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NATIONAL BUREAU OF STANDARDS REPORT

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8536

FINAL REPORT

BONDING TO TREATED CONCRETE SURFACES

NBS

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

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NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT | **NBS REPORT**

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FINAL REPORT

BONDING TO TREATED CONCRETE SURFACES

by

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Sponsored by

Office of the Chief of Engineers
Department of the Air Force
Bureau of Yards and Docks

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

FINAL REPORT

BONDING TO TREATED CONCRETE SURFACES

1. INTRODUCTION

The present study of the adhesion of resilient floor coverings and organic coatings to concrete surfaces treated with curing, parting, sealing, hardening, and other agents was initiated during 1962. Occasionally problems are reported with resilient flooring installations in which the cause is suspected to be some treatment applied to the concrete on which the floor is laid. Some paint failures might also be ascribed to concrete treatments on floors, walls, and ceilings because membrane curing agents or form oil have been used on these areas. The first task in this project was to develop information on concrete treatments and classify them according to their effect on the adhesion of resilient flooring or paint to concrete substrates. The second task was to develop methods for determining in the field whether a particular concrete surface is suitable for the application of adhesives or organic coatings. A third task was to devise methods of treating unsatisfactory surfaces to make them suitable for the application of adhesives or organic coatings.

2. BACKGROUND

During the course of this project we have cooperated with other Government agencies and with private associations, such as the Rubber Manufacturers Association and the Asphalt and Vinyl Asbestos Tile Institute, in collecting information on commercial curing agents. The private associations mentioned have made some of this information available to industry in the form of reports.

Some information on practical tests for adhesion of resilient floor coverings is available from industry and Government. The only practical field test which has been published and was available to us appears to be the procedure mentioned on page 33 of "Technical Data 1964-65", Armstrong Cork Company, Floor Division, Lancaster, Pennsylvania. In this test, 3-foot squares of flooring material are installed approximately 50 feet apart in the manner to be used in the final installation. The panels are examined for secure bonding after two weeks. The material is considered to be securely bonded if "unusual force is required to lift it from the subfloor and if, after doing so, adhesive clings to both the subfloor and the back of the material."

We have also investigated the possibility of a field test in which bond strength could be measured and expressed numerically. Such a quantitative test is written in NAVDOCKS SPECIFICATION 13Yf, December 1960, CONCRETE CONSTRUCTION, Section 2.13.7, page 12.

3. MATERIALS AND LABORATORY TEST PROCEDURES

3.1 Adhesion of Organic Coatings to Concrete

In testing for adhesion of organic coatings, 108 3- by 5-inch cement mortar panels, 5/8-inch thick were prepared from approximately 2 parts "high early strength" portland cement, 6 parts of concrete sand, and 1 part of water, by weight. The panels were steam cured for 28 days. Some of the panels were left untreated and some were coated with various concrete curing agents. The panels to be treated were first soaked in water for 24 hours, then curing agents were applied by brush. After a suitable drying period, organic coatings were applied to the panels.

Treatments and coatings were applied to the test panels as indicated in Table 1. In Table 1, curing agents are identified by capital letters and organic coatings by Roman numerals. Information on the formulation of the curing agents and organic coatings is found in Appendix 3 of this report.

After the organic coatings had dried for 24 hours, each of the test panels was divided into four sections from top to bottom as shown in the photographs. These sections were marked off with indelible ink. A section or area 1/4- by 3-inches at the top of each panel was reserved for numbering. This was followed by three areas, equal in size, each 1-1/2- by 3-inches. Within each area or section, the top portion was reserved for the adhesive tape test and the bottom portion for the thumb nail and knife blade tests. The bottom area was tested 1 day after applying the organic coating; the middle or second area was tested after 7 days; and the top area was tested after 30 days.

In the thumb nail test, the thumb nail was scraped across the width of the panel. In the knife blade test, paint was scraped off the width of the panel (horizontally in the photograph) with a Bard Parker knife at about a 30° angle to the panel.

The adhesive tape test was a modification of that described by Henry A. Gardner and George G. Sward, Paint Testing Manual, 12th edition, March, 1962, p. 165. The procedure is as follows:

Scribe a cross about 1/2- by 1-inch in the coating. Apply pressure sensitive tape to the cut and parallel to the long arm of the cross all the way across the width (horizontal) of the panel (3 inches). Rub to firm adherence with the fingers. Pull the tape off with a sharp pull as nearly as possible in the plane of the surface. Observe the removal of coating visually.

Table 1. ASSIGNMENT OF TREATMENTS TO TEST PANELS

Paint	A	B	C	D	E	F	G	H	I
	Treatment								
	Panel Number								
I	1	2	3	4	5	6	7	8	9
II	10	11	12	13	14	15	16	17	18
III	19	20	21	22	23	24	25	26	27
IV	28	29	30	31	32	33	34	35	36
V	37	38	39	40	41	42	43	44	45
VI	46	47	48	49	50	51	52	53	54
VII	55	56	57	58	59	60	61	62	63
VIII	64	65	66	67	68	69	70	71	72
IX	73	74	75	76	77	78	79	80	81
X	82	83	84	85	86	87	88	89	90
XI	91	92	93	94	95	96	97	98	99
XII	100	101	102	103	104	105	106	107	108

KEY TO THE CURING AGENTS AND ORGANIC COATINGS

Curing Agents

A. Untreated (water cured). As with the other panels, cured for 28 days in a steam curing room but no membrane curing agent applied.
B. Commercial curing agent, based on butadiene-styrene copolymer.
C. Commercial curing agent, based on petroleum hydrocarbon resin.
D. Commercial curing agent, a resin type.
E. Commercial curing agent, a wax-resin type.
F. Butadiene-styrene copolymer formulation prepared at the NBS.
G. Petroleum hydrocarbon resin formulation prepared at the NBS.
H. Chlorinated rubber-chlorinated paraffin formulation prepared at the NBS.
I. Wax and oil mixture prepared at the NBS.

