

NATIONAL BUREAU OF STANDARDS REPORT

2809

**Laboratory Evaluation of Six Selected
Commercially Available Materials
as Stabilizers for Asphalt Roofing**

By

Sidney H. Greenfeld



**U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS**

U. S. DEPARTMENT OF COMMERCE

Sinclair Weeks, Secretary

NATIONAL BUREAU OF STANDARDS

A. V. Astin, Director



THE NATIONAL BUREAU OF STANDARDS

The scope of activities of the National Bureau of Standards is suggested in the following listing of the divisions and sections engaged in technical work. In general, each section is engaged in specialized research, development, and engineering in the field indicated by its title. A brief description of the activities, and of the resultant reports and publications, appears on the inside of the back cover of this report.

Electricity. Resistance Measurements. Inductance and Capacitance. Electrical Instruments. Magnetic Measurements. Applied Electricity. Electrochemistry.

Optics and Metrology. Photometry and Colorimetry. Optical Instruments. Photographic Technology. Length. Gage.

Heat and Power. Temperature Measurements. Thermodynamics. Cryogenics. Engines and Lubrication. Engine Fuels. Cryogenic Engineering.

Atomic and Radiation Physics. Spectroscopy. Radiometry. Mass Spectrometry. Solid State Physics. Electron Physics. Atomic Physics. Neutron Measurements. Infrared Spectroscopy. Nuclear Physics. Radioactivity. X-Rays. Betatron. Nucleonic Instrumentation. Radiological Equipment. Atomic Energy Commission Instruments Branch.

Chemistry. Organic Coatings. Surface Chemistry. Organic Chemistry. Analytical Chemistry. Inorganic Chemistry. Electrodeposition. Gas Chemistry. Physical Chemistry. Thermochemistry. Spectrochemistry. Pure Substances.

Mechanics. Sound. Mechanical Instruments. Aerodynamics. Engineering Mechanics. Hydraulics. Mass. Capacity, Density, and Fluid Meters.

Organic and Fibrous Materials. Rubber. Textiles. Paper. Leather. Testing and Specifications. Polymer Structure. Organic Plastics. Dental Research.

Metallurgy. Thermal Metallurgy. Chemical Metallurgy. Mechanical Metallurgy. Corrosion.

Mineral Products. Porcelain and Pottery. Glass. Refractories. Enameled Metals. Concreting Materials. Constitution and Microstructure. Chemistry of Mineral Products.

Building Technology. Structural Engineering. Fire Protection. Heating and Air Conditioning. Floor, Roof, and Wall Coverings. Codes and Specifications.

Applied Mathematics. Numerical Analysis. Computation. Statistical Engineering. Machine Development.

Electronics. Engineering Electronics. Electron Tubes. Electronic Computers. Electronic Instrumentation.

Radio Propagation. Upper Atmosphere Research. Ionospheric Research. Regular Propagation Services. Frequency Utilization Research. Tropospheric Propagation Research. High Frequency Standards. Microwave Standards.

Ordnance Development. These three divisions are engaged in a broad program of research and development in advanced ordnance. Activities include basic and applied research, engineering, pilot production, field testing, and evaluation of a wide variety of ordnance matériel. Special skills and facilities of other NBS divisions also contribute to this program. The activity is sponsored by the Department of Defense.

Missile Development. Missile research and development: engineering, dynamics, intelligence, instrumentation, evaluation. Combustion in jet engines. These activities are sponsored by the Department of Defense.

● Office of Basic Instrumentation

● Office of Weights and Measures.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

NBS REPORT

1004-10-1017

September 23, 1953

2809

Laboratory Evaluation of Six Selected Commercially Available Materials 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

Asphalt Roofing Industry Bureau



U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

The publication, re-
unless permission is
25, D. C. Such per-
cially prepared if t

Approved for public release by the
Director of the National Institute of
Standards and Technology (NIST)
on October 9, 2015.

In part, is prohibited
andards, Washington
port has been specifi-
eport for its own use.

LABORATORY EVALUATION OF SIX SELECTED COMMERCIALY
AVAILABLE MATERIALS AS STABILIZERS FOR ASPHALT ROOFING

	<u>Page</u>
ABSTRACT	1
1. INTRODUCTION	2
2. MATERIALS	3
2.1 Materials Tested as Stabilizers	3
2.2 Asphalts	4
3. EQUIPMENT	4
3.1 Aluminum Panels	4
3.2 Panel Making	4
3.3 Panel Exposure	4
3.4 Panel Inspection	4
4. PROCEDURE	8
4.1 Panel Preparation	8
4.2 Panel Exposure	9
4.3 Panel Inspection	10
5. RESULTS	14
5.1 Accelerated Durability Test Data	14
5.2 Physical Test Data	20
6. DISCUSSION OF RESULTS	20
6.1 Accelerated Durability Tests	20
6.2 Physical Tests on Coatings	31
7. CONCLUSIONS	35
8. APPENDIX A.	37
8.1 Volume Composition	38
8.2 Specific Gravity	38
8.3 Shatter Test	39
8.4 Water Absorption Tests	40
9. REFERENCES	48

TABLES

<u>TABLE NO.</u>		<u>PAGE</u>
I.	Physical and Chemical Data on Mineral Materials	6
II.	Properties of Asphalts	7
III.	Dominant Spectral Lines in Asphalt Ash	8
IV.	Failure Classification	11
V.	PRINCIPAL SERIES - Durability Data by Accelerated Test	15 - 17
VI.	Repeat Tests	18
VII.	Physical Tests of Unstabilized Asphalt Coatings Used as Controls	21
VIII.	Physical Tests of Coatings	23
IX.	Physical Tests of Coatings	25
X.	Physical Tests of Coatings	26
XI.	Effect of Mineral Matter on Softening Points and Shatter of Coatings	34
XII.	Specific Gravities	39
XIII.	Water Absorption Data	
	A. Water Absorbed in One Year	44
	B. Weight Increase in 20 Months	45
	C. Volume Increase in 20 Months	46

ILLUSTRATIONS

<u>FIGURE NO.</u>		<u>PAGE</u>
1.	Materials Tested as Stabilizers	5
2.	Crack Patterns "A" To "D"	12
3.	Crack Patterns "E" To "H"	13
4.	Durability Probability	19
5.	Accelerated Durability Data - Blue Black Slate	19
6.	Accelerated Durability Data - Florida Clay	19
7.	Accelerated Durability Data - Niagara Dolomite	19
8.	Accelerated Durability Data - Low Carbon Fly Ash	19
9.	Accelerated Durability Data - Lake Erie Silica	19
10.	Water Absorption Results	22
11.	Water Absorption Tests - Volume Increase vs. Weight Increase	24
12.	Particle Size Distributions - Sieve Range	47
13.	Particle Size Distributions - Sub-Sieve Range	47

LABORATORY EVALUATION OF SIX SELECTED COMMERCIALY
AVAILABLE MATERIALS AS STABILIZERS FOR ASPHALT ROOFING

by

Sidney H. Greenfield

ABSTRACT

The effect of six finely divided mineral materials in three concentrations (35, 50 and 60%) 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 mineral-asphalt system had to have its characteristics evaluated individually, the following generalizations are valid:

- (1) For every combination tested, the durability of the coating increased with film thickness.
- (2) Blue black slate and Tennessee mica increased the durability of all three asphalts at all concentrations and film thicknesses tested.
- (3) Niagara dolomite increased the durability of the coatings in many cases, but in others had no effect.
- (4) Low carbon fly ash increased the durability of the coatings in many cases, but did not affect it in others; however, because the coatings containing fly ash could be rated only by visual inspection, the durability reported may be high.
- (5) Lake Erie silica and Florida clay increased the durability only in some instances, primarily in the thicker films with asphalts II and III.
- (6) All the materials studied increased the softening point, viscosity, water absorption, and impact resistance of the coatings progressively with increasing concentration, but without a systematic relationship with durability.

(7) There is some indication that plate-like particles effect the greatest increase in durability.

1. INTRODUCTION

During the late 1920's and 1930's numerous investigators were examining means of conducting accelerated tests of the durabilities of bituminous materials. In 1928 Hickson and Walker [1] reported on the development of an accelerated weathering machine to produce the rapid deterioration by light and spray water of organic coating materials. In the following year Shelley [2] reported on the accelerated weathering characteristics of Oklahoma asphalts, and in 1930 Strieter [3] published the first of a series of articles on accelerated tests on roofing asphalts. Six years later two more papers by Strieter dealt with the relation between the chemical nature of asphalts and their weathering characteristics [4] and the effects of mineral additives on the durability of asphalts under accelerated test [5]. Snoke and Strieter [6] and Strieter [7] in 1937 recommended test procedures for asphalts. In February 1938, Research Paper No. 1073, by Dr. O. G. Strieter [8], was published as the final report on this early work on the effects of finely divided mineral matter on the durability of coatings used in the manufacture of asphalt roofing. 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 material came under consideration for blending with the asphalts to improve their durability. Improved methods of manufacturing roofing made possible higher concentrations of mineral stabilizer in coating asphalt. In order to take full advantage of these new materials and improved techniques, and to assist in the choice of materials for stabilizer use, the Research Committee of the Asphalt Roofing Industry Bureau, with representatives of the National Bureau of Standards, outlined a program to expand the study of many of the factors involved in the durability of coating asphalts and to find how these factors affect the suitability of the available materials.

This program was specifically designed to answer a number of questions which always arise when a material is considered for use as a stabilizer in prepared roofing and ultimately to provide a basis for a specification for the selection of stabilizers. The work covered in this report was planned to answer the following questions:

- (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?

Three concentrations of six materials in three different asphalts at three coating thicknesses were evaluated in accelerated durability machines. This series of exposures is designated as the Principal Series. A number of tests were also made on these materials, the individual asphalts and the blended coatings in order to integrate the accelerated durability data with more fundamental concepts.

Although the scope of the project embraces all of these questions, this report covers only the results of accelerated tests of some coatings on aluminum panels and of the physical tests made on these coatings and the materials that went into them. Later reports will deal with the results of aluminum- and felt-based specimens exposed outdoors and further accelerated durability tests of aluminum-based specimens.

2. MATERIALS

2.1 Materials Tested as Stabilizers

Six finely divided minerals were selected from the large number of those currently available to 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 available in convenient form. The properties and attributes of these six minerals are tabulated in Table I. Figure 1 contains photomicrographs of these materials; they are included to show the particle shape only and because each is at a different magnification, direct comparison for particle size cannot be made.

2.2 Asphalts

The asphalts subjected to test were obtained from three sources and are widely available for use in manufacturing 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 II.

Portions of Product 1 of each asphalt were ignited at 1800°F to determine the inorganic matter present. Spectrographic analyses of the ash are summarized in Table III.

3. EQUIPMENT

3.1 Aluminum Panels

Aluminum panels 2 3/4 x 6 x 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.

3.2 Panel Making

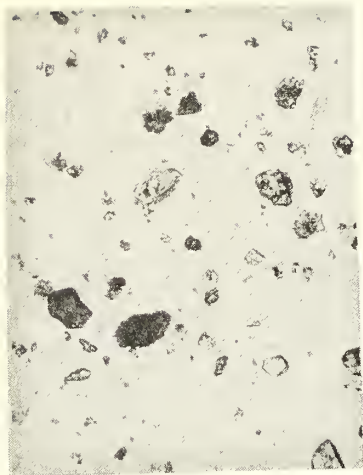
The exposed panels were made with the aid of a hydraulic press, as described elsewhere [10].

3.3 Panel Exposure

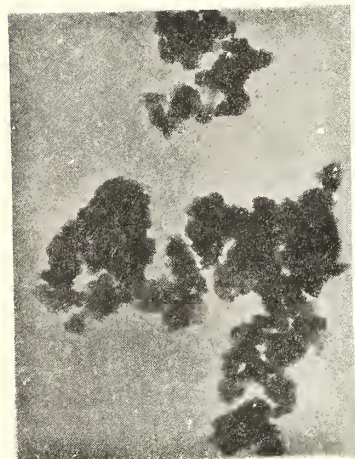
The panels were exposed in six accelerated durability machines constructed at the National Bureau of Standards and described in [11].

