NATIONAL BUREAU OF STANDARDS REPORT

3616

LABORATORY STUDIES OF SEVERAL FACTORS AFFECTING THE BEHAVIOR OF SOME MINERALS AS STABILIZERS FOR ASPHALT ROOFING

By

Sidney H. Greenfeld

U. S. DEPARTMENT OF COMMERCE
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LABORATORY STUDIES OF SEVERAL FACTORS AFFECTING THE BEHAVIOR OF SOME MINERALS AS STABILIZERS FOR ASPHALT ROOFING

By
Sidney H. Greenfeld
Research Associate
Asphalt Roofing Industry Bureau

Floor, Roof and Wall Coverings Section
Building Technology Division

Sponsored by
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TABLES

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LABORATORY STUDIES OF SEVERAL FACTORS AFFECTING THE BEHAVIOR OF SOME MINERALS AS STABILIZERS FOR ASPHALT ROOFING

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SUMMARY

The effects of a number of variables inherent in asphalts and minerals available for use in asphalt roofings that were not reported in "Laboratory Evaluation of Six Selected Commercially Available Materials as Stabilizers for Asphalt Roofings" are covered in this report. The influence of softening point of the asphalts; particle size distribution of the mineral matter; particle shape of the mineral matter; natural variations in three of the minerals; and the thoroughness of mixing on the durability of the coatings were measured. The following generalizations resulted:

(1) The durability of the three asphalts decreased as their softening points increased.
(2) The greater durability of the lower softening point asphalts was readily apparent with mineral concentrations of 50% or greater.
(3) None of the three clays evaluated had any appreciable effect on the durability.
(4) Fly ash samples could not be evaluated on the same basis as other samples because the spark method of determining failure could not be used. The indications are, however, based on visual determination of failure, that the effect of fly ash on durability is variable, that sample being designated as "low carbon" being least beneficial.
(5) Of the four silicas, only the special silica, characterized by its relatively greater fineness, (and with Asphalt II, Ottawa Silica) increased the durability of the asphalts.
(6) Thorough mixing is necessary to obtain the most uniform and optimum benefits from the use of minerals as stabilizers.
(7) Commercial blue black slate, Niagara dolomite and Lake Erie Silica were fractionated into a number of particle size ranges and reconstituted into five

synthetic size distributions. The durabilities of coatings made from 35% of each of these with each of the asphalts were in the following descending order:

<u>Classification with U. S. Standard Sieves</u>

Passing 200 mesh
Commercial Gradation
Passing 325 mesh
Equal Size (approximately equal weights on each sieve)
Uniform Size (Passing 140 - Retained on 230)
Passing 60 - Retained on 200 mesh

(8) Minerals with flat, plate-like particles (blue black slate, mica, and oyster shell) were most beneficial as stabilizers.

1. INTRODUCTION

During the planning of the mineral stabilizer research program it was recognized that there were several variables involved in stabilization of asphalts against degradation, that were not covered by the exposures of the main series[1] to be made. Supplementary exposure series were planned to determine whether any relationship could be established between some of these properties of the materials and durability and between some processing variables and durability. The following series of exposures were made:

(1) The Effect of Softening Point on the Durability of Asphalt.

(2) The Effect of the Softening Point of the Base Asphalts on the Durability of Coatings Containing Mineral Additives.

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[1] Greenfeld, S. H., "Laboratory Evaluation of Six Selected Commercially Available Materials as Stabilizers for Asphalt Roofing", Sept. 23, 1953.

(3) Selected Minerals - The Effect of Some Natural Variations of Clay, Fly Ash, and Silica on the Durability of Coatings.
(4) The Effect of the Degree of Mixing of Minerals with Asphalt on the Durability of Coatings.
(5) The Effect of the Particle-Size Distribution of the Mineral Matter on the Durability of Coatings.
(6) The Effect of the Particle Shape of the Mineral Matter on the Durability of Coatings.

With the exception of the Selected-Materials and Particle-Shape Series, all of the properties studied are of the type that can be modified in the laboratory or plant.

2. MINERAL MATERIALS

2.1 Materials Tested as Stabilizers

Because of the numerous materials tested as stabilizers and the limited number of tests performed on each, the mineral materials will be described in connection with the series of exposures with which each is connected. Table I is a general tabulation of the properties of some of these minerals.

2.2 Asphalts

The asphalts used were the same as those reported in "Laboratory Evaluation of Six Selected Commercially Available Materials as Stabilizers for Asphalt Roofing". The asphalt products were blended to produce a base asphalt which, when mixed with the desired mineral matter, resulted in coating with a softening point in the range of 217 to 227°F.

3. EQUIPMENT AND PROCEDURE

The equipment and procedures for making the exposure panels and exposing and evaluating the coatings were as described in the above-mentioned report.

TABLE I. PROPERTIES OF MINERALS

PROPERTY	BBS	FLA. CLAY	N. DOL.	L.E. SIL.	TENN. MICA	O.S.
Moisture, %	0.2	2.7	0.1	0.2	0.2	0.4
Loss on Ignition at 1000°F, %[a]	2.1	11.8	1.8	0.7	0.9	0.9
Loss on Ignition at 1800°F, %[a]	5.4	13.3	43.7	2.5	4.4	43.5
Water Solubility, %[a]	0.0	0.0	0.0	0.0	0.5	0.5
Free Alkali, %[a]	0.0	0.0	0.0	0.0	0.0	0.0
Density, g/cc[b]	2.94	2.64	2.87	2.68	3.01	2.68
Surface Area, M^2/g[c]	1.0	27.6	2.0	2.5	2.7	2.23
Oil Absorption, g/100g[d]	29.5	63.9	19.4	19.5	97.2	22.6
Water Absorption, g/100g[d]	32.7	36.4	18.5	20.2	61.5	23.0
Particle Size Distribution:[a]						
% Passing U.S. #60	99.8	100.0	99.9	99.9	100.0	97.7
% Passing U.S. #80	99.3	99.9	99.9	98.2	96.0	93.6
% Passing U.S. #100	97.9	99.8	99.6	80.8	91.0	88.3
% Passing U.S. #120	96.2	99.8	99.3	53.7	85.6	84.4
% Passing U.S. #170	91.3	99.6	96.6	15.3	65.9	75.5
% Passing U.S. #200	86.9	99.3	93.4	8.1	56.8	69.5
% Passing U.S. #230	83.6	99.1	89.9	5.4	50.3	65.2
% Passing U.S. #325	76.7	98.9	81.0	3.4	37.8	57.5
% Finer than 40 microns[e]	68	97	73	9	65	----
% Finer than 20 microns	54	12	39	6	22	----
% Finer than 10 microns	8	2	26	4	4	----
% Finer than 4 microns	2	1	13	1	2	----
% Finer than 2 microns	1	----	7	----	----	----
Chemical Analysis:[f]						
SiO_2, %	56	47	6	98+	50	
R_2O_3, %	32	38	1		35	0.2
CaO + MgO, %			49		1	
$K_2O + Na_2O$, %	4				10	
C, %	2					
Mixture with Asphalt[a][g]:						
Ease of Mixing	Good	Poor	Fair	Good	Good	Good
Soft. Point Increase, °F	15	28	13	5	40	15

(FOR FOOTNOTES, SEE NEXT PAGE.)

[a] Method described in "A Survey of Materials Available for Use as Mineral Stabilizers", A.R.I.B., Oct. 1952.

[b] Isopropyl Alcohol Displacement.

[c] Nitrogen Adsorption - B.E.T. Method.

[d] A.S.T.M. Method D281-31, Using Primol D and Water.

[e] Sedimentation in Isopropyl Alcohol.

[f] Supplier's Data for Typical Material.

[g] 40% Mineral Matter in Asphalt III (S.P. = 223°F).

4. EXPOSURE SERIES

4.1 The Effect of the Softening Point of the Asphalts on Their Durability

Coatings, 25 mils thick, were made from each of the asphalt products and exposed in the accelerated durability machines to determine the effect of softening point on the durability of the asphalts.

The durabilities of these products are tabulated in Table II and shown graphically in Figure 1. The general trend is for the durability to decrease as the softening point of the asphalt increases. It must be stated, however, that there was an occasional specimen that deviated from the general trend with an abnormally low or high durability. Some differences in durability were the result of exposing half of the panels in one machine (odd ones) and half in another (even ones). In most cases the agreement was good, but in two instances, there was considerable divergence between the results of the two exposures.