Organic Coatings

I. Cement water type concrete paint.
II. Cement water concrete paint.
III. Chlorinated rubber in solvent.
IV. Butadiene-styrene copolymer in solvent.
V. Butadiene-styrene and acrylic resin emulsion.
VI. Butadiene-styrene copolymer emulsion.
VII. Polyvinyl acetate emulsion.
VIII. Alkyd modified vinyl resin emulsion.
IX. Oil-alkyd modified latex paint.
X. Acrylic resin emulsion.
XI. Alkyd enamel.
XII. Alkyd enamel.

The numerical rating system described in the original test was too cumbersome and, since there were no replicates, it would be impossible to determine the precision of the method.

3.2 Adhesion of Resilient Flooring to Concrete

3.2.1 Qualitative Tests Using Cement Mortar Panels

A number of qualitative adhesion tests were run on cement mortar panels 4-1/2- x 9-inches x 5/8-inch thick. These panels were made from approximately 2 parts "high early strength" portland cement, 6 parts of concrete sand, and 1 part of water, by weight. In some cases, additional water was added to make the mix workable. The object of this part of the program was to evaluate concrete curing agents for adhesion of floor coverings and to devise a suitable test for such evaluation.

Water-cured panels were steam cured for 28 days and allowed to dry for several days before applying floor coverings or gauze. Membrane-cured panels were prepared by applying the curing agents to the "green" or fresh cement mortar surface with a camel's hair brush and storing in a constant temperature-constant humidity room at 73°F., 50% relative humidity for 28 days. After curing and drying, the panels were coated with the desired adhesives, using a notched trowel, as in actual practice, and the selected floor covering was applied. The majority of tests were made with vinyl asbestos tile, using either an asphalt cutback or an asphalt emulsion adhesive.

Since tests with floor coverings are slow, an attempt was made to devise a quicker test, using cotton gauze strips in place of floor coverings. The open structure of the gauze would permit the adhesive solvent to evaporate more rapidly. The gauze strip test was based on an earlier method for testing the adhesion of paint films, reported on page 176 of Henry A. Gardner and George G. Sward, "Physical and Chemical Examination of Paints, Varnishes, Lacquers, and Colors", Henry A. Gardner Laboratory, Bethesda, Maryland, 11 edition, 1950. In this method, strips of silk cloth were pressed on paint films while still tacky. After the paint had dried, the strips were pulled away from the substrate with a portable tensile testing instrument. The instrument used was similar to the "Gardner Tensile Strength and Elongation Apparatus", Gardner Laboratory, Inc., Bethesda, Maryland, Catalog No. TG-1500. It was hoped that this portable instrument might be used to test adhesion of floor coverings, as in NAVDOCKS Specification 13Yf. In the NBS tests, observations with the cotton gauze strips were qualitative and were intended to be exploratory, with quantitative evaluation as the goal. In the qualitative tests, gauze strips one-inch wide were applied to the cement mortar panels, using asphalt adhesive. The adhesive was spread with a putty knife, scraping off the excess, so that as thin a film was applied as consistent with efficient wetting of the gauze and cement mortar surface.

3.2.2 Quantitative Straight-pull Adhesion Tests Using Asphalt Tile and Cement Mortar Cubes

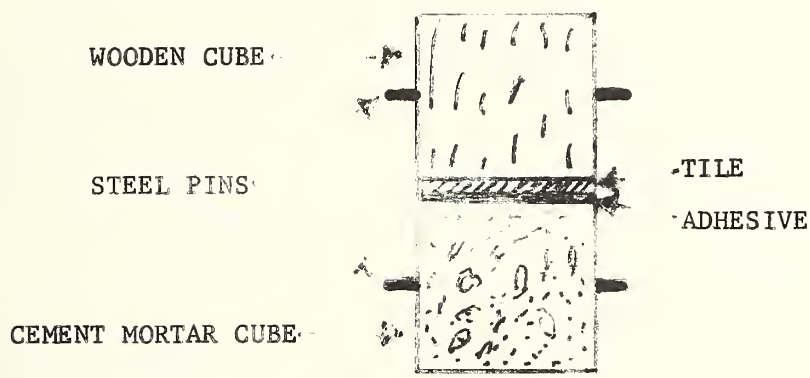
Both the straight-pull and the stripping-pull tests were adapted from those described by Percy A. Sigler and Robert I. Martens, Building Materials and Structures Report BMS 59, "Properties of Adhesives for Floor Coverings", National Bureau of Standards, 19 September 1940.

Specimens for this series of tests were 2-inch cement mortar cubes, cast in modified "Bowen Expansible Cube Molds", U. S. Patent 2,061,137, Bowen and Company, Inc., Bethesda, Maryland. The molds were modified by drilling holes, 17/64-inch in diameter in a position such that the three completed cubes from each mold would each have a 1/4-inch diameter steel pin through the center of two faces adjacent to the top or troweled surface under test. The pins were inserted through the drilled holes before casting the cement mortar cubes. The cubes were cast in accordance with ASTM C-109 except that concrete sand was used in place of graded Ottawa sand and the mixer accordingly set with greater clearance. Also, the cement mortar cubes were not leveled immediately, but were placed in the curing cabinet for about an hour with the mortar heaped up and then finished with a steel trowel. This was similar to the procedure in finishing concrete floors. Cubes to be water cured were placed in a moist cabinet overnight; removed from the molds; then placed under water in the moist cabinet for 6 more days. The cubes were then placed in a constant temperature-humidity room at 73°F. and 50% relative humidity for 21 days or more. Cubes which were to be membrane cured were coated with membrane curing agents immediately after troweling, using a camel's hair brush. The membrane coating was applied only to the top surfaces of the cubes, which had been troweled. The cubes were then placed immediately in polyethylene bags, which were tied and placed with the tied ends inside other polyethylene bags and the outer bags then tied. The cubes were allowed to cure in the double polyethylene bags at 73°F. for at least 28 days.

After curing, the test cubes were coated with asphalt cut-back or emulsion adhesive at 73°F. and 50% relative humidity, air dried under these conditions for approximately 90 minutes, then 2-inch squares of OTAT applied. OTAT refers to Official Test Asphalt Tile (for 1963) of the Chemical Specialties Manufacturers Association (CSMA), 50 East 41st Street, New York, N.Y. 10017. After laying the asphalt tile, pressure was applied to the top surface of the tile. In some of the tests, the tile was bonded at a pressure of approximately 0.2 pounds per square inch (psi) for one hour. A steel plate was placed on 9 specimens at a time, so that the pressure was about 2 psi. This was adapted from

Federal Specifications MMM-A-00115 and SS-A-128. In other tests, the tile was bonded by placing the specimen in a Carver hydraulic laboratory press, such that the pressure was 625 psi and load applied for 30 seconds. After laying the tile and pressing, the specimens were stored in the constant temperature-humidity room for the desired period of time for curing or setting of the adhesive.