3.4 Panel Inspection

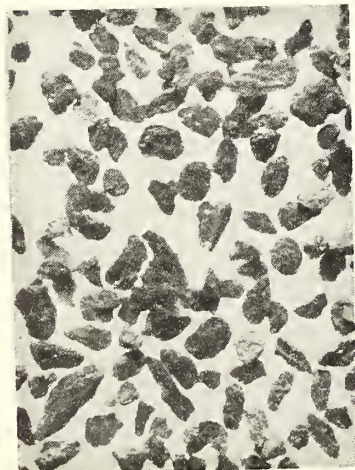
The panels were inspected weekly, both visually and with a high-voltage probe [11]. Representative types of failures were photographed with a Speed Graphic View Camera containing a 6-in. F6.8 Goerz Dagor lens.



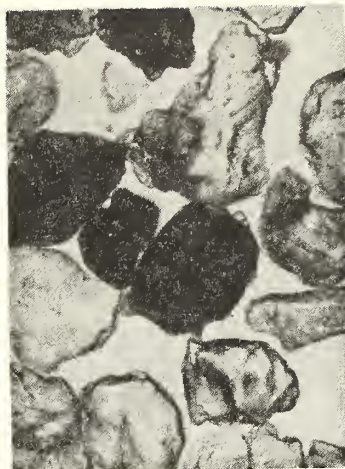
NIAGARA DOLOMITE
X 90



FLORIDA CLAY
X 13,500



BLUE BLACK SLATE
X 11



LAKE ERIE SILICA
X 80



TENNESSEE MICA
X 80



LOW CARBON FLY ASH
X 80



MATERIALS TESTED AS STABILIZERS
(FOR PARTICLE SHAPE ONLY)

FIG. 1

TABLE 1. PHYSICAL AND CHEMICAL DATA ON MINERAL MATERIALS

STABILIZER:	Blue Black Slate	Florida Clay ^{a/}	Niagara Dolomite	Low Carbon Fly Ash	Tennessee Mica	Lake Erie Silica
SOURCE:	#50 Quarry Delta, Pa.	Mine Edgar, Fla.	Quarry Joliet, Ill.	Philadelphia Electric Co.	#160 Pit Mine Johnson City, Tenn.	Dredged Lake Erie
PARTICLE SIZE, $\frac{1}{2}$ % FINER THAN						
Mils						
250	99.8	100.0	99.9	99.6	100.0	99.9
177	99.3	99.9	99.9	98.9	96.0	98.2
149	97.9	99.8	99.6	97.5	91.0	80.8
125	96.2	99.8	99.3	96.7	85.6	53.7
88	91.3	99.6	96.6	94.2	65.9	15.3
74	86.9	99.3	93.4	92.3	56.8	8.1
62	83.6	99.1	89.9	90.6	50.3	5.4
44	76.7	98.9	81.0	84.8	37.8	3.4
40	68	97	73	73	65	9
20	54	12	39	60	22	6
10	8	2	26	43	4	1
4	2	1	13	15	2	1
0.16	1	-	7	2	-	-
0.08	2.94	2.64	2.87	2.62	3.01	2.68
SPECIFIC GRAVITY ^{b/}	1.0	27.2	2.0	2.0	2.7	2.5
SURFACE AREA, $\frac{1}{2}$ m ² /g	29.5	63.9	19.4	30.0	97.2	19.5
OIL ABSORPTION, lb/100 lb	32.7	36.4	18.5	33.8	61.5	20.2
WATER ABSORPTION, lb/100 lb	2.1	11.8	1.8	4.9	0.9	0.7
LOSS ON IGNITION AT 1000°F, $\frac{1}{2}$ %	5.4	13.3	43.7	7.3	4.4	2.5
LOSS ON IGNITION AT 1800°F, $\frac{1}{2}$ %	0.2	2.7	0.1	0.4	0.2	0.2
MOISTURE, $\frac{1}{2}$ %	0.00	0.04	0.00	5.90	0.46	0.00
SOLUBILITY, $\frac{1}{2}$ %	0.0	0.0	0.00	0.00	0.0	0.0
FREE ALKALI, $\frac{1}{2}$ %			0.00	0.00		
CHEMICAL ANALYSIS, $\frac{1}{2}$ %						
SiO ₂	56	47	6	40	50	98+
R ₂ O ₃ (Al ₂ O ₃ + Fe ₂ O ₃)	32	38	1	48	35	
CaO + MgO			49	2.5	1	
K ₂ O + Na ₂ O	4				10	
SO ₃					0.5	
Carbon	2			7.6		
MIXTURE WITH ASPHALT ^{c/} - (40% MINERALS)						
EASE OF MIXING	GOOD	POOR	FAIR	FAIR TO GOOD	GOOD	GOOD
SOFTENING POINT INCREASE, °F	15	28	13	20	40	5

^{a/} Plasticity Index = 34. ASTM Method D424-39.^{b/} Sedimentation in Isopropyl alcohol.^{c/} Turns phenolphthale pink in aqueous solution.^{d/} Supplier's Analyses.^{e/} Isopropyl Alcohol Displacement.^{f/} See Report on a Survey of Materials Available for Use as Mineral Stabilizers for Method.^{g/} Low Temperature Nitrogen Adsorption - B.E.T. Method.^{h/} ASTM D281-31, using a mineral oil and water instead of the specified oil.

TABLE II. PROPERTIES OF ASPHALTS

ASPHALT	I			II			III		
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 F.S.			286 F.S.			595 F.S.	
Flash Point, °F (COC)		445			580			620	

^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 D5-52.

^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 III. DOMINANT SPECTRAL LINES
IN ASPHALT ASH

ELEMENTS											
Asphalt	% Ash	Al	Ca	Cu	Fe	Mg	Na	Ni	Pb	Ti	V
I	0.2%	S	S	W	VS	S	S	S	W	M	S
II	0.1%	S	S	W	VS	S	S	S	W	M	S
III	0.1%	S	S	W	VS	S	M	S	W	M	S
VS = Very Strong						M = Moderate					
S = Strong						W = Weak					

4. PROCEDURE

4.1 Panel Preparation

All coatings were blended from adjacent asphalt products (Table II) and the desired percentage of mineral matter to have softening points in the range of 217-227°F. In some instances 227°F was exceeded even though only the softest product was used, because that particular concentration of that material resulted in a very large softening point increase. Preliminary to preparing coating mixtures, the softening point increase for the minerals at their test concentrations were determined. These increases were used to estimate the base softening point required for a given mixture. Suitable proportions of the asphalt products straddling this value were blended to form the base.

This asphalt was melted, the mineral matter added, and the mixture stirred continually by hand 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, and viscosity measurements were made at about 10 degree intervals after thorough stirring. The mix was reheated to its working range for the preparation of exposure panels as described elsewhere [10].

Just prior to making the first panel, two specimens for softening point determination [11] 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. On random occasions ash determinations were made to assure uniform mineral distribution in the molten coating asphalt.

4.2 Panel Exposure

Thirteen aluminum-base and five felt-base panels were made from each of the 48 coatings. The thirteen aluminum-base panels comprised four each of three test thicknesses, i.e., 13, 25 and 43 mils. Of each group of four, two were subjected to accelerated durability tests and two were exposed outdoors. An additional panel, 25 mils thick, was retained as a reference specimen. The five felt-base panels comprised two of 13 and two of 25 mils thickness, all of which were exposed outdoors, and an additional 25-mil panel retained as a reference specimen. Felt-base, granule-surfaced specimens of 27 of these coatings were also made for outdoor exposure. The outdoor tests were set up to provide a basis for further correlation of the accelerated durability results with weather exposure performance [9]. Accelerated durability tests were conducted so that individual panels of each

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

duplicate pair were exposed in different accelerated durability machines in order to average possible machine differences. The 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. This cycle was selected for its effectiveness, based on a comparison of numerous combinations of light, water spray, and refrigeration, reported elsewhere [12]. This cycle provides the frequency and quantity of cold water needed for removal of the water-soluble weathering products, and also induces frequent thermal shocks, which make a low temperature refrigeration period unnecessary. It also has the advantage of yielding consistent results regardless of the portion of the cycle in which the inspection is made, providing the panels are dry when inspected.

A temperature control thermostat was embedded in an asphalt-coated panel and rotated with the test specimens. This thermostat operated forced air circulation to maintain the panel in which it was mounted at 140 - 145°F for about half of each cycle. During the 30 minutes in which the panels were not between 140 and 145°F , their temperatures increased from 50°F at the end of the spray period through about 20 minutes of warming up. The precise length of each temperature period varied with the number of panels in the machine and ambient conditions.

4.3 Panel Inspection

The exposure panels were examined weekly, both visually and with a high-voltage probe [13], and spark pictures were taken whenever there was any break in the coating. The patterns 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 spark patterns appeared on the photograph in a minimum of 30 of these squares, i.e., 50% of total test area, the panel was considered failed, removed from the machine, and filed away for future reference. Each panel was classified into one of eight types of crack patterns, as listed in Table IV, according to its appearance at the time of failure.^{2/} The spark pattern classifications are typified by a series of photographs of failed panels in Figures 2 and 3. For each type of crack pattern, a photograph of the coating is on the left and the corresponding spark picture of the crack pattern is on the right. The spark pictures are the larger of the two because they cover the entire aluminum base of the panel. The spark pictures, which are normally mirror images of the coatings, have been reversed to permit a direct comparison of the crack patterns on them with those on the photographs of the coatings.

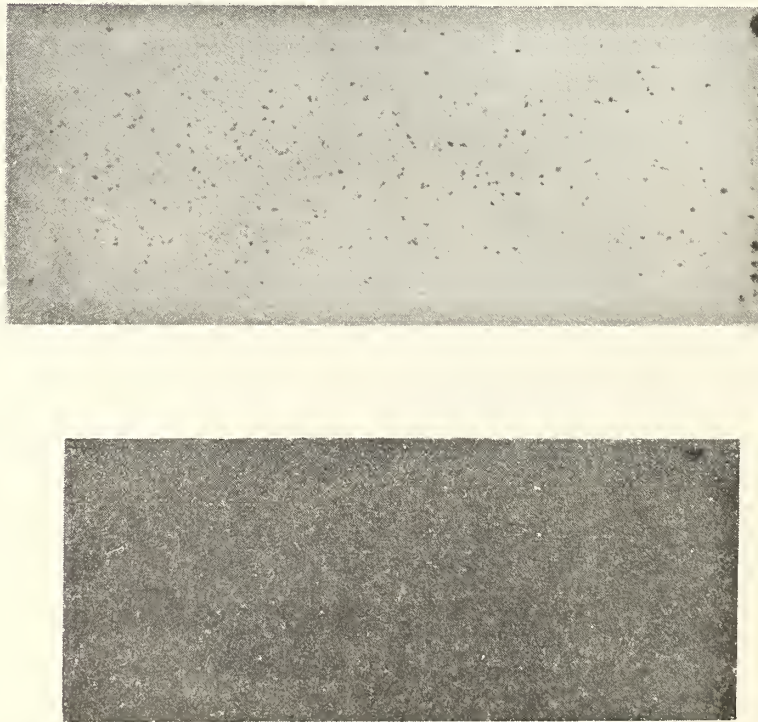
TABLE IV. FAILURE CLASSIFICATION

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.

^{2/}

Because coatings containing fly ash were conductive, only visual inspection was used on them.