4.2 The Effect of the Softening Point of the Base Asphalt on the Durability of Coatings Containing Mineral Additives

Table III and Figure 2 are comparisons of coatings made from a common base (Product C of Asphalt II) asphalt with those made to a definite softening point range, employing progressively softer asphalt as the quantity of mineral additive was increased. The coatings containing the common base seemed to be slightly more durable in the lower mineral concentrations, but the difference was very small and of the same order of magnitude as the differences found in replicate panels exposed at the same time. Thus, variation in the softening point of coatings containing mineral additives seems to be less significant than in asphalt coatings without additives.

TABLE II. DURABILITY OF ASPHALT PRODUCTS

PRODUCT	A	B	C	D	E
Asphalt I					
Softening Point, °F	192	205	215	224	237
Penetration at 25°C	24	21	19	17	15
Acc. Dur., Days[a]					
Panel No. 1	44	37	29	29	28
Panel No. 7	30	37	29	29	28
Panel No. 2	52	38	36	36	28
Panel No. 6	52	38	36	29	28
Average	44	37	33	31	28
Failure Pattern[b]	C	C	C	C	C
Asphalt II					
Softening Point, °F	196	212	225	234	239
Penetration at 25°C	25	20	17	17	15
Acc. Dur., Days[a]					
Panel No. 1	108	83	82	81	48[c]
Panel No. 7	83	83	69	61	81
Panel No. 2	100	84	84	83	83
Panel No. 6	100	84	84	63	83
Average	98	83	80	72	82
Failure Pattern[b]	CA	A	AC	EA	AED
Asphalt III					
Softening Point, °F	195	195	208	235	239
Penetration at 25°C	19	19	19	15	15
Acc. Dur., Days[a]					
Panel No. 1	95	95	81	85	64
Panel No. 7	86	95	81	85	57
Panel No. 2	87	87	81	46	37
Panel No. 6	87	87	41[c]	46	37
Average	89	91	81	66	48
Failure Pattern[b]	AC	AC	A	AF	A

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[a] Odd numbered panels were in one machine; even in another.

[b] Patterns are described in "Laboratory Evaluation of Six Selected Materials as Stabilizers in Asphalt Roofing".

[c] Not counted in average.

TABLE III. EFFECT OF THE SOFTENING POINT OF THE BASE ASPHALT ON THE DURABILITY OF COATINGS[a]

CONCENTRATION OF MINERAL, %	PANEL NO.[b]	COMMON BASE SOFT. PT. °F BASE	COMMON BASE SOFT. PT. °F MIX	COMMON BASE DUR. DAYS	VARIABLE BASE SOFT. PT. °F BASE	VARIABLE BASE SOFT. PT. °F MIX	VARIABLE BASE DUR. DAYS
Asphalt II	1	225	225	82	228	228	74
	7	"	"	69	"	"	74
	2	"	"	84	"	"	76
	6	"	"	84	"	"	76
	Ave.	"	"	80	"	"	75
35% B.B.S.	1	"	231	177	211	222	145
	7	"	"	177	"	"	---
	2	"	"	163	"	"	158
	6	"	"	163	"	"	---
	Ave.	"	"	170	"	"	152
50% B.B.S.	1	"	246	211	202	217	169
	7	"	"	211	"	"	---
	2	"	"	209	"	"	215
	6	"	"	198	"	"	---
	Ave.	"	"	209	"	"	192
60% B.B.S.	1	"	270	233	190	237	207
	7	"	"	227	"	"	---
	2	"	"	224	"	"	270
	6	"	"	224	"	"	---
	Ave.	"	"	227	"	"	238
35% N. Dol.	1	"	230	152	217	225	98
	7	"	"	134	"	"	---
	2	"	"	110	"	"	103
	6	"	"	110	"	"	---
	Ave.	"	"	126	"	"	101
50% N. Dol.	1	"	236	152	206	219	142
	7	"	"	152	"	"	---
	2	"	"	145	"	"	161
	6	"	"	145	"	"	---
	Ave.	"	"	149	"	"	152
60% N. Dol.	1	"	248	140	197	---	163
	7	"	"	146	"	---	---
	2	"	"	137	"	---	187
	6	"	"	130	"	---	---
	Ave.	"	"	138	"	---	175

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[a] In Asphalt II - 25 mils thick.

[b] Odd numbered panels were exposed in one machine; even numbered ones, in another.

FIGURE 1

DURABILITY OF ASPHALT PRODUCTS

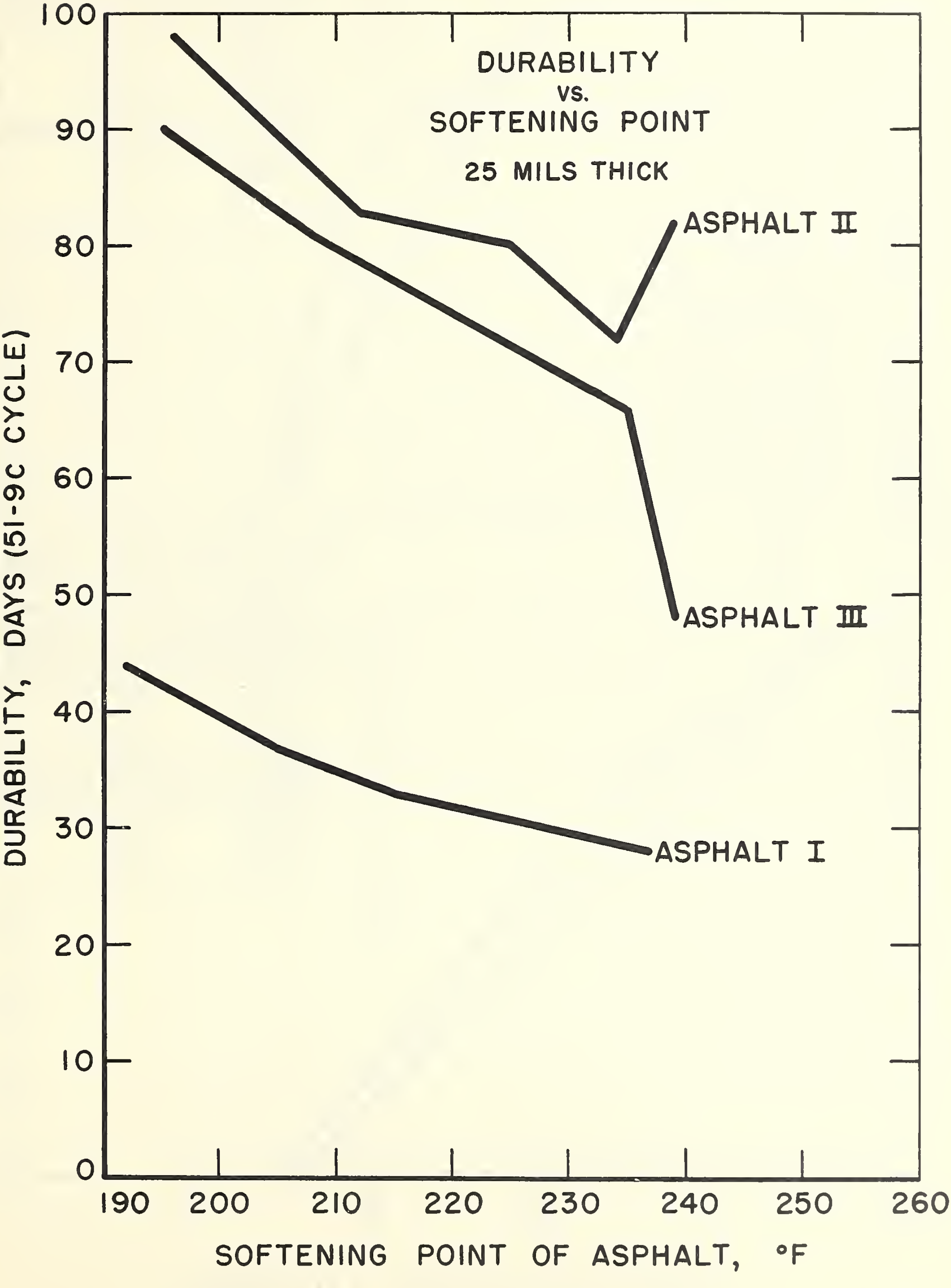
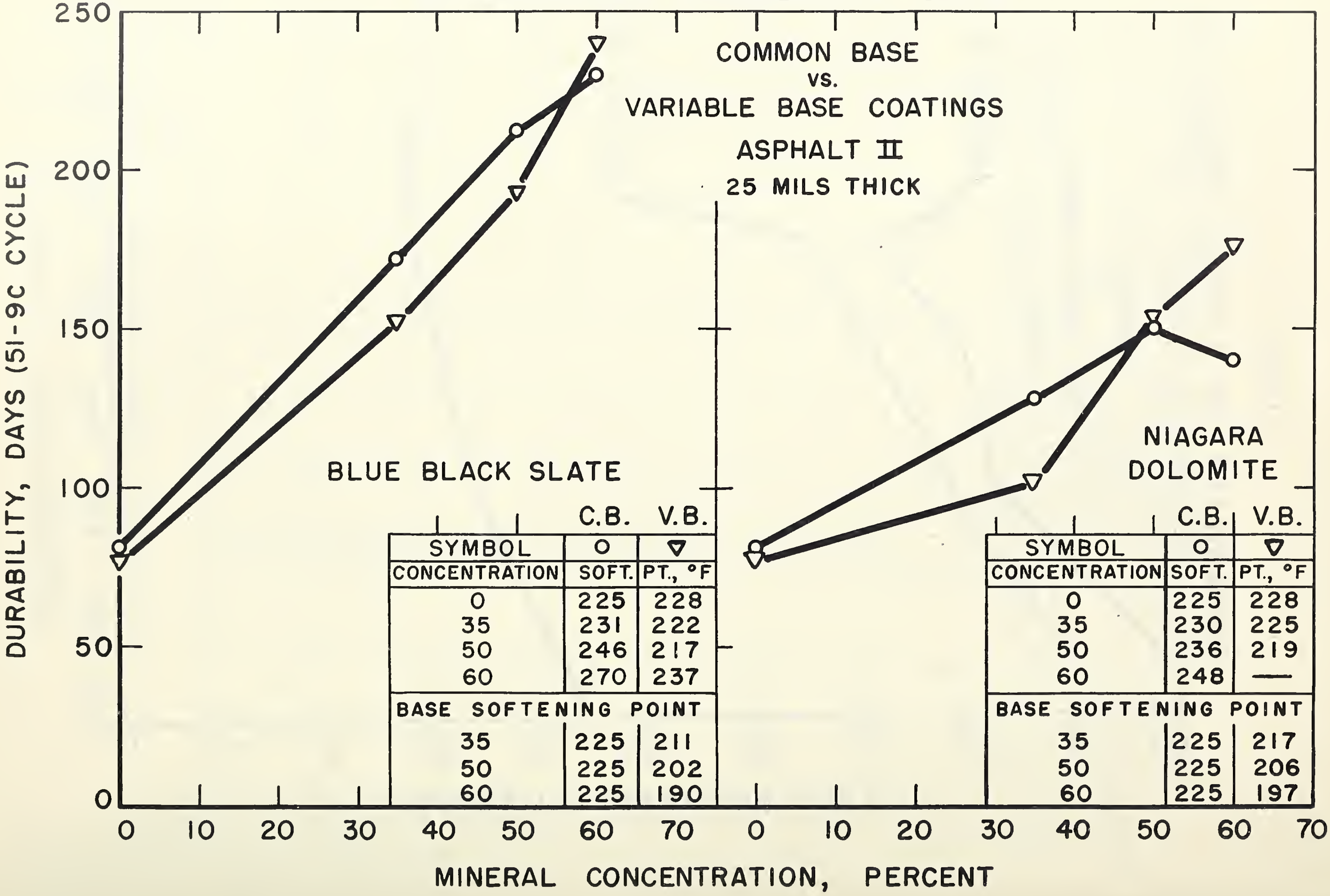


