

2236

NATIONAL BUREAU OF STANDARDS REPORT

2236

FIRST DRAFT

ACCELERATED DURABILITY TESTS OF ASPHALT ROOF COATINGS

I THE PRINCIPAL SERIES

by

Sidney H. Greenfeld



U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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NBS REPORT

1004-10-1017

January 15, 1953

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Sponsored by

Asphalt Roofing Industry Bureau



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ACCELERATED DURABILITY TESTS OF
ASPHALT ROOF COATINGS
I THE PRINCIPAL SERIES

ABSTRACT

The effect of six finely divided mineral materials in three concentrations on the durability of three asphalts was determined in accelerated durability machines. Numerous physical tests were also made on the individual asphalts and minerals and on their various blends. Although each system had to be evaluated individually, in general, the following trend was noted:

- (1) Blue Black Slate and Mica improved the durability of all three asphalts at all concentrations and film thicknesses tested.
 - (2) Schundlers Dolomite and Low Carbon Fly Ash increased the durability for many combinations, but for some they did not change the durability appreciably.
 - (3) Erie Silica and Florida Clay generally did not modify the durability, but in a few coatings increased it slightly and in others decreased it somewhat.
-

1. INTRODUCTION

In February 1938, Research Paper No. 1073, by Dr. O. G. Strieter, was published as the final report on the early work on the effects of finely divided mineral matter on the durability of asphalt roof coatings. During the following few years great strides were made in the petroleum industry, uncovering new fields and developing new refining processes; new asphalts became available for roofing manufacture, and many of the older sources disappeared. New types of mineral

matter came under consideration for blending with the asphalts to improve their durability. Improved methods of manufacturing roofing made possible higher concentrations of mineral matter in roof coatings. In order to take advantage of these new materials and improved techniques, and to prevent the possible introduction of inferior materials, the Research Committee on the Asphalt Roofing Industry Bureau outlined a program to study many of the factors involved in the durability of roof coatings and to find how these factors determine the suitability of the current raw materials for use in roofing.

This program was specifically designed to answer a number of questions which always arise when a new material is considered for use in prepared roofing. These problems may be summarized briefly as follows:

- (1) What is the effect of the concentration of the mineral matter on the durability of the coating?
- (2) What is the effect of the thickness of the coating on its durability?
- (3) What is the effect of the mineral matter on the impact resistance, or brittleness, of the coating?
- (4) What is the effect of the mineral matter on the adhesion of the coating to the saturated felt base and the granules?

While the data being obtained on the project will eventually answer all of these questions, this report covers only the early results of the accelerated tests of the durability of the coatings and the physical tests made on the coatings and materials that went into them. The answers to these questions for felt-based specimens and aluminum-based specimens exposed out-of-doors will be reported later.

Three concentrations of six types of stabilizer were evaluated in three different asphalts at three coating thicknesses in accelerated durability machines. A number of tests were also made on both the individual asphalts and stabilizers and on the blended coatings in order to correlate the accelerated durability data with the remainder of the program and with more fundamental concepts.

2. MATERIALS

2.1 Asphalts

The three coating asphalts subjected to test were typical of those used in making prepared roofing in the United States. Each asphalt was commercially processed from the crude oil into six products: a flux and five different softening point materials, as shown in Table I.

Portions of Product 1 of each asphalt were ignited in order to determine the inorganic matter present. Table II lists the dominant lines in the ash spectra.

TABLE I. PROPERTIES OF ASPHALTS

ASPHALT: CODE DESIGNATION:	CALIFORNIAN C			MID-CONTINENT M			VENEZUELAN V		
Date	9/49	1/51 ^a	4/52	9/49	1/51 ^a	4/52	9/49	1/51 ^a	4/52
<u>PRODUCT 1</u>									
S.P., °F ^b	187	189	192	185	190	196	185	190	195
Pen. ^c	31	28	24	29	27	25	25	22	19
Sp. Gr. ^d	1.013			0.995			1.015		
<u>PRODUCT 2</u>									
S.P., °F ^b	197	201	205	196	204	212	189	192	195
Pen. ^c	25	23	21	25	23	20	25	22	19
<u>PRODUCT 3</u>									
S.P., °F ^b	211	213	215	210	217	225	207	207	208
Pen. ^c	22	21	19	22	20	17	21	20	19
<u>PRODUCT 4</u>									
S.P., °F ^b	213	218	224	221	227	234	218	226	235
Pen. ^c	20	19	17	19	18	17	18	17	15
<u>PRODUCT 5</u>									
S.P., °F ^b	223	230	237	231	239 ^e	239	224	232	239
Pen. ^c	19	14	15	17	15	15	17	16	15
Sp. Gr. ^d	1.017			1.003			1.021		
<u>FLUX</u>	Viscosity at 210°F, 84 Furol Seconds, Flash Point (COC) 445°F			Gravity, °API 12.3, Flash Point (COC) 580°F, Viscosity at 210°F 286 Furol Seconds			Steam refined to 105°F Softening Point & 160 Pen.		

^aBecause of the progressive increase in the hardness of all of the products, the softening points and penetrations changed progressively. The time-weighted averages of the determinations of these properties measured in 9/49 and 4/52 were used in estimating them when the products were used.

^bSoftening Point, Ring and Ball - ASTM Method D36-26.

^cPenetration at 77°F, 100 g, 5 sec. - ASTM Method D5-49.

^dThe specific gravity at 77°F was determined only on Products 1 and 5.

^eThis product was not used because of its high softening point.

TABLE II. SPECTRAL ANALYSES OF ASPHALT ASH

Asphalt	% Ash	Elements									
		Al	Ca	Cu	Fe	Mg	Na	Ni	Pb	Ti	V
California	0.2%	S	S	W	VS	S	S	S	W	M	S
Mid-Continent	0.1%	S	S	W	VS	S	S	S	W	M	S
Venezuelan	0.1%	S	S	W	VS	S	M	S	W	M	S

VS = Very Strong M = Moderate
S = Strong W = Weak

2.2 Stabilizers

Six mineral stabilizers were selected from the large number of materials currently being used in the roofing industry. These particular ones were chosen because they were believed to cover the complete spectrum of performance, from very poor to excellent, and were readily available and reproducible in form. The properties and attributes of these six stabilizers are tabulated in Table III. Figure 1 contains photomicrographs of these materials; they are included to show the particle shape only and because each is at a different magnification must not be used to compare the particle sizes of the various materials.

2.3 Aluminum Panels

Aluminum panels $2\frac{3}{4} \times 6 \times 0.064$ in. were used to support the coatings during exposure. These panel bases were all made from Kaiser aluminum alloy 2S - 1/2 H.

3. EQUIPMENT

3.1 Panel Making

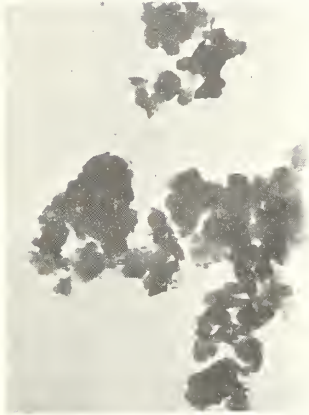
The exposed panels were made with the aid of a hydraulic press, as described elsewhere (1).

3.2 Panel Exposure

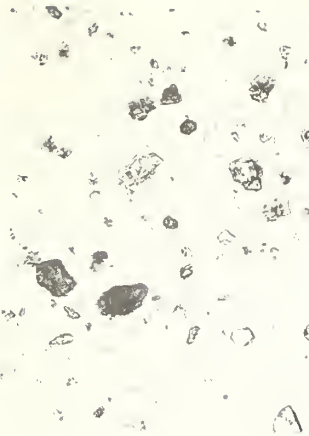
The panels were exposed in six accelerated durability machines constructed at the N.B.S. and described in (2).



BLUE BLACK SLATE
X 11



FLORIDA CLAY
X 13,500



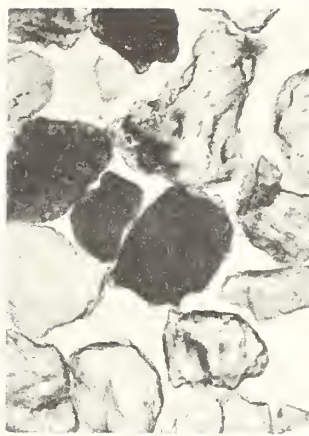
SCHUNDLERS DOLOMITE
X 90



LOW CARBON FLY ASH
X 80



MICA
X 80



ERIE SILICA
X 80

PHOTOMICROGRAPHS OF STABILIZERS

FIG. I

TABLE III. PROPERTIES AND ATTRIBUTES OF STABILIZERS

Stabilizer:	#50		Schundlers
	Blue Black	Florida Clay ^a	Dolomite
	Slate		
Code Designation	A	B	C
Source	Quarry Delta, Pa.	Mine Edgar, Fla.	Quarry Joliet, Ill.
Particle Shape	Plates	Blocky, Rounded Edges	Blocky, Sharp, Irregular
Particle Size, % Passing			
Mils Microns U.S. No.			
9.8 250 60	99.8	100.0	99.9
7.0 177 80	99.3	99.9	99.9
5.9 149 100	97.9	99.8	99.6
4.9 125 120	96.2	99.8	99.3
3.5 88 170	91.3	99.6	96.6
2.9 74 200	86.9	99.3	93.4
2.4 62 230	83.6	99.1	89.9
1.7 44 325	76.7	98.9	81.0
1.5 40 Sed. ^b	68	97	73
0.8 20 "	54	12	39
0.4 10 "	8	2	26
0.02 4 "	2	1	13
0.01 2 "	1	-	7
Specific Gravity	2.9 ⁴	2.6 ⁴	2.8 ⁷
Surface Area, m ² /g	1.0	27.2	2.0
Oil Absorption, lb/100 lb	29.5	63.9	19.4
Water Absorption, lb/100 lb	32.7	36.4	18.5
Loss on Ignition at 1000°F	2.1	11.8	1.8
Loss on Ignition at 1800°F	5.4	13.3	43.7
Moisture, %	0.2	2.7	0.1
Solubility, %	0.00	0.0 ⁴	0.00
Free Alkali, %	0.0	0.0	0.0 ^c
Chemical Analysis, %			
SiO ₂	56	47	6
R ₂ O ₃	32	38	1
CaO + MgO			49
K ₂ O + Na ₂ O	4		
SO ₃			
Carbon	2		
Mixture With Asphalt			
Ease of Mixing	G	P	F
Softening Point Increase, °F	15	28	13

TABLE III. PROPERTIES AND ATTRIBUTES OF STABILIZERS (CONTINUED)

Stabilizer:	Low Carbon		Erie Silica		
Code Designation	Fly Ash	#160 Mica			
Source	D	E	F		
Particle Shape	Philadelphia Electric	Pit Mine Johnson City	Dredged Lake Erie		
Particle Size, % Passing	Spherical, Irregular, Sharp	Plates	Blocky, Round Corners		
Mils	Microns	U.S. No.			
9.8	250	60	99.6	100.0	99.9
7.0	177	80	98.9	96.0	98.2
5.9	149	100	97.5	91.0	80.8
4.9	125	120	96.7	85.6	53.7
3.5	88	170	94.2	65.9	15.3
2.9	74	200	92.3	56.8	8.1
2.4	62	230	90.6	50.3	5.4
1.7	44	325	84.8	37.8	3.4
1.5	40	Sed. ^b	73	65	9
0.8	20	"	60	22	6
0.4	10	"	43	4	4
0.02	4	"	15	2	1
0.01	2	"	2	-	-
Specific Gravity	2.62		3.01		2.68
Surface Area, m ² /g	2.0		2.7		2.5
Oil Absorption, lb/100 lb	30.0		97.2		19.5
Water Absorption, lb/100 lb	33.8		61.5		20.2
Loss on Ignition at 1000°F	4.9		0.9		0.7
Loss on Ignition at 1800°F	7.3		4.4		2.5
Moisture, %	0.4		0.2		0.2
Solubility, %	5.90		0.46		0.00
Free Alkali, %	0.0 ^c		0.0		0.0
Chemical Analysis, %					
SiO ₂	40		50		
R ₂ O ₃	48		35		
CaO + MgO	2.5		1		
K ₂ O + Na ₂ O			10		
SO ₃			0.5		
Carbon	7.6				
Mixture With Asphalt					
Ease of Mixing	G		G		G
Softening Point Increase, °F	20		40		5

^aPlastic Index = 34. Plastic Limit = 34.