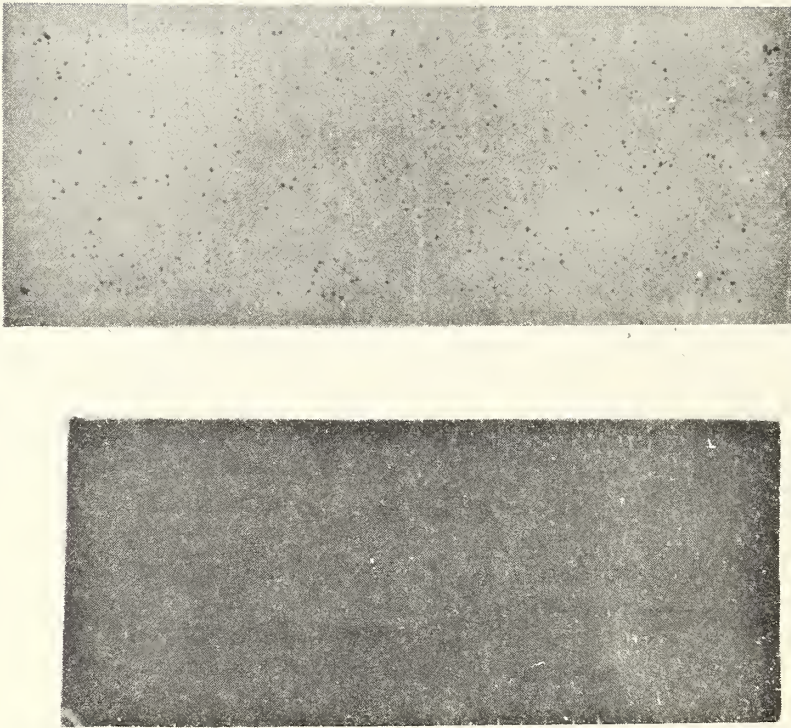
60 PERCENT LAKE ERIE SILICA IN ASPHALT III - 25 MILS



TYPE A

LITTLE OR NO VISIBLE CRACKING.

35 PERCENT LOW CARBON FLY ASH IN ASPHALT I - 13 MILS



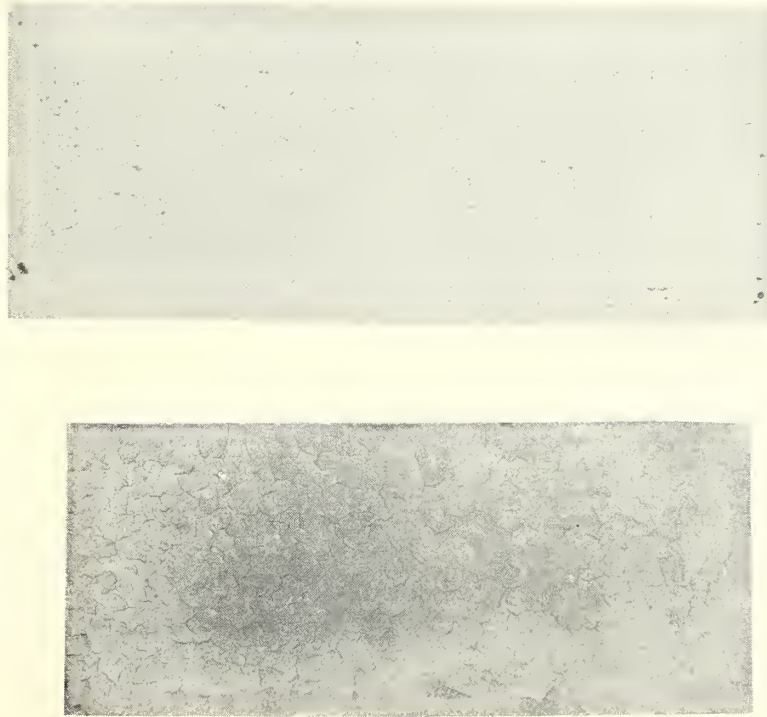
TYPE B

FINE MAP CRACKS, LESS THAN 3/16" BETWEEN INTERSECTIONS.



FIGURE 2 CRACK PATTERNS "A" AND "B"

35 PERCENT FLORIDA CLAY IN ASPHALT I - 25 MILS



TYPE C

FINE MAP CRACKS, 3/16" - 3/8" BETWEEN INTERSECTIONS.

35 PERCENT NIAGARA DOLOMITE IN ASPHALT I - 43 MILS



TYPE D

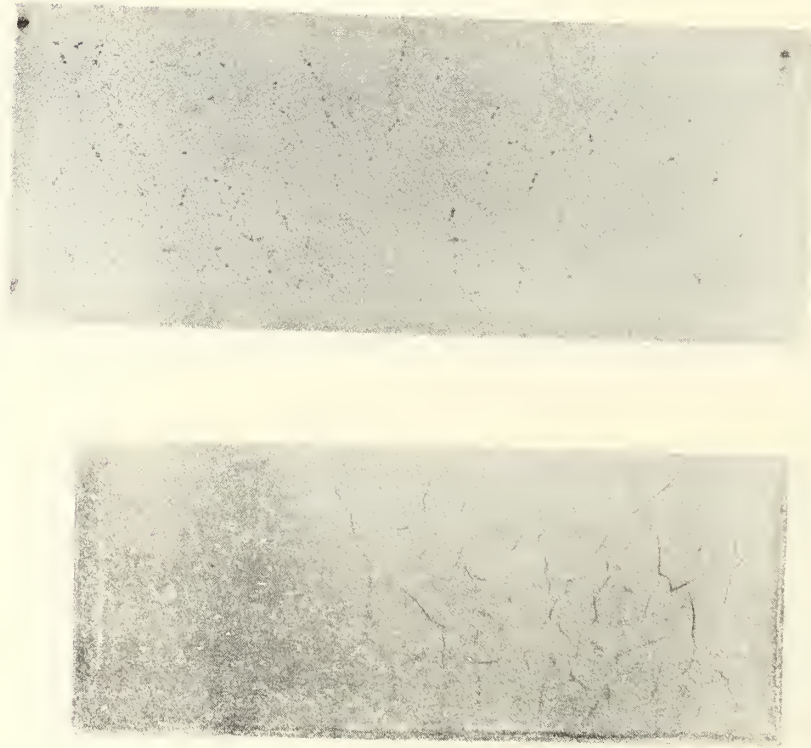
COARSE MAP CRACKS, 3/16" - 3/8" BETWEEN INTERSECTIONS.

FIGURE 2 CRACK PATTERNS



"C" AND "D"

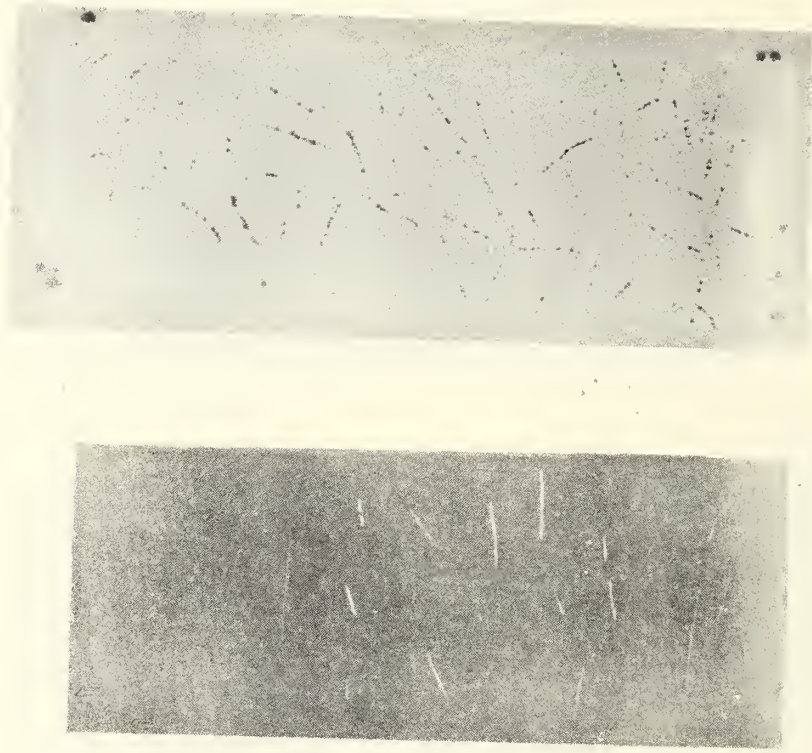
ASPHALT II - 43 MILS



TYPE E

MAP CRACKS WITH LARGE UNCRACKED AREAS (OVER 3/8").

ASPHALT III - 25 MILS



TYPE F

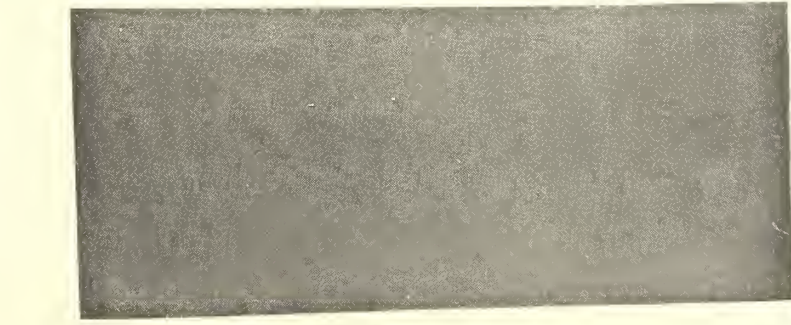
STRAIGHT LINE CRACKS.

FIGURE 3 CRACK PATTERNS "E" AND "F"



50 PERCENT BLUE BLACK SLATE IN ASPHALT I - 43 MILS

50 PERCENT NIAGARA DOLOMITTE IN ASPHALT II - 43 MILS



TYPE G

ORDERED CRACKS (CRACKS SEEM TO FORM DEFINITE PATTERNS, RATHER THAN BEING RANDOM).

TYPE H

LARGE PRINCIPAL CRACK WITH SMALLER TRIBUTARIES.

NBS

"G" AND "H"

FIGURE 3 CRACK PATTERNS

5. RESULTS

The data obtained in this investigation are: (1) accelerated durability machine test results, (2) results of physical tests on the coatings, and (3) correlations, where possible, between items (1) and (2).

5.1 Accelerated Durability Test Data

The results of the exposure of aluminum-base panels in the accelerated durability machines are given in Tables V and VI. The averages of the durability of duplicate exposures are reproduced on graphs in Figures 5 to 9. 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 4. (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 additional data for averaging. The results of these exposures are reported in Table VI, 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 the two machines. These results were used with the original exposures to figure the average durability of the coatings plotted in Figures 5 to 9.