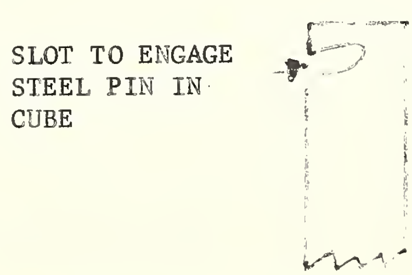
After the adhesive had cured for the desired time, 2-inch wooden cubes were cemented to the top surfaces of the tile, using commercial epoxy cement containing a filler. The wooden cubes were also fitted with steel pins, 1/4-inch in diameter, so that the completed specimen appeared as in the following sketch:



SIDE VIEW OF SPECIMEN (NOT TO SCALE)

In order to secure the specimen to a tensile testing instrument, special yokes were used. On yoke was clamped in the upper jaw of the instrument and engaged the steel pin in the wooden cube. The other yoke was clamped in the lower jaw of the instrument and engaged the steel pin in the cement mortar cube. One of the yokes is illustrated in the following sketch:

TOP VIEW



DETAIL, SIDE VIEW OF ONE ARM OF YOKE

(NOT TO SCALE)

The instrument used for the test was an Instron Tensile Testing Instrument, Model TT-B, standard speed, 1,000 lbs. capacity, crosshead speed 0.02 - 20 inches per minute, Instron Engineering Corporation, 2500 Washington Street, Canton, Massachusetts. A crosshead speed of 0.05 inches per minute was used. To determine the variation with different adhesives, three asphalt cut-back adhesives of three manufacturers and three emulsion adhesives of the same manufacturers were used.

3.2.3 Quantitative Stripping-pull Tests Using Flexible Resilient Tile, Asphalt Adhesives, and Cement Mortar Prisms

A number of 90° or stripping-pull tests were performed on water cured cement mortar prisms in order to compare the stress-strain or load-elongation relationship with that from the straight-pull tests. For this purpose, cement mortar prisms, 1.575- by 1.575- by 6.3-inches, were made from modified prism molds as described in Federal Test Method Standard 158a, Method 2701.1, Section 2.3. Holes, 17/64-inch in diameter, were drilled in each mold, centers equidistant from the ends of the side plates and 5/8-inch from the bottom. In casting, stainless steel pins, 1/4-inch in diameter, were inserted through the holes to serve as handles. Each finished prism had a steel pin 5/8-inch from the bottom and equidistant from each end. The pin was used to hold the prism in the stripping-pull test. The cement mortar mix and finishing and curing procedure were similar to those used for the 2-inch cement mortar cubes except for the amount of mix and filling and tamping procedure outlined in Method 2701.1, Federal Test Method Standard No. 158a. The prisms were all water cured in the same manner as the 2-inch cement mortar cubes.

After curing for 28 days, pieces of flexible tile, 1/8-inch gauge, 1-5/8 inches wide, and 9 inches long, were cemented to the top or troweled surfaces of the prisms, using asphalt cut-back or emulsion adhesive, Brand A. The flexible tile was supplied by the Rubber Manufacturers Association and was the same as that used in their laboratory adhesion tests. An area of tile at the end of each specimen, 1-5/8 inches wide and 2-1/2 inches long (4 sq. in.), was pressed in a Carver laboratory hydraulic press at 625 psi for 30 seconds. A line was drawn at the end of the pressed area, 2-1/2 inches from the end of the piece of tile. Following this line, another area of the same size was pressed in the same manner and a second line drawn, 5 inches from the end of the piece of tile. The second line was, in each case, positioned in testing such that it was aligned with the bottom edge of the upper jaw of the tensile testing instrument.

In performing stripping-pull tests, each specimen was secured in a special stainless steel holder as shown in the sketch. The vertical plate was clamped in the lower jaw of the Instron tensile testing instrument and the strip of tile was clamped in the upper jaw of the instrument. Pull tests were run to constant stress or until the strip was free of the prism.

4. TEST RESULTS

4.1 Adhesion of Organic Coatings to Concrete (See 3.1)

The results of the tests of adhesion of organic coatings to cement mortar panels are shown in the following three photographs. The numbers refer to those given in Table 1. For example, all specimens in the first column of each page are controls not treated with curing agents. The rows represent the same paint applied to panels treated with different curing agents. In examining the photographs it must be remembered that the bottom section of each panel was used for tests one day, the middle section seven days, and the top section thirty days after application of the paint. Also, the lower half of each section was used for the thumb nail and knife tests and the upper half for the adhesive tape test. Aside from the appearance of the panels, which can be seen in the photographs, it is necessary to make the following observations regarding the thumb nail test. Examination of the photographs will not distinguish between the thumb nail and knife blade tests.

Coatings on the following panels were easily removed with the thumb nail one day after application and also after 7 days and 30 days:

Nos. 2, 3, 4, 5, 6, 7, 8, 13, 14, 15, 16, 17, 18, 54, 63, 72, 81, 90.