FIGURE 2

ACCELERATED DURABILITY DATA



COMMON BASE
vs.
VARIABLE BASE COATINGS

ASPHALT II

25 MILS THICK

BLUE BLACK SLATE

	C.B.	V.B.
SYMBOL	O	▽
CONCENTRATION	SOFT.	PT., °F
0	225	228
35	231	222
50	246	217
60	270	237
BASE SOFTENING POINT		
35	225	211
50	225	202
60	225	190

NIAGARA DOLOMITE

	C.B.	V.B.
SYMBOL	O	▽
CONCENTRATION	SOFT.	PT., °F
0	225	228
35	230	225
50	236	219
60	248	—
BASE SOFTENING POINT		
35	225	217
50	225	206
60	225	197

4.3 Selected Minerals - The Effect of Some Natural Variations of Clay, Fly Ash and Silica on the Durability of Coatings (All coatings tested in this series were 25 mils thick and contained 35% mineral matter.)

The three types of materials investigated in this series (described in Table IV) are representative of the wide variety of each type available. Despite the fact that the three clays tested varied from a disordered kaolinite (Florida Clay) to a mullite, there was little difference in their effects on the durability of any of the asphalts (Table V and Figure 3). Because of the flocculated nature of the Florida and Bradbury clays, the reported size distributions are probably not accurate. These clays were not dispersed readily in the asphalts. However, even in the Kyanite, which was mixed easily with the asphalts, there was no appreciable improvement in durability. In general, therefore, all three clays produced no decrease in the durability of asphalts II and III and only a small decrease in asphalt I.

Fly ash contains some components which are conductors of electricity; therefore, the high voltage probe could not be used to determine failures in coatings containing fly ash. Therefore, all end-point determinations on coatings containing fly ash were made by visual inspection.

Because fly ash is a by-product of the combustion of coal, its composition, both physical and chemical, varies with the source of the coal, the way it is burned, and the efficiency of the combustion. Therefore, its effect on

TABLE IV. PROPERTIES OF CLAYS, FLY ASHES, AND SILICAS

PROPERTY[a]	FLA. CLAY	KYA-NITE	BRAD-BURY CLAY	L.C. FLY ASH	H.C. FLY ASH	CHI. FLY ASH
Moisture, %	2.7	0.0	1.6	0.4	0.4	0.4
Loss on Ignition at 1000°F, %	11.8	0.1	4.4	4.9	22.8	2.1
" " " " 1800°F, %	13.3	0.1	5.7	7.3	25.9	4.2
Water Solubility, %	0.0	0.5	1.0	5.9	4.9	5.6
Free Alkali, %	0.0	0.0	0.0	0.0	0.1	0.6
Particle Size Distribution:						
% Passing U.S. #60	100.0	99.9	100.0	99.6	98.7	99.9
% Passing U.S. #80	99.9	99.0	99.7	98.9	97.4	99.7
% Passing U.S. #100	99.8	95.8	99.1	97.5	95.5	99.4
% Passing U.S. #120	99.8	91.9	98.8	96.7	93.9	99.1
% Passing U.S. #170	99.6	80.3	97.9	94.2	89.2	98.2
% Passing U.S. #200	99.3	72.4	97.3	92.3	85.4	97.4
% Passing U.S. #230	99.1	65.8	96.8	90.6	82.5	96.6
% Passing U.S. #325	98.9	58.9	95.0	84.8	72.9	94.0
Mixture with Asphalt:						
Ease of Mixing	Poor	Good	Fair	Good	Good	Good
Soft. Point Increase, °F	28	5	4	20	17	22

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[a] Method is described in "A Survey of Materials Available for Use as Mineral Stabilizers", A.R.I.B., Oct. 1952. (40% stabilizer in Asphalt III, S.P. = 223°F)

(TABLE CONTINUED ON NEXT PAGE)

TABLE IV. PROPERTIES OF CLAYS, FLY ASHES, AND SILICAS (CONTINUED)

PROPERTY[a]	LAKE ERIE SILICA	BANK SILICA	OTTAWA SILICA	SPECIAL SILICA
Moisture, %	0.2	0.1	0.0	0.6
Loss on Ignition at 1000°F, %	0.7	0.2	0.1	0.7
" " " " 1800°F, %	2.5	0.4	0.2	1.4
Water Solubility, %	0.0	0.2	0.1	3.0
Free Alkali, %	0.0	0.0	0.0	0.0
Particle Size Distribution:				
% Passing U.S. #60	99.9	100.0	100.0	98.2
% Passing U.S. #80	98.2	97.8	99.0	98.1
% Passing U.S. #100	80.8	85.8	96.8	98.0
% Passing U.S. #120	53.7	72.2	90.2	97.8
% Passing U.S. #170	15.3	42.7	65.0	94.3
% Passing U.S. #200	8.1	34.3	51.7	91.2
% Passing U.S. #230	5.4	30.8	43.0	87.9
% Passing U.S. #325	3.4	19.9	27.8	83.1
Mixture with Asphalt:				
Ease of Mixing	Good	Good	Good	Good
Soft. Point Increase, °F	5	7	7	20