TABLE V-A. PRINCIPAL SERIES
DURABILITY DATA BY ACCELERATED TEST

COATING THICKNESS, MILS:												
STABILIZER, %:												
13												
25												
43												
BLUE BLACK SLATE												
ASPHALT I												
1, Days _a /	33	54	40	66	52	61	81	115	72	118	117	171
2, Days _a /	32	52	33 _b /	45 _b /	51 _b /	75	81	115	72	124	---	170
Average, Days	33	53	37 _b /	55 _b /	51 _b /	68	81	115	72	121	---	170
Ratio	1	1.54	1.41	2.06	1	1.58	1.88	2.67	1	1.68	1.62	2.36
Crack Pattern	B	BA	AA	A	C	BG	G	G	D	DG	G	E
ASPHALT II												
1, Days _a /	74	90	66	140	91	145	169	207	96	186	202	221
2, Days _a /	61	102	64	157	96	158	215	270	103	192	236	251
Average, Days	68 _b /	96	65 _b /	148	94 _b /	152	192	238	99 _b /	189	219	236
Ratio	1	1.75	2.07	2.69	1	2.03	2.56	3.18	1	2.01	2.33	2.51
Crack Pattern	B	A	A	A	AC	C	GH	G	E	G	HG	H
ASPHALT III												
1, Days _a /	67	116	101	111	67	188	187	203	124	195	299	276
2, Days _a /	69 _b /	100	85 _b /	123	81 _b /	159	210	197	115	186	269	270
Average, Days	68 _b /	108	93 _b /	117	74 _b /	174	199	200	120 _b /	190	284	273
Ratio	1	1.80	1.78	1.95	1	2.32	2.65	2.66	1	1.61	2.41	2.31
Crack Pattern	C	A	A	A	F	A	A	E	F	G	G	G
FLORIDA CLAY												
Asphalt I												
1, Days _a /	33	20	25	---	52	33	38	---	72	54	59	---
2, Days _a /	32	21	26	---	51 _b /	42	40	---	72	70	82	---
Average, Days	33	21	26	---	51 _b /	38	39	---	72	62	71 _b /	---
Ratio	1	0.63	0.79	---	1	0.87	0.91	---	1	0.87	0.89	---
Crack Pattern	B	B	B	---	C	C	C	---	D	D	D	---
ASPHALT II												
1, Days _a /	74	55	47	---	91	80	79	---	96	94	113	---
2, Days _a /	61 _b /	64	56	---	96	91	90	---	103 _b /	112	117	---
Average, Days	68 _b /	60	51	---	94 _b /	86	85	---	99 _b /	103	115	---
Ratio	1	1.09	0.93	---	1	1.15	1.13	---	1	1.10	1.22	---
Crack Pattern	B	A	EA	---	AC	E	E	---	E	E	F	---
ASPHALT III												
1, Days _a /	67	42	35	---	67	76	81	---	124	144	170	---
2, Days _a /	69 _b /	30 _b /	36	---	81 _b /	75	89	---	115	124	156	---
Average, Days	68 _b /	30 _b /	36	---	74 _b /	76	85	---	120 _b /	134	163	---
Ratio	1	0.80	0.60	---	1	1.01	1.13	---	1	1.13	1.38	---
Crack Pattern	C	CE	AE	---	F	E	F	---	F	F	F	---

^a/ Panel No. and Days to Final Failure.

^b/ See "Repeat Tests", Table VI, for average used in calculating ratios.

TABLE V-B. PRINCIPAL SERIES
DURABILITY DATA BY ACCELERATED TEST

COATING THICKNESS, MILS: STABILIZER, %:	13			25			43					
	0	35	50	60	0	35	50	60	0	35	50	60
NIAGARA DOLOMITE												
ASPHALT I												
1, Days ^a	33	39	36	34	52	45	48	60	72	73	69	67
2, Days ^a	32	38	34	33	51 ^b	59	62	61	72	101	90	96 ^b
Average, Days	33	38	35	33	51 ^b	52	55	61	72	87 ^b	80	82 ^b
Ratio	1	1.18	1.07	1.02	1	1.20	1.28	1.41	1	1.29	1.11	1.29
Crack Pattern	B	AB	A	B	C	C	C	C	D	D	D	D
ASPHALT II												
1, Days ^a	74	77	79	92	91	98	142	163	96	147	157	176
2, Days ^a	61 ^b	76	91	104	96 ^b	103	161	187	103 ^b	166	185	201
Average, Days	68 ^b	76	85	98	94 ^b	101	152	175	99 ^b	156	173	188
Ratio	1	1.38	1.55	1.78	1	1.35	2.02	2.33	1	1.66	1.84	2.00
Crack Pattern	B	A	A	A	AC	A	E	EA	E	C	H	E
ASPHALT III												
1, Days ^a	67	64	63	92	67	122	115	118	124	227	226	206
2, Days ^a	69	74	101	69	81 ^b	108	135	137	115	174	173	194
Average, Days	68 ^b	69	82 ^b	80	74 ^b	115	125	128	120 ^b	200	200	200
Ratio	1	1.15	1.43	1.33	1	1.53	1.67	1.71	1	1.69	1.69	1.69
Crack Pattern	C	A	A	A	F	A	E	E	F	DE	CE	HE
LOW CARBON FLY ASH ^c												
ASPHALT I												
1, Days ^a	33	34	51	50	52	51	57	57	72	68	78	64
2, Days ^a	32	34	33	33	51 ^b	40	36	38	72	61	64	85
Average, Days	33	34	42	41	51 ^b	46	48 ^b	48 ^b	72	65	71	74
Ratio	1	1.15	1.28	1.27	1	1.06	1.01	0.95	1	0.90	0.99	1.03
Crack Pattern	B	B	B	B	C	C	C	C	D	D	D	D
ASPHALT II												
1, Days ^a	74	86	63	66	91	111	101	97	96	119	125	112
2, Days ^a	61	101	75	70	96 ^b	122	141	95	103	146	148	116
Average, Days	68 ^b	95	69	68	94 ^b	116	121 ^b	96	99 ^b	133	137	114
Ratio	1	1.73	1.26	1.24	1	1.55	1.60	1.28	1	1.41	1.46	1.21
Crack Pattern	B	B	B	C	AC	E	E	E	E	G	E	E
ASPHALT III												
1, Days ^a	67	103	82	76	67	114	102	97	124	149	148	97
2, Days ^a	69	101	107	82	81 ^b	126	125	103	115	154	166	128
Average, Days	68 ^b	102	94	79	74 ^b	119	113	100	120 ^b	151	157	112
Ratio	1	1.20	1.57	1.32	1	1.59	1.51	1.33	1	1.28	1.33	0.95
Crack Pattern	C	B	C	C	F	E	E	E	F	E	E	E

^a Panel No. and Days to Final Failure. ^b See "Repeat Tests", Table VI, for average used in calculating ratios.

^c Failure determined by visual inspection.

TABLE V-C. PRINCIPAL SERIES
DURABILITY DATA BY ACCELERATED TEST

COATING THICKNESS, MILS: STABILIZER, %:	13					25					43				
	0	35	50	60	75	0	35	50	60	75	0	35	50	60	75
TENNESSEE MICA															
ASPHALT I															
1, Days ^a /	33	188	---	---	---	52	614	---	---	---	72	---	---	---	---
2, Days ^a /	32	161	---	---	---	51	531	---	---	---	72	---	---	---	---
Average, Days	33	175	---	---	---	51b/	572	---	---	---	72	---	---	---	---
Ratio	1	5.35	---	---	---	1	13.3	---	---	---	1	---	---	---	---
Crack Pattern	B	A	---	---	---	C	A	---	---	---	D	---	---	---	---
ASPHALT II															
1, Days ^a /	74	278	---	---	---	91	---	---	---	---	96	---	---	---	---
2, Days ^a /	61	460	---	---	---	96b/	---	---	---	---	103b/	---	---	---	---
Average, Days	68b/	369	---	---	---	94b/	---	---	---	---	99b/	---	---	---	---
Ratio	1	6.71	---	---	---	1	---	---	---	---	1	---	---	---	---
Crack Pattern	B	A	---	---	---	AC	---	---	---	---	E	---	---	---	---
ASPHALT III															
1, Days ^a /	67	276	---	---	---	67	---	---	---	---	124	---	---	---	---
2, Days ^a /	69	526	---	---	---	81b/	---	---	---	---	115	---	---	---	---
Average, Days	68b/	402	---	---	---	74b/	---	---	---	---	120b/	---	---	---	---
Ratio	1	6.60	---	---	---	1	---	---	---	---	1	---	---	---	---
Crack Pattern	C	A	---	---	---	F	---	---	---	---	F	---	---	---	---
LAKE ERIE SILICA															
ASPHALT I															
1, Days ^a /	33	20	19	15	15	52	33	32	49	---	72	54	53	84	---
2, Days ^a /	32	20	26b/	16	16	51	41	33	44	---	72	62	68	79	---
Average, Days	33	20	22b/	15	15	51b/	37	32	46	---	72	58	56	82	---
Ratio	1	0.61	0.68	0.47	0.47	1	0.85	0.75	1.07	---	1	0.81	0.77	1.14	---
Crack Pattern	B	B	B	A	A	C	C	B	B	---	D	D	C	B	---
ASPHALT II															
1, Days ^a /	74	58	57	37	37	91	91	97	99	---	96	120	161	181	---
2, Days ^a /	61	62	61	41	41	96b/	118	117	118	---	103	145	171	193	---
Average, Days	68b/	60	59	39	39	94b/	105	107	108	---	99b/	133	166	187	---
Ratio	1	1.09	1.07	0.71	0.71	1	1.40	1.43	1.44	---	1	1.41	1.76	1.99	---
Crack Pattern	B	A	A	A	A	AC	AC	A	A	---	E	C	AC	A	---
ASPHALT III															
1, Days ^a /	67	39	35	32	32	67	80	82	124	---	124	141	164	201	---
2, Days ^a /	69	32	30	27	27	81	72	75	120	---	115	105	150	205	---
Average, Days	68b/	36	33	30	30	74b/	76	79	122	---	120b/	122	157	203	---
Ratio	1	0.60	0.55	0.50	0.50	1	1.01	1.05	1.63	---	1	1.03	1.33	1.72	---
Crack Pattern	C	A	A	A	A	F	A	A	A	---	F	AC	A	A	---

^a/ Panel No. and Days to Final Failure.

^b/ See "Repeat Tests", Table VI, for average used in calculating ratios.

TABLE VI. REPEAT TESTS

DESCRIPTION	DATE	SP ^o F	#1	#7	#2	#6	SUB AVERAGE	FINAL AVERAGE
ASPHALT I - 25 MILS	2/2/51	223	52	---	50	---	51	
	9/7/51	219	52	52	34	34	43	43
	10/25/51	---	41	41	35	42	40	
50% BBS IN I - 13 MILS	2/6/51	226	40	---	33	---	37	
	3/19/52	218	35	68	50	50	51	46
60% BBS IN I - 13 MILS	2/7/51	240	66	---	45	---	55	
	3/20/52	243	67	74	77	69	72	66
50% CLAY IN I - 43 MILS	3/1/51	226	59	---	82	---	71	
	3/24/52	244	63	51	65	65	61	64
35% DOLOMITE IN I - 43 MILS	2/15/51	220	73	---	101	---	87	
	3/25/52	217	115	115	79	72	95	93
60% DOLOMITE IN I - 43 MILS	2/21/51	224	67	---	96	---	82	
	3/3/52	221	109	109	80	94	98	93
50% FLY ASH ^a IN I - 25 MILS	2/13/51	219	57	---	39	---	48	
	3/20/52	220	41	41	42	42	41	44
60% FLY ASH ^a IN I - 25 MILS	2/14/51	256	57	---	39	---	48	
	3/24/52	250	37	37	38	38	37	41
50% SILICA IN I - 13 MILS	3/8/51	226	19	---	26	---	22	
	3/26/52	228	21	21	22	22	22	22
ASPHALT II - 13 MILS	12/21/50	224	74	---	61	---	68	
	3/13/52	228	47	47	49	49	48	55
ASPHALT II - 25 MILS	12/21/50	224	91	---	96	---	94	
	11/23/51	222	70	56	68	74	68	
	3/13/52	228	74	74	76	76	75	75
ASPHALT II - 43 MILS	12/21/50	224	96	---	103	---	99	
	3/13/52	228	92	92	91	91	92	94
50% BBS IN II - 13 MILS	1/8/51	217	66	---	64	---	65	
	3/19/52	219	149	183	106	114	138	114
50% FLY ASH ^a IN II - 25 MILS	1/11/51	225	101	---	141	---	121	
	3/31/52	221	125	125	102	128	120	120
ASPHALT III - 13 MILS	11/28/50	227	66	---	69	---	68	
	4/10/51	227	55	---	55	---	55	61
ASPHALT III - 25 MILS	11/28/50	227	66	---	81	---	74	
	4/10/51	227	69	---	81	---	75	75
ASPHALT III - 43 MILS	11/28/50	227	124	---	115	---	120	
	4/10/51	227	---	---	110	---	110	118
50% BBS IN III - 13 MILS	11/30/50	227	101	---	85	---	93	
	3/18/52	227	145	138	84	90	114	107
35% CLAY IN III - 13 MILS	3/19/51	221	42	---	30	---	36	
	3/25/52	221	61	61	49	42	53	48
50% DOLOMITE IN III - 13 MILS	12/14/50	222	63	---	101	---	82	
	3/26/52	216	109	94	76	76	89	86

^a/ VISUAL INSPECTION ONLY.