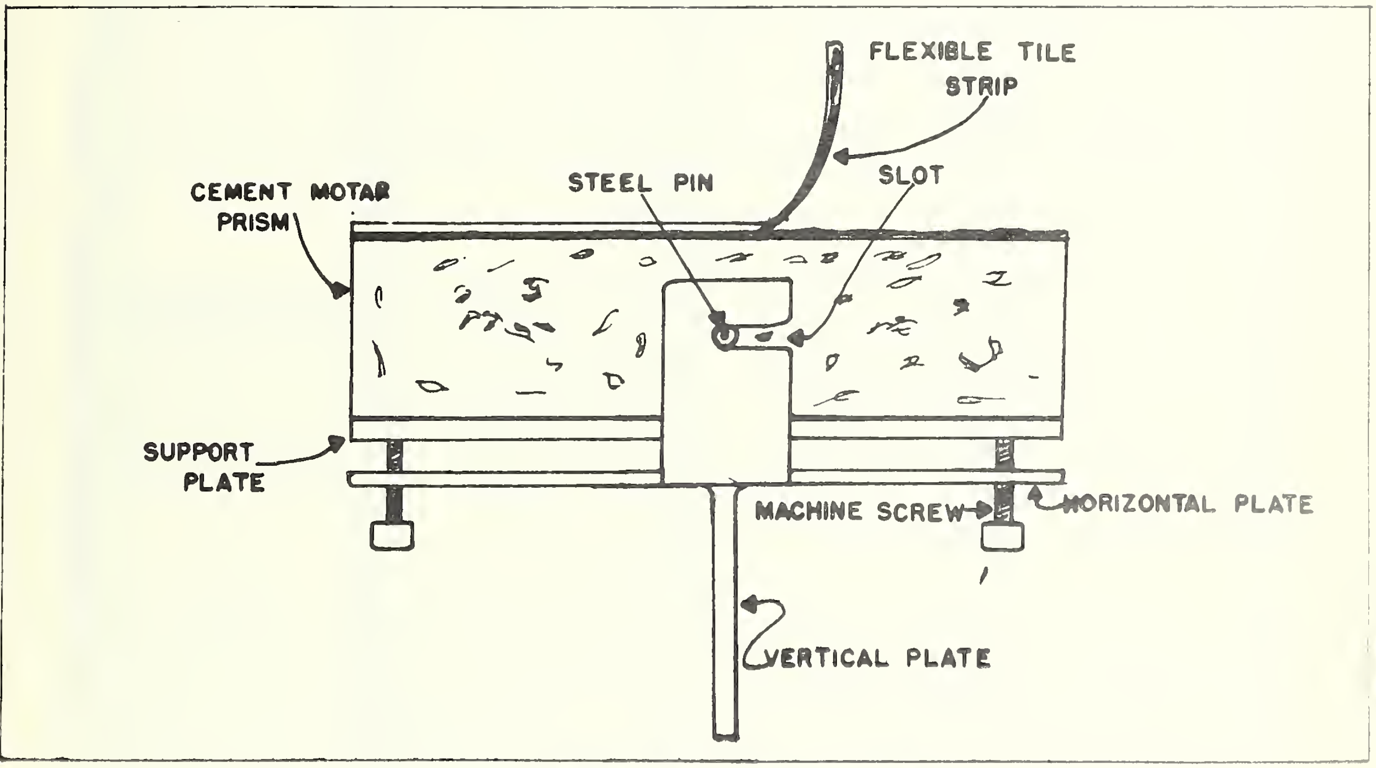
Coatings on the following panels were easily removed after 1 day and 7 days but not after 30 days:

Nos. 66, 75, 84, 99.

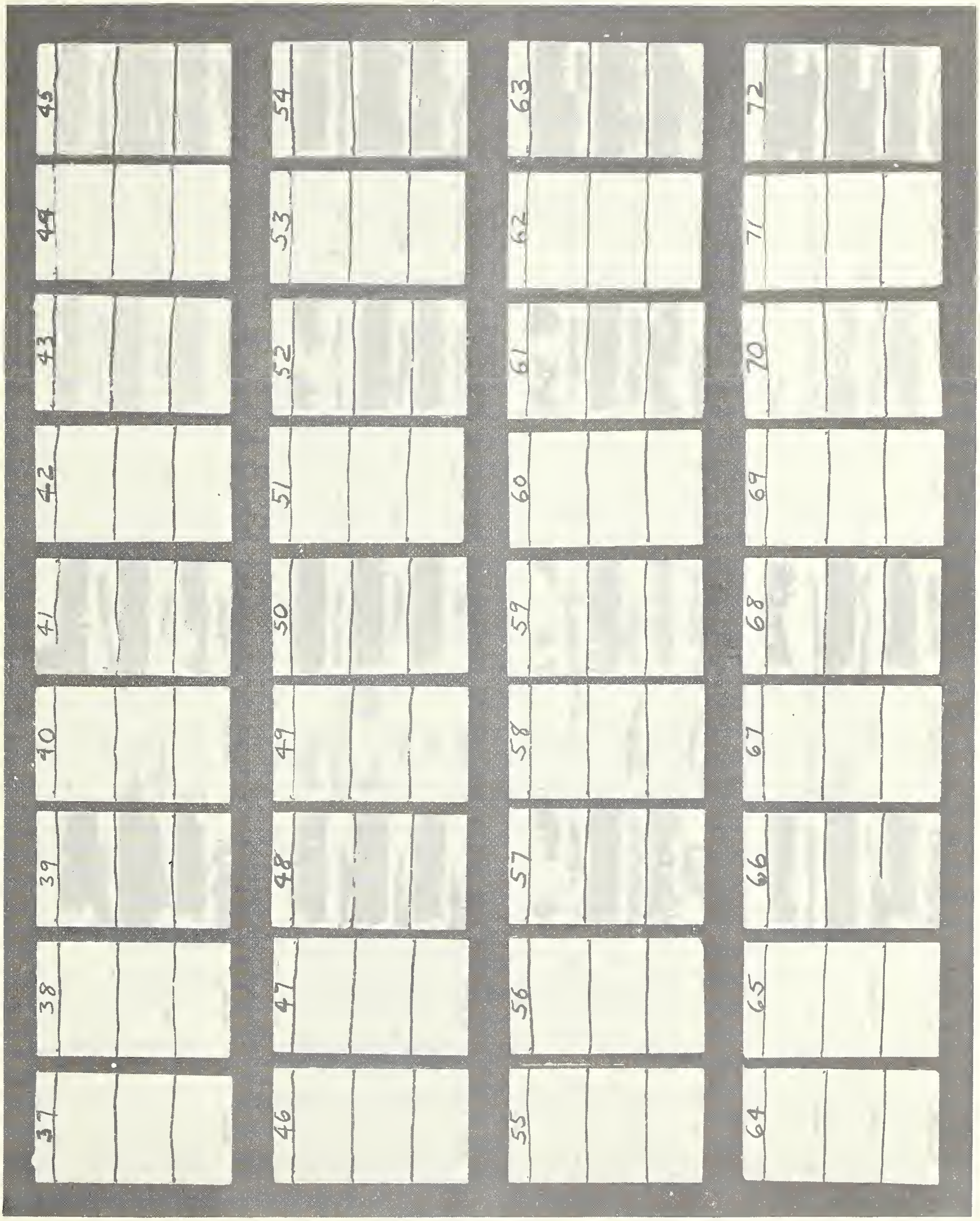
Coatings on the following panels were easily removed after 1 day but not after 7 or 30 days:

Nos. 76, 80, 103, 108.