[a] Method is described in "A Survey of Materials Available for Use as Mineral Stabilizers", A.R.I.B., Oct. 1952. (40% Stabilizer in Asphalt III, S.P. = 223°F)

TABLE V. DURABILITY DATA

SELECTED MINERALS TESTED AS STABILIZERS[a]

ASPHALT	STABILIZER						
	NONE	FLA. CLAY	KYA-NITE	BRAD-BURY CLAY	L.C. FLY ASH[b]	H.C. FLY ASH[b]	CHI. FLY ASH[b]
Asphalt I							
Panel No. 1, Dur., Days	52	33	39	31	51	60	47
" No. 7, " "	52	--	39	31	--	60	60
" No. 2, " "	34	42	48	33	40	61	62
" No. 6, " "	34	--	48	33	--	61	62
Average	43	38	43	32	46	60	58
Ratio[c]	1	0.88	1	0.74			
Crack Pattern	C	C	C	C	C	C	C
Softening Point, °F	219	219	219	219	223	219	220
Soft. Point Base, °F	219	199	214	210	205	205	205
Pen. at 25°C, Base	17	24	20	21	22	22	22
Impact, Inches	6.3	8.7	6.0	6.0	7.8	6.3	4.8
Asphalt II							
Panel No. 1, Dur., Days	74	80	73	86	111	132	114
" No. 7, " "	74	--	87	79	---	132	114
" No. 2, " "	76	91	122	103	122	136	144
" No. 6, " "	76	--	110	96	---	131	144
Average	75	86	98	91	116	133	129
Ratio[c]	1	1.15	1.31	1.21			
Crack Pattern	AC	E	A	EA	E	BEC	EB
Softening Point, °F	228	221	224	224	219	220	219
Soft. Point Base, °F	228	206	223	212	205	209	208
Pen. at 25°C, Base	17	23	18	20	23	25	26
Impact, Inches	8.3	8.3	6.3	6.8	7.3	6.3	4.0
Asphalt III							
Panel No. 1, Dur., Days	66	76	109	103	114	126	169
" No. 7, " "	69	--	116	103	---	153	169
" No. 2, " "	81	75	75	89	126	136	150
" No. 6, " "	81	--	82	89	---	133	136
Average	75	76	96	96	120	137	156
Ratio[c]	1	1.01	1.28	1.28			
Crack Pattern	F	E	A	A	E	EC	BCE
Softening Point, °F	227	221	224	222	224	222	220
Soft. Point Base, °F	227	205	215	206	205	204	204
Pen. at 25°C, Base	14	20	19	20	20	20	20
Impact, Inches	2.7	9.3	5.0	6.8	5.3	5.0	5.5

[a] 35% Mineral Matter - 25 mils thick.

[b] End point determined by visual inspection, therefore no ratio reported.

[c] Ratio = $\frac{\text{Durability of coating}}{\text{Durability of asphalt}}$

TABLE V. DURABILITY DATA (CONTINUED)

SELECTED MINERALS TESTED AS STABILIZERS[a]

ASPHALT	STABILIZER			
	LAKE ERIE SILICA	BANK SILICA	OTTAWA SILICA	SPECIAL SILICA
Asphalt I				
Panel No. 1, Dur., Days	33	31	40	47
" No. 7, " "	--	31	33	47
" No. 2, " "	41	40	49	43
" No. 6, " "	--	40	42	43
Average	37	35	41	45
Ratio	0.86	0.82	0.95	1.05
Crack Pattern	C	CB	B	C
Softening Point, °F	---	226	222	219
Soft. Point, Base, °F	213	221	214	205
Pen. at 25°C, Base	21	18	20	22
Impact, Inches	7.3	4	6	7.3
Asphalt II				
Panel No. 1, Dur., Days	91	65	97	127
" No. 7, " "	--	76	97	107
" No. 2, " "	117	89	123	129
" No. 6, " "	---	89	134	129
Average	104	80	113	123
Ratio	1.40	1.07	1.50	1.64
Crack Pattern	AC	A	A	EA
Soft. Point, °F	217	221	220	225
Soft. Pt., Base, °F	207	211	211	214
Pen. at 25°C, Base	23	21	21	20
Impact, Inches	11.7	5.8	6.0	9.0
Asphalt III				
Panel No. 1, Dur., Days	80	76	96	179
" No. 7, " "	--	83	103	172
" No. 2, " "	72	--	89	144
" No. 6, " "	--	78	89	124
Average	76	79	94	155
Ratio	1.01	1.05	1.25	2.07
Crack Pattern	A	A	A	AE
Soft. Point, °F	226	221	227	222
Soft. Point, Base, °F	206	217	219	205
Pen. at 25°C, Base	20	18	18	19
Impact, Inches	6.3	4.5	5.8	5.0

[a] 35% Mineral Matter - 25 mils thick. [b] End point determined by visual inspection.

the durability of asphalt roofing varies considerably. The three samples of fly ash tested varied from one produced under very efficient combustion conditions (2% loss on ignition) to one with almost 18% loss on ignition. All three materials were very finely divided and had appreciable amounts of water-soluble material present; only two had small amounts of free alkali. Because failure was determined visually on these coatings, their durability cannot be compared directly with the unstabilized asphalts or the other coatings in which failure was measured by means of a high voltage probe. (Where comparisons between the two methods for determining failure are possible, visible inspection always results in a somewhat longer coating life.) Therefore, only comparisons among the fly ash specimens can legitimately be made. The material designated as Low Carbon Fly Ash contributes the least to the durability of the coatings.

Two of the samples of silica tested were in their natural form while the other two (Ottawa and Special) had been processed through a grinding and sieving operation. Further, the Special Silica had been used to polish plate glass and in addition to the small particles of glass present, the corners and edges of the particles were rounded. With the exception of this material, which was appreciably soluble in water, the silicas were rather coarse and inert.

None of the silicas affected the durability of asphalt I; both of the processed silicas and Lake Erie Silica increased the durability of asphalt II; and only the Special Silica appreciably increased the durability of asphalt III. The efficacy of the Special Silica is probably due to its fine degree of subdivision, 82% being minus 44 microns (325-mesh sieve).

4.4 The Effect of the Degree of Mixing of Minerals with Asphalt on the Durability of Roof Coatings

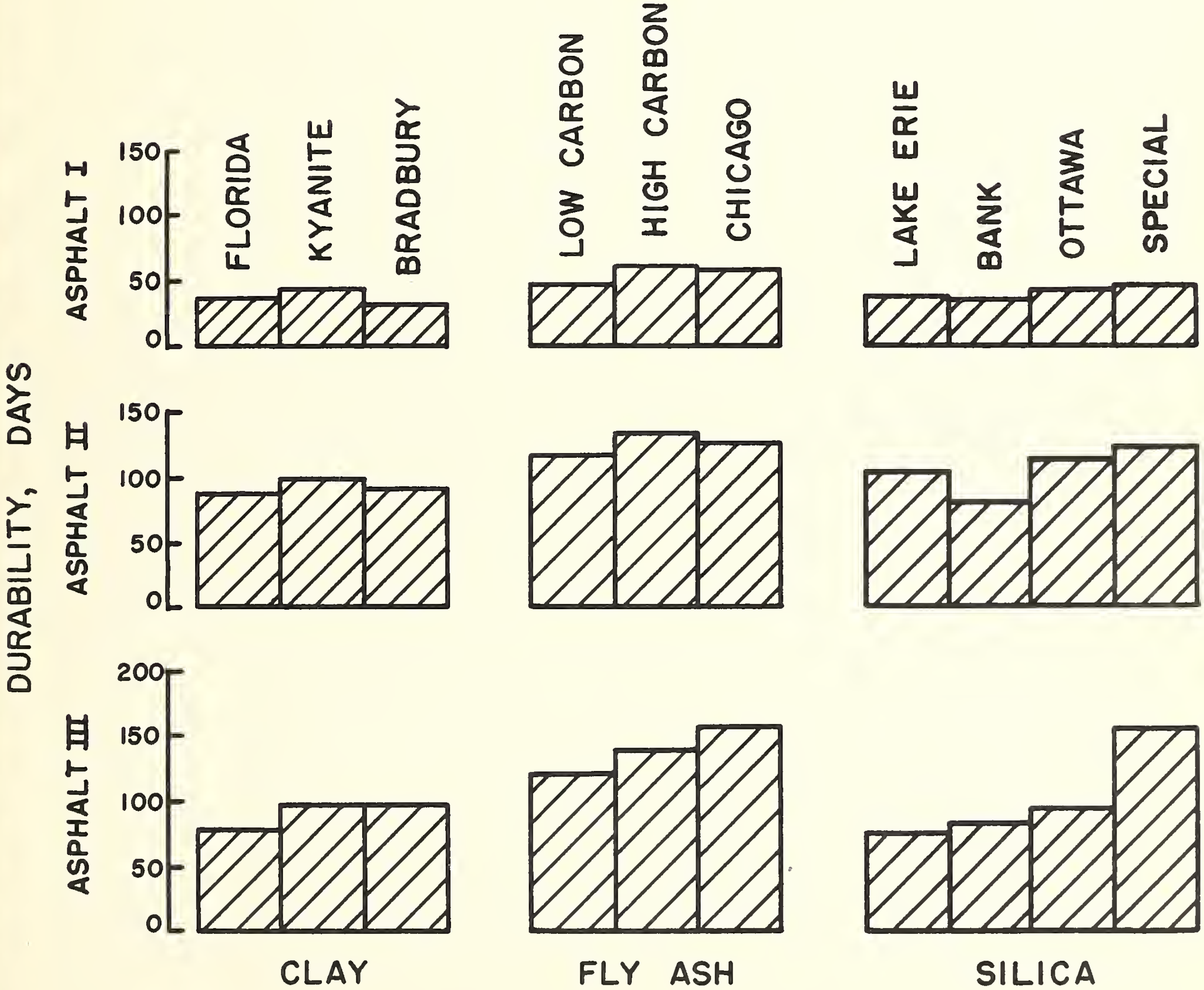
The effect of the thoroughness with which the mineral matter was mixed with the asphalt on the durability of the coating was investigated. Three mineral additives were used: 1) Blue black slate, representing a material which mixes readily and easily with asphalt; 2) Niagara