^bSedimentation in Isopropyl alcohol.

^cTurns phenolphthalein pink in aqueous solution.

3.3 Panel Inspecting

The panels were inspected weekly, both visually and with a high-voltage probe (2). Representative types of failures were photographed with a Speed Graphic View Camera containing a 6-in. F6.8 Goerz Dagor lens.

4. PROCEDURE

All coatings were blended from adjacent asphalt products (Table I) and the desired percentage of stabilizer to have softening points in the range of 217 to 227°F. In those mixtures in which the softening point was in excess of 227°F, only Product 1 was used; the softening point rise produced by the stabilizer was too large to keep the softening point within the range.

After the proportions of the two asphalt products were determined, the asphalt was melted, the stabilizer added, and the mixture stirred continually at about 420-430°F until the surface became free from foam and bubbles. The temperature was increased to 450°F and the viscosity measured with a Brookfield viscometer. The temperature was permitted to drop slowly to 190°F, viscosity measurements being made at approximately every 10°F. The mix was reheated to its working range for the preparation of exposure panels (1).

Just prior to making the first panel, two specimens for the softening point determination (2) and one shatter specimen

were poured. Halfway through the panel-making procedure, another shatter specimen and a water-absorption^{1/} specimen were poured. A third shatter and two additional softening point specimens were poured at the completion of the panel-making.

Although thirteen aluminum-based panels and five felt-based panels were made from each of the 48 coatings^{2/}, only six aluminum-based panels were exposed in the accelerated durability machines (2). (One felt- and one aluminum-based panel were kept for reference, and the remainder were exposed out-of-doors.) These represented the coating thicknesses, 0.013, 0.025, and 0.043 in. Each of the duplicate panels of each thickness was exposed in a different machine in order to average the differences among the accelerated durability machines. For, even though the six machines were calibrated with an integrating light meter and set to give approximately the same exposure conditions before the exposures were started, the normal wear in the parts of the machines continually changed their operating characteristics. Exposure of duplicate panels in two machines served to average the constantly changing differences. As described in (2), the

^{1/} For a description of the water absorption and shatter tests, refer to Appendix A.

^{2/} Felt-based granule-surfaced panels were also made from 27 of these coatings and exposed out-of-doors.

panels were inverted in their supports every other day and the supports were inverted on the intervening days.

The accelerated durability machines were operated 22 hours a day, seven days a week, during the entire course of exposures. The exposure cycle consisted of 51 minutes of radiation followed by nine minutes of radiation and cold ($40 \pm 2^\circ\text{F}$) demineralized water spray, delivered at 25 ± 5 psig. Although the temperature program varied somewhat, depending on the number of panels in the machines and the ambient temperature, it ranged from a low of about 50°F at the end of a spray period to 140 - 145°F about 20-25 minutes after a spray period and maintained until the beginning of the next spray period. The exact conditions of each panel were different because of the wide range in light absorption of the coating surfaces, their shades varying from a matte gray-white (clay stabilizer showing) to a glossy black, but the conditions were maintained for the straight asphalt from which all of the coatings in that machine were made, for the temperature control was coated with that asphalt and revolved with the coatings.

The exposure panels were examined weekly, both visually and with a high-voltage probe (3), and spark pictures were taken whenever there was any break in the coating. The pictures were examined through a transparent grid of 60

squares covering the central 2-x 5-in. portion of the coating (1/8 in. around the edge of the coating was not counted). When exposed areas appeared on the photograph in a minimum of 30 of these squares, the panel was removed from the machine and filed away for future reference. Each panel was classified into one of eight types of panel failure, as listed in Table IV, according to the crack pattern which resulted in its failure.

This classification according to crack patterns was designed to supplement the spark pictures in identifying failure types. Its purpose is two-fold:

- (1) Spark pictures cannot be used if the coating contains material that conducts electricity (fly ash).
- (2) There is little correlation between visual and electric-probe failures in many cases because of the large number of breaks in the coating that are not visible to the unaided eye, and some correlation must be maintained among all coatings. For example, a coating containing fly ash may have failed with a type A pattern, but it cannot be removed from the machine until a visual failure occurs, because the electric probe is nullified by the conductivity of the fly ash. Hence the durability of coating containing fly ash may be exaggerated.

TABLE IV. FAILURE CLASSIFICATION

Failure Type	Description
A	Little or no visible cracking.
B	Fine map cracks - less than $3/16$ " between intersection.
C	Fine map cracks - $3/16$ "- $3/8$ " between intersection.
D	Coarse map cracks - $3/16$ "- $3/8$ " between intersection.
E	Map cracks with large uncracked areas.
F	Straight line cracks.
G	Ordered cracks (cracks seem to follow a definite pattern).
H	Large principal crack with smaller tributaries.

Panels that fail in patterns toward the lower end of the classification, E, F, G, H, generally have a large span of time between the first appearance of a break in the coating and their removal from the machines. Their durability is very closely related to the arbitrary endpoint, which has been set as failures in 50% of the grid areas for this paper. However, those panels failing toward the upper end of the classification, A, B, or C, tend to fail rather suddenly, in many cases spontaneously, and their durability would be almost the same whether the end point was taken at 10% or 90% of the grid areas. Classification D is the intermediate between these two extremes.

5. RESULTS

The data obtained in this investigation are of two types: (1) Physical tests on the coating asphalts, and (2) Accelerated durability tests.

5.1 Tests on Coatings

Each of the 48 coatings subjected to accelerated durability tests was examined rather critically in an effort to find some property or combination of properties which would aid in predicting the ultimate life of roofing made from that coating, with a minimum expenditure of time. In addition to the measurements made on the individual asphalts (Tables I

and II) and stabilizers (Table III), a number of other tests were made on each coating that was exposed. Table V summarizes the data on the three asphalts exposed without stabilizer.

The tests made on the stabilized coatings are reported in Tables VI to VIII, according to stabilizer:

- Table VI)-(Blue Black Slate
)-(Florida Clay
- Table VII)-(Schundlers Dolomite
)-(Low Carbon Fly Ash
- Table VIII)-(Mica
)-(Erie Silica

Several calculated quantities are reported in Tables VI to VIII along with the measured data. The volume composition was calculated from the weight composition and the specific gravity of the components as follows:

W = weight	<u>Subscripts</u>
V = volume	A = asphalt
d = specific gravity	S = stabilizer
	C = coating

$$\frac{100 W_S}{W_A + W_S} = \% S \text{ by weight}$$

$$\frac{W_S}{d_S} = V_S \qquad \frac{W_A}{d_A} = V_A$$

$$\frac{100 V_S}{V_A + V_S} = \% S \text{ by volume}$$

TABLE V. PROPERTIES OF UNSTABILIZED ASPHALT COATINGS

ASPHALT:	VENEZUELAN	MID-CONTINENT	CALIFORNIA
<hr/>			
<u>TEST</u>			
Softening Point ¹	227	224	223
Penetration at 32°F ²	11.0	11.2	10.4
" at 77°F	13.5	17.0	17.0
" at 115°F	20.8	26.0	30.1
Penetration Index ²	4.5	4.7	4.7
Susceptibility ²	0.73	0.87	1.16
Loss on Heating ³	0.10	0.03	0.22
Penetration after Heating ²	13.9	17.1	16.6
Specific Gravity at 77°F	1.018	0.999	1.015
Viscosity cps at 400°F ⁴	375	420	280
" " at 450°F	130	140	100
" " at 500°F	28	53	25
Water Absorption, g/ft ²			
at 28 days ⁵	0.34	0.43	0.67
at 56 days ⁵	0.52	0.70	1.00
at 280 days ⁵	1.61	2.20	3.00
at 609 days ⁵	2.29	2.97	4.62
Δ Vol., cc/ft ² at 609 days	0.70	1.63	4.11
Shatter ⁶ , inches (1)	2.5	8	5
" " (2)	3.0	9	7
" " (3)	---	8	7
" " Ave.	2.7	8.3	6.3

¹ASTM Method D36-26.

²ASTM Method D5-25.

³ASTM Method D6-39T.

⁴Brookfield Viscometer.

⁵3- x 3/16-inch specimens submerged 1/4-inch in distilled water at 70°F. See Appendix for absorption curves and method.

⁶1/2-lb. weight on 3- x 3/16-inch specimen in mixed ice and water bath. See Appendix.