Coatings on panels 39 and 45 were not completely removed by scraping with the thumb nail after one day but could be removed completely with the thumb nail after 7 days or 30 days. The coating on panel No. 48 could be partially removed after 1 day or 30 days but was completely removed by thumb nail after 7 days.



SPECIMEN AND HOLDER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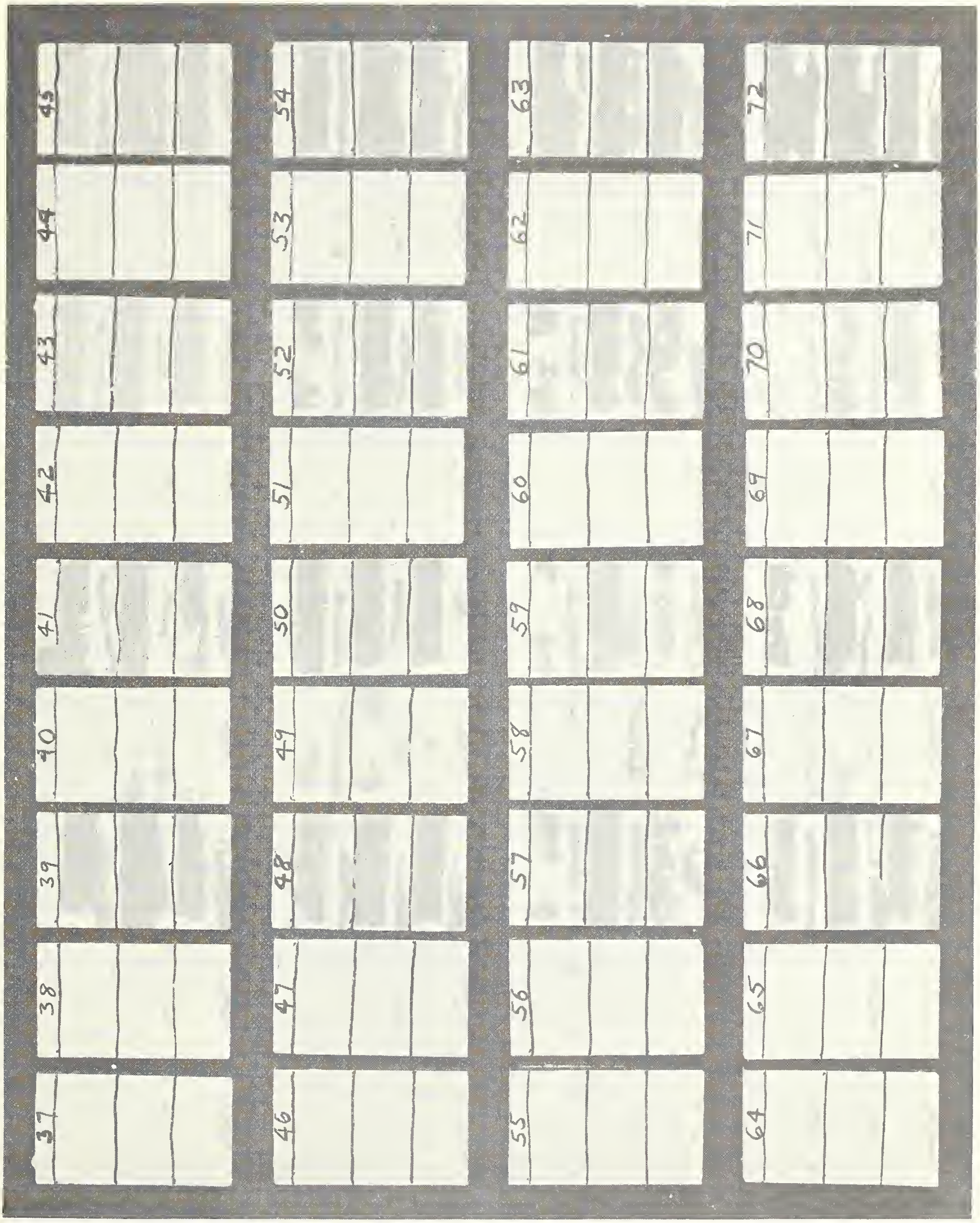
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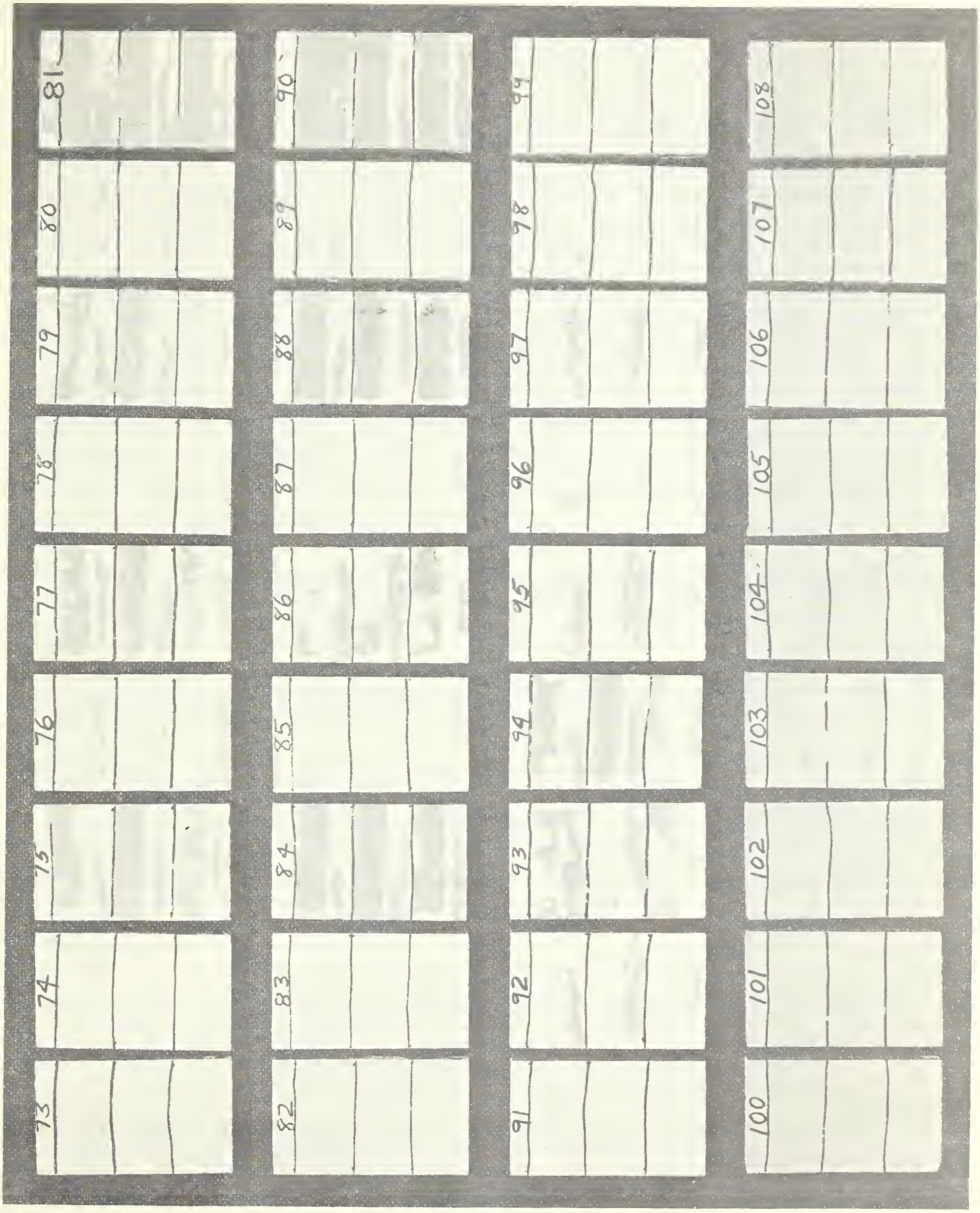
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4.2 Adhesion of Resilient Flooring to Concrete