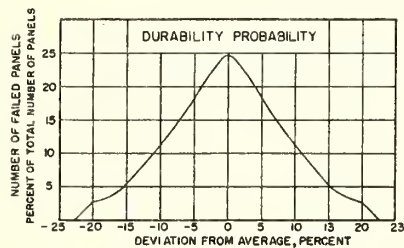


FIG. 4

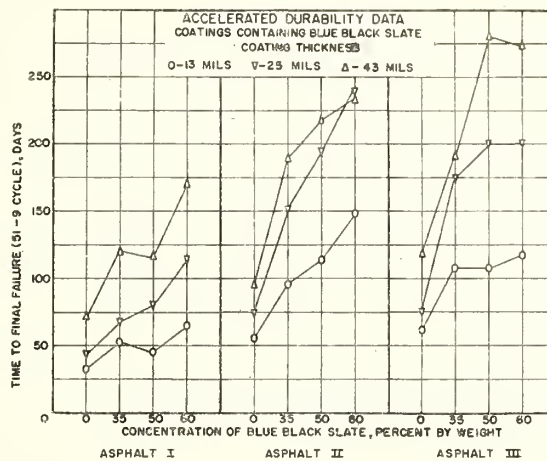


FIG. 5

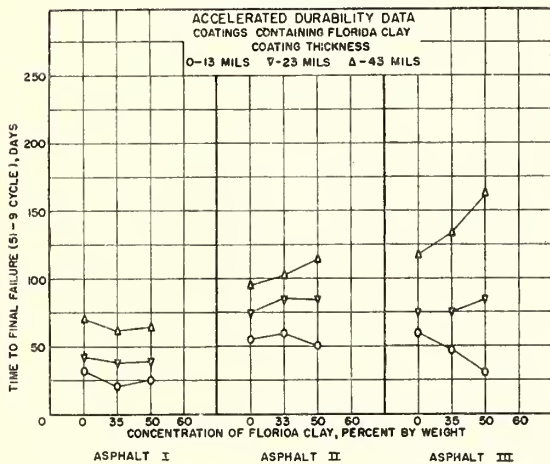


FIG. 6

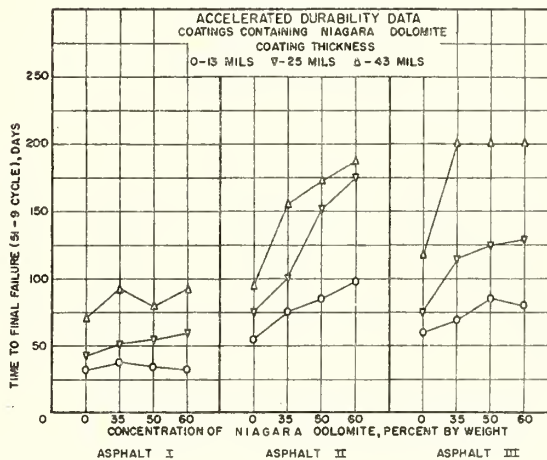


FIG. 7

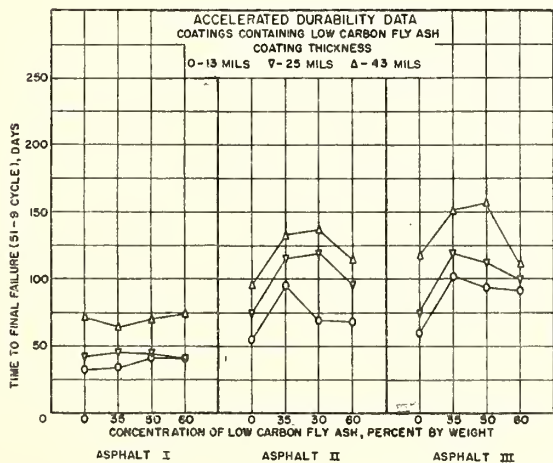


FIG. 8

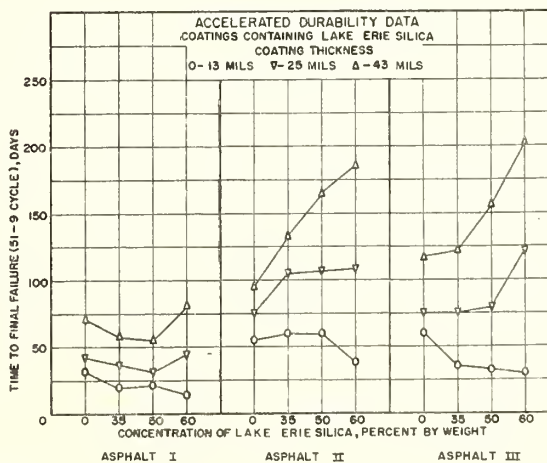


FIG. 9

5.2 Physical Test Data

(a) Measured Data

The results of the physical tests performed on the coatings are reported in Tables VII to X. Tabulations of the water absorption data at 28, 56, 280 and 609 days are in Tables VIII to X and compilation at 12 and 20 months are in Table XV, while representative curves of 35% of each mineral in each asphalt are in Figure 10.

The temperatures of the molten coatings during panel making are reported under "Temperature of Preparation".

(b) Calculated Results

Several calculated quantities appear in Tables VII to X. The methods of calculation are shown in the appendix.

6. DISCUSSION OF RESULTS

6.1 Accelerated Durability Tests

The data in Table VI show that even in the repeat exposures some duplicate panels failed widely apart, but except in six instances, the averages of the original exposures were 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 13 mils thick, as were most of the coatings in which large differences in durability were present in duplicate exposures. Because the top size of the mineral particles is of the same order of magnitude as this film thickness, particle dispersion and orientation are critical in determining the durability of these thin coatings, and may be a partial explanation for these few large discrepancies.

TABLE VII. PHYSICAL TESTS OF UNSTABILIZED ASPHALT COATINGS USED AS CONTROLS

ASPHALT:	I	II	III
TEST			
Softening Point ^{1/}	223	224	227
Penetration at 32°F ^{2/}	10	11	11
" at 77°F	17	17	14
" at 115°F	30	26	21
Penetration Index ^{2/}	4.7	4.7	4.5
Susceptibility ^{2/}	1.16	0.87	0.73
Loss on Heating ^{3/}	0.22	0.03	0.10
Penetration after Heating ^{2/}	17	17	14
Specific Gravity at 77°F	1.015	0.999	1.018
Viscosity, cp at 400°F ^{4/}	280	420	375
" " at 450°F	100	140	130
" " at 500°F	25	53	28
Water Absorption, g/sq.ft.			
at 28 days ^{5/}	0.67	0.43	0.34
at 56 days ^{5/}	1.00	0.70	0.52
at 280 days ^{5/}	3.00	2.20	1.61
at 609 days ^{5/}	4.62	2.97	2.29
Δ Vol., cc/sq.ft. at 609 days	4.11	1.63	0.70
Shatter ^{6/} , inches (1)	5	8	2.5
" " (2)	7	9	3.0
" " (3)	7	8	
" " (Ave.)	6.3	8.3	2.7

^{1/} ASTM Method D36-26.

^{2/} ASTM Method D5-52. P.I. from Nomograph, and S =

$$\frac{\text{Pen. 115°F} - \text{Pen. 32°F}}{\text{Pen. 77°F}} \quad [14]$$

^{3/} ASTM Method D6-39T.

^{4/} Brookfield Viscometer.

^{5/} Specimens, 3- x 3/16-inch, submerged 1/4-inch in distilled water at 70°F. See Appendix for method.

^{6/} A 1/2-lb. weight is dropped on a 3- x 3/16-inch specimen in a mixed ice and water bath. See Appendix for method.