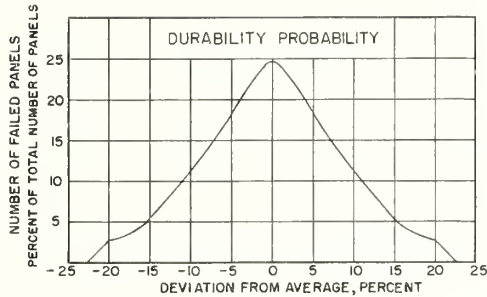


FIG. 8

TABLE VI

ASPHALT	PHYSICAL TESTS OF COATINGS											
	VENEZUELAN		MID-CONTINENT		CALIFORNIAN							
Per Cent Stabilizer	0	3.5	5.0	6.0	0	3.5	5.0	6.0				
A Blue Black Slate												
Volume Per Cent *	0	15.7	25.7	34.2	0	15.5	25.4	32.8	0	15.7	25.7	34.1
Density of mixture *	1.018	1.32	1.37	1.48	0.999	1.30	1.49	1.66	1.015	1.32	1.51	1.68
Softening Point, °F	227	218	221	256	224	222	217	237	223	222	226	240
S.P. of Base, °F **	227	205	191	190	224	217	202	190	223	210	202	199
Pen. at Base at 77°F	14	20	22	22	17	22	22	27	17	21	23	22
Temp. of Preparation	401-437	410-437	448-473	464-490	410-464	437-446	446-464	462-472	365-418	410-428	410-428	446-482
Viscosity, CPS @ 400°F	375	580	1450	14,500	420	640	1,480	13,500	280	540	1,210	11,000
@ 450°F	130	224	535	6,200	140	280	600	4,150	100	180	580	3,900
@ 500°F	28	87	244	2,350	53	130	275	1,850	25	58	260	2,100
Water Absorption												
@ 28 Days, g/ig. ft.	0.34	0.53	0.66	0.70	0.43	0.70	0.91	0.99	0.67	0.77	0.92	1.02
@ 56 Days	0.12	0.29	1.08	1.19	0.70	2.10	1.36	1.59	1.90	1.78	1.97	1.55
@ 180 Days	1.21	3.14	4.45	5.55	2.20	3.62	4.68	6.51	3.00	4.20	6.51	7.11
@ 609 Days (no mes)	2.29	4.06	6.93	10.29	2.97	5.61	7.65	12.35	4.62	6.49	12.66	13.22
Volume Increase %/ft	0.70	5.34	7.68	11.69	1.63	6.28	6.10	14.34	4.11	7.02	12.37	13.22
Shatter inches												
#1 (1" Dia x 1/4" ht)	2.5	8	14.5	22.1	8	12	18.5	22.1	5	12.0	—	22.1
#2	3.0	10.5	18	22.1	8	12	18	22.1	7	12.0	18	22.1
#3	—	9	15	22.1	8	12	18	22.1	7	12.0	18.5	22.1
Average	2.7	9.2	15.5	22.1	8.3	12.1	18.1	22.1	6.3	12.7	18.3	22.0
Air Content (by grav)												
Florida Clay												
Volume Per Cent *	0	17.3	27.8	36.7	0	16.9	27.5	36.3	0	17.3	27.8	36.6
Density of mixture *	1.018	1.30	1.47	1.61	0.999	1.28	1.45	1.59	1.015	1.30	1.47	1.61
Softening Point, °F	227	221	227	—	224	221	220	—	223	219	226	—
S.P. of Base, °F **	227	205	190	—	224	206	191	—	223	199	199	—
Pen. at Base at 77°F	14	20	22	—	17	22	27	—	17	24	24	—
Temp. of Preparation	401-437	437-464	446-482	—	410-464	437-455	446-482	—	365-418	410-428	437	—
Viscosity, CPS @ 400°F	375	990	5000	—	420	820	3600	—	280	540	3000	—
@ 450°F	130	235	2100	—	140	340	1550	—	100	225	1400	—
@ 500°F	28	117	850	—	53	135	700	—	25	94	570	—
Water Absorption												
@ 28 Days, g/ig. ft.	0.34	1.88	1.91	—	0.43	8.43	2.18	—	0.67	1.22	1.96	2.04
@ 56 Days	0.53	2.35	2.80	—	0.70	8.02	3.17	—	1.00	1.99	3.43	3.32
@ 180 Days	1.11	4.60	6.40	—	2.20	4.70	7.60	—	3.00	5.90	7.80	10.00
@ 609 Days (no mes)	2.29	8.81	10.57	—	2.97	5.72	7.78	—	4.62	7.89	12.07	9.35
Volume Increase %/ft	0.70	7.60	7.32	—	1.63	5.27	10.12	—	4.11	5.83	10.46	9.86
Shatter inches												
#1 (1" Dia x 1/4" ht)	2.5	10	7	—	8	8	15.5	—	5	8	10	—
#2	3.0	8	6.5	—	8	8	18.5	—	7	11	9	12
#3	—	18	5.5	—	8	8	18.5	—	7	8	8	—
Average	2.7	9.3	6.3	—	8.3	8.3	15.7	—	5.7	8.7	9.0	12.0

* Calculated = Race Slow
 ** Estimated

The specific gravity of each stabilized asphalt coating was calculated from the composition of the coating and the specific gravity of the individual components.

$$d_C = \frac{W_A + W_S}{V_A + V_S} = \frac{W_A + W_S}{\frac{W_A}{d_A} + \frac{W_S}{d_S}}$$

In order to check these calculations, for they are again used in determining the volume changes taking place in the water-absorption specimens, a number of specific gravity measurements were actually made on some of the stabilized coatings. Table IX compares the calculated and observed specific gravity for 13 coatings.

The softening points and penetrations of the base asphalts were estimated from those of the five products from each source and the softening-point rise expected for each concentration of each stabilizer. Because this rise is slightly different for each of the products of an asphalt, being a function of the softening point of that asphalt, and because the asphalts hardened progressively in storage in the period during which these studies were made, the final softening point of a stabilized coating could not be pinpointed closer than within a 10°F range, and the estimated softening points and penetrations of the base asphalts would, consequently, be subject to the same order of variation, their being calculated from the measured softening point of the stabilized coating.

TABLE IX. SPECIFIC GRAVITIES

COMPOSITION		SPECIFIC GRAVITY	
		Calculated	Observed
California	- 50% BBS	1.51	1.50
"	- 35% Clay	1.30	1.30
"	- 50% Clay	1.47	1.48
"	- 60% Dolomite	1.66	1.66
"	- 35% Silica	1.30	1.29
"	- 50% Silica	1.47	1.47
Mid-Cont.	- 50% BBS	1.49	1.50
"	- 50% Fly Ash	1.44	1.39
"	- 35% Silica	1.28	1.24
"	- 50% Silica	1.45	1.43
Venezuelan	- 50% Clay	1.47	1.47
"	- 50% Dolomite	1.50	1.49
"	- 60% Silica	1.62	1.62

The viscosity values reported at 400 and 450°F are values interpolated from a semi-logarithmic viscosity vs. temperature curve of measurements made in the range of 390 to 480°F with a Brookfield viscometer. The 500°F value was extrapolated from the same curve.

The water absorption measurements were made on specimens three inches in diameter and approximately 3/16-in. thick. The exact thickness was subject to slight variation because the specimens were cast in an open-top mold and were not trimmed. The specimens were submerged in distilled water to a depth of 1/4 in. and were weighed weekly for three months, monthly for three months, and then quarterly until the test was discontinued at 20 months.^{3/} The specific gravity also was determined at the final weighing of each specimen.

From the physical dimensions of each specimen the surface area was calculated and the absorption data reported as grams of water absorbed per square foot of specimen surface at 28, 56, 280, and 609 days. The change in volume for the 609 days (20 months) of immersion was calculated from the final measured volume and the original volume, which was calculated from the original weight and specific gravity. Figure 2 is

^{3/}The absorption curves are in Appendix A.

a graph of the change in volume vs. the change in weight of the coatings at the end of 20 months submersion.

The shatter determinations were made on three specimens of the same size as those used for the water absorptions. One was poured when the first exposure panel was made; the second, between the making of the aluminum- and felt-based specimens, and the third, when all of the exposure panels were completed. In this manner, any changes in the coating that occurred during the panel-making process were taken into consideration.

The temperature ranges in which the exposure panels were made are reported under "Temperature of Preparations".

5.2 Accelerated Durability Results

(a) The results of the exposure of the aluminum-based panels in the accelerated durability machines are given in Tables X and XI, as follows:

<u>Table No.</u>		<u>Stabilizer in Coatings</u>
X	-	(Blue Black Slate (Florida Clay (Schundlers Dolomite (Low Carbon Fly Ash
XI	-	(Mica (Erie Silica

The averages of the durability of the duplicate exposures are reproduced on graphs in Figures 3 to 7.

**TABLE X
PRINCIPAL SERIES
ACCELERATED DURABILITY DATA**

Coating Thickness mil	0.012				0.025				0.042				0.075				0.142							
	0	35	50	60	0	35	50	60	0	35	50	60	0	35	50	60	0	35	50	60				
A Blue Black Stain																					C Schmalzer's Delomite			
Venezuelan, Days	67	104	181	111	67	178	187	203	124	195	399	276	67	104	181	111	67	178	187	203	124	195	399	276
Average Ratio	6.8	10.8	9.3	11.7	7.4	15.8	2.0	19.2	11.5	18.6	26.9	37.0	6.8	10.8	9.3	11.7	6.8	10.8	9.3	11.7	11.5	18.6	26.9	37.0
Crack Pattern	C	A	A	A	F	A	A	A	F	A	A	A	C	A	A	A	F	A	A	A	F	A	A	A
Mid-Continent, Days	74	90	66	180	91	195	169	207	96	186	302	231	74	90	66	180	91	195	169	207	96	186	302	231
Average Ratio	6.7	10.4	6.8	17.7	9.6	19.5	15.7	22.0	10.9	21.6	37.6	33.1	6.7	10.4	6.8	17.7	6.7	10.4	6.8	17.7	10.9	21.6	37.6	33.1
Crack Pattern	B	A	A	A	C	A	A	A	F	A	A	A	B	A	A	A	C	A	A	A	F	A	A	A
Californian, Days	33	54	40	66	52	61	81	115	72	118	117	171	33	54	40	66	52	61	81	115	72	118	117	171
Average Ratio	3.2	5.2	3.7	4.5	5.1	7.5	8.1	11.5	7.2	12.8	12.8	17.8	3.2	5.2	3.7	4.5	3.2	5.2	3.7	4.5	5.1	7.5	8.1	11.5
Crack Pattern	B	A	A	A	C	A	A	A	C	A	A	A	B	A	A	A	C	A	A	A	C	A	A	A
B Florida Clay																					D Lion Carbon Fly Ash			
Venezuelan, Days	67	42	35	—	67	76	81	—	124	144	170	—	67	104	181	111	67	178	187	203	124	195	399	276
Average Ratio	6.8	3.6	3.6	—	7.4	7.8	8.9	—	12.4	12.4	15.6	—	6.8	10.8	9.3	11.7	6.8	10.8	9.3	11.7	11.5	18.6	26.9	37.0
Crack Pattern	C	C	C	—	F	F	F	—	F	F	F	—	C	A	A	A	F	A	A	A	F	A	A	A
Mid-Continent, Days	74	55	47	—	91	80	79	—	96	96	117	—	74	90	66	180	91	195	169	207	96	186	302	231
Average Ratio	6.8	6.0	5.1	—	9.6	8.6	8.5	—	9.6	10.9	11.5	—	6.7	10.4	6.8	17.7	6.7	10.4	6.8	17.7	10.9	21.6	37.6	33.1
Crack Pattern	B	A	A	—	A	C	C	—	E	E	F	—	B	A	A	A	C	A	A	A	C	A	A	A
Californian, Days	33	20	25	—	52	32	38	—	72	52	57	—	33	54	40	66	52	61	81	115	72	118	117	171
Average Ratio	3.2	2.0	2.6	—	5.1	3.8	4.6	—	7.2	5.2	5.7	—	3.2	5.2	3.7	4.5	3.2	5.2	3.7	4.5	5.1	7.5	8.1	11.5
Crack Pattern	B	B	B	—	C	C	C	—	D	D	D	—	B	A	A	A	C	A	A	A	C	A	A	A

□ Failure determined by visual inspection * See "Duplication of Results" Table XI for Average using Calculating Ratios