FIGURE 3

EFFECTS OF SELECTED MATERIALS ON DURABILITY

35 % MINERAL MATTER

25 MILS THICK



dolomite, representing a material which is incorporated with asphalt with moderate difficulty; and 3) Florida Clay, representing a material extremely difficult to mix with asphalt. These materials were mixed in concentrations of 35, 50, and 60% with asphalt III by three different procedures:

1) Prolonged Hand Mixing - The melt was stirred continually for about one hour until all bubbling and foaming ceased.

2) Continuous Mechanical Mixing - The mixture was stirred for 30 minutes with a high-speed laboratory stirrer after the mineral matter was added to the molten asphalt.

3) Brief Hand Mixing - Panels were made as soon as the mineral matter was folded into the asphalt. Only enough stirring was used to keep the mineral matter suspended.

The three minerals are described in Table I. Table VI and Figure 4 cover the results of the accelerated durability tests on these coatings. The data in Figures 4 and 5 are plotted on a ratio basis, i.e., the durability of the coating divided by the durability of the corresponding asphalt without mineral additives. In this way, it is possible to see at a glance the magnitude of the change in durability produced by the additive, and the effects of the additive are reduced to a comparable basis. It can be seen that with blue black slate, the mineral which mixes readily with asphalt, there was little difference in the durability of the panel made by the three procedures. The same was true of the coatings containing dolomite, except that there was a little more spread among the three procedures. However, with clay, the material that is difficult to mix with asphalt, the only procedure that yielded coatings as durable as the unstabilized asphalt was the prolonged hand mixing, i.e., thorough mixing. Despite the fact that the average durability was not appreciably changed by the degree of mixing in most cases, the data show that with both the 30-minute mechanical mixing and with the brief hand mixing, the spread in durability of replicate panels exposed in different machines was greater than with prolonged hand mixing. Therefore, it must be recommended strongly that thorough mixing be employed to obtain the most uniform and optimum benefits from a mineral additive.

TABLE VI. MIXING PROCEDURES IN ASPHALT III[a]

PROCEDURE	PROLONGED HAND MIXING				30-MIN. MECH. MIX.			BRIEF HAND MIX.		
PERCENT MINERAL	0	35	50	60	35	50	60	35	50	60
Blue Black Slate (Easy Mixing)										
Panel No. 1, Dur., Days	66	188	188	203	152	217	216	152	225	231
" " 7, " "	69	---	---	---	173	217	216	173	225	231
" " 2, " "	81	159	210	197	139	171	184	139	171	177
" " 6, " "	81	---	---	---	125	171	171	125	178	177
Average	75	174	199	199	147	194	197	147	200	202
Ratio	1	2.32	2.66	2.66	1.96	2.59	2.63	1.96	2.67	2.69
Crack Pattern	F	A	A	E	A	A	A	A	A	A
Softening Point, °F	227	218	221	256	225	226	249	224	221	253
Soft. Pt. of Base, °F	227	205	191	190	206	194	193	206	194	193
Pen. at 25°C of Base	14	20	22	22	19	20	20	19	20	20
Niagara Dolomite (Average Mixing)										
Panel No. 1, Dur., Days	66	122	115	118	149	95	76	121	98	138
" " 7, " "	69	---	---	---	149	95	76	121	95	165
" " 2, " "	81	108	135	137	115	126	128	102	126	128
" " 6, " "	81	---	---	---	102	126	122	114	126	128
Average	75	115	125	128	129	111	100	114	112	140
Ratio	1	1.53	1.67	1.71	1.72	1.48	1.33	1.52	1.49	1.87
Crack Pattern	F	A	E	E	A	A	AE	A	A	E
Softening Point, °F	227	221	222	224	219	226	226	217	225	225
Soft. Pt. of Base, °F	227	204	202	194	205	202	195	205	202	195
Pen. at 25°C of Base	14	20	21	22	19	19	20	19	19	20
Florida Clay (Difficult Mixing)										
Panel No. 1, Dur., Days	66	76	81	---	42	47	---	42	47	---
" " 7, " "	69	---	---	---	42	47	---	42	55	---
" " 2, " "	81	75	89	---	63	62	---	82	75	---
" " 6, " "	81	---	---	---	63	---	---	63	62	---
Average	75	76	85	---	52	52	---	57	60	---
Ratio	1	1.01	1.13	---	0.69	0.69	---	0.76	0.80	---
Crack Pattern	F	E	F	---	AEC	DE	---	AE	DE	---
Softening Point, °F	227	221	227	---	225	234	---	225	241	---
Soft. Pt. of Base, °F	227	205	190	---	206	193	---	206	193	---
Pen. at 25°C of Base	14	20	22	---	19	20	---	19	20	---

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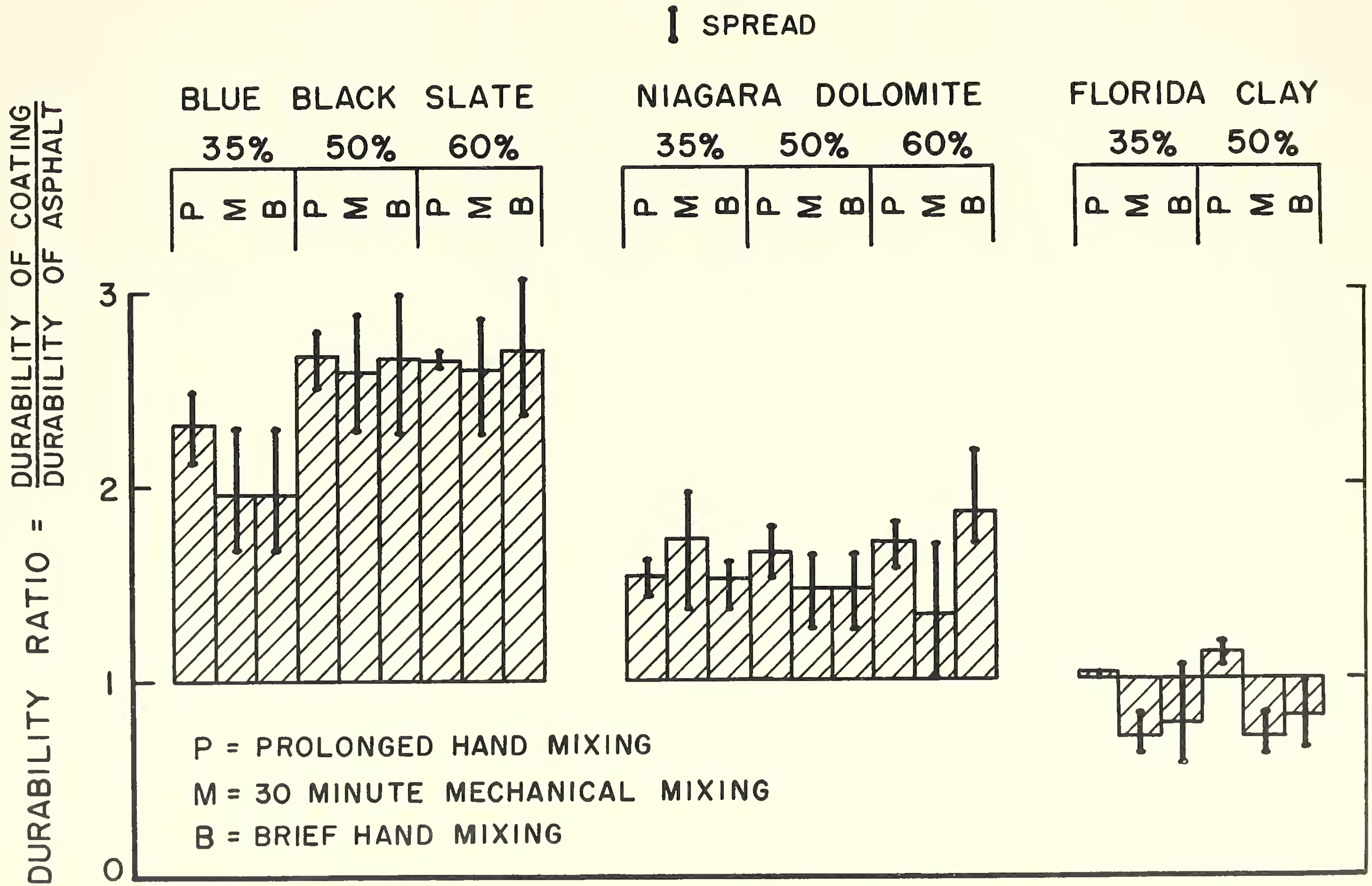
[a] All Coatings 25 mils thick.

