	Retained (%)	Original (°F)	Recovered (°F)	Increase (%)	Original (cm)	Recovered (cm)	Decrease (%)	Original (x 10 ⁶)	Recovered (x 10 ⁶)	Increase (%)
I	20	A	1	BCBC	390	5.6	93	49	52.7	111.7	128.3	14.9	150+	138	-	1.18	-	-
	20A	A	1	BCBC	390	-	93	65	69.9	111.7	119.1	8.6	150+	150+	-	1.18	2.3	88.5
II	22	A	1	1-2 WS	390	-	93	41	44.1	112.6	132.6	17.8	150+	80	-	1.14	10.0	775
	22A	A	1	1-2 WS	390	-	93	63	67.7	112.6	120.6	7.1	150+	150+	-	1.14	3.7	224
III	25	A	1	1-2 WS	280	-	93	71	76.3	-	-	-	-	-	-	-	-	-
	25A	A	1	1-2 WS	280	-	93	77	82.8	-	-	-	-	-	-	-	-	-
IV	27	B	2	Mod F-2 WS	388	-	96	38	39.6	109.4	131.4	20.1	150+	111	-	1.08	5.4	400
	27A	B	2	Mod F-2 WS	388	-	96	54	56.3	109.4	119.8	9.5	150+	150+	-	1.08	4.5	317
V	33	C	2	Mod F-2 WS	255	-	91	60	65.9	109.9	120.4	9.6	150+	150+	-	-	3.5	-
	32A	C	2	Mod F-2 WS	255	-	91	61	67.0	109.9	119.5	8.7	150+	-	-	-	3.0	-
	31	C	2	Mod F-2 WS	397	-	91	34	37.4	109.9	135.5	23.3	150+	96	-	-	8.8	-
	31A	C	2	Mod F-2 WS	397	-	91	56	61.5	109.9	121.6	10.6	150+	150+	-	-	3.6	-
VI	36	C	2	SAWS	417	6.6	91	24	28.4	111.6	151.0	35.3	150+	7	-	-	7.1	-
	36A	C	2	SAWS	417	-	91	51	56.0	111.6	123.3	10.5	150+	150+	-	-	-	-
XI	55	B	2	Mod F-2 WS	349	-	96	49	51.0	-	-	-	-	-	-	-	-	-
	55A	B	2	Mod F-2 WS	349	2.45	96	60	62.5	-	-	-	-	-	-	-	-	-
XVII	82	B	2	SAWS	298	-	90	52	57.8	-	-	-	-	-	-	-	3.5	-
	82A	B	2	SAWS	298	-	90	52	57.8	-	-	-	-	-	-	-	-	-
	79	B	2	SAWS	304	0.50	90	52	57.8	-	-	-	-	-	-	-	3.0	-
	79A	B	2	SAWS	304	-	90	52	57.8	-	-	-	-	-	-	-	-	-
	80	B	2	SAWS	356	-	90	44	48.9	-	-	-	-	-	-	-	6.8	-
	80A	B	2	SAWS	356	-	90	52	57.8	-	-	-	-	-	-	-	-	-
	81	B	2	SAWS	412	-	90	30	33.3	-	-	-	-	-	-	-	19.0	-
	81A	B	2	SAWS	412	-	90	50	55.6	-	-	-	-	-	-	-	-	-

*Samples designated by A were quenched in water

TABLE 5
TEST RESULTS ON QUENCHED AND UNQUENCHED SAMPLES TAKEN BETWEEN PLANTS AND PAVERS

Group	Sample No.	Plant	Type of Mix	Mixing Temp. (°F)	Ash (%)	Penetration			Remarks
						Original (0.1 mm)	Recovered (0.1 mm)	Retained (%)	
III	25	A	1-2 WS	280	-	93	71	76.3	At plant site
	25B	A	1-2 WS	275	-	93	71	76.3	After 35 min in truck
	25A	A	1-2 WS	280	-	93	77	82.8	Quenched at plant site
	25C	A	1-2 WS	275	-	93	72	77.4	Quenched after 35 min in truck
XI	55	B	Mod. F-2 WS	349	-	96	49	51.0	At plant site
	55B	B	Mod. F-2 WS	349	-	96	42	43.7	After 25 min in truck
	55D	B	Mod. F-2 WS	349	-	96	46	47.9	After 45 min in truck
	55A	B	Mod. F-2 WS	349	2.45	96	60	62.5	Quenched at plant site
	55C	B	Mod. F-2 WS	349	-	96	52	54.2	Quenched after 25 min in truck
	55E	B	Mod. F-2 WS	349	-	96	53	55.2	Quenched after 45 min in truck

Toward the end of the project, one of the unquenched samples was prepared to study the effect of storage. The sample was divided into four portions with each portion being stored as follows: the first portion was broken up and spread out in a layer approximately 2 in. deep; the second portion was broken up and sealed in a 1-gal container in the presence of air; the third portion was broken up and stored in a 1-gal container in the presence of carbon dioxide; and the fourth portion was left unbroken in the cardboard box. All of these portions were subjected to the extraction and recovery process at the end of 30 days. The fourth portion was also subjected to the same process at the end of 1, 9, 15, 21, 29, 62, and 120 days.