**TABLE XI
PRINCIPAL SERIES
ACCELERATED DURABILITY DATA**

Coating Thickness mil	0.012				0.025				0.042				DUPLICATION OF RESULTS											
	0	35	50	60	0	35	50	60	0	35	50	60	Date	SPH	#1	#2	#3	#4	#5	#6	#7	#8	#9	#10
E Mica																								
Venezuelan, Days	67	276	—	—	67	—	—	—	124	—	—	—	11/28/50	227	66	—	69	—	68	—	—	—	—	—
Average Ratio	6.8	4.02	—	—	7.4	—	—	—	12.4	—	—	—	4/10/51	227	55	—	55	—	55	67	—	—	—	
Crack Pattern	C	A	—	—	F	—	—	—	F	—	—	—	4/10/51	227	66	—	81	—	75	65	—	—	—	
Mid-Continent, Days	74	278	—	—	91	—	—	—	96	—	—	—	11/15/50	227	64	—	—	—	—	—	—	—	—	
Average Ratio	6.8	3.69	—	—	9.6	—	—	—	10.3	—	—	—	3/18/52	227	145	118	84	90	116	107	—	—	—	
Crack Pattern	B	A	—	—	A	—	—	—	99	—	—	—	3/28/52	227	61	61	49	42	36	48	—	—	—	
Californian, Days	33	188	—	—	52	614	—	—	72	—	—	—	12/15/50	224	96	—	101	—	82	—	—	—	—	
Average Ratio	3.2	1.8	—	—	5.1	6.7	—	—	7.2	—	—	—	3/24/52	224	100	86	76	76	77	86	—	—	—	
Crack Pattern	B	A	—	—	C	A	—	—	D	—	—	—	3/13/52	224	96	—	109	—	99	—	—	—	—	
F Erie Silica																								
Venezuelan, Days	67	39	35	32	67	80	82	124	124	141	164	201	3/18/52	228	78	—	78	78	—	—	—	—	89	
Average Ratio	6.8	3.6	3.3	3.0	7.4	7.6	7.9	12.4	12.4	15.6	18.6	23.1	3/18/52	227	55	183	104	114	114	114	—	—	—	
Crack Pattern	C	A	A	A	C	A	A	A	F	A	A	A	3/18/52	227	66	125	104	128	120	120	—	—	—	
Mid-Continent, Days	74	52	57	37	91	91	99	96	120	141	164	121	1/11/51	219	101	—	50	—	51	—	—	—	—	
Average Ratio	6.8	6.2	6.1	4.1	9.6	11.7	12.8	10.9	12.4	15.6	18.6	23.1	3/18/52	227	66	125	104	128	120	—	—	—	—	
Crack Pattern	B	A	A	A	C	A	A	A	F	A	A	A	2/7/52	219	66	125	104	128	120	—	—	—	—	
Californian, Days	33	20	19	15	52	32	32	52	72	54	57	84	3/18/52	228	78	—	78	78	—	—	—	—	—	
Average Ratio	3.2	2.0	2.2	1.5	5.1	3.2	3.2	5.1	7.2	5.2	5.7	8.1	3/18/52	228	78	—	78	78	—	—	—	—	—	
Crack Pattern	B	B	B	A	C	C	B	B	D	D	C	B	3/18/52	227	66	125	104	128	120	—	—	—	—	

* See "Duplication of Results" Table XI for Average using Calculating Ratios

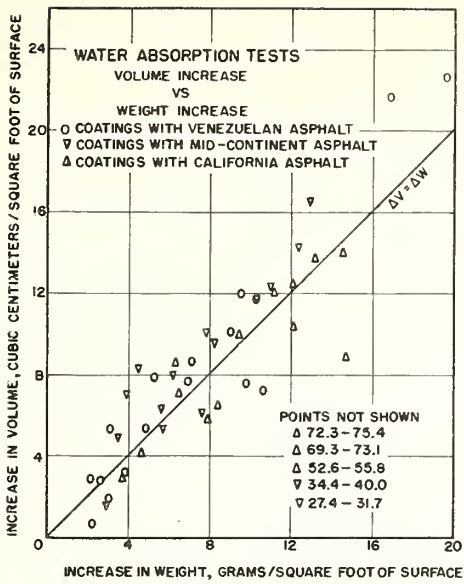


FIG. 2

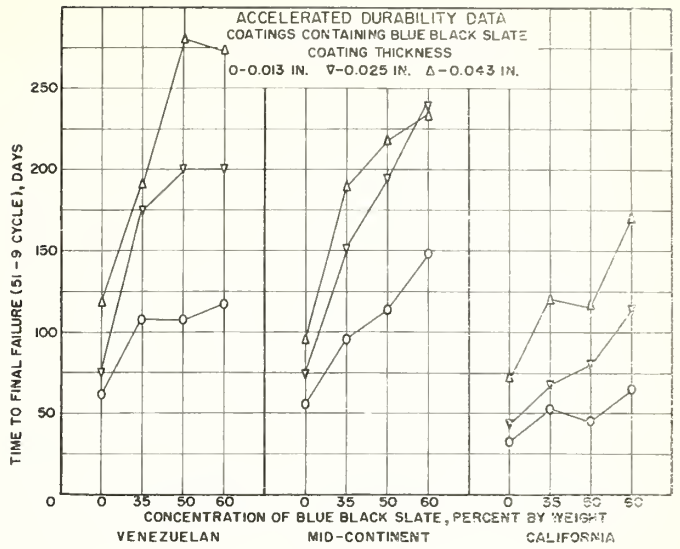


FIG. 3

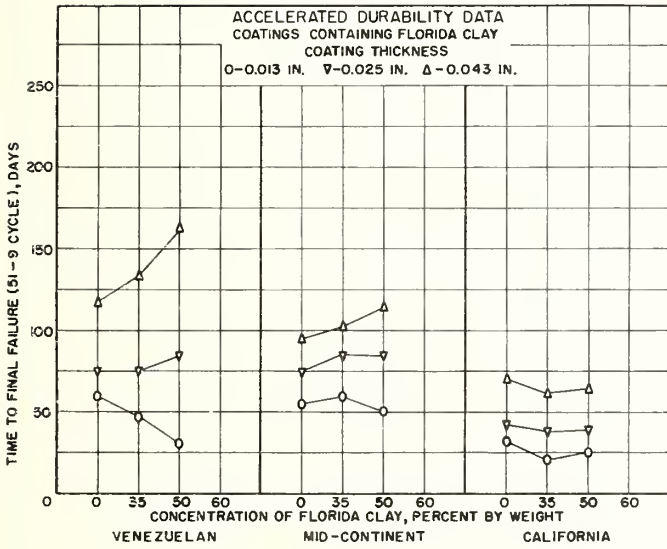


FIG. 4

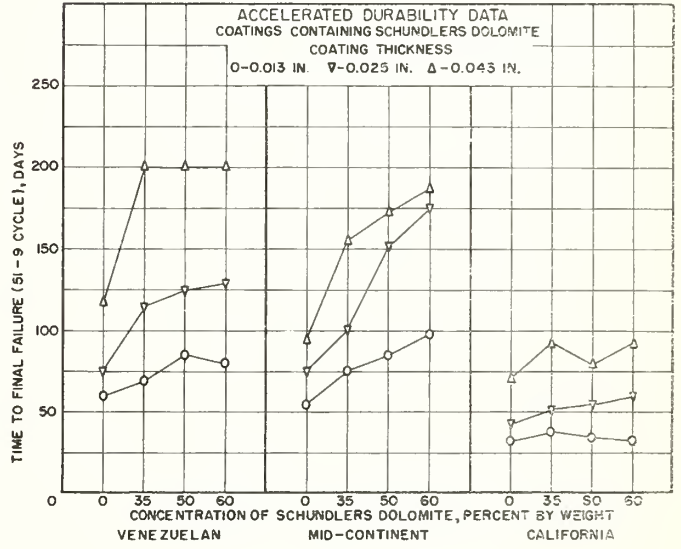


FIG. 5

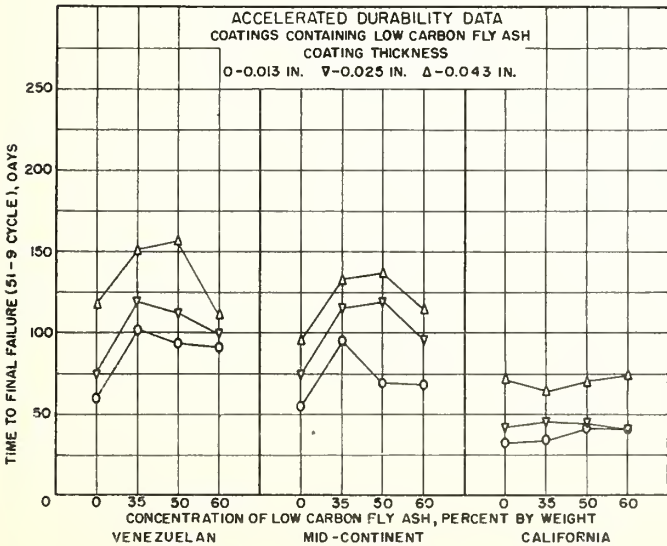


FIG. 6

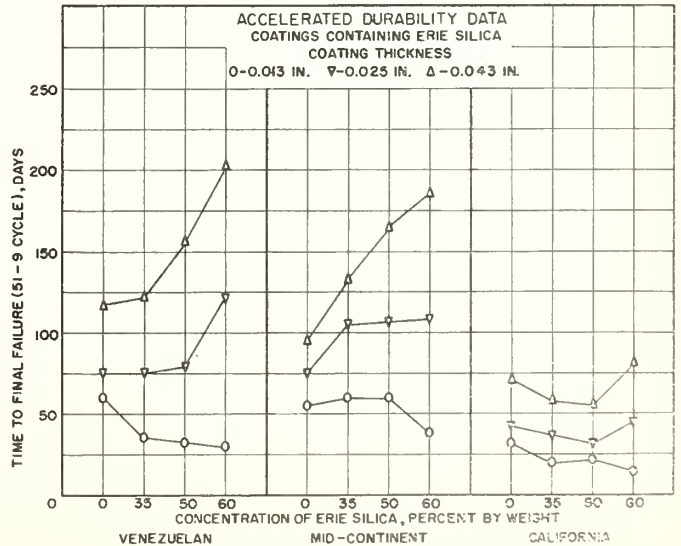


FIG. 7

The air content of a few of the coatings was determined but proved to be too small to be measured by the techniques employed.

(b) When the data were analysed to determine the degree of difference between the duplicate coatings exposed in different machines, it was found that in 92% of the cases, the individual panels failed within $\pm 15\%$ of their average durability. Figure 8 is a graphical presentation of the failure probability curve showing the percentage of the total number of panels^{failed}/vs. the percent deviation from their average. All of the sets falling outside the $\pm 15\%$ limits were repeated; the results are reported in Table XI.

6. DISCUSSION OF RESULTS

Since the durability data must be considered in respect to the asphalts and stabilizers from which the coatings were made, the results of the tests on these materials will be considered first. The asphalts were obtained from three roofing plants, where each had been processed into five different products from the fluxes described in Table I. These products, differing only in the length of time the asphalt was blown, were classified according to their softening points, as shown in Table I. During storage, the asphalts hardened progressively, but not uniformly, making it difficult to pinpoint the exact softening point to which a coating

was blended. However, preliminary softening point determinations made shortly before a coating was to be prepared for exposure permitted the estimation of which adjacent products to blend to enable the coating to have a softening point close to the 220-225°F range customarily followed commercially and arbitrarily selected for these laboratory exposures. The above considerations serve to explain why, in Tables VI to VIII, some of the softening points reported are as low as 217°F and others as high as 227°F.

In a few instances where it was physically impossible to obtain a softening point as low as 227°F, because the softening point rise produced by that particular concentration of stabilizer was very great, all of Product 1 was used and the softening point was accepted even though, as in the case of 35% mica, it was very much higher.

The spectral analyses reported in Table II show that all three asphalts had strong lines present for such metals as nickel, copper, and vanadium. The consequences of the presence of these catalytically active metals are not certain.