FIGURE 4

EFFECTS OF MIXING ON DURABILITY

ASPHALT III - 25 MILS THICK

I = 75 DAYS



P = PROLONGED HAND MIXING

M = 30 MINUTE MECHANICAL MIXING

B = BRIEF HAND MIXING

4.5 The Effect of the Particle-Size Distribution of the Mineral Matter on the Durability of Coatings

Because the particle-size distribution is one of the attributes of mineral matter which may seriously affect its performance, its effects on durability were evaluated in all three asphalts. Three minerals, blue black slate, Niagara dolomite and Lake Erie silica, were sieved into eight fractions and reconstituted into five different synthetic size distributions, as follows:

1) +200 Mesh - Coarse material retained on a No. 200 U. S. Standard Sieve.

2) -200 Mesh - Material passing a No. 200 U. S. Standard Sieve.

3) -325 Mesh - Material passing a No. 325 U. S. Standard Sieve.

4) Uniform Size - Material passing a No. 140 but retained on a No. 230 U. S. Standard Sieve.

5) Equal Size - Approximately equal weights retained on a square-root of two series of U.S. Standard Sieves.

The geometry of irregular particles being separated by means of sieves with square openings introduced some deviations from the distributions desired.

Exposure panels (25 mils thick) were made from coatings containing 35 per cent of materials containing each of these distributions. The particle-size distributions are recorded along with the accelerated durability data in Table VII. Average durability figures are shown graphically in Figure 5.

TABLE VII. PARTICLE SIZE DISTRIBUTION SERIES*

ASPHALT	BLUE BLACK SLATE						
	None	Regular	+200	-200	-325	-140 +230	Equal Size
<u>Asphalt I</u>							
Panel No. 1, Dur., Days	52	61	58	58	75	53	52
" " 7, Dur., Days	52	--	58	58	75	59	52
" " 2, Dur., Days	34	75	65	72	80	55	54
" " 6, Dur., Days	34	--	65	72	80	42	54
Average	43	68	62	65	77	52	53
Durability Ratio	1	1.58	1.44	1.51	1.79	1.21	1.23
Crack Pattern	C	BG	B	G	G	BC	C
Softening Point, °F	219	222	226	220	222	227	230
Base Soft. Point, °F	219	210	220	207	207	212	214
Base Pen. at 77°F	18	21	18	21	21	20	20
<u>Asphalt II</u>							
Panel No. 1, Dur., Days	74	146	131	161	149	140	122
" " 7, Dur., Days	74	---	131	161	128	133	129
" " 2, Dur., Days	76	158	131	147	---	182	152
" " 6, Dur., Days	76	---	138	161	191	168	153
Average	75	152	133	157	156	158	143
Durability Ratio	1	2.01	1.77	2.09	2.08	2.11	1.91
Crack Pattern	AC	C	A	A	A	A	A
Softening Point, °F	228	222	225	227	224	225	220
Base Soft. Point, °F	228	211	217	215	211	217	215
Base Pen. at 77°F	17	22	19	19	22	19	19
<u>Asphalt III</u>							
Panel No. 1, Dur., Days	66	188	122	185	177	145	153
" " 7, Dur., Days	69	---	122	171	163	138	133
" " 2, Dur., Days	81	159	120	198	161	134	149
" " 6, Dur., Days	81	---	100	173	154	134	129
Average	75	174	116	182	164	137	141
Durability Ratio	1	2.32	1.56	2.43	2.19	1.83	1.88
Crack Pattern	F	A	A	AH	A	A	A
Softening Point, °F	227	218	226	221	227	223	223
Base Soft. Point, °F	227	205	221	205	207	205	209
Base Pen. at 77°F	14	20	17	20	20	20	20
% Passing U. S. 60		99.8	99.1	100.0		100.0	100.0
80		99.3	94.7	100.0		100.0	98.7
100		97.9	85.0	100.0		94.7	95.1
120		96.2	74.1	99.9		92.4	90.2
170		91.3	35.2	99.9		72.9	66.6
200		86.9	5.7	99.3		40.2	54.5
230		83.1	1.0	95.4		13.5	42.8
325		76.7	0.7	85.5	100.0	4.1	21.7

*35% Mineral Additive Exposed in Films 25 mils thick on Aluminum.