4.2.1 Qualitative Tests (See 3.2.1)

Three photographs which follow show the results of qualitative adhesion tests on 4-1/2- by 9- by 5/8-inch cement mortar panels. One-inch wide strips of flooring or cotton gauze were removed after various periods of time of curing or setting of the adhesive. The photographs show the cement mortar surfaces after removal of the strips. Table 2 is a description and interpretation of the pictures. Panel numbers in the table correspond to numbers in the panels in the photographs. The order of removal of the strips was from top to bottom of each panel at time intervals indicated in order in the table. Panels omitted from the table were those tested with cotton gauze. Tests with cotton gauze were inconclusive and were therefore not included in the table. Information concerning the curing agents designated by capital letters in this table and also later in the report is given in Appendix 3.

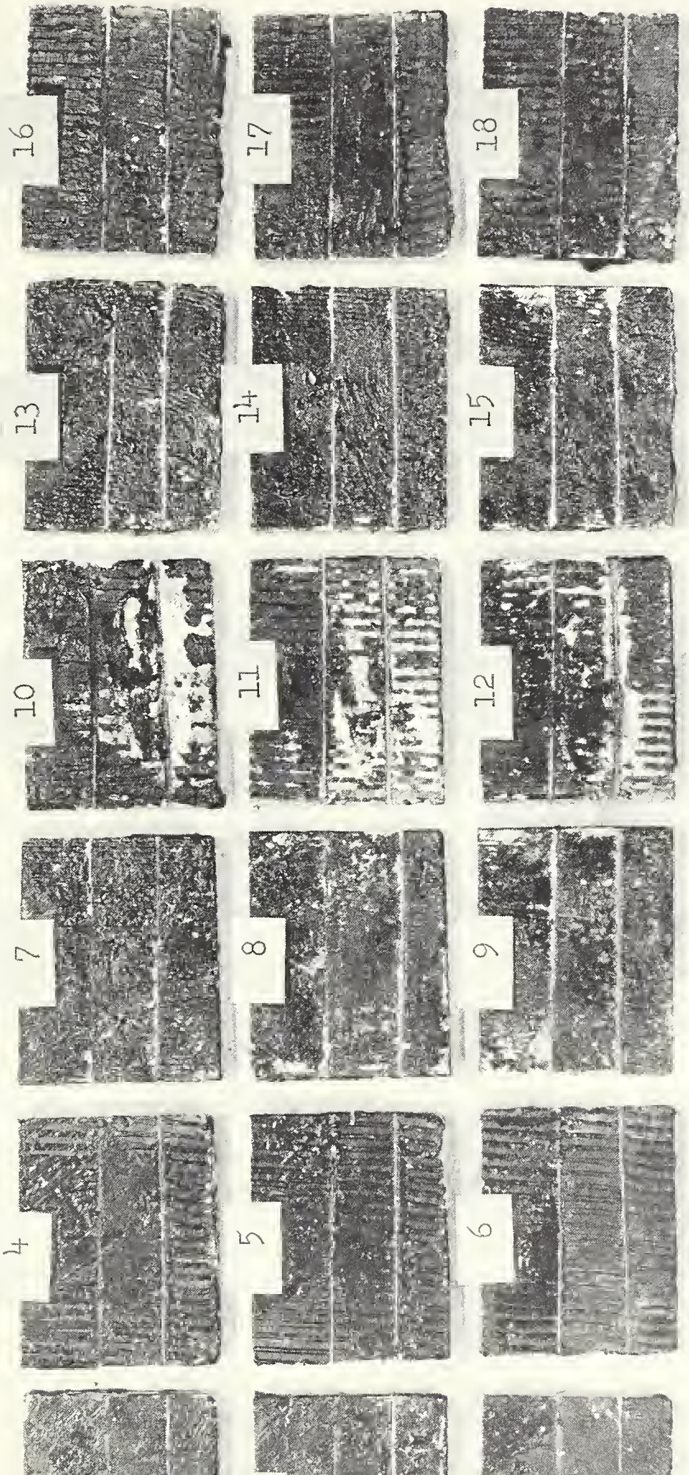
4.2.2 Quantitative Straight-pull Tests with Asphalt Tile and Cement Mortar Cubes (See 3.2.2)

The data from the quantitative straight-pull tests are summarized in Table 3, which was prepared with the assistance of Dr. David Hogben of the Statistical Engineering Section, using an OMNITAB computer.