The stabilizers which were blended with the asphalts for these studies were examined rather thoroughly and the results reported in Table III. All of these materials were selected on the basis of past experience to include stabilizers which were known to perform well, materials which had caused

trouble, and materials which were used in large quantities, but whose performance was reported to be variable. The relations between each stabilizer and the durability of the coatings made with it will be discussed later.

The physical properties of each of the unstabilized asphalt coatings exposed are listed in Table V. These show that all three asphalts are of the normal coating grade, of high softening point and low penetration. The penetration indices of all three are about the same and greater than 2, testifying to the fact that they are highly blown. The lowest susceptibility, shown by the Venezuelan asphalt, indicates that it should withstand temperature variations best, followed by Mid-Continent and California asphalts, in order.

The California asphalt had the greatest heat loss at 163°C; none of the penetrations changed appreciably during the five-hour exposure.

Although the viscosity measurements do not indicate it, the California asphalt had a sharper melting point than the other two. However, the Mid-Continent asphalt was the most tacky as well as the most viscous.

The Venezuelan asphalt had the lowest water absorption during any particular time interval and also the smallest increase in volume during submersion. The shatter tests

showed it to be the most brittle of the three. However, while the Mid-Continent asphalt fell between the Venezuelan and California on most determinations, it was the least brittle as well as the most viscous of the three. In general, the properties of the unstabilized asphalts do not align themselves in a manner that would be a positive indication of their relative durabilities.

When finely divided mineral matter is added to an asphalt it stiffens the asphalt and increases the temperature to which it must be heated to induce flow. The stabilizers behave differently in each asphalt and in the different asphalts. In Tables VI to VIII are listed the softening points of the coatings and the estimated softening points of the base asphalts. By subtracting the two (see Table XII), it can be seen that the softening point rise increased with concentration for each stabilizer. However, the effect was different in each asphalt, being least pronounced in Mid-Continent and most in Venezuelan. The viscosity data show that all of the stabilizers are influential in making the coatings progressively more viscous as their concentration is increased, but the effect is not so systematic as for the softening point rise. To state this condition differently, the temperature coefficient of viscosity is a function of the stabilizer-asphalt combination. The same stiffening effect

TABLE XII. EFFECT OF STABILIZERS ON SOFTENING POINT AND SHATTER OF COATINGS

Asphalt:	Softening Point Rise ^a			Shatter Resistance Increase ^b		
	Ven.	M.C.	Cal.	Ven.	M.C.	Cal.
Coating	deg Fahr	deg Fahr	deg Fahr	in.	in.	in.
35% BBS	13	11	12	6.5	3.7	5.4
50% BBS	30	15	24	12.8	9.8	12.0
60% BBS	66	47	51	>18.3	>12.7	>14.7
35% Clay	16	15	20	6.6	0	3.0
50% Clay	37	29	37	3.6	7.4	3.3
35% Dolomite	17	8	11	4.6	7.4	0
50% Dolomite	20	13	19	5.6	7.2	-0.5
60% Dolomite	30	--	28	9.3	10.0	7.7
35% Fly Ash	19	14	18	2.6	-1.0	2.1
50% Fly Ash	33	28	30	8.0	3.7	---
60% Fly Ash	76	63	67	10.0	12.7	5.8
35% Mica	>80	>80	>80	>18.3	>12.7	>14.7
35% Silica	20	10	--	3.6	3.4	1.6
50% Silica	22	17	18	4.6	11.7	9.0
60% Silica	31	25	21	5.6	9.4	11.0

^aThe softening point rise is the difference between the softening point of the coating and that of the base asphalt.

^bThe shatter resistance increase is the difference between the shatter of the coating and that of the corresponding unstabilized asphalt. The shatter of the unstabilized asphalts are: Ven. = 2.7; M.C. = 8.3; Cal. = 6.3.

can be seen upon examination of the increase in shatter reported in Table XII. In all but four instances the stabilizers increased the shatter resistance of the coating; but again, no quantitative correlation is apparent.

The water absorptions determined on 3- by 3/16-in. specimens of each coating immersed beneath one-quarter inch of distilled water revealed that all stabilizers increased the water absorption of the asphalts progressively with increasing concentration, but not always in direct proportion to the concentration. The Venezuelan asphalt, itself, absorbed water at the lowest rate and all of its stabilized coatings absorbed water more slowly than the corresponding coatings made from the other asphalts. The Mid-Continent asphalt fell between the low rate of the Venezuelan asphalt and the high rate of the California asphalt.

For comparison purposes the water absorptions at one year have been listed in Table XIII-A along with ratio figures to indicate the increase in absorption produced by the addition of the stabilizer. These data show that although the Venezuelan asphalt coatings continued to have the lowest water absorption when stabilizers were mixed with it, the increase in absorption produced by the addition of stabilizer was greater than for the other two asphalts in the case of Blue Black Slate, Clay and Dolomite, less in Fly Ash, and equivalent

TABLE XIII. WATER ABSORPTION DATA^a

A. WATER ABSORBED IN ONE YEAR

ASPHALT:	Venezuelan		Mid-Continent		California	
	g/ft ²	ratio	g/ft ²	ratio	g/ft ²	ratio
Unstabilized	1.85	1	2.50	1	3.38	1
35% BBS	3.72	2.0	4.28	1.7	5.25	1.6
50% BBS	5.44	2.9	5.55	2.2	7.96	2.4
60% BBS	6.75	3.7	8.05	3.2	8.64	2.6
35% Clay	5.01	2.7	4.84	1.9	6.80	2.0
50% Clay	6.90	3.7	7.52	3.0	8.67	2.6
60% Clay	--	---	--	---	11.33	3.4
35% Dolomite	4.29	2.3	4.94	2.0	6.60	2.0
50% Dolomite	5.54	3.0	6.31	2.5	7.96	2.4
60% Dolomite	7.00	3.8	7.86	3.1	10.15	3.0
35% Fly Ash	6.00	3.2	7.45	3.0	51.70	15.3
50% Fly Ash	11.05	6.0	14.30	5.7	57.30	17.0
60% Fly Ash	12.20	6.6	23.60	9.4	42.90	12.7
35% Mica	2.46	1.3	3.19	1.3	5.21	1.5
35% Silica	2.47	1.3	2.80	1.1	3.81	1.1
50% Silica	2.66	1.4	3.02	1.2	6.28	1.9
60% Silica	2.97	1.6	3.45	1.4	11.29	3.3

^aThe complete water absorption curves are in the Appendix.

TABLE XIII. WATER ABSORPTION DATA^a

B. WEIGHT INCREASE IN 20 MONTHS

ASPHALT:	Venezuelan		Mid-Continent		California	
	g/ft ²	ratio	g/ft ²	ratio	g/ft ²	ratio
Unstabilized	2.29	1	2.97	1	4.62	1
35% BBS	4.86	2.1	5.61	1.9	6.49	1.4
50% BBS	6.93	3.0	7.65	2.6	12.06	2.6
60% BBS	10.27	4.5	12.35	4.2	13.23	1.9
35% Clay	9.81	4.3	5.72	1.9	7.89 ^b	1.7
50% Clay	10.57	4.6	7.78 ^b	2.6	12.07	1.6
60% Clay	--	---	--	---	9.35 ^b	2.0
35% Dolomite	5.34	2.3	6.19	2.1	9.36	2.0
50% Dolomite	7.16	3.1	8.24	2.8	11.14	1.4
60% Dolomite	9.03	4.0	10.32	3.5	14.68	3.2
35% Fly Ash	9.56	4.2	12.90	4.3	69.30	15.0
50% Fly Ash	16.87	7.4	27.38	5.9	72.26	15.6
60% Fly Ash	19.60	8.6	34.44	11.6	52.55	11.4
35% Mica	3.06	1.3	3.94	1.3	6.34	1.4
35% Silica	3.04	1.3	3.48	1.2	3.71 ^b	0.8
50% Silica	2.70 ^b	1.2	3.80	1.3	8.36 ^b	1.8
60% Silica	2.18	1.0	4.53	1.5	14.47 ^b	3.1

^aThe complete water absorption curves are in the Appendix.

^bUnderwent a weight loss during the last 100-200 days of immersion.

TABLE XIII. WATER ABSORPTION DATA^a

C. VOLUME INCREASE IN 20 MONTHS

ASPHALT:	Venezuelan		Mid-Continent		California	
	cc/ft ²	ratio	cc/ft ²	ratio	cc/ft ²	ratio
Unstabilized	0.70	1	1.63	1	4.11	1
35% BBS	5.34	7.6	6.28	3.9	7.07	1.7
50% BBS	7.68	11.0	6.10	3.7	12.37	3.0
60% BBS	11.69	16.7	14.34	8.8	13.72	3.3
35% Clay	7.60	10.9	5.27	3.2	5.83 ^b	1.4
50% Clay	7.32	10.5	10.12 ^b	6.2	10.44	2.5
60% Clay	--	--	--	--	9.86 ^b	2.4
35% Dolomite	7.91	11.3	7.99	4.9	10.03	1.4
50% Dolomite	8.66	12.4	9.59	5.9	12.11	2.9
60% Dolomite	10.16	14.5	11.75	7.2	8.94	2.2
35% Fly Ash	12.00	17.1	16.61	10.2	73.10	17.8
50% Fly Ash	21.64	30.9	31.68	19.4	75.40	18.3
60% Fly Ash	22.70	32.4	39.97	24.5	55.80	13.6
35% Mica	5.38	7.7	7.02	4.3	8.58	2.1
35% Silica	1.93	2.8	4.85	3.0	2.94 ^b	0.7
50% Silica	3.76 ^b	3.9	3.22	2.0	6.52 ^b	1.6
60% Silica	2.90	4.1	8.26	5.1	14.02 ^b	3.4

^aThe complete water absorption curves are in the Appendix.

^bUnderwent a weight loss during the last 100-200 days of immersion.

in Mica and Silica. These data also show the difficulty involved in trying to draw generalized conclusions for all stabilizers and asphalts; each system must be considered on its own merits.

When the water absorption data at 20 months are examined (Table XIII-B), it is found that while the rate of absorption generally had increased, the ratio of absorptions of the stabilized coatings to unstabilized asphalt had remained the same. The exceptions to this rule were the fly ash-stabilized coatings, in which the ratio increased for the Venezuelan and Mid-Continent asphalts and decreased for the California asphalts.

Table XIII-C also shows the volume changes involved for the 20-month immersion and the ratio of the changes in the stabilized coatings to those of the straight asphalts. Again, though the volume changes in the Venezuelan coatings tended to be lower than in the coatings containing the other two asphalts, because of the extremely low volume increase of the straight Venezuelan asphalt, the relative volume changes in the stabilized coatings were highest.

During the last 100-200 days of immersion several of the specimens underwent a loss in weight, while the remainder continued to gain. Three of these contained clay and four, silica. There was no apparent reason for the deviation of these specimens from the general trend.