FIGURE 5

EFFECT OF PARTICLE SIZE DISTRIBUTION ON DURABILITY

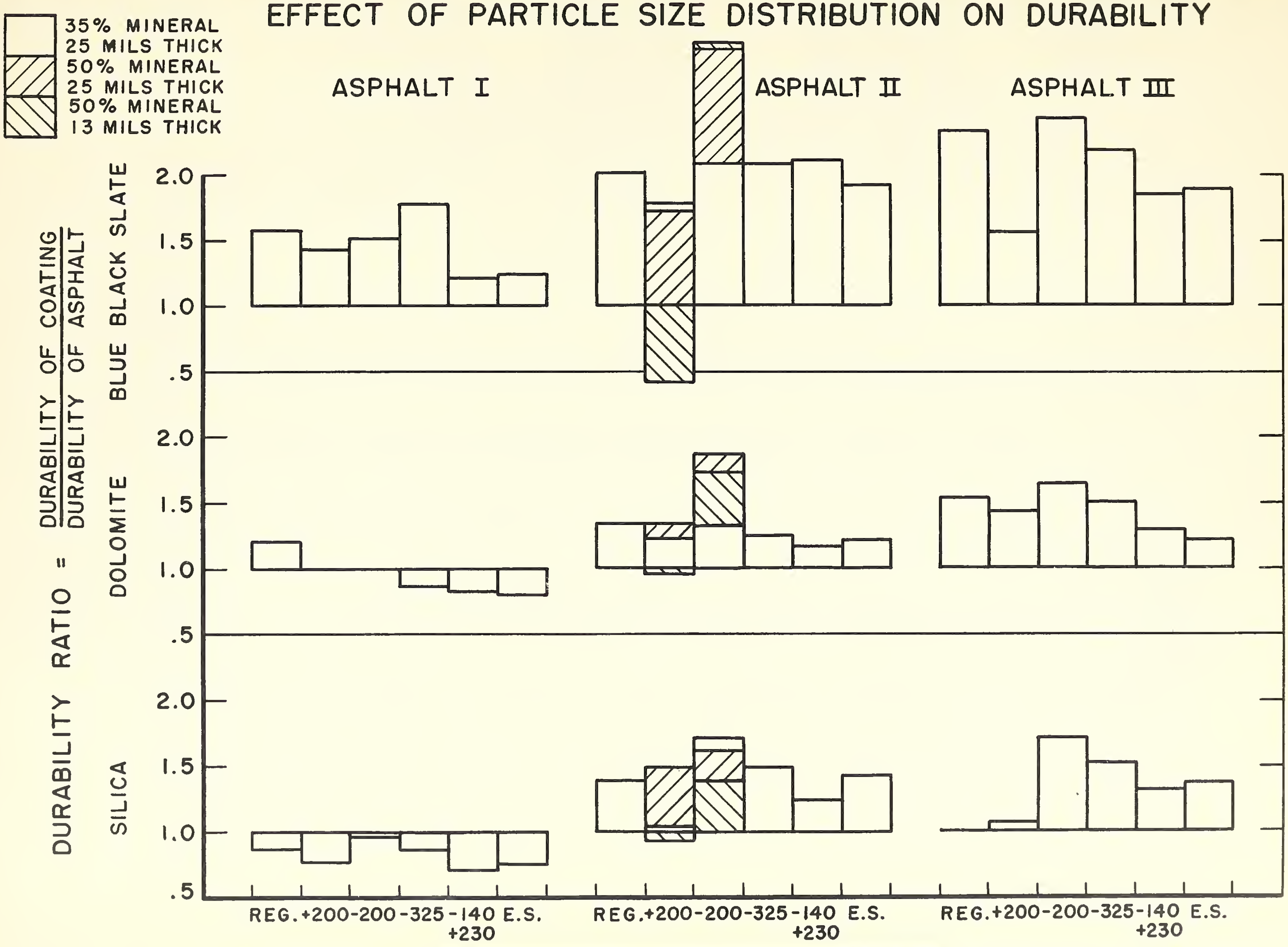


TABLE VII. PARTICLE SIZE DISTRIBUTION* (CONT'D)

ASPHALT	NIAGARA DOLOMITE					
	Regular	+200	-200	-325	-140 +230	Equal Size
Asphalt I						
Panel No. 1, Dur., Days	45	43	43	38	36	35
" " 7, " "	--	43	36	38	36	35
" " 2, " "	59	42	50	37	36	35
" " 6, " "	--	42	42	37	36	35
Average	52	43	43	37	36	35
Durability Ratio	1.21	1.00	1.00	0.86	0.84	0.81
Crack Pattern	C	B	C	BC	C	C
Softening Point, °F	220	226	221	218	223	224
Base Soft. Point, °F	209	219	211	207	212	214
Base Pen. at 77°F	21	18	20	21	20	20
Asphalt II						
Panel No. 1, Dur., Days	98	87	88	99	91	99
" " 7, " "	--	80	88	87	84	99
" " 2, " "	103	89	114	93	83	85
" " 6, " "	--	103	104	93	90	78
Average	101	90	99	93	87	91
Durability Ratio	1.35	1.20	1.32	1.24	1.16	1.21
Crack Pattern	A	AC	AE	A	A	A
Softening Point, °F	225	223	226	219	223	223
Base Soft. Point, °F	217	214	219	214	214	221
Base Pen. at 77°F	19	19	18	19	19	18
Asphalt III						
Panel No. 1, Dur., Days	122	112	134	109	95	87
" " 7, " "	--	112	134	102	95	87
" " 2, " "	108	108	128	116	98	110
" " 6, " "	--	94	110	116	98	76
Average	115	107	127	111	97	90
Durability Ratio	1.53	1.43	1.69	1.48	1.29	1.20
Crack Pattern	A	A	A	A	A	A
Softening Point, °F	221	226	220	223	222	223
Base Soft. Point, °F	204	219	215	206	210	214
Base Pen. at 77°F	20	18	19	20	20	19
% Passing U.S. 60	99.9	99.6	100.0			
80	99.9	98.6	100.0			100.0
100	99.6	95.8	100.0			97.9
120	99.3	91.1	100.0		100.0	94.5
170	96.6	61.4	99.9		93.0	79.8
200	93.4	20.5	99.9		74.1	70.0
230	89.9	5.9	97.9		41.2	50.0
325	81.0	0.7	87.0	100.0	9.5	19.1

*35% Mineral Additive Exposed in Films 25 Mils Thick on Aluminum.

TABLE VII. PARTICLE SIZE DISTRIBUTION SERIES*(CONT'D)

	LAKE ERIE SILICA					
ASPHALT	Regular	+200	-200	-325	-140 +230	Equal Size
Asphalt I						
Panel No. 1, Dur., Days	33	28	49	41	29	37
" " 7, " "	--	28	49	41	36	30
" " 2, " "	41	42	35	26	29	30
" " 6, " "	--	35	35	40	29	30
Average	37	33	42	37	31	32
Durability Ratio	0.86	0.77	0.98	0.86	0.72	0.75
Crack Pattern	C	BC	C	C	C	C
Softening Point, °F	---	222	226	227	223	225
Base Soft. Point, °F	213	212	210	208	213	213
Base Pen. at 77°F	20	20	21	21	20	20
Asphalt II						
Panel No. 1, Dur., Days	91	68	132	112	93	120
" " 7, " "	--	68	132	112	93	109
" " 2, " "	117	84	118	112	93	101
" " 6, " "	---	98	132	112	93	101
Average	104	80	128	112	93	107
Durability Ratio	1.39	1.07	1.71	1.49	1.24	1.43
Crack Pattern	AC	AC	A	A	A	A
Softening Point, °F	217	219	223	227	225	226
Base Soft. Point, °F	207	214	215	217	215	216
Base Pen. at 77°F	23	19	19	19	19	19
Asphalt III						
Panel No. 1, Dur., Days	80	93	122	108	95	100
" " 7, " "	--	93	122	108	87	100
" " 2, " "	72	81	143	123	105	103
" " 6, " "	--	81	136	123	105	103
Average	76	87	129	115	98	102
Durability Ratio	1.01	1.08	1.72	1.53	1.31	1.36
Crack Pattern	A	A	A	A	A	A
Softening Point, °F	226	222	223	224	226	219
Base Soft. Point, °F	206	215	210	207	216	210
Base Pen. at 77°F	20	19	20	20	19	20
% Passing U. S. 60	99.9	100.0	100.0			
80	98.2	99.3	100.0			100.0
100	80.8	92.7	99.9			97.9
120	53.7	76.9	99.9		100.0	93.1
170	15.3	26.5	99.7		43.7	71.2
200	8.1	7.7	98.2		18.8	56.5
230	5.4	1.8	85.7		7.3	46.7
325	3.4	1.1	52.5	100.0	1.2	28.2

*35% Mineral Additive Exposed in Films 25 Mils Thick on Aluminum.

The durability was a function of both the asphalt and the mineral additive. However, the results will be discussed primarily in terms of the minerals. While the finer distributions of blue black slate increased the durability more than the coarse ones in asphalts I and III, in asphalt II the durability was independent of the size distribution. The coatings made with silica, however, indicated that the finer distributions yielded the more durable coatings.

The indecisive nature of the above results led to the conclusion that the quantity of mineral matter present was insufficient for the effect of particle-size distributions to be observed in coatings 25 mils thick. Accordingly, the two extreme size ranges, +200 and -200, were formulated at a concentration of 50% in asphalt II and exposed in films 13 and 25 mils thick. The results are presented in Table VIII and Figure 5.

The effects of size distribution were clearly evident with 50% mineral matter in coatings 13 mils thick. For example, +200 blue black slate lasted only 42% as long as the asphalt alone, but the coating with the -200 blue black slate was three times as durable, making it produce over seven times the durability of the +200 material. At 25 mils, the same trend, though not so extreme, was maintained, the -200 mesh material producing a coating twice as durable as the +200 mesh. With dolomite, there was a factor of about two in both cases, but in the thin film it shifted the durability from below to above that of the asphalt alone. In the case of silica, however, the increase in durability produced by going to the finer size was small.

In summary, the -200 mesh fraction of blue black slate, Niagara dolomite, and Lake Erie silica seem to be most effective in increasing the durability of asphalt roof coatings in concentrations of 35 and 50 percent. The material retained on the 200 mesh sieve is the least effective, while the regular (commercial), -325, equal size, and uniform size (-140 +230) follow in descending order, with little difference between adjacent distributions.