Extraction and Recovery

Paving samples were extracted with Faulwetter extractors. Some samples were

TABLE 6
TEST RESULTS ON MIX STORAGE SAMPLES

Group	Sample No	Plant	Type of Mix	Mixing Temp (°F)	Ash (%)	Days of Storage	Penetration			Remarks
							Original (0.1 mm)	Recovered (0.1 mm)	Retained (%)	
III	25 w/t	A	I-2 WS	280	-	3	93	66	71 0	Mix sample stored in cardboard box, original AC stored in 3-oz can
	25 w/t (aged)	A	I-2 WS	280	-	120	-	50	-	
XIX	90	H	I-2 WS	315	-	1	-	55	-	Mix sample stored in cardboard box, original AC stored in 3-oz can
	90-1	H	I-2 WS	315	-	6	-	52	-	Mix sample stored in cardboard box, original AC stored in 3-oz can
	90-2	H	I-2 WS	315	-	15	90	47	52 2	Mix sample stored in cardboard box, original AC stored in 3-oz can
	90-3	H	I-2 WS	315	-	21	-	48	-	Mix sample stored in cardboard box, original AC stored in 3-oz can
	90-4	H	I-2 WS	315	-	29	87	48	55 2	Mix sample stored in cardboard box, original AC stored in 3-oz can
	90-5	H	I-2 WS	315	-	62	87	47	54 0	Mix sample stored in cardboard box, original AC stored in 3-oz can
	90-6	H	I-2 WS	315	-	120	85	42	49 4	Mix sample stored in cardboard box, original AC stored in 3-oz can
	90-4	H	I-2 WS	315	-	29	87	48	55 2	Unbroken sample stored in cardboard box
	90A	H	I-2 WS	315	-	28	87	48	55 2	
	90B	H	I-2 WS	315	-	30	87	48	55 2	Sample sealed in air in 1-gal container
	90C	H	I-2 WS	315	-	31	87	51	58 6	Sample sealed in CO ₂ in 1-gal container

TABLE 7
TEST RESULTS ON ASPHALT STORAGE SAMPLES

Group	Sample No	Plant	Type of Mix	Mixing Temp. (°F)	Days of Storage			Penetration		
					Mix	A. C. ^a	Total	Original (0.1 mm)	Recovered (0.1 mm)	Retained (%)
XIX	90	H	I-2 WS	315	1	1	2	-	-	-
	90a	H	I-2 WS	315	1	15	16	90	55	61.1
	90b	H	I-2 WS	315	1	29	30	87	52	59.8
	90c	H	I-2 WS	315	1	62	63	87	55	63.2
	90d	H	I-2 WS	315	1	95	96	87	55	63.2
	90e	H	I-2 WS	315	1	120	121	85	54	63.5
	90-2	H	I-2 WS	315	15	1	16	90	47	52.2
	(90-2)a	H	I-2 WS	315	15	15	30	87	45	51.7
	(90-2)b	H	I-2 WS	315	15	48	63	87	48	55.2
	(90-2)c	H	I-2 WS	315	15	81	96	87	45	51.7
	(90-2)d	H	I-2 WS	315	15	106	121	85	45	52.9
	88-92	H	-	-	-	-	^b 121	86	-	-

^aStored in 3-oz penetration containers.

^bStored in 1-gal container.

extracted with Fisher reagent grade benzene and others with Perm A Chlor-NA trichloroethylene from Detrex Chemical Company of Detroit, Mich. The extract was centrifuged at a centrifugal force of 445 times gravity for 15 min, and the asphalt recovered by the Abson method.

The penetration, ductility, and softening point were determined for the recovered asphalts using ASTM standard methods. Ash contents of some samples were determined. The absolute viscosity of a few of the asphalts was measured with a sliding plate viscosimeter.

The penetration, softening point, ductility, and Bureau of Public Roads thin film oven test values were obtained for the original asphalts. The viscosity of these samples was checked with a Saybolt viscosimeter and found to be in good agreement with viscosity information furnished by the asphalt supply sources.

RESULTS

The results of tests made on the original and recovered asphalts are given in Tables 2 through 8. Manufacturers tests are given in Table 9 and in Figure 1. Characteristics of the pugmills used are given in Table 10. Data on the mixes and aggregates are given in Table 1.

Effect of Mixing Temperature

The effect of mixing temperature on percent retained penetration is shown in Figure 2.

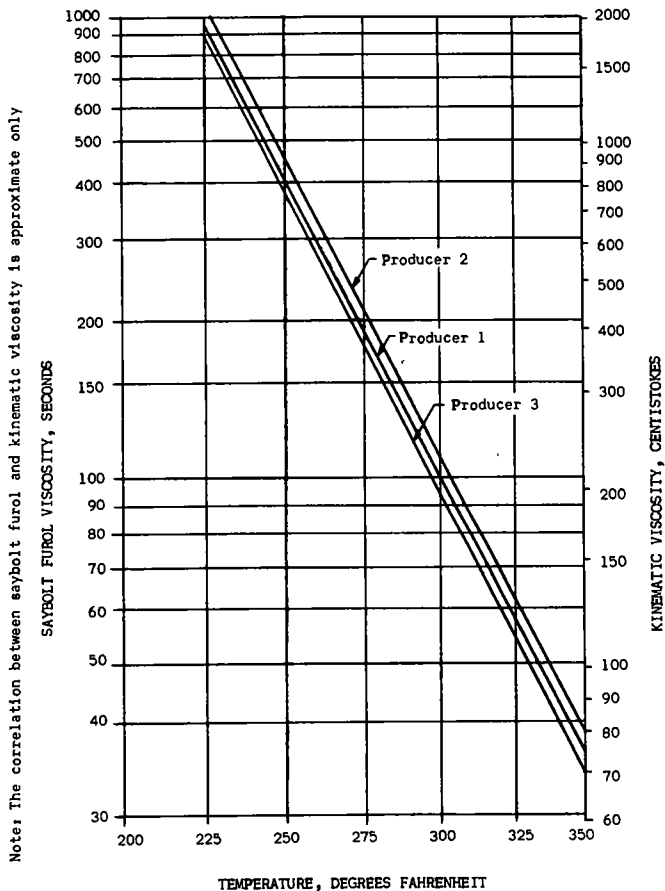


Figure 1. Typical viscosity-temperature relationships as supplied by the producers.

The percent retained penetration decreases about 2 percent for each 10 F increase in mixing temperature until a mix temperature of 340 F is obtained. At temperatures in excess of 340 F, the rate of decrease in retained penetration becomes more severe.

Figure 3 shows a comparison of the data of this project with that of other investigations (8, 12, 15). The asphalts used in this project behave similarly to those shown by Schaub and Parr (15).