Forty-eight coatings were subjected to accelerated durability tests, each in duplicate, in three film thicknesses. Because all of these could not be exposed simultaneously in one accelerated durability machine and because no two machines, or indeed no individual machine, operate identically at all times, duplicate panels were exposed in two different machines (odd-numbered panels in one machine and even-numbered ones in the other) to average the machine differences. When the coatings had been removed from the machines and the results tabulated, it was found that differences in durability between the two panels of each thickness of each coating never exceeded 22.5% of their average, as shown in Figure 8. (This graph is the failure probability distribution curve of all of the panels exposed in the Principal Series, in which the number of panels that failed at any particular percentage of their average, expressed as a percentage of the total number of panels, is plotted against that percentage.) It was decided to make additional sets of the 8% of the coatings falling in the heels of the curve, outside of the $\pm 15\%$ limits, in order to get more precise data on their durabilities. The results of these exposures are reported in Table XI, along with additional exposures of all of the unstabilized asphalts and a few of the coatings of which the durabilities seemed to be questionable. In these "check" exposures, four panels of each

coating were exposed, two in each of two machines. These results were used with the original exposures to figure the average durability of the coatings plotted in Figures 3 to 7. The data in Table XI show that even in these check exposures some duplicate panels failed widely apart, but except in six instances, the average was not shifted over nine days. In one set of coatings, which had apparently failed prematurely in both machines, possibly because of some fault in the panel preparation, the average durability was increased 49 days. It should be noted that this coating was only 0.013-in. thick, as were most of the coatings in which large differences in durability were present in duplicate exposures. Because the top size of the stabilizer particles is of the same order of magnitude as this film thickness, particle dispersion and orientation would be critical in determining the durability of the coating, and may be a partial explanation for these few large discrepancies.

Of course, the nature of the method by which failure is judged is probably the factor of greatest influence in determining the precise durability of a coating and possible differences in duplicate coatings. Inspection periods were seven days apart and a difference of as little as one pin hole in the entire panel area might make that much (seven days) difference in the reported durability of two panels. The

random nature of the cracking of the coating, just as in the breaking of a sheet of glass, in many instances will relieve the strains in one panel with greater than 50% of the area showing cracks and in its duplicate with somewhat less than that number. It may require several hundred hours before additional strain is produced sufficient to induce more cracking. Thus, if the final failure crack level had been selected at some other degree, the concordance would have been reported differently.

The type of cracking, as has been discussed previously (page 8), also entered into the determination of final failure. In the coatings in which types A to C crack patterns appeared, the exact failure level was of little consequence, for cracks appeared simultaneously throughout the major portion of the coating; but in those coatings which show types E through H crack patterns, cracking was slow and progressive, and the durability would be very closely related to the fractional area affected and adjudged to be final failure. All of the above considerations must be kept in mind when reviewing the durability data.

In interpreting the durability data there are several ways in which they must be considered. The absolute durability is the primary consideration -- How long will each particular coating last? Secondly, the way the durability

of an asphalt is modified by the addition of stabilizer -- What is the ratio of the durability of a stabilized coating to that of a film of the same thickness of the same unstabilized asphalt? And finally, the film thickness must be considered -- Will a thin film perform as satisfactorily as a normal or thick film? These points will be discussed for each stabilizer and its effects on each asphalt.

The three asphalts used in this investigation came from widely separated sources and varied considerably in their durability. The California asphalt was a shorter lived material than the Mid-Continent and Venezuelan asphalts, which were of approximately the same durability. Only in the 0.043-inch thick films did the Venezuelan prove to be appreciably more durable than the Mid-Continent. When stabilizers were added to these asphalts, the same order of spread was maintained.

In terms of absolute durability as determined in the Accelerated Durability Machines, the coatings stabilized with 35% mica have proved to be best. After more than 650 days, all of the normal and thick films, except for the 0.025-inch ones containing California asphalt, show no signs of failing; the thin mica-stabilized films with California asphalt failed in 175 days, with Mid-Continent asphalt failed in 369 days, and with Venezuelan asphalt failed in 402 days.

These are greater than the durability of any other corresponding coating of the asphalts tested even in the 0.043-in. thickness range. But, of course, the California asphalt stabilized with 35% mica, 0.013-in. thick, was not more durable than some of the other stabilized asphalts of greater film thickness.

The California coating, 0.025-inch thick, with 35% mica, failed in 572 days, which is more than 13 times the durability of the unstabilized asphalt of equivalent thickness. The 0.013-inch coating lasted only 6.7 times as long as the straight California asphalt, indicating that not only was the durability greatly increased by doubling the coating thickness, but the increase in durability was more than proportional to the increase in thickness. All of the mica-stabilized coatings that have failed did so in the Type A pattern, with no visible signs of cracking.

The stabilizer that produced the next greatest durability was blue black slate; the magnitude of the durabilities of the coatings made with blue black can be seen in Table X and Figure 3, and the ratios of these durabilities to that of the corresponding straight asphalts and the failure crack patterns are also reported in Table X. Again the California-based coatings were the least durable and the Venezuelan and Mid-Continent ones about the same. However,

in all instances the stabilizer increased the durability of the coating. The smallest increase was 41% for 50% blue black slate in California asphalt 0.013-inch thick; the greatest increase was 315%, with blue black slate in Mid-Continent asphalt 0.025-inch thick.

Another way of looking at these data, since each stabilizer-asphalt system behaves differently and must be considered separately, is to calculate the thickness or weight of stabilized coating equivalent in durability to some standard material. Since in commercial practice unstabilized coating asphalt on shingles is about 0.025-inch thick, Table XIV contains these calculations referred to the straight asphalts 0.025-inch thick. It can be seen that for the Mid-Continent and Venezuelan asphalts, coatings only 40% as thick (limited by the top particle size of the stabilizer) containing blue black slate stabilizer and between 52 and 67% as heavy provided the same durability as the 0.025-inch thick straight asphalts. For the stabilized California asphalt it was necessary to have 52% of the thickness and between 68 and 86% of the weight, depending on the concentration of the stabilizer.

Since commercial roofing is made to a given weight, rather than thickness, it would be of practical interest to compare the durability of coatings equivalent in weight to

TABLE XIV. STABILIZED COATING THICKNESS EQUIVALENT IN DURABILITY TO STRAIGHT ASPHALT COATINGS 25 MILS THICK

ASPHALT:	<u>Thickness</u>								
	Venezuelan (75 Days)			Mid-Continent (75 Days)			California (51 Days)		
	35%	50%	60%	35%	50%	60%	35%	50%	60%
CONCENTRATION:	35%	50%	60%	35%	50%	60%	35%	50%	60%
Stabilizer	Mils	Mils	Mils	Mils	Mils	Mils	Mils	Mils	Mils
BBS	10(6)*	10	10(7)*	10	11	10(4)*	13	13	13
Clay	25	25	--	22	23	--	35	33	--
Dolomite	13	13	13	13	12	10	25	25	21
Fly Ash	10(4)*	10	10	10(5)*	16	16	25	25	25
Silica	25	25	19	18	18	20	31	40	31

*The figures in parenthesis are those actually obtained by extrapolation, but because the maximum particle size of the stabilizers is ten mils, that has been taken as the minimum coating thickness.

TABLE XIV. WEIGHT OF STABILIZED COATING EQUIVALENT IN DURABILITY TO STRAIGHT ASPHALT COATINGS 25 MILS THICK

ASPHALT:	<u>Weight per 100 sq. ft.</u>								
	Venezuelan* (75 Days)			Mid-Continent* (75 Days)			California* (51 Days)		
	35%	50%	60%	35%	50%	60%	35%	50%	60%
CONCENTRATION:	35%	50%	60%	35%	50%	60%	35%	50%	60%
Stabilizer	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.	lb.
BBS	6.9	7.9	8.7	6.8	8.5	8.6	8.9	10.2	11.4
Clay	16.9	19.2	--	14.6	17.3	--	23.6	25.2	--
Dolomite	8.9	10.1	11.2	8.7	9.2	8.5	17.0	19.6	18.2
Fly Ash	6.7	7.6	8.3	6.6	12.0	13.2	16.8	19.0	20.8
Silica	16.9	19.1	16.0	12.0	13.6	16.6	21.0	30.6	26.1

*Weight of straight asphalt coatings 0.025-in. thick.

Ven. = 13.20 lb/100 sq.ft. - M.C. = 12.95 lb/100 sq.ft.

Cal. = 13.15 lb/100 sq.ft.

an unstabilized asphalt coating 0.025-inch thick. In Table XV are listed the thicknesses of the stabilized coatings of this weight and their durability. For lack of data to make more precise calculations, these figures were obtained from straight line interpolations between the durabilities of the 0.013- and 0.025-inch coatings. On this uniform-weight basis, the trends indicated by the accelerated durability tests are somewhat different from those reported on a film-thickness basis. For the blue black slate in Mid-Continent asphalt, the durability still increased with stabilizer concentration, despite the fact that the coating thickness was decreased through 40 percent. However, the durability of the coatings with the other two asphalts now appears to be almost independent of the stabilizer concentration.

Blue black slate and mica are the only two stabilizers that increased the durability of all three asphalts under all of the test conditions. The behavior of clay, dolomite, fly ash and silica varied somewhat among the three asphalts. They will be considered in descending order of their general effects.

Dolomite and fly ash were about equivalent in their effects on the durability of the three asphalts. However, because of the large number of type A failures and because

TABLE XV.

DURABILITY OF COATINGS EQUIVALENT IN WEIGHT TO THEIR
UNSTABILIZED ASPHALT 25 MILS THICK

ASPHALT:	Venezuelan (75 Days)			Mid-Continent (75 Days)			California (51 Days)		
COATING WEIGHT, lb/100 sq.ft.:	13.2			13.0			13.2		
CONCENTRATION:	35%	50%	60%	35%	50%	60%	35%	50%	60%
<u>Stabilizer</u>	Days	Days	Days	Days	Days	Days	Days	Days	Days
BBS	141	138	131	124	140	163	60	58	74
Clay	64	52	--	75	62	--	31	30	--
Dolomite	92	99	88	88	107	111	45	42	38
Fly Ash	112	100	84	107	86	75	41	43	41
Silica	59	48	53	86	75	56	32	25	23

STABILIZED COATING EQUIVALENT IN WEIGHT TO STRAIGHT
ASPHALT COATING 25 MILS THICK

ASPHALT:	<u>Thickness</u>								
	Venezuelan (75 Days)			Mid-Continent (75 Days)			California (51 Days)		
CONCENTRATION:	35%	50%	60%	35%	50%	60%	35%	50%	60%
<u>Stabilizer</u>	Mils	Mils	Mils	Mils	Mils	Mils	Mils	Mils	Mils
BBS	19	17	15	19	17	15	19	17	15
Clay	20	17	16	20	17	16	20	17	16
Dolomite	19	17	15	19	17	15	19	17	15
Fly Ash	20	17	16	20	17	16	20	17	16
Mica	19	17	15	19	17	15	19	17	15
Silica	20	17	16	20	17	16	20	17	16

fly ash had to be inspected visually only (precluding the possibility of a type A failure and thus prolonging the reported life of the coating), dolomite was probably more active than fly ash in prolonging the durability of the asphalts. As a matter of fact, dolomite and fly ash were the intermediate stabilizers, between the excellent qualities of mica and blue black slate and the rather doubtful qualities of silica and clay.

Dolomite showed marked improvement in the durability of the Mid-Continent (Ratios from 1.34-2.32) and the Venezuelan (Ratios from 1.43-1.71) asphalts. However, the durability of Venezuelan asphalt with 35% of dolomite was increased only 14% and only one combination of California asphalt and dolomite exceeded the durability of unstabilized California asphalt more than 30%. The coatings stabilized with dolomite, equivalent in durability to the corresponding 0.025-inch straight asphalts, ranged from 0.015 to 0.025 inch thick. As in the case of blue black slate, only in Mid-Continent asphalt did the durability continue to increase with concentration when the film thickness was adjusted so that the coating had the same weight as the unstabilized asphalt coating. Again, in Venezuelan asphalt, the durability was independent of the stabilizer

concentration; but, in the California asphalt, the durability was slightly below that of the straight asphalt.