TABLE VIII. PARTICLE-SIZE DISTRIBUTION SERIES
50% MINERAL MATTER IN ASPHALT II*

MINERAL	BLUE BLACK SLATE		NIAGARA DOLOMITE		LAKE ERIE SILICA	
Size Range	+200	-200	+200	-200	+200	-200
Softening Pt., °F	220	226	219	217	219	223
13 Mils Thick						
No. 1, Dur., Days	23	178	54	104	64	80
No. 7, Dur., Days	23	178	61	90	50	86
No. 2, Dur., Days	23	167	53	93	50	71
No. 6, Dur., Days	23	160	48	102	43	64
Ave., Dur., Days	23	171	54	97	52	75
Durability Ratio	0.42	3.10	0.98	1.76	0.94	1.36
Crack Pattern	A	A	A	A	A	A
25 Mils Thick						
No. 1, Dur., Days	120	225	110	144	134	129
No. 7, Dur., Days	135	230	110	139	120	129
No. 2, Dur., Days	145	223	102	137	98	113
No. 6, Dur., Days	120	233	102	143	98	113
Ave., Dur., Days	129	228	106	141	112	121
Durability Ratio	1.72	3.04	1.41	1.88	1.49	1.61
Crack Pattern	A	EG	A	E	A	A

*Durability of Asphalt II: 13 Mils - 55 Days
25 Mils - 75 Days

4.6 The Effect of Particle Shape of Minerals on the Durability of Coatings

Because particle shape was the one property of the minerals studied in the Principal Series[1] of exposures that seemed to be related to the efficacy of their stabilizing action, some quantitative method of expressing particle shape was sought. A relation between a bulk density and the true density of the minerals seemed worth considering for this purpose. Franz Pöpel,[2] in 1929, proposed a relation of this type, which he called "fineness factor", and defined as

$$f = \frac{\text{density - compacted wt.}}{\text{compacted wt.}} = \frac{d-b}{b}$$

where the compacted weight is an empirical bulk density determined under any set of conditions that would yield reproducible results.

Rather than a compacted weight, the bulk density used was obtained by fluffing the mineral matter by letting it flow slowly through a column of one-inch inside diameter, 21 inches long. Baffles set at an angle of 45° were placed 12 and 16 inches from the top of the column and the column was constricted to a diameter of 3/4 inch at the bottom. The column discharged into a 100-ml, weighed and calibrated, graduated cylinder from a point 1/4 inch above the cylinder. In practice the mineral matter was poured slowly into the column until it just overflowed the receiver. The excess was struck off level with the top of the receiver and the receiver and contents weighed. The weight of the mineral

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[1] "Laboratory Evaluation of Six Commercially Available Minerals as Stabilizers for Asphalt Coatings", S. H. Greenfeld.

[2] Franz Pöpel, Der Moderne Asphalstrassenbau (Strassenbau-Verlag Martin Boerner, Halle 1929).

matter divided by its volume is termed the "fluffed bulk density" or simply, the "fluff density". A correlation of fineness factor based on this bulk density is presented in Figure 6. The individual points are tabulated in Table IX.

It is readily seen that for each asphalt this factor correlated well with durability. The only materials that deviate considerably from the curves are oyster shell and mica, both being materials characterized by flat, platelike particles. Since the deviations are in the direction of greater durability than indicated by the lines, the lines may be taken as conservative indications of durability.

Despite the rather good correlations indicated by Figure 6, it must be emphasized that these data were obtained for specific materials under very specific test conditions and that the results may vary under other conditions. The fact that a different line is plotted for each asphalt emphasizes the importance of the asphalt itself in respect to durability.

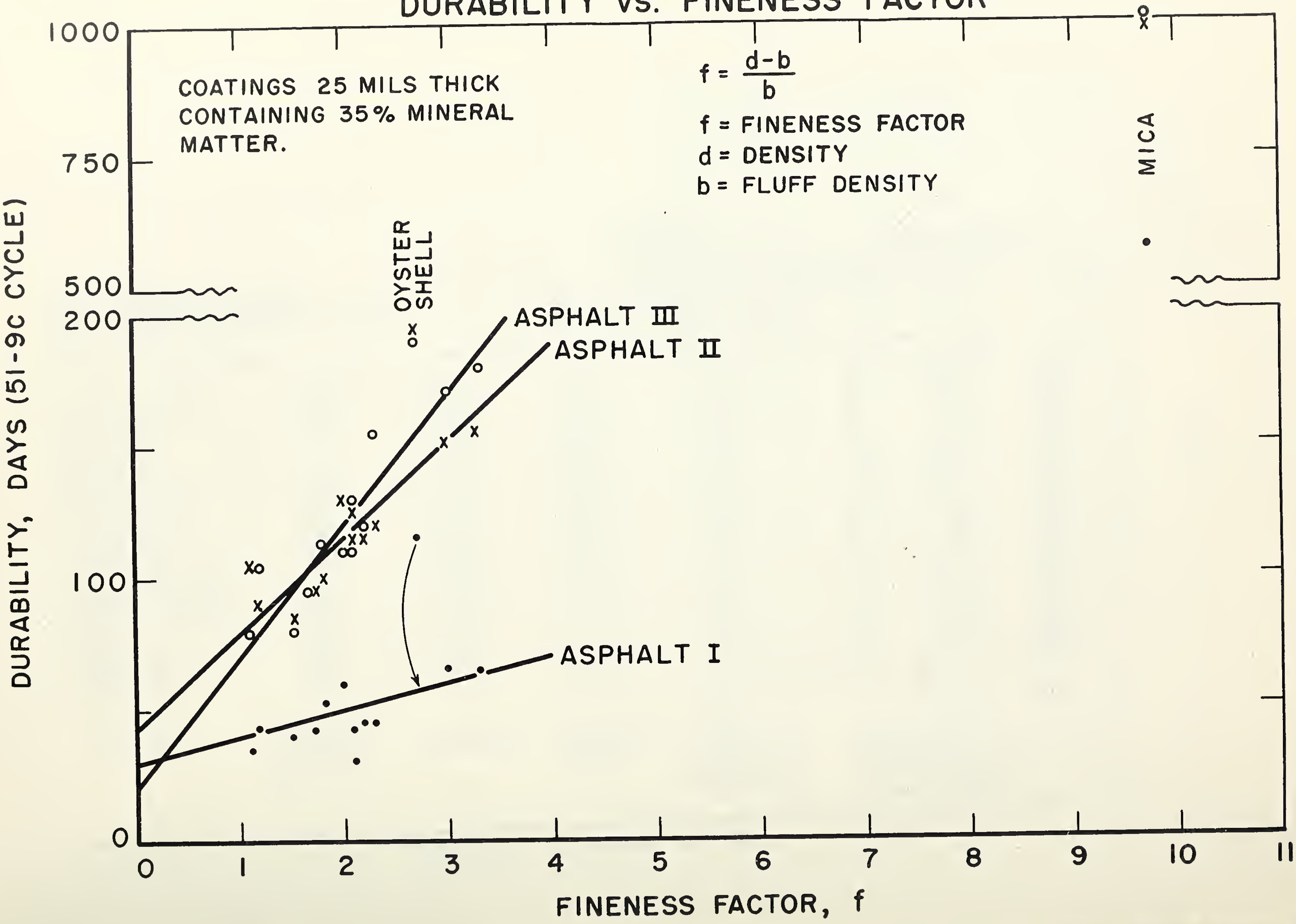
TABLE IX. FINENESS FACTOR

Mineral	Particle Shape (Visual)	Density d	Fluff Density b	Fineness Factor f $\frac{d-b}{b}$	Durability Days to F.F. 35% Mineral in Asphalts		
					I	II	III
Mica	FP	3.01	0.29	9.7	572	>1000	>1000
-200 BBS	FP	2.94	0.68	3.3	65	157	182
BBS	FP	2.94	0.73	3.0	68	152	174
Oyster Shell	AP	2.68	0.72	2.7	115	198	193
Spec. Silica	BR	2.58	0.78	2.3	45	123	155
L.C. Fly Ash	H	2.62	0.62	2.2	46	116	119
-200 Dol.	BS	2.87	0.94	2.1	43	99	127
-325 Dol.	BS	2.87	0.93	2.1	37	93	111
+200 BBS	FP	2.94	0.99	2.0	62	133	116
Niag. Dol.	BS	2.87	1.03	1.8	52	101	115
Kyanite	I	3.60	1.34	1.7	43	98	96
F. Clay	I	2.64	1.04	1.5	38	86	76
+200 Dol.	BS	2.87	1.29	1.2	43	90	107
L.E. Silica	BR	2.68	1.31	1.1	37	105	76

FP = Flat Plates
BR = Blocky, Rounded Corners
H = Heterogeneous

AP = Acicular Plates
BS = Blocky, Sharp Corners
I = Indefinite (Requires higher magnification than on light microscope)

FIGURE 6
DURABILITY VS. FINENESS FACTOR



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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 ($1.25) and its Supplement ($0.75), available from the Superintendent of Documents, 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.


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