Analysis of variance of the test results showed no significant differences between brands, but possibly a significant difference between asphalt cut-back and emulsion adhesive (Table 3a).

From Table 3b it is apparent that increased bonding pressure causes considerable increase in bond strength of asphalt emulsion adhesive, but does not affect asphalt cut-back adhesive. Longer curing time up to 30 days increases the bond strength of asphalt emulsion adhesive when bonded at 625 psi, but not when bonded at only 0.2 psi. Asphalt cut-back adhesive shows very low bond strength after one day curing; this is increased considerably after 14 days with little change after 30 days.

According to Table 3c, all membrane curing agents lessen the adhesion of asphalt emulsion adhesive, although curing agent N is not significantly different and M is only barely significant. Curing agents A, B, C, D, E, and F definitely lessen adhesion of asphalt emulsion adhesive to concrete. In the case of asphalt cut-back adhesive, curing agent B actually shows better adhesion. There is no significant change with curing agents A, C, D, E, F, M, and N. This confirms previous observations that curing agents are generally compatible with asphalt cut-back adhesive and not with asphalt emulsion adhesive.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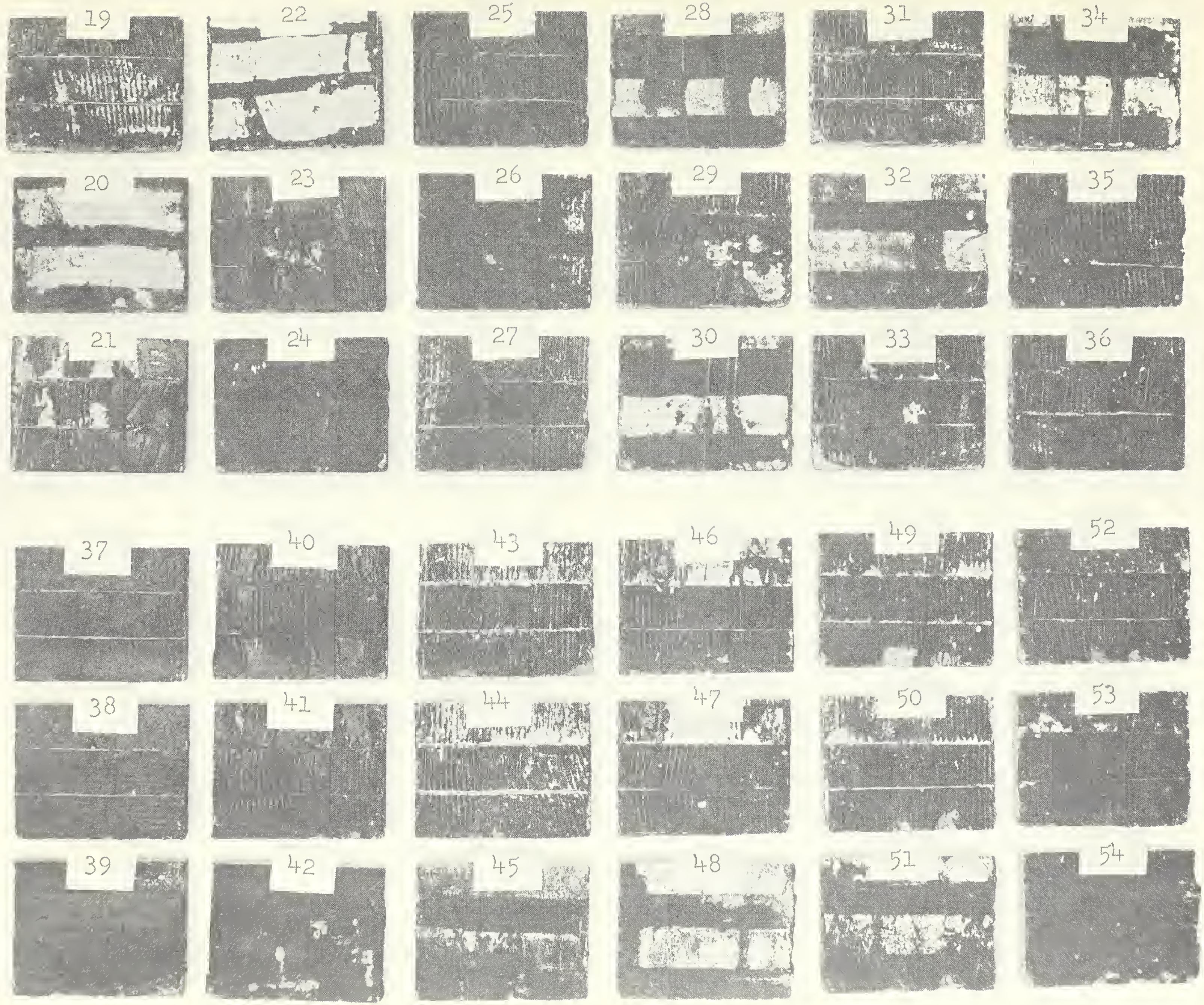
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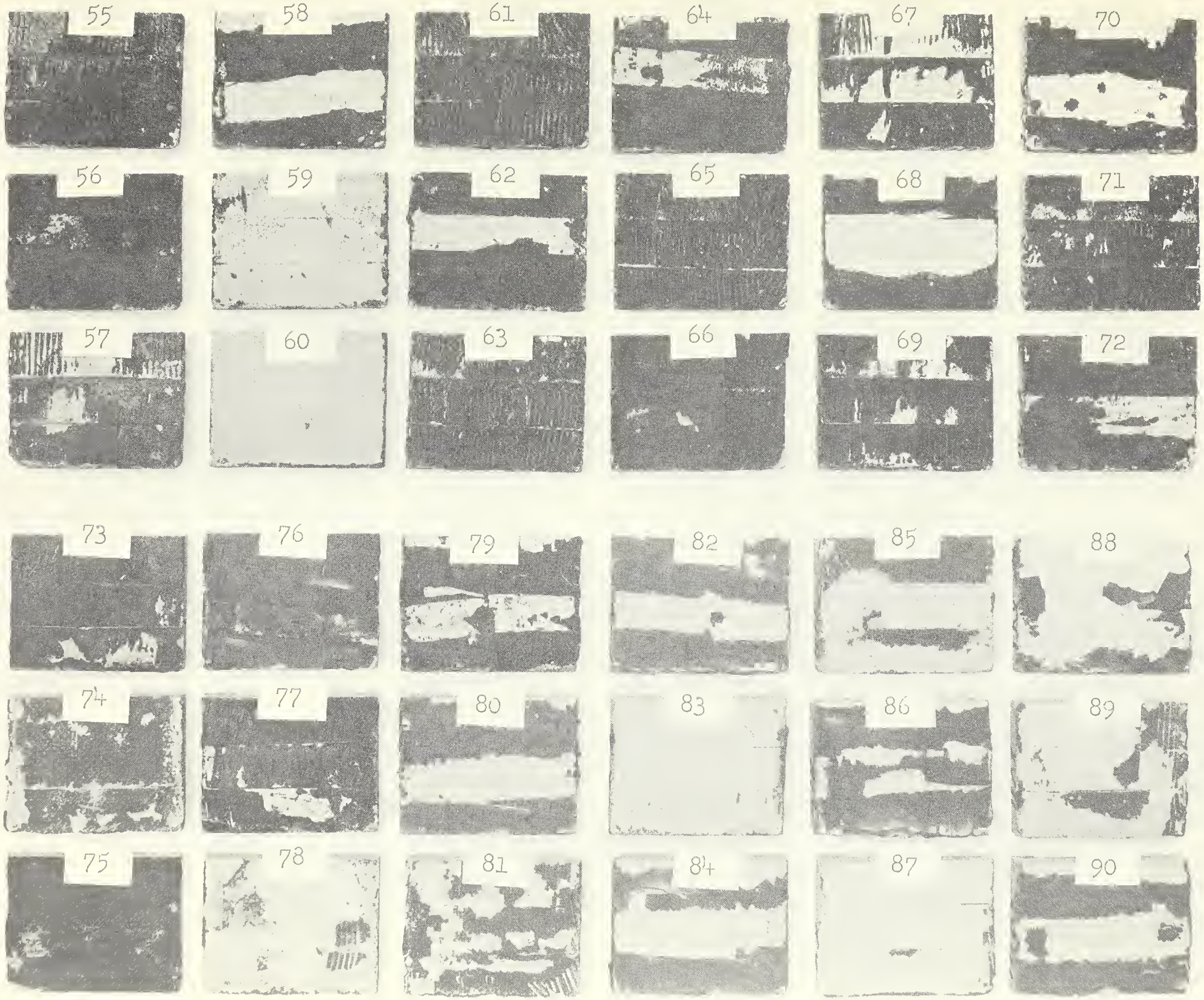
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Table 2.