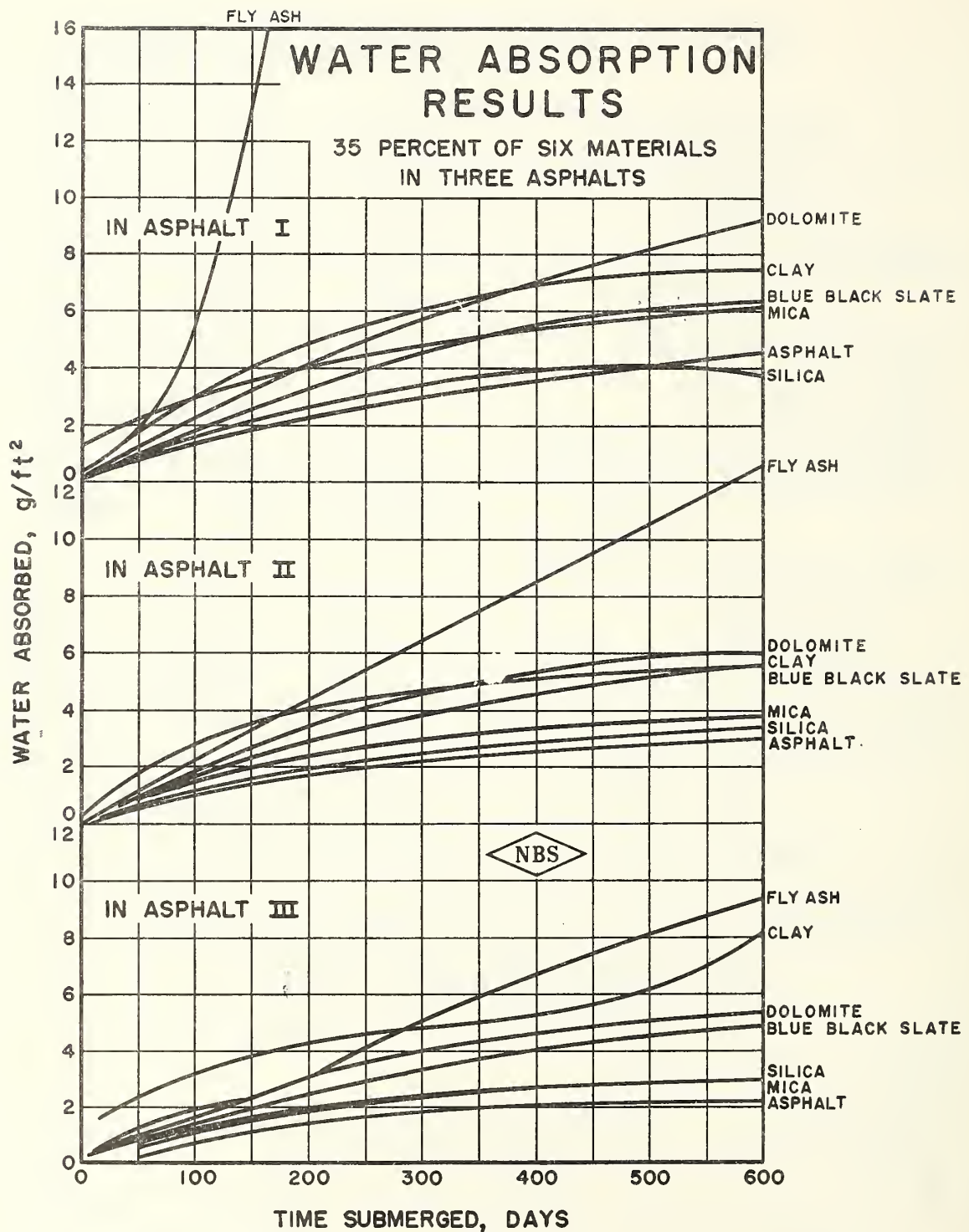


FIG. 10

TABLE VIII. PHYSICAL TESTS OF COATINGS

ASPHALT:	ASPHALT I				ASPHALT II				ASPHALT III			
STABILIZER, %	0	35	50	60	0	35	50	60	0	35	50	60
BLUE BLACK SLATE												
Volume Percent ^a / Density of Mixture ^a / Softening Point, °F	0 1.015	15.7 1.32	25.7 1.51	34.1 1.63	0 0.999	15.5 1.30	25.4 1.49	33.8 1.66	0 1.018	15.7 1.32	25.7 1.51	34.2 1.68
S.P. of Base, °F ^b	223	222	226	240	224	222	217	237	227	218	221	256
Pen. of Base, 10 ^b	223	210	202	189	224	211	202	190	227	205	191	190
Temp. of Preparation, °F	365-428	410-428	410-428	446-482	410-464	437-446	446-464	467-473	401-437	410-437	448-473	464-490
Viscosity, cp. at 400°F	280	540	1210	11,100	420	640	1480	13,500	375	580	1450	14,500
Viscosity, cp. at 450°F	100	180	580	3,900	140	290	600	4,150	130	224	535	6,200
Viscosity, cp. at 500°F	25	58	260	2,100	53	130	275	1,850	28	87	240	2,350
Water Absorption, g/sq.ft.												
at 28 days	0.67	0.77	0.92	0.92	0.43	0.70	0.91	0.99	0.34	0.53	0.66	0.70
at 56 days	1.00	1.18	1.47	1.55	0.70	1.10	1.36	1.59	0.52	0.84	1.08	1.19
at 280 days	3.00	4.20	6.51	7.11	2.20	3.67	4.68	6.51	1.61	3.16	4.45	5.55
at 609 days (20 mo.)	4.62	6.49	12.06	13.23	2.97	5.61	7.65	12.35	2.29	4.86	6.93	10.24
Volume Increase, cc/sq.ft.	4.11	7.07	12.37	13.72	1.63	6.28	6.10	14.34	0.70	5.34	7.68	11.69
Shatter ^c , Inches												
#1	5	12.0	-----	21	8	12	18.5	21	2.5	8	14.5	21
#2	7	12.0	18	21	9	12	18	21	3.0	10.5	15	21
#3	7	11.0	18.5	21	8	12	18	21	-----	9	17	21
Average	6.3	11.7	18.3	21.0	8.3	12.0	18.1	21	2.7	9.2	15.5	21
Air Content, ml/25 g.				10.01				10.01				
FLORIDA CLAY												
Volume Percent ^a / Density of Mixture ^a / Softening Point, °F	0 1.015	17.3 1.30	27.8 1.47	36.6 1.61	0 0.999	16.9 1.28	27.5 1.45	36.3 1.59	0 1.018	17.3 1.30	27.8 1.47	36.7 1.61
S.P. of Base, °F ^b	223	219d	226	-----	224	221	220	-----	227	221	227	-----
Pen. of Base, 10 ^b	223	199	189	-----	224	206	191	-----	227	205	190	-----
Temp. of Preparation, °F	365-428	410-428	437	-----	410-464	428-455	446-482	-----	14	20	22	-----
Viscosity, cp. at 400°F	280	590	3000	-----	420	830	3600	-----	401-437	437-464	446-482	-----
Viscosity, cp. at 450°F	100	225	1400	-----	140	340	1550	-----	375	990	5000	-----
Viscosity, cp. at 500°F	25	94	570	-----	53	135	700	-----	130	335	2100	-----
Water Absorption, g/sq.ft.												
at 28 days	0.67	1.32	1.96	2.04	0.43	1.42	2.18	-----	0.34	1.88	1.99	-----
at 56 days	1.00	1.99	3.03	3.32	0.70	2.02	3.17	-----	0.52	2.35	2.80	-----
at 280 days	3.00	5.90	7.80	10.00	2.20	4.50	7.60	-----	1.61	4.60	6.40	-----
at 609 days (20 mo.)	4.62	7.89	12.07	9.35	2.97	5.72	7.78	-----	2.29	9.81	10.57	-----
Volume Increase, cc/sq.ft.	4.11	5.83	10.44	9.86	1.63	5.27	10.12	-----	0.70	7.60	7.32	-----
Shatter ^c , Inches												
#1	5	8	10	-----	8	8	15.5	-----	2.5	10	7	-----
#2	7	11	9	12	9	8	18.5	-----	3.0	8	6.5	-----
#3	7	7	8	-----	8	9	13.5	-----	-----	10	5.5	-----
Average	5.7	8.7	9.0	12.0	8.3	8.3	15.7	-----	2.7	9.3	6.3	-----
a/ Calculated.	b/ Estimated.				c/ 3- X 3/16-in. disk.				d/ Rate slow.			

^a/Calculated. ^b/Estimated. ^c/3- X 3/16-in. disk. ^d/Rate slow.

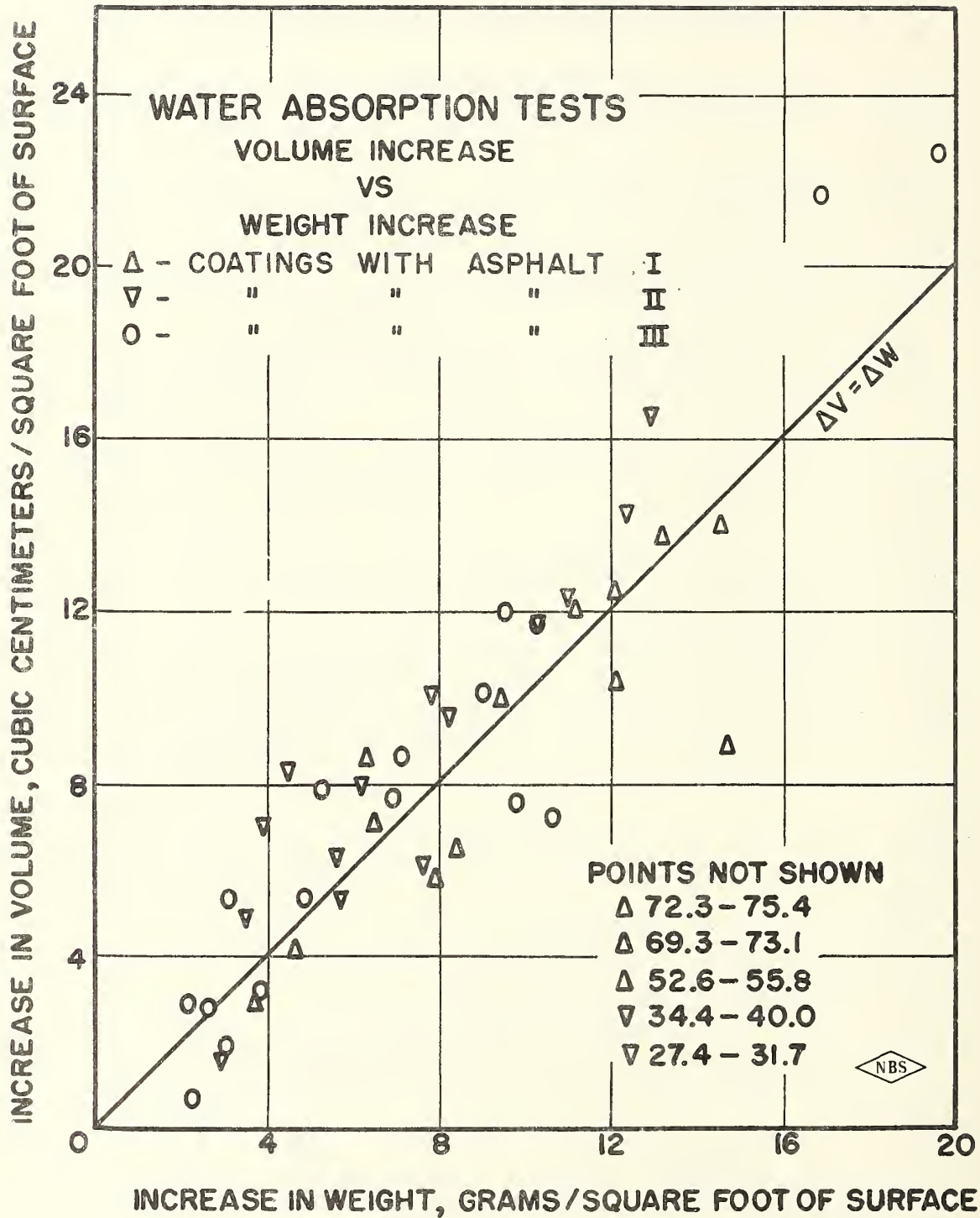


FIGURE II

TABLE IX. PHYSICAL TESTS OF COATINGS

ASPHALT: STABILIZER, %:	ASPHALT I				ASPHALT II				ASPHALT III			
	0	35	50	60	0	35	50	60	0	35	50	60
NIAGARA DOLOMITE												
Volume Percent ^a / Density of Mixture ^a / Softening Point, °F	0	16.0	26.1	34.7	0	15.8	25.8	34.3	0	16.0	26.1	34.7
S.P. of Base, °F ^b	1.015	1.31	1.50	1.66	0.999	1.29	1.48	1.64	1.018	1.31	1.50	1.66
Pen. of Base at 77°F	223	220	224	226	224	225	219	197	227	221	222	224
Temp. of Preparation, °F	223	209	205	198	224	217	206	197	227	204	202	194
Viscosity, cp. at 400°F	17	22	22	25	17	20	23	25	14	20	21	22
Viscosity, cp. at 450°F	365-428	428-437	419-455	455-464	410-464	428-464	395-410	401-419	401-437	437-464	446-482	436-455
Viscosity, cp. at 500°F	280	500	780	1380	420	690	880	1380	375	590	900	1280
Water Absorption, g/sq.ft.	100	175	295	485	140	240	360	550	130	222	275	450
at 28 days	25	90	125	225	53	100	180	260	28	80	140	230
at 56 days	0.67	0.88	1.07	1.30	0.43	0.86	1.15	1.41	0.34	0.91	1.15	1.26
at 280 days	1.08	1.50	1.82	2.15	0.70	1.32	1.75	2.19	0.52	1.32	1.74	1.95
Volume Increase, cc/sq.ft.	3.00	5.52	6.84	8.65	2.20	4.36	5.63	6.91	1.61	3.75	4.89	6.21
Shatter ^c , Inches	4.11	10.03	11.14	14.68	2.97	6.19	8.24	10.32	2.29	5.34	7.16	9.03
Average	5	5	5.5	16	8	17	16	14	2.5	7	10	13
Air Content, ml/25 g.	7	8	6.5	12	8	15	15	20	3.0	8	7	11
LOW CARBON FLY ASH	7	6	5.5	14	8	15	15.5	21	---	7	8	12
Volume Percent ^a / Density of Mixture ^a / Softening Point, °F	0	17.2	27.9	36.8	0	17.0	27.6	36.4	0	17.3	28.0	36.8
S.P. of Base, °F ^b	1.015	1.29	1.46	1.60	0.999	1.27	1.44	1.58	1.018	1.29	1.46	1.60
Pen. of Base at 77°F	223	223	219	256	224	219	225	273	227	224	224	266
Temp. of Preparation, °F	223	205	189	189	224	205	197	190	227	205	191	190
Viscosity, cp. at 400°F	17	22	28	28	17	23	25	27	14	20	22	22
Viscosity, cp. at 450°F	365-428	410-473	437-455	464-482	410-464	435-464	437-455	428-437	401-437	419-446	457-475	509-527
Viscosity, cp. at 500°F	280	490	1500	18,500	420	560	---	---	375	820	2200	33,000
Water Absorption, g/sq.ft.	100	205	590	7,750	140	200	---	---	130	320	860	14,500
at 28 days	25	96	255	4,300	53	120	---	---	28	94	365	5,700
at 56 days	0.67	0.95	1.12	1.14	0.43	0.88	1.08	1.23	0.34	0.70	0.93	1.08
at 280 days	1.00	1.97	2.20	2.61	0.70	1.35	1.70	1.89	0.52	1.09	1.34	1.41
Volume Increase, cc/sq.ft.	3.00	41.00	45.50	35.80	2.20	5.85	10.39	16.21	2.29	4.79	8.73	9.37
Shatter ^c , Inches	4.62	69.30	72.26	52.55	2.97	12.90	27.38	34.44	2.29	9.56	16.87	19.60
Average	4.11	73.10	75.40	55.80	1.63	16.61	31.68	39.97	0.70	12.00	21.64	22.70
at 28 days	5	7.5	---	12	8	7	14	21	2.5	6.5	12	12
at 56 days	7	9	---	11	8	7.5	12	21	3.0	5	10	13
at 280 days	7	7	---	---	8	7.5	10	21	---	4.5	10	13
Volume Increase, cc/sq.ft.	5.7	7.8	---	11.5	8.3	7.3	12.0	21	2.7	5.3	10.7	12.7
Average	5.7	7.8	---	11.5	8.3	7.3	12.0	21	2.7	5.3	10.7	12.7

^a/Calculated ^b/Estimated. ^c/3- X 3/16-in. disk.