TABLE 8
COMPARISON OF RECOVERED PENETRATIONS WITH THIN FILM OVEN
TEST PENETRATIONS

Group	Plant	Type of Mix	Producer	Percent Penetration Retained		
				Mix at 325 F	T. F. O. Test ¹	Difference
XVIII	F	SAWS	1	53.5	56.0	2.5
II	A	I-2 WS	1	57.0	54.8	2.2
IX	E	H binder	1	54.3	52.7	1.6
XII	F	H binder	1	52.5	52.1	0.4
XIV	E	SABC	1	60.5	50.0	10.5
V	C	Mod. F-2 WS	2	54.3	56.0	1.7
IV	B	Mod. F-2 WS	2	51.4	55.2	3.8
VIII	D	Mod. F-2 WS	2	48.3	53.8	5.5
VII	C	I-2 WS	2	52.0	53.2	1.2
XVII	B	SAWS	2	54.6	51.1	3.5
XIX	H	I-2 WS	3	59.0	60.0	1.0
XV	G	Mod. F-2 WS	3	55.5	58.9	3.4

¹At 325 F, 5 hr.

TABLE 9
TYPICAL TEST RESULTS AS SUPPLIED BY THE PRODUCERS

Test	Result		
	Producer 1	Producer 2	Producer 3
Specific gravity at 60 F	1.037	1.0328	1.03
Flash point (COC) (°F)	510	555	565
Solubility in CCl ₄ (%)	99.92	99.80	99.83
Penetration at 77 F, 100 g, 5 sec	92	91	96
Softening point (R & B) (F)	119	117	114
Ductility at 77 F (cm)	150+	-	-
Viscosity at 275 F, SSF. (sec)	237	200	162
Loss on heating at 325 F, 50 g, 5 hr:			
Loss by weight (%)	0.053	0.1822	0.03
Penetration of residue at 77 F	87.0	-	88.5
Ductility at 77 F (cm)	150+	-	100+
Thin film oven test:			
Loss by weight (%)	0.64	-	-
Penetration of residue at 77 F, percent of original	58.0	54.1	-
Ductility at 77 F (cm)	150+	-	100+

The results indicate that from the standpoint of hardening, it would be advantageous to mix the asphalts used in North Carolina at lower mix temperatures than now used. However, the hardening occurring in the mixing operation is only one of the factors that must be considered in selecting the proper mix temperature.

The samples mixed at temperatures lower than 350 F retained ductilities of 150 CM +.

TABLE 10
PUGMILL INFORMATION ON THE VARIOUS PLANTS

Plant	Pugmill			Shaft Speed (rpm)	Periph. Speed of Paddles (fpm)	Area of Each Paddle (sq in.)	Angle of Paddle to Shaft (deg)	Method of Introducing Asphalt to Pugmill
	Length (ft)	Width (ft)	Volume (cu ft)					
A	7.00	5.92	59.40 ^a	55	460	32	45	Sprayed under pressure by spray bar with fan-type nozzles
B	6.42	5.25	39.60 ^a	61	390	30	45	Spray bar with 20 fan-type nozzles in center of mixer, asphalt sprayed under 50 lb pressure
C	5.92	5.92	50.20 ^a	55	460	32	45	Sprayed under pressure by spray bar with fan-type nozzles
D	3.17	7.31	23.00 ^a	36	435	72	45	Pressure spray, one spray bar running width of pugmill
E	7.83	6.00	58.85 ^a	56	568	49	45	Two rows of gravity "fans" (11 per shaft) running length of pugmill
F	5.75	6.00	107.00 ^b	45	365	30.25	45	Spray bar with 30 fan-type nozzles in center of mixer, asphalt sprayed under 50 lb pressure
G	6.42	5.25	39.60 ^a	61	390	30	45	Spray bar with 20 fan-type nozzles in center of mixer; asphalt sprayed under 50 lb pressure
H	8.00	3.83	47.30 ^c	68	427	22.25	30	Pressure spray, one spray bar running width of pugmill in spray chamber at aggregate inlet point

^aVolume below centerline of shaft.

^bTotal net volume.

^cVolume at top of liner plates.

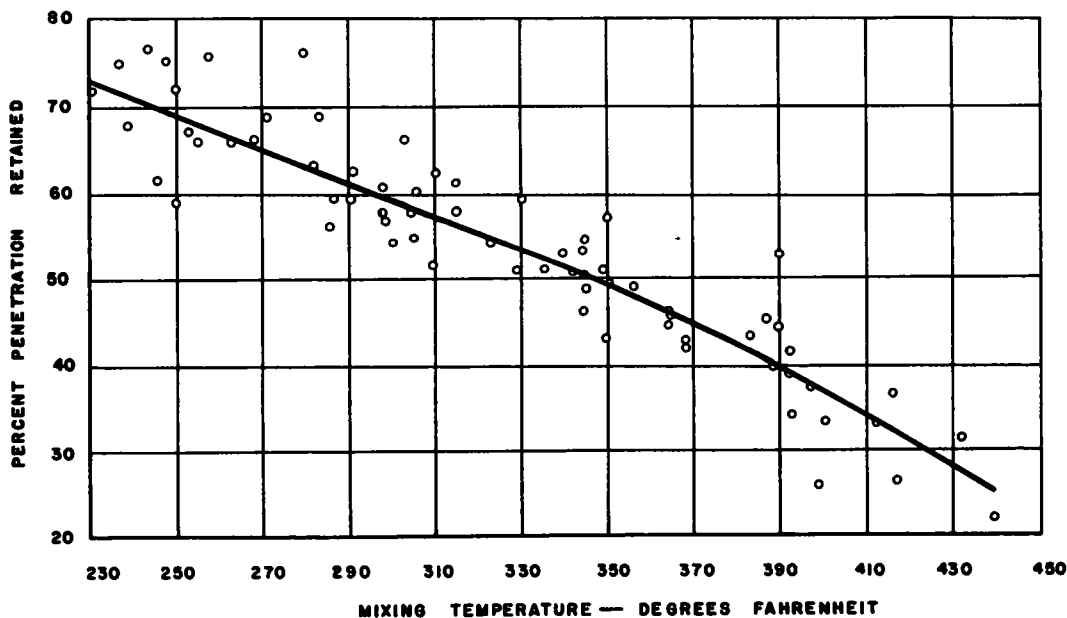


Figure 2. Relation between percent penetration retained and mixing temperature.