The low carbon fly ash improved the durability of both Mid-Continent and Venezuelan asphalts, but did not alter it for the California asphalt, when the results are compared on an equal film thickness basis. In all instances the durability increased with film thickness for any given stabilizer concentration, but went through a maximum around 50% stabilizer. When compared on an equal weight basis, the California asphalt coatings with fly ash reacted similarly to those containing dolomite, but the durability of both the Mid-Continent and Venezuelan coatings decreased with increasing stabilizer concentration.

The Florida clay and Erie silica, although vastly different in properties, behaved similarly in influencing the durability of asphalt coatings. While the durability in all cases increased with film thickness, only in the 0.043-inch coatings with Venezuelan and Mid-Continent asphalts did it increase appreciably. For these two materials, except in the thickest films, durability was almost independent of stabilizer concentration. The data in Table XV indicate that on an equal-weight basis, at best, these materials did not decrease the durability at lower

concentrations in Mid-Continent asphalt, but were definitely deleterious in all other combinations tested.

As constantly reiterated throughout this discussion, it is extremely hazardous to attempt to draw generalized conclusions from the data in this report. However, a very rough listing of the additives tested would place them in the following descending order of their stabilizer merits:

- | | | |
|------|----|--------------------------|
| Good | -- | (1) Mica |
| | | (2) #50 Blue Black Slate |
| Fair | -- | (3) Schundlers Dolomite |
| | | (4) Low Carbon Fly Ash |
| Poor | -- | (5) Erie Silica |
| | | (6) Florida Clay |

No matter how the results of the accelerated durability tests are considered, the mica and blue black slate were always beneficial in all three asphalts. The dolomite and fly ash increased the durability in most instances, but in some did not affect it. Their relative order is opposite to that in the above listing in some combinations. Similarly, the order of clay and silica varied with their various combinations with asphalt.

In attempting to predict the performance of coatings made with these asphalts and minerals, any properties or combination of properties of the minerals which would separate them into the above three categories would be

useful in estimating the value of a new material which might become available. The only characteristic that fulfills this requirement is particle shape. The stabilizers designated as "good" all have flat, plate-like particles; the "fair" materials have sharp, blocky particles; and the "poor" materials have rounded corners and edges. Thus a new material would be expected to have sharp, irregular to flat, platy particles to be considered for use as a stabilizer. Of course, the other properties would have to be considered as well. The particle size and size distribution would have to be considered as would the moisture and free alkali content, loss on ignition, and solubility; for, obviously, an inert material of suitable firmness is required. Further work, to be reported later, will deal with these characteristics.

7. CONCLUSIONS

Because the stabilizers behaved differently in each asphalt, it was not possible to draw general conclusions on the effects of the six stabilizers on the three asphalts tested. However, several broad generalizations can be stated:

- (1) For every stabilizer-asphalt combination, the durability of the coating increased with film thickness.

(2) All stabilizers increased the softening point, viscosity, water absorption, and impact resistance of the coatings progressively with increasing concentration.

(3) California asphalt and stabilized coatings made with it were the least durable, while coatings containing Mid-Continent and Venezuelan asphalts were approximately equivalent in durability.

(4) Blue black slate and mica increased the durability of all three asphalts at all concentrations and film thicknesses tested.

(5) Dolomite and fly ash increased the durability of the coatings in many cases, but in others had no appreciable effect on it.

(6) Silica and clay increased the durability only in some instances, mainly in the thicker films with Mid-Continent and Venezuelan asphalt.

APPENDIX A.

SHATTER TEST ON COATINGS

Preparation of Specimen:

Several disks of asphalt 3" in diameter and 3/16" thick are cast using a suitable amalgamated or glycerine coated brass mold. After pouring to an excess thickness, and cooling, the specimens are trimmed down to exact thickness using a hot knife.

Test Method:

The test apparatus consists of a means for dropping a constant weight from a variable and measured height on the cast disk as prepared above, recording the height of drop required to split the specimen in one or more places, each split extending from the center to the edge of the specimen.

The apparatus consists of a 20" vertical brass tube 1" in internal diameter, a solenoid sliding within the tube and adjustable to any height up to 20", an electrical connection to a standard 110 volt line (either A.C. or D.C.) with the solenoid in series with a 60 watt lamp and a switch for shorting the solenoid, a falling steel weight 15/16" in diameter and weighing exactly 1/2 lb., and a stationary steel contact rod 15/16" in diameter, weighing exactly 1/2 lb. and having a hemispherical end contacting the asphalt.

In operation the specimen is brought to a temperature of 40°F. by submersion in a bath of ice and water for a period of not less than one hour. It is then placed under the vertical brass tube, being submerged in water at 40°F. during the test, and the contact rod placed in the tube and in contact with the center of the specimen. The circuit is closed to the solenoid and the falling 1/2 lb. weight raised to a height of 1" by raising the solenoid holding it. The solenoid is then shorted, the weight allowed to drop, the specimen removed and quickly examined for fracture. If it has not split it is replaced, the weight raised to 1-1/2" and the drop again made. This procedure is repeated with 1/2" increments in height until the specimen fails. Subsequent specimens should be started at a height 1" below the failing height of the first test. At least three determinations shall be made on each asphalt.

Results:

Failure is recorded when the specimen splits, in one or more places, from the center to the edge. Fractures that do not extend to the edge of the disk are ignored. The average height of drop required to break the specimen is recorded as its impact resistance.

Test Method No. 17134
Johns-Manville Research Laboratories
Manville, New Jersey.

WATER ABSORPTION OF ASPHALT
DISC METHOD

Application:

This method is applicable to all asphalts having Softening Points (R & B) of 170°F or over.

Apparatus and Materials Required:

1. Brass mould 3/16" thick, 3" diameter hole.
2. Brass plate - 4" x 4".
3. Glycerine.
4. Hot plate.
5. Red marking pencil.
6. 100 grams of asphalt to be tested.
7. Pyrex glass tray, 1-1/2" deep, any convenient length and width.
8. Distilled water.

Preparation of Specimen:

Apply glycerine to the surface of the clean brass plate and mould which will come in contact with the asphalt. Assemble the mould and place it on the brass plate. Carefully heat the sample of asphalt to be tested until fluid and free from air bubbles. If the sample contains filler, the sample should be stirred slowly with a piece of stiff wire so as to keep the filler properly suspended yet without incorporating air bubbles.

Pour a sufficient amount of the sample to barely fill the mould. The pouring must be done with care so that air bubbles are not created. The surface may be flamed lightly to remove a few which might form. Not more than 1/16" of the sample should show above the top of the mould. After the specimen has cooled thoroughly, remove it from the mould and wash thoroughly to remove the attached glycerine. Allow the specimen to dry and mark identification on both sides with the red marking pencil.

Procedure:

Weigh the specimen and record the weight. Place the specimen in the glass tray and fill with sufficient distilled water to submerge the specimen at least 1/4".

Procedure continued:

Place the glass tray and specimen in a dark cabinet at room temperature.

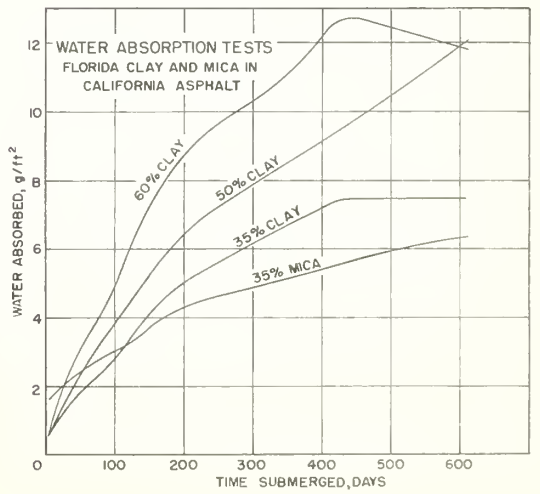
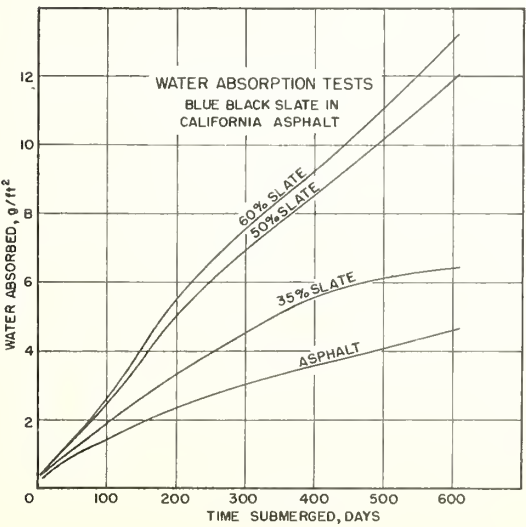
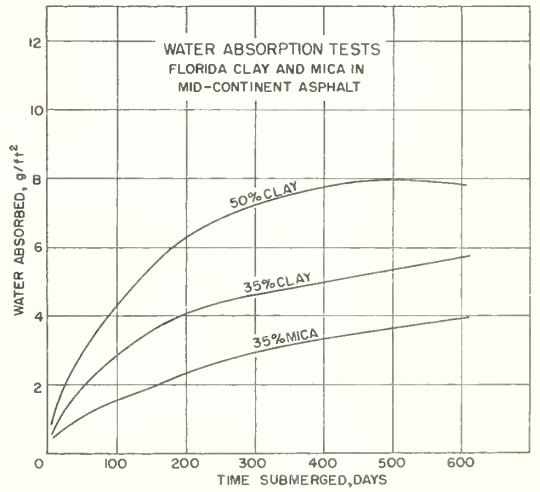
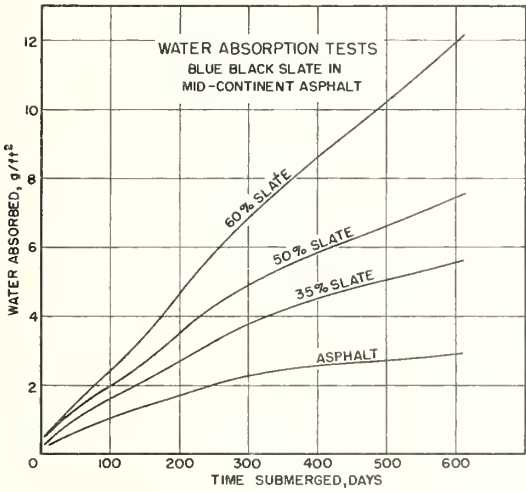
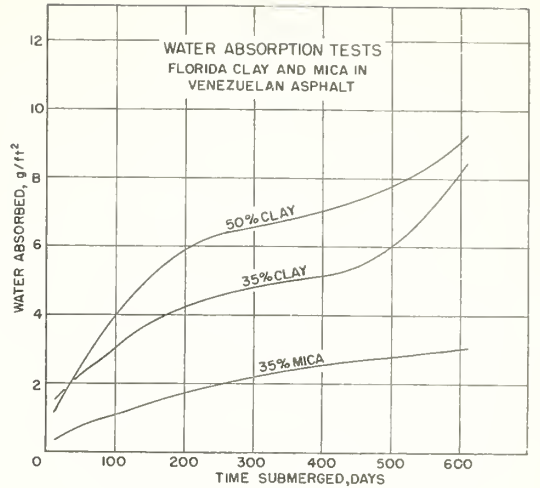
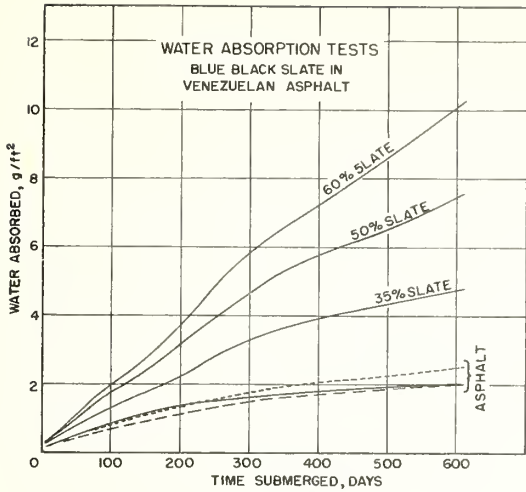
Make periodic weighings to determine the amount of water absorbed as follows:

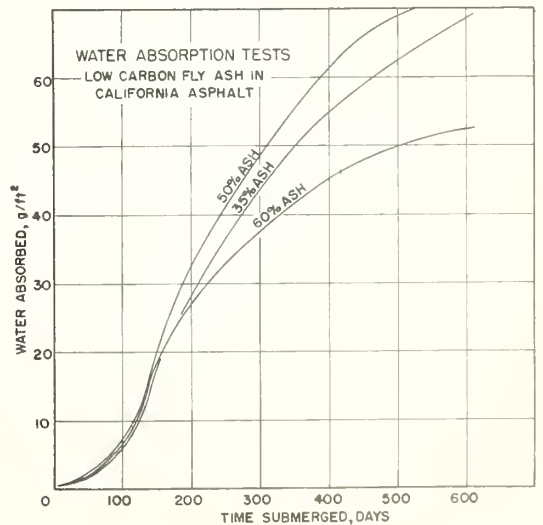
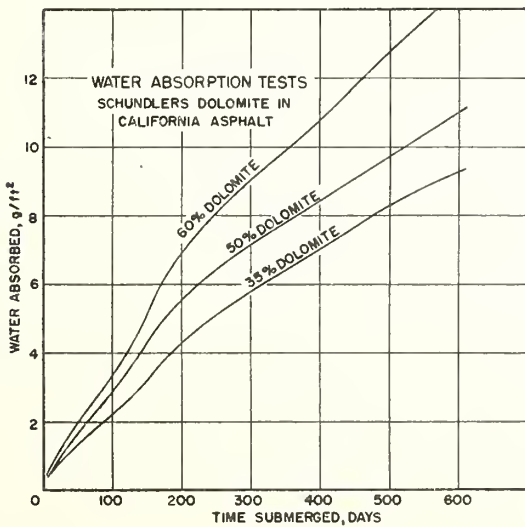
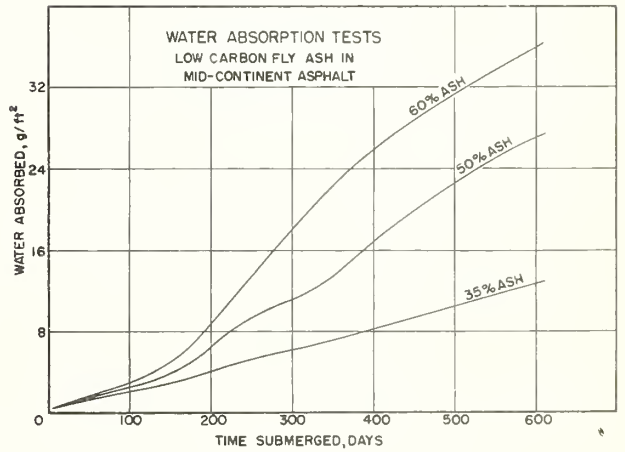
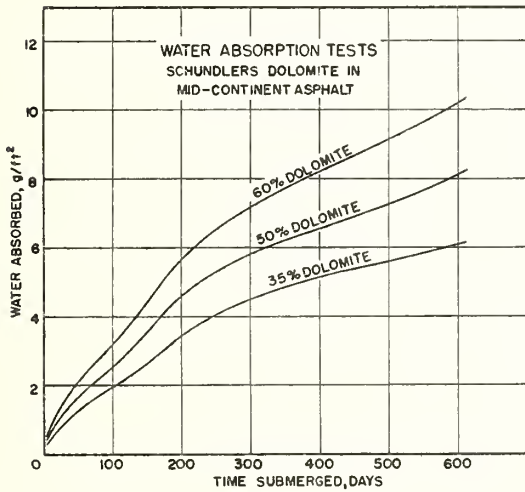
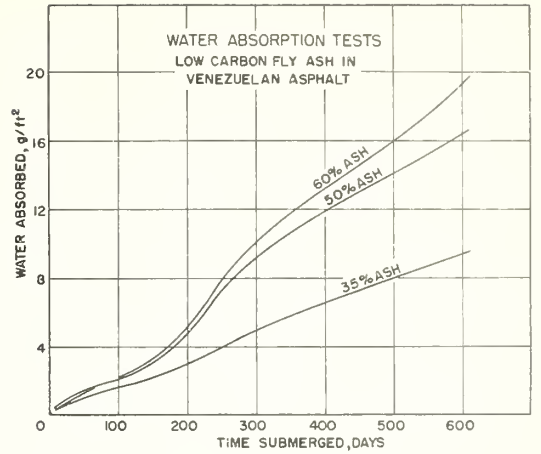
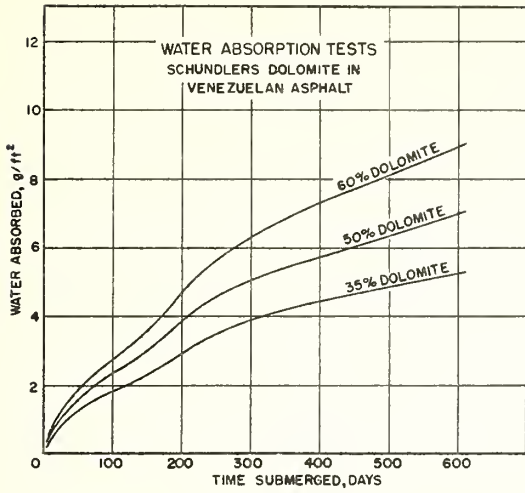
First 3 months	---	Weekly
Next 9 months	---	Monthly
Thereafter	---	Each 6 months.

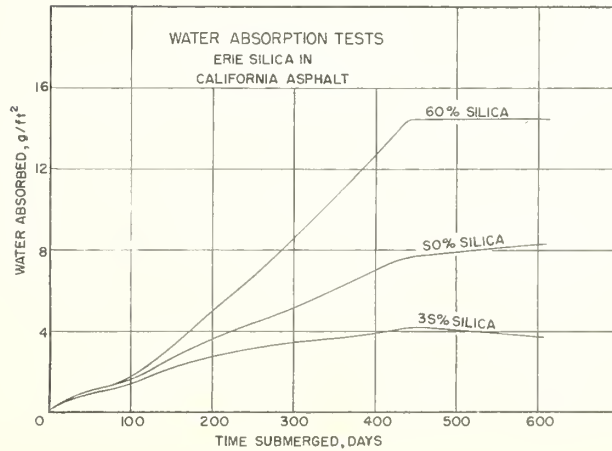
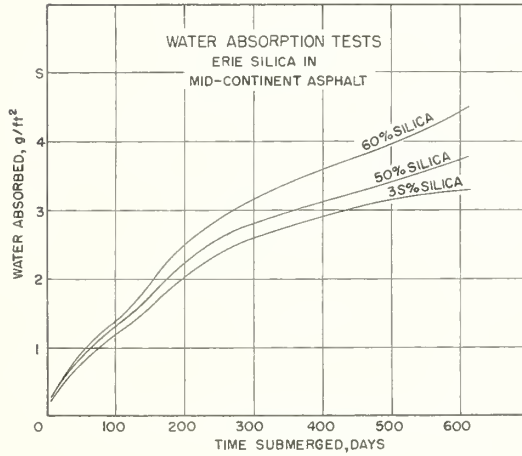
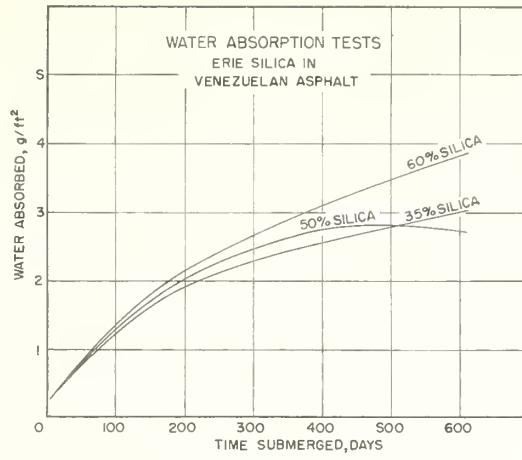
Remove the specimen from the water at the end of each specified period. Do not wipe but blot both sides and edges carefully so that each surface is as uniformly dry as possible. Weigh the specimen and record the weight. Return the specimen to the distilled water tray. Renew with fresh water at each weighing.

Compute the water absorbed and convert the result to grams of water absorbed per square foot of asphalt surface exposed.

Test Method: WA-2
Johns-Manville Research Laboratories
Manville, New Jersey.







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THE NATIONAL BUREAU OF STANDARDS

Functions and Activities

The functions of the National Bureau of Standards are set forth in the Act of Congress, March 3, 1901, as amended by Congress in Public Law 619, 1950. These include the development and maintenance of the national standards of measurement and the provision of means and methods for making measurements consistent with these standards; the determination of physical constants and properties of materials; the development of methods and instruments for testing materials, devices, and structures; advisory services to Government Agencies on scientific and technical problems; invention and development of devices to serve special needs of the Government; and the development of standard practices, codes, and specifications. The work includes basic and applied research, development, engineering, instrumentation, testing, evaluation, calibration services and various consultation and information services. A major portion of the Bureau's work is performed for other Government Agencies, particularly the Department of Defense and the Atomic Energy Commission. The scope of activities is suggested by the listing of divisions and sections on the inside of the front cover.

Reports and Publications

The results of the Bureau's work take the form of either actual equipment and devices or published papers and reports. Reports are issued to the sponsoring agency of a particular project or program. Published papers appear either in the Bureau's own series of publications or in the journals of professional and scientific societies. The Bureau itself publishes three monthly periodicals, available from the Government Printing Office: The Journal of Research, which presents complete papers reporting technical investigations; the Technical News Bulletin, which presents summary and preliminary reports on work in progress; and Basic Radio Propagation Predictions, which provides data for determining the best frequencies to use for radio communications throughout the world. There are also five series of nonperiodical publications: The Applied Mathematics Series, Circulars, Handbooks, Building Materials and Structures Reports, and Miscellaneous Publications.

Information on the Bureau's publications can be found in NBS Circular 460, Publications of the National Bureau of Standards (\$1.00). Information on calibration services and fees can be found in NBS Circular 483, Testing by the National Bureau of Standards (25 cents). Both are available from the Government Printing Office. Inquiries regarding the Bureau's reports and publications should be addressed to the Office of Scientific Publications, National Bureau of Standards, Washington 25, D. C.