TABLE X. PHYSICAL TESTS OF COATINGS

ASPHALT:	ASPHALT I				ASPHALT II				ASPHALT III			
STABILIZER, %:	0	35	50	60	0	35	50	60	0	35	50	60
TENNESSEE MICA												
Volume Percent ^a / Density of Mixture ^a / Softening Point, °F ^b / S.P. of Base, °F ^b / Pen. of Base at 77°F ^b / Temp. of Preparation, °F ^b / Viscosity, cp. at 400°F ^b / 100 at 450°F ^b / 25 at 500°F ^b	0 1.015 223 223 17 365-428 280 100 25	15.4 1.33 270 189 28 428-446 ---- ---- ----	25.2 1.52 ---- ---- ---- ---- ---- ---- ----	33.6 1.69 ---- ---- ---- ---- ---- ---- ----	0 0.999 224 224 17 410-464 420 140 53	15.2 1.31 270 190 27 482-491 ---- ---- ----	24.9 1.50 ---- ---- ---- ---- ---- ---- ----	33.2 1.67 ---- ---- ---- ---- ---- ---- ----	0 1.018 227 227 14 401-437 375 130 28	15.4 1.33 270 190 22 491-518 ---- ---- ----	25.2 1.52 ---- ---- ---- ---- ---- ---- ----	33.6 1.69 ---- ---- ---- ---- ---- ---- ----
Water Absorption, g/sq.ft. at 28 days at 56 days at 280 days at 609 days (20 mo.)	0.67 1.00 3.00 4.62 4.11	2.05 2.56 4.75 6.34 8.58	---- ---- ---- ---- ----	33.6 1.69 ---- ---- ---- ---- ---- ---- ----	0.43 0.70 2.20 2.97 1.63	0.83 1.11 2.97 3.94 7.02	---- ---- ---- ---- ----	33.2 1.67 ---- ---- ---- ---- ---- ---- ----	0.34 0.52 1.61 2.29 0.70	0.52 0.83 2.12 3.06 5.38	---- ---- ---- ---- ----	33.6 1.69 ---- ---- ---- ---- ---- ---- ----
Volume Increase, cc/sq.ft. Shatter ^c , Inches #1 #2 #3 Average	5 7 7 6.3	21 21 21 21	---- ---- ---- ----	33.6 1.69 ---- ---- ---- ---- ---- ---- ----	8 9 8 8.3	21 21 21 21	---- ---- ---- ----	33.2 1.67 ---- ---- ---- ---- ---- ---- ----	2.5 3.0 ---- 2.7	21 ---- ---- 21	---- ---- ---- ----	33.6 1.69 ---- ---- ---- ---- ---- ---- ----
LAKE ERIE SILICA												
Volume Percent ^a / Density of Mixture ^a / Softening Point, °F ^b / S.P. of Base, °F ^b / Pen. of Base at 77°F ^b / Temp. of Preparation, °F ^b / Viscosity, cp. at 400°F ^b / 100 at 450°F ^b / 25 at 500°F ^b	0 1.015 223 223 17 365-428 280 100 25	16.9 1.30 ---- 213 21 410-419 600 200 50	27.5 1.47 226 208 22 410-437 790 250 77	36.2 1.62 217 ^d / 196 25 419-446 419-437 790 250 77	0 0.999 224 224 17 410-464 420 140 53	16.7 1.28 217 ^d / 207 23 383-401 480 220 92	27.2 1.45 217 ^d / 200 24 392-410 860 300 190	35.8 1.60 222 197 25 383-419 383-419 860 300 190	0 1.018 227 227 14 401-437 375 130 28	16.9 1.30 226 206 20 401-410 600 215 65	27.5 1.47 227 205 20 419-446 1140 485 200	36.2 1.62 222 191 22 446-464 ---- ---- ----
Water Absorption, g/sq.ft. at 28 days at 56 days at 280 days at 609 days (20 mo.)	0.67 1.00 3.00 4.62 4.11	0.64 0.98 3.30 3.71 2.94	0.73 1.09 4.42 4.36 6.52	36.2 1.62 217 ^d / 196 25 419-446 419-437 790 250 77	0.43 0.70 2.20 2.97 1.63	0.56 0.85 2.51 3.48 4.85	0.62 0.92 2.74 3.80 3.22	0.65 0.98 3.11 4.53 8.26	0.34 0.52 1.61 2.29 0.70	0.63 0.86 2.15 3.04 1.93	0.59 0.82 2.86 2.70 2.76	0.59 0.86 2.43 2.18 2.90
Volume Increase, cc/sq.ft. Shatter ^c , Inches #1 #2 #3 Average	5 7 7 5.7	8 7 7 7.3	15 15 14 14.7	36.2 1.62 217 ^d / 196 25 419-446 419-437 790 250 77	8 9 8 8.3	12 12 11 11.7	21 21 18 20.0	16 16 18 17.7	2.5 3.0 ---- 2.7	6 7 6 6.3	7 8 7 7.3	8 8.5 ---- 8.3
a/ Calculated.	b/ Estimated.	c/ 3- X 3/16-in. disk.	d/ Rate slow - probably 3-4°F higher.									

Of course, the nature of the method by which failure is judged is another important factor affecting the reported 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 days before additional strain sufficient to induce more cracking is produced. 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 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.

Some mineral additives do not lend themselves to being spark photographed when in asphalt, because they are electrically conductive. Those coatings containing fly ash had to be inspected visually for this reason, and any type A failures were thus precluded. The lack of correlation between the spark photograph and panel appearance is illustrated in the type B failure in Figure 6, which is 35% fly ash in Asphalt I.

In interpreting the durability data there are several ways in which they must be considered. The relative durability is the primary consideration--What is the relative life of each coating? 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 thinner films perform as satisfactorily as a thicker 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 durability. Asphalt I was a shorter lived material than II and III, which were of approximately the same durability. Only in the 43-mil thick films did Asphalt III prove to be appreciably more durable than II. When stabilizers were added to these asphalts, the same order of spread was maintained.

In terms of relative 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 25-mil and 43-mil films, except for the 25-mil ones containing Asphalt I, show no signs of failing; the 13-mil, mica-stabilized films with Asphalt I failed in 175 days, with Asphalt II failed in 369 days, and with Asphalt III failed in 402 days. These are greater than the durability of any other coating of the corresponding asphalts tested even in the 43 mil thickness range, except for the thicker films containing mica, of course.

Asphalt I coating, 25 mils thick, with 35% mica, failed in 572 days, which is more than 13 times the durability of the unstabilized asphalt of equivalent thickness. The 13-mil coating lasted only 5.4 times as long as the unstabilized Asphalt I, 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 slate can be seen in Table V and Figure 5, and the ratios of these durabilities to that of the corresponding straight asphalts and the failure crack patterns are also reported in Table V. Again the Asphalt I-base coatings were the least durable and the coatings with Asphalts II and III about the same. However, in all instances, the stabilizer increased the durability of the coating. The smallest ratio was 1.4 for 50% blue black slate in Asphalt I, 13 mils thick; the greatest ratio was 3.2, with blue black slate in Asphalt II, 25 mils thick.

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 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 effective than fly ash in increasing the durability of the asphalts.

Dolomite in many instances showed marked improvement in the durability of Asphalt II (Ratios from 1.4-2.3) and Asphalt III (Ratios from 1.3-1.7). However, in one instance (13 mils), the durability of Asphalt III with 35% of dolomite was increased only 15% and only one combination of Asphalt I and dolomite exceeded the durability of unstabilized asphalt more than 30% (Ratio 1.4).

The low carbon fly ash improved the durability of both Asphalts II and III, but did not alter it for Asphalt I, when the results are compared on the basis of equal film thickness. 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, Asphalt I coatings with fly ash reacted similarly to those containing dolomite, but the durability of both Asphalts II and III coatings decreased with increasing stabilizer concentration.

The clay and 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 43-mil coatings with Asphalts II and III did it increase appreciably. For these two materials, except in the thickest films, durability was almost independent of stabilizer concentration.

The above discussion indicates quite clearly that these data do not lend themselves to precise conclusions. However, a rough listing of the materials tested would place them in the following descending order of their merits as stabilizers:

- | | | |
|------|----|------------------------|
| Good | == | (1) Tennessee Mica |
| | | (2) Blue Black Slate |
| Fair | == | (3) Niagara Dolomite |
| | | (4) Low Carbon Fly Ash |
| Poor | == | (5) Lake 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.

6.2 Physical Tests on Coatings

These accelerated durability data must be considered in their relation to the materials from which the coatings were made and to the properties of the coatings.

(a) Stabilizers

The materials which were blended with the asphalts for these studies were examined rather thoroughly and the results reported in Table I. 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 whose performance was reported to be variable.

(b) Asphalts

The asphalts were obtained from three roofing plants, where each had been processed into five different products from the fluxes described in Table II. These products, differing only in the length of time the asphalt was blown, were classified according to their softening points, as shown in Table II.

The spectral analyses reported in Table III show that all three asphalts had strong lines present for such metals as nickel and vanadium. However, the effect of these catalytically active metals on the durability of asphalts is not known.

The physical properties of each of the unstabilized asphalt coatings exposed are listed in Table VII. These show that all three asphalts are of the normal air blown coating grade. 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 Asphalt III, indicates that it should withstand temperature variations best, followed by Asphalt II and Asphalt I, in order.

Asphalt I had the greatest heat loss at 163°C; none of the penetrations changed appreciably during the five-hour heating period.

Although the viscosity measurements do not indicate it, Asphalt I had a sharper melting point than the other two. However, Asphalt II was the most tacky as well as the most viscous.

Asphalt III 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 Asphalt II fell between III and I 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.

(c) Effects of the Addition of Mineral Materials

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. These materials behave differently in each asphalt and in the different asphalts. In Tables VIII to X are listed the softening points of the coatings and the

estimated softening points of the base asphalts. By subtracting the two (See Table XI) it can be seen that the softening point rise increased with concentration for each material. However, the effect was different in each asphalt, being least pronounced in II and most in III. The viscosity data show that all of the materials 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 can be seen upon examination of the increase in shatter reported in Table XI. In all but four instances the materials 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 the materials increased the water absorption of the asphalts progressively with increasing concentration, but not always in direct proportion to the concentration. Asphalt III, itself, absorbed water at the lowest rate and all of its coatings absorbed water more slowly than the corresponding coatings made from the other asphalts. Asphalt II fell between the low rate of III and the high rate of Asphalt I.

For comparison purposes, the water absorption curves of the coatings containing 35% mineral matter are shown in Figure 10, the complete data on water absorptions being in Tables VIII to X and XIII. Although the water absorption in all cases increased progressively with increasing proportions of mineral matter, there was no relation between this increase and the results obtained in the accelerated durability machines.

TABLE XI. EFFECT OF MINERAL MATTER ON SOFTENING POINTS AND SHATTER OF COATINGS

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

^{a/}The softening point rise is the difference between the softening point of the coating and that of the base asphalt.

^{b/}The 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: I = 6.3; II = 8.3; III = 2.7.