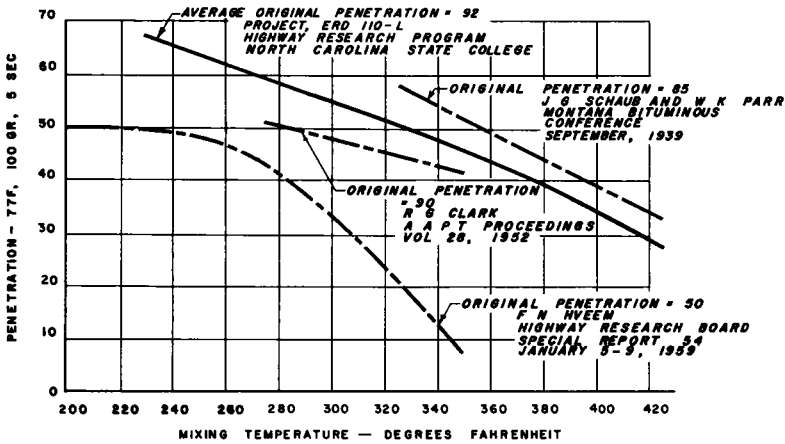


Figure 3. Comparison of penetration-mixing temperature relation with those of other studies.

At extremely high mix temperatures, ductilities decreased. One sample mixed at 440F showed a ductility of only 5 cm.

A limited number of the recovered asphalts were tested for absolute viscosity with the sliding plate viscosimeter. The shear rate-viscosity curves were extrapolated to a shear rate of 0.05 sec^{-1} . Most of the recovered asphalts showed non-Newtonian behavior. The determinations were rather scattered and followed roughly the trend of the relationship set forth by Pheffer (13).

$$\text{Viscosity (poises)} = \frac{1.58 \times 10^{10}}{\text{penetration}} 2.16$$

For the most part, viscosities determined were higher than those found from this relationship. This formula applies to Newtonian asphalts.

Effect of Other Factors

Aggregates.—Considerable difficulty was found in getting aggregates of one mineralogical type in the paving mixtures. This was brought about because many plants use a mineral filler of ground limestone and because many mixes contained sand along with the other types of aggregates so that siliceous and limestone material formed a part of many mixes. Some mixes of granite only were obtained and variations in aggregate type were made from plant to plant. There appeared no noticeable difference in the hardening obtained with different aggregates.

An effort was made to compare the characterizing factors* of the original and recovered asphalts as suggested by Kinnaird (9). In some instances, this factor remained constant. In others, there was as much as 50 percent increase though the change was more normally 10 and 20 percent. The value in two instances showed 10 to 15 percent decreases. Both of these batches were made with sandy limestone aggregates.

On this basis, the work of Kinnaird would indicate hardening from effects other than oxidation, but whether this is from aggregate type of other variables is not known.

Plants.—Plants of several manufacturers were represented. There appeared to be no clear cut evidence that the type of plant had appreciable effect on hardening obtained (e.g., Fig. 4). Figure 5, for example, shows the hardening obtained for an I-2 wearing surface from five different plants and the results are not very different. Some of the variations shown could have been caused by asphalt type or a difference in aggregates.

*For a more complete discussion of the characterizing factors, see Appendix.

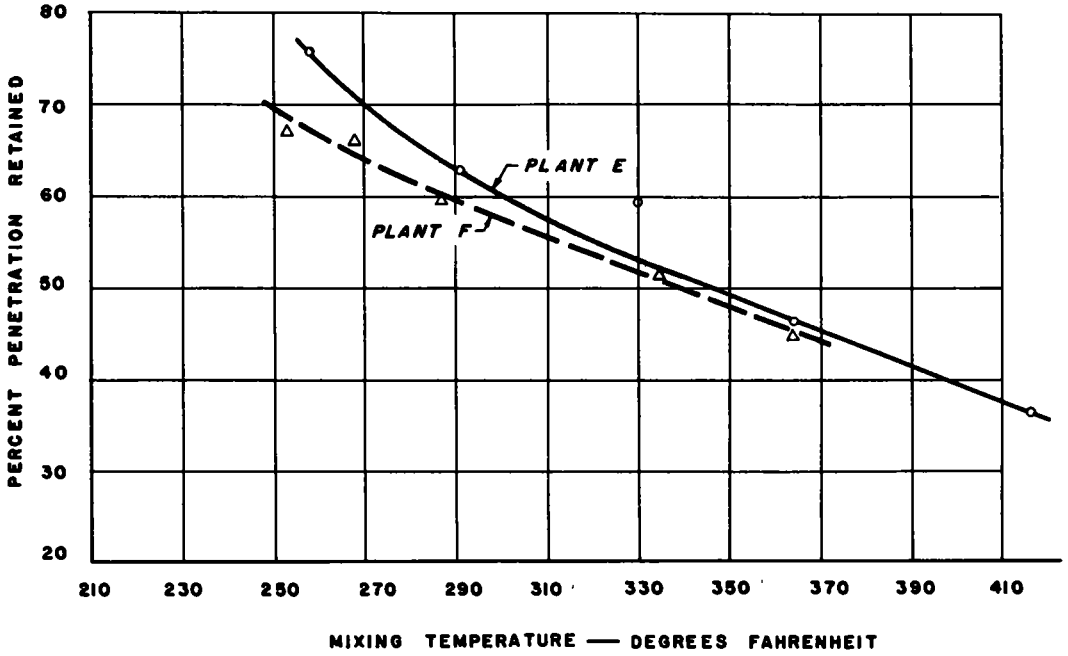


Figure 4. Percent penetration retained for H-binder mix from designated plants.