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 three categories on page 27 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 materials designated as "good" both have flat, plate-like particles; the "fair" materials have sharp, blocky particles; and the "poor" materials have rounded corners and edges. This would indicate that a satisfactory 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 materials tested behaved differently in each asphalt, it is not possible to draw definite conclusions on the effects of these six materials on the three asphalts tested. However, the following broad generalizations are indicated by the results of these accelerated tests:

- (1) For every combination tested, the durability of the coating increased with film thickness.
- (2) Blue black slate and Tennessee mica increased the durability of all three asphalts at all concentrations and film thicknesses tested.
- (3) Niagara dolomite increased the durability of the coatings in many cases, but in others had no effect.

- (4) Low carbon fly ash increased the durability of the coatings in many cases, but did not affect it in others; however, because the coatings containing fly ash could be rated only by visual inspection, the durability reported may be high.
- (5) Lake Erie silica and Florida clay increased the durability only in some instances, primarily in the thicker films with asphalts II and III.
- (6) All the materials studied increased the softening point, viscosity, water absorption, and impact resistance of the coatings progressively with increasing concentration, but without a systematic relationship with durability.
- (7) There is some indication that plate-like particles effect the greatest increase in durability.

8. APPENDIX A.

8.1 Volume Composition

Several calculated quantities are reported in Tables VIII to X 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

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

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

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

8.2 Specific Gravity

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 XII compares the calculated and observed specific gravity for 13 coatings.

TABLE XII. SPECIFIC GRAVITIES

COMPOSITION		SPECIFIC GRAVITY	
		Calculated	Observed
Asphalt I	- 50% Blue Black Slate	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
Asphalt II	- 50% Blue Black Slate	1.49	1.50
"	- 50% Fly Ash	1.44	1.39
"	- 35% Silica	1.28	1.24
"	- 50% Silica	1.45	1.43
Asphalt III	- 50% Clay	1.47	1.47
"	- 50% Dolomite	1.50	1.49
"	- 60% Silica	1.62	1.62

8.3 Shatter Test

SHATTER TEST ON COATINGS [15]

Preparation of Specimens:

Several disks of asphalt 3 inches in diameter and 3/16-inch thick are cast using a suitable glycerine-coated brass mould. (See section 8.4 for specimen preparation.)

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 21-inch vertical brass tube, 1 inch in internal diameter, a solenoid sliding within the tube and adjustable to any height up to 21 inches, 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 inch in diameter and weighing exactly 1/2 lb., and a stationary steel contact rod 15/16 inch 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 about 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 inch 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 inches and the drop again made. This procedure is repeated with 1/2-inch increments in height until the specimen fails. Subsequent specimens should be started at a height 1 inch 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.

8.4 Water Absorption Tests

WATER ABSORPTION OF ASPHALT DISK METHOD [15]

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 $3/16$ inch thick, 3 inch diameter hole.
2. Brass plate - 4- x 4-inches.
3. Glycerine.
4. Hot plate.
5. Red marking pencil.
6. 100 grams of asphalt to be tested.
7. Pyrex glass tray, $1\ 1/2$ inch 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 mineral matter, the sample should be stirred slowly with a piece of stiff wire to keep the matter properly suspended without incorporating air bubbles.

Pour a sufficient amount of the sample to fill the mould. The pouring must be done with care in order that air bubbles are not occluded. The surface may be flamed lightly to remove a few which might form. Not more than $1/16$ inch of the sample should show above the top of the mould. After the specimen has cooled thoroughly, remove it from the mould and wash it 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 to 0.001 gram and record the weight. Place the specimen in the glass tray and fill with sufficient distilled water to submerge the specimen at least $1/4$ inch. Place the glass tray and specimen in a dark cabinet at room temperature.

Procedure (continued):

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

First 3 months	-- Weekly
Next 3 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 until 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.

NOTE: The water absorption measurements were made on specimens three inches in diameter and approximately 3/16-inch 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 inch and were weighed weekly for three months, monthly for three months, and then quarterly until the test was discontinued at 20 months. 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.

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 Asphalt III 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 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 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 Asphalts II and III and decreased for Asphalt I.

Table XIII-C 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 Asphalt III coatings tended to be lower than in the coatings containing the other two asphalts, because of the extremely low volume increase of the unstabilized Asphalt III, the relative volume changes in these 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.

To summarize the results of the water absorption tests, it must be noted that despite the fact that all the mineral additives increased the water absorption of the asphalts, many of them also increased the durability as well and, there is no correlation between durability and water absorption.

TABLE XIII. WATER ABSORPTION DATA

A. WATER ABSORBED IN ONE YEAR

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

TABLE XIII. WATER ABSORPTION DATA
B. WEIGHT INCREASE IN 20 MONTHS

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

^{a/} Underwent a weight loss during the last 100-200 days of immersion.

TABLE XIII. WATER ABSORPTION DATA
C. VOLUME INCREASE IN 20 MONTHS

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

^{a/} Underwent a weight loss during the last 100-200 days of immersion.

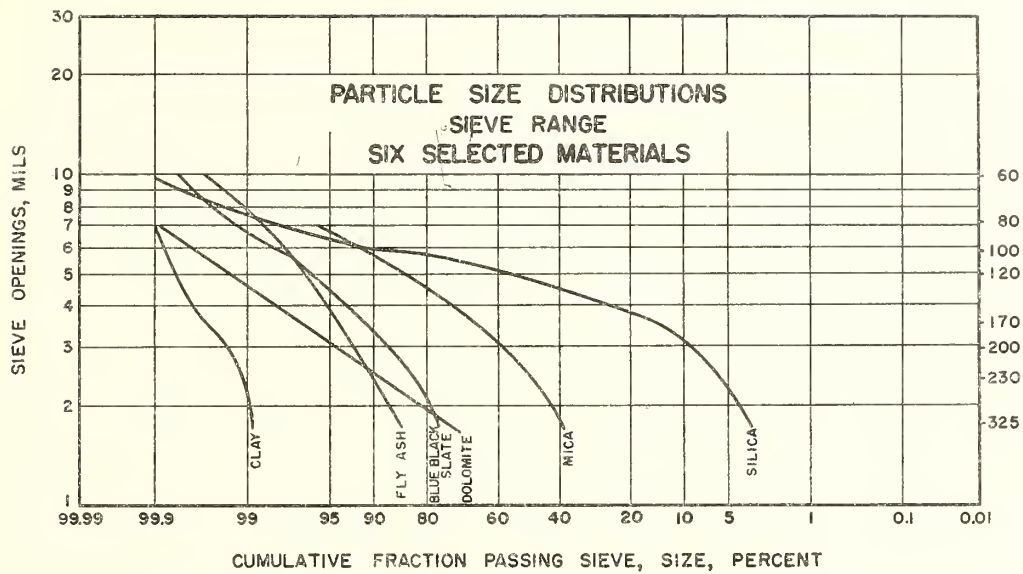


FIG. 12

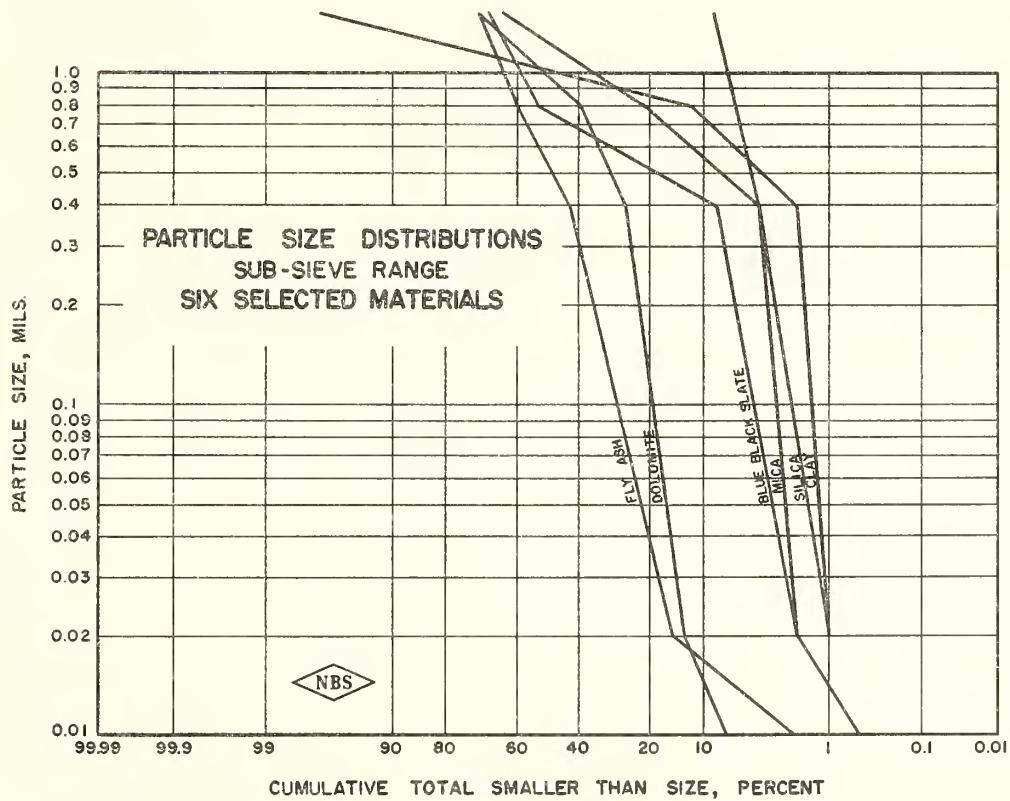


FIG. 13

9. REFERENCES

- 1 "Accelerated Tests of Organic Protective Coatings", P. H. Walker and E. F. Hickson. B.S. J. Res. 1, 1, (1928); Ind. Eng. Chem. 20, 591, (1928).
- 2 "Accelerated Weathering Properties of Oklahoma Asphalts", P. G. Shelley, Circ. #19, Oklahoma Geological Survey, Norman, Oklahoma.
- 3 "Accelerated Tests of Asphalts", O. G. Strieter, B.S. J. Res. 5, 247, (1930) (RP 197).
- 4 "The Accelerated Weathering Test and the Chemical Nature of Asphalts", O. G. Strieter, B.S. J. Res. 16, 481, (1936).
- 5 "The Effect of Mineral Fillers on the Serviceability of Coating Asphalts", O. G. Strieter, Proc. A.S.T.M. 36, Part II, 486, (1936).
- 6 "A Modified Accelerated Weathering Test for Asphalts and Other Materials", O. G. Strieter and H. R. Snoke, B.S. J. Res. 16, (1936).
- 7 "Method of Test for Evaluating Asphalts", O. G. Strieter, B.S. J. Res. 17, 276, (1937).
- 8 "Weathering Tests of Filled Coating Asphalts", O. G. Strieter, B.S. J. Res. 20, 159-171, (1938) (RP 1073).
- 9 "Mineral Stabilizers in Asphalt Roofing", L. Kirschbraun, R. H. Cubberley, F. W. Yeager. A.S.T.M. Symposium on Accelerated Durability Testing of Bituminous Materials (Spec. Tech. Pub. #94) (1949).
- 10 "A Method of Preparing Uniform Films of Bituminous Materials", S. H. Greenfeld. Presented at the A.S.T.M. 1953 Annual Meeting.
- 11 "The Effects of Refrigeration on the Accelerated Testing of the Durability of Asphalts", S. H. Greenfeld. Progress Report of July 12, 1950.

- 12 "Effects of Thermal Shock on the Durability of Asphalt Coatings Under Accelerated Test", S. H. Greenfeld. Presented at the A.S.T.M. 1953 Annual Meeting.
- 13 "A New Method for Evaluating Failure of Bituminous Materials Due to Weathering", J. B. Hunter, F. C. Gzinski, and L. Laskaris. A.S.T.M. Symposium on Accelerated Durability Testing of Bituminous Materials, 1949.
- 14 "The Properties of Asphaltic Bitumen", J. P. Pfeiffer, Elsevier Publishing Co., New York, 1950, 157-158.
- 15 Test Methods 1713⁴ and WA-2 of the Johns-Manville Co.

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.