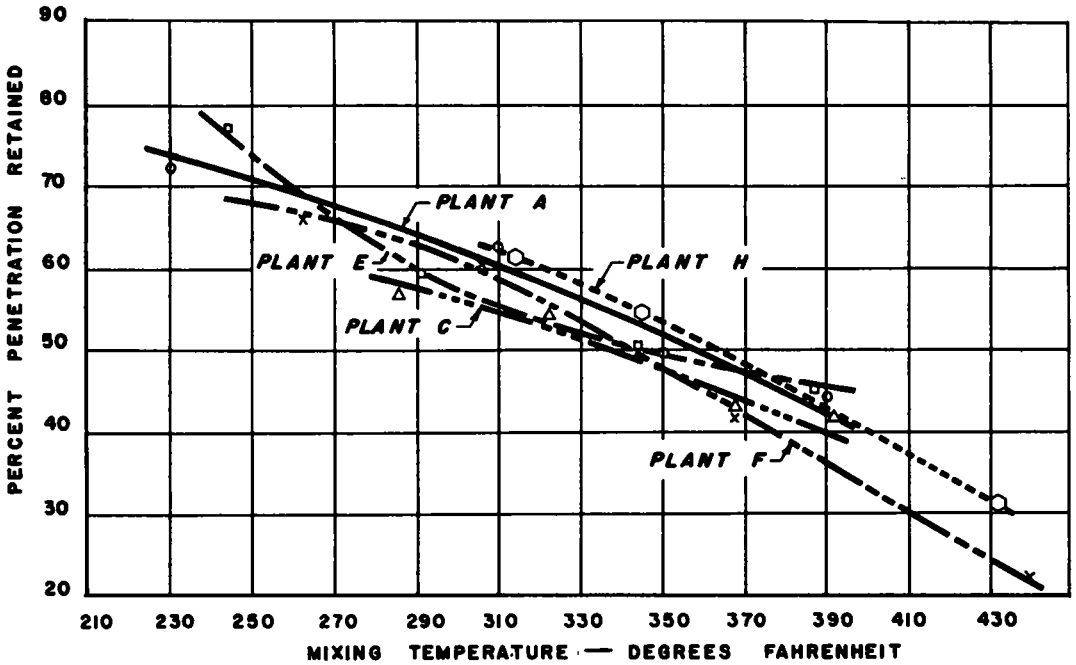


Figure 5. Percent penetration retained for I-2 WS mix from designated plants.

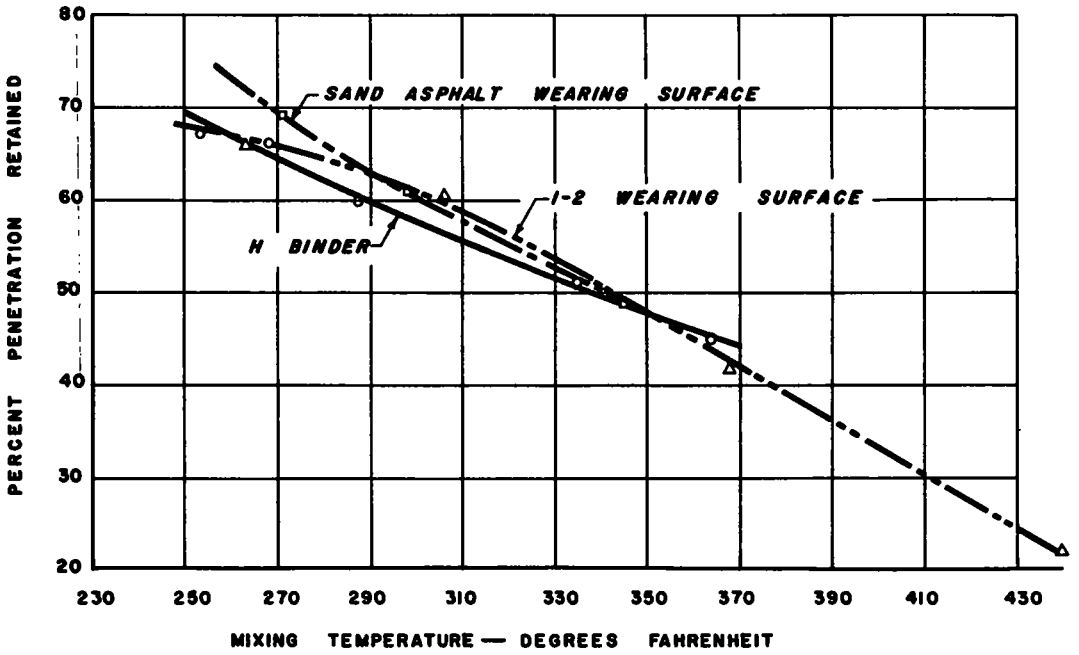


Figure 6. Percent penetration retained for mixes from Plant F.

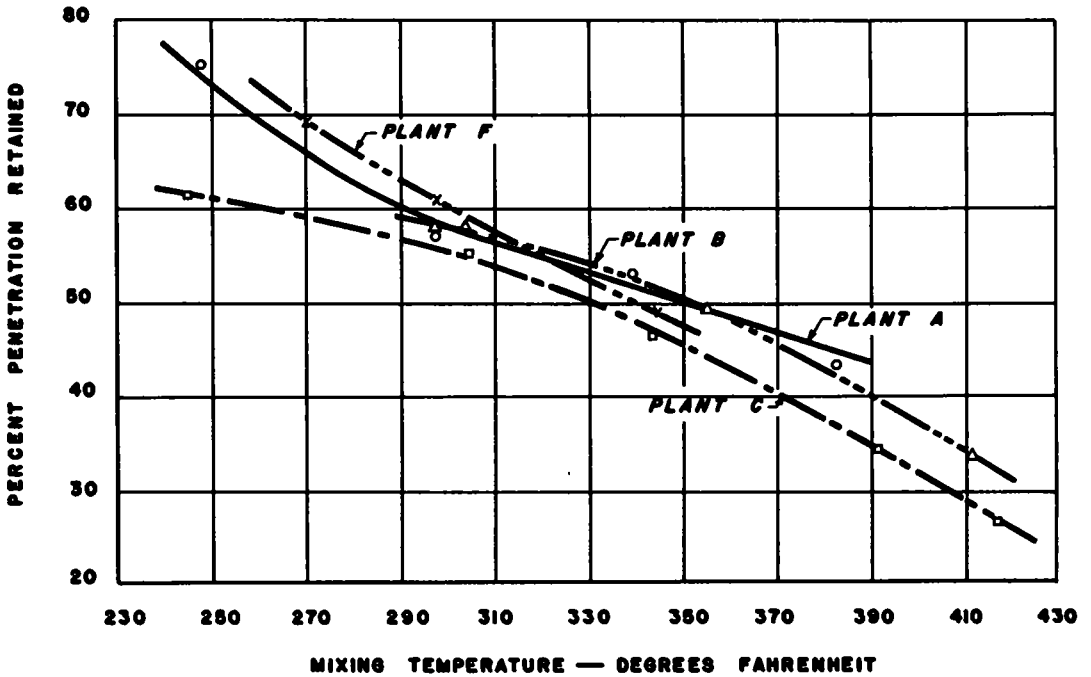


Figure 7. Percent penetration retained for SAWS mix from designated plants.

Samples taken from a continuous mixer at standard mix temperatures showed about the same amount of hardening as batch plants.

Mixes.—The results of three different mixes made at Plant F are shown in Figure 6. There is no significant difference in the hardening obtained with the different mixes. Data from other plants indicate no definite effect on hardening of mix composition. Another example is shown in Figure 7.

Asphalts.—The asphalts used in this project were from three different sources but thin film oven tests, viscosities, and other properties varied little. Table 8 gives some typical test results that were supplied by the producers for their 85-100 penetration grade asphalts, and Figure 1 shows typical viscosity-temperature relationships as supplied by the same producers.

There appears to be some difference in viscosity values at 275 F between the table values and the curves. This is brought about by variations in the asphalt from time to time as the tables or charts were compiled. Tests made on the viscosity of the asphalts indicate the curves in Figure 1 represent the material used in the mixes of this project.

Table 9 gives the results of the thin film oven tests compared with hardening obtained in the plant mixes at 325 F. This temperature probably approaches the maximum for acceptable operations in construction. The retained penetrations of the laboratory tests and of the field mixing are in excellent agreement.

The thin film oven tests and other laboratory tests of the asphalts do not vary greatly and it would not be expected that there would be any marked difference in the behavior of the asphalts when mixed in the field. The effect of asphalt type on hardening was somewhat difficult to evaluate because it was impossible to separate this variable completely from the effect of plants and aggregates. However, there appeared to be no great difference in hardening that could be attributed to type of asphalt. Had asphalts

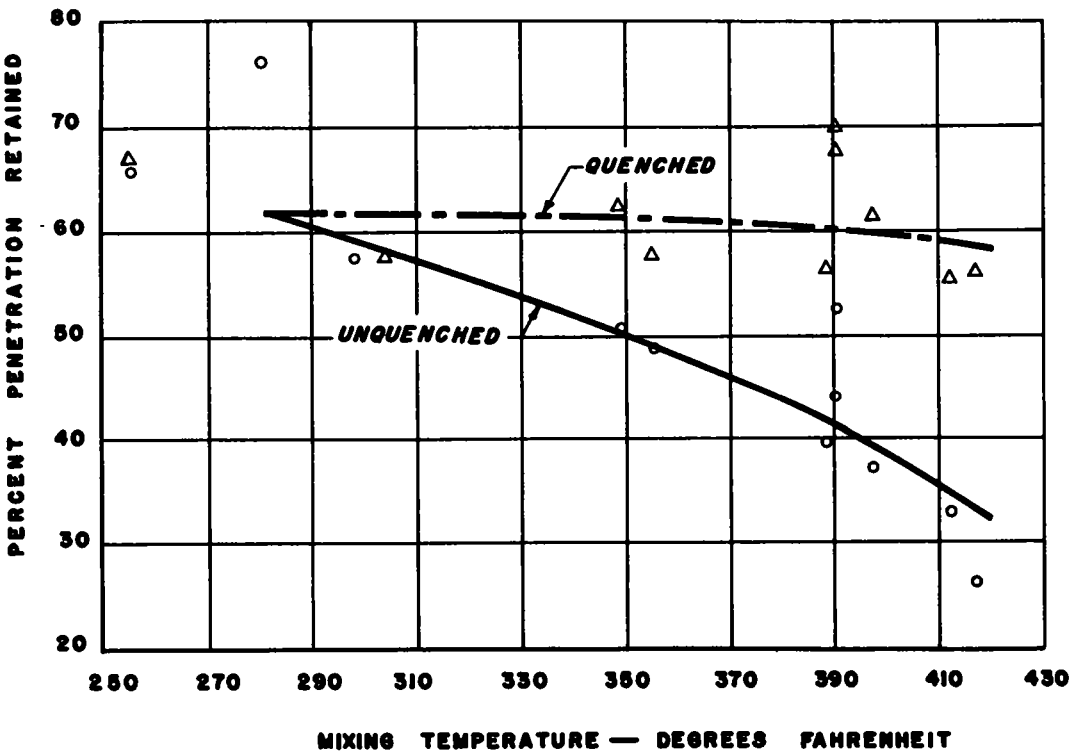


Figure 8. Comparison of percent penetration retained for all quenched and unquenched samples.

less resistant to hardening been used, it is quite likely that greater hardening would have occurred in the plant.

Rate of Cooling.—In measuring hardening in the mixer, a problem arose as to the proper procedure to use in cooling samples. Some investigators (19) have found that samples cooled quickly showed less penetration loss than those cooled slowly. This behavior was borne out by the present investigation. A study of Figures 8 and 9 shows this effect quite clearly.

The quenched samples show little change in hardening when the temperature was increased from about 300 to 400 F. Because large changes took place in the slowly cooled samples, indications are that the hardening is both a time- and temperature-controlled phenomenon. The mixes made at about 300 F show less difference between quenched and unquenched samples than do the mixes made at higher temperatures. This would mean that the hardening occurring after mixing is not serious with mixing temperatures normally used. Moreover, under present day practice, it is difficult to see what could be done about the situation to prevent such hardening. The finding is interesting from the theoretical aspect in that it shows appreciable hardening occurring long after the violent agitation of the mixture has ceased.

Some attempts were made to follow trucks enroute from the plant to the paver and take samples at intervals during the hauling period. Such results indicate some hardening enroute but results were spotty and it was difficult to draw conclusions.

None of the off-specification batches were laid or compacted, so taking road samples was impossible. Others (1, 11) found no difference between samples taken at the plant and those taken from the newly laid pavement. On this project, slowly cooled samples taken at the plant gave results similar to samples taken at the paving machine, so it is assumed that the slowly cooled samples approached the values that would have been obtained if paving samples had been available.

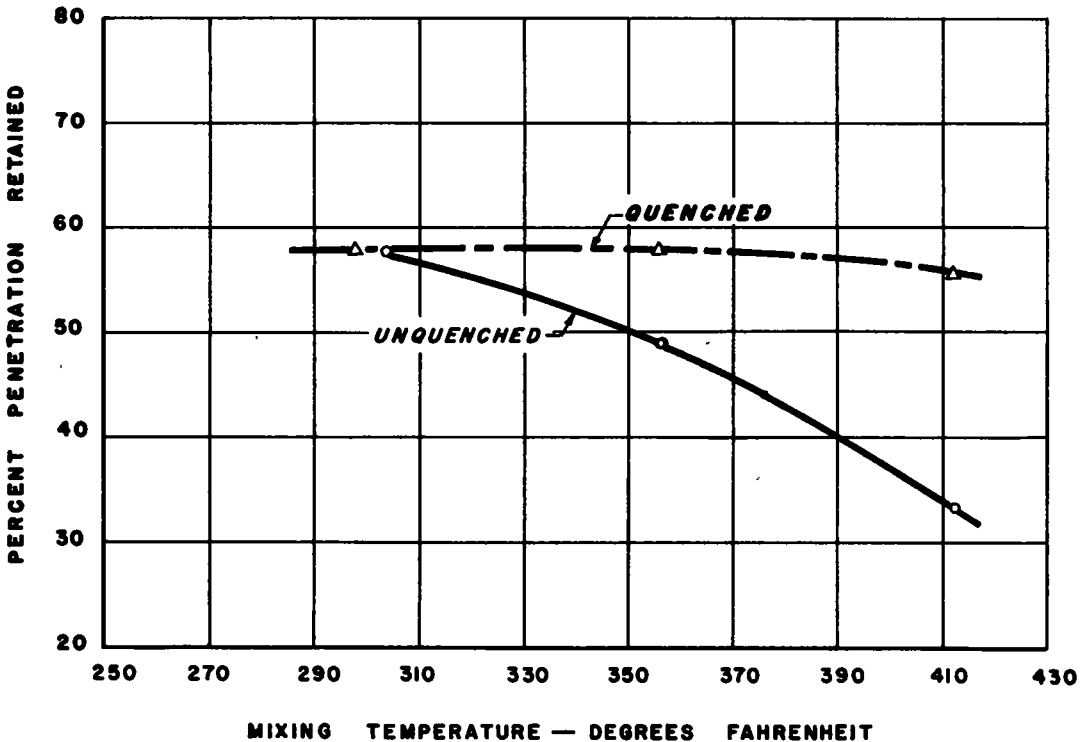


Figure 9. Comparison of percent penetration retained for quenched and unquenched samples within Group XVII.

The literature seldom describes the method of cooling samples when these samples are taken of the loose paving mixture at the plant. Because it was felt that the penetration after laydown was of practical significance in evaluating the effect of the mixing operation on hardening, the major portion of the samples were cooled slowly.

Storage.—In connection with taking samples of paving mixture, two other questions arose: (a) how long after mixing does this hardening occur, and (b) what are the effects of various methods of storage? In an effort to find answers to these questions, storage tests were performed on samples of an I-2 WS that had been mixed at 315 F. Table 6 shows that these tests indicated hardening of the mixture continued long after the mixing process with the hardening occurring most rapidly during the first 15 days after mixing. This effect may have had some detrimental effects on some of the test results of this study because some of the samples were allowed to set for a few days before the asphalt was extracted from the mixture.

The decrease in penetration after 30 days was the same for the sample that was stored unbroken in the cardboard box, the sample that was broken up and spread out, and the sample that was sealed in the container in the presence of air. The sample stored in the sealed container in the presence of carbon dioxide showed a slightly higher penetration than the other samples at the end of the 30-day period. This would support the belief that oxidation and volatilization have an effect on the hardening of the mixture.

As shown in Tables 6 and 7, it was found that a slight hardening occurred in the original and recovered asphalts over the same period as that of the mixture; but this was so small that the difference may have been due to errors in testing rather than a difference in the hardness of the asphalt. However, when penetration tests were rerun on several of the asphalt samples that had been tested during the early months of the project, it was found that the penetration had dropped 4 to 6 points after being stored for about 200 days, thus indicating additional hardening over a longer period of time. These samples were normally stored in 3-oz tins and were reheated in each case before being tested.

To summarize briefly, this investigation shows that a very serious increase in hardening results as the mixing temperature is raised and that the differences in the asphalts, aggregates, and mixes used in North Carolina are not major factors in the hardening process. This investigation also shows that the manner in which samples are cooled is very important in determining this hardening effect and, due to serious hardening of the loose paving mixture during the first 15 days after sampling, the samples should be extracted as soon as possible after they are taken.

CONCLUSIONS

From the results of this test, the following conclusions were formed concerning the effect of mixing temperature and other related factors on the hardening of asphaltic binder in hot bituminous concrete mixes normally used in North Carolina.

1. The tests performed on recovered asphalts indicated a relatively constant hardening with increased mixing temperature of 2 percent loss in retained penetration for each 10 F rise in temperature to about 350 F at which point this hardening becomes accelerated.
2. Although it would be advantageous from the standpoint of hardening to decrease mixing temperatures from 285 to 250 F, factors such as aggregate drying and coating would likely dictate that this change not be made.
3. The type of aggregate, asphalt, mix, and pugmill showed little effect on hardening.
4. Samples of paving mixture that were cooled slowly in air showed approximately the same penetration loss at 300 F and approximately 20 percent greater penetration loss at 400 F than those quenched in water.
5. Samples of loose paving mixture showed an additional loss in penetration of approximately 13 percent during the first 15 days after mixing. Hardening of the samples continued after this time but at a much slower rate.
6. The hardening characteristics, as measured by the thin film oven test, were similar for all asphalts used in the project and any findings are limited to asphalts of these particular characteristics.

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Appendix

MIXING OPERATION AND ASPHALT HARDENING

The subject of hardening of the asphaltic binder in the mixing operation of hot bituminous concrete manufacture and the slower hardening that occurs as the road is used, requires some explanation and discussion. Likewise, the factors that influence choice of mixing temperatures need clarifying inasmuch as these factors are vital to the choice of a proper mixing temperature.

The importance of the hardening that occurs is illustrated best by the fact that even though the subject has been studied for many years, the 1958 meeting of the Association

of Asphalt Paving Technologists devoted an entire symposium to the subject, and at the 1959 Annual Meeting of the Highway Research Board the subject was discussed in several of the papers.

Hardening as it occurs in the mixing operation or in the road is normally measured by recovering the asphalt from the pavement by the Abson method and comparing the penetration, softening point, ductility, and viscosity with these same properties as they existed in the original asphalt.

The hardening that occurs takes place in two steps. First, there may be a decrease in penetration during the mixing operation of as much as 50 percent of the original penetration. Mixing temperature and the kind of asphalt usually have the greatest influence on this penetration loss. Second, there is a rather slow decrease in penetration with time. It has been shown (1) that this hardening follows a hyperbolic curve so that the hardening becomes less and less with time. However, it must be remembered that as the road becomes older the mechanism of hardening may undergo change and cease to follow the curve indicated by the results of the early years.

In general, it appears that, when the penetration of asphalt recovered from pavement is above 30, little cracking occurs. Between 20 and 30, cracking may occur, and below 20, it usually does occur. These limits are approximate and are by no means rigid. The loss that occurs in ductility is considered by some (2,16) to indicate the asphalt has lost much of its value as a binder. However, asphalt technologists do not appear to agree as to just how serious this loss of ductility may prove to be in the performance of the road.

The hardening phenomenon is not thoroughly understood. When asphalt is heated in bulk, even for rather long periods of time, no large amount of hardening occurs. When the same asphalt is mixed with hot aggregate, serious hardening results even in as short a period as 30 sec to 1 min. This hardening is usually attributed to the fact that the asphalt spreads out over the hot aggregate surface in films only a few microns thick and offers excellent opportunities for oxidation and volatilization.

It has been shown definitely that there are oxidation effects. Thurston et al. (17), Ebberts (4), and others have studied such oxidation effects, and Van Oort (18) studied the durability of asphalts and showed that oxygen diffused into the asphalt films a distance of only a few microns.

However, when asphalt films are heated in atmospheres of N_2 or CO_2 gages serious hardening often results so that changes other than oxidation must take place. This hardening is attributed mainly to volatilization and some investigators (2,3) believe volatilization to be the most important hardening effect in the mixing operation.

Stevens (5) heated asphalt in sealed tubes in the absence of oxygen and with aggregate that had oxygen removed from the surface; he found appreciable hardening which varied with aggregate. This would indicate some hardening effect from the aggregate surfaces possibly some catalytic effect. Though other reasons can possibly account for this hardening, it would seem that the effect of aggregate surface should receive further study.

There is a strong possibility that the hardening mechanism at 275 to 375 F and that at 90 to 130 F may be different. It is not likely that the amount of hardening resulting from mixing is related to the additional hardening that occurs in the road.

Kinnaird (9) found that there was a definite relationship between penetration and softening point and established a characterizing factor for each asphalt:

$$P_{77} = aQ \frac{(SP-77)^{-4.25}}{(100)^{3\sqrt{aQ}}}$$

in which

- P_{77} = penetration at 77 F, 100 g, 5 sec;
- S. P. = ring and ball softening point, F; and
- aQ = characterizing factor.

Kinnaird found that asphalts that hardened by oxidation retained a constant characterizing factor, but when factors other than oxidation entered into the hardening the characterizing factor changed.

Numerous laboratory methods have been developed for predicting the behavior of the asphalt during the mixing operation and in the road. An excellent resume of these is given by Heithaus and Johnson (6) and is not repeated here. No attempt is made here to evaluate the value of these tests. The thin film oven test developed by the Bureau of Public Roads, however, is simple to perform, requires simple equipment, and yields results that can be correlated with most of the other tests used to predict the asphalt behavior.

Efforts have been made to separate the asphalts into various fractions by solvents and by adsorption methods. These fractions can be studied as to their behavior with oxygen and as to their quantities in the asphalt recovered from paving as compared with the original asphalt. Such separations offer promise of a better understanding of the hardening reaction, but they have not reached the point where they are generally accepted as forming the basis for asphalt specifications.

In attempting to establish a proper mixing temperature a number of factors must be considered. The hardening occurring in the mixer is a serious problem but the mix temperatures cannot be established solely on this basis.

First, the aggregate must be dried. It is thought that wet aggregates cause trouble. To dry aggregate with present equipment, somewhat high temperatures are obtained. Because much of the heat capacity of the batch lies in the aggregate, the temperature of this material largely determines mix temperatures. This would mean that if low mix temperatures were to be obtained along with dry aggregate either the aggregate would need to be heated at lower temperatures for much longer time intervals than now used or the aggregate would have to be cooled after heating to the higher temperatures required for drying. Neither plan would be feasible with present equipment.

The mixing temperature affects the coating of the aggregate by the liquid asphalt. Although visual inspection is the principal means of evaluating proper mixing, and this method may not be entirely satisfactory, still there appears little argument that when uncoated stone appears in the finished mix the resulting pavement will probably prove faulty. As temperatures are decreased, the asphalt viscosity increases and the stone is not coated in the normal mixing time. Power costs are probably also increased as a result of the increased viscosity of the asphalt.

Mix temperatures must be sufficiently high so that the material will perform satisfactorily in the paving machine and be compacted to the proper densities. Although proper laydown and compaction temperatures and the variables affecting them have not been too well established, they are tied very closely to mixing temperatures.

Finally, the mix temperature may be closely related to the final road performance of the bituminous concrete. Such information can only be obtained by roads laid with material mixed at various temperatures, say, from 225 to 400 F. Such tests are expensive as well as time consuming and, therefore, only a small amount of information is available on tests made over this wide range of mixing temperatures.

It can thus be seen that proper mixing temperature involves a balancing of several factors. Only when complete knowledge of all such factors is available can a correct answer be supplied.