Panel No.	Adhesive Asphalt	Concrete Treatment	Remarks
1,2,3	Cut-back	Water cured	Adhesion excellent - no bare spots
4,5,6	Emulsion	Water cured	Adhesion excellent
7,8,9	Cut-back	Paraffin wax and kerosene mixture	Some bare spots indicating loss of adhesion
10,11,12	Emulsion	Paraffin wax and kerosene mixture	Large areas of bare concrete indicating poor adhesion
13,14,15	Cut-back	Chlorinated rubber-chlorinated paraffin	Some bare spots but adhesion very good on the whole
16,17,18	Emulsion	Chlorinated rubber-chlorinated paraffin	Adhesion very good
19	Emulsion	Left half water cured and right half treated with paraffin wax and kerosene mixture	Adhesion excellent after 14 days. Considerable bare concrete after 30 & 90 days. Little difference between treated & untreated halves
21	Emulsion	Left half water cured and right half treated with chlorinated rubber-chlorinated paraffin	Large bare areas, mostly on water cured half after 14 and 30 days. Adhesion excellent after 90 days on both halves.
23	Emulsion	Left half water cured and right half treated with commercial curing agent B	Adhesion excellent except for bare spots in center on both halves after 30 days

Table 2. (Cont'd) - 2

Panel No.	Adhesive Asphalt	Concrete Treatment Initial	Concrete Treatment Subsequent	Remarks
25	Emulsion	Left half water cured and right half treated with commercial curing agent C	None	Adhesion excellent over entire panel
27	Cut-back	Water cured, then four coats of paste wax	None	Adhesion fairly good with some streaks of bare concrete
29	Emulsion	Water cured then four coats of paste wax	None	Adhesion good after 14 days with few bare spots. Large areas of bare concrete after 30 & 90 days
31	Cut-back	Water cured, then four coats of paste wax	Stripped with xylene	Adhesion fairly good with some bare spots
33	Emulsion	Water cured, then four coats of paste wax	Stripped with xylene	Adhesion fair with bare spots scattered over the panel at 14, 30 & 90 days
35	Emulsion	Water cured, then four coats of paste wax	Sanded, then coated with asphalt primer	Adhesion excellent over entire panel
36	Emulsion	Water cured, then four coats of paste wax	Coated with asphalt primer	Adhesion excellent over entire panel
37,38	Cut-back	Water cured	None	Adhesion excellent over entire panels
40,41	Emulsion	Water cured	None	Adhesion excellent

Table 2. (Cont'd) - 3

Panel No.	Adhesive Asphalt	Concrete Treatment	Remarks
43,44	Cut-back	Commercial curing agent C	In both panels, adhesion was rather poor after 10 days, with a number of bare spots and streaks. Adhesion fair after 30 & 90 days, with occasional bare spots
46,47	Emulsion	Commercial curing agent C	Poor adhesion after 10 days with large bare area. Good adhesion after 30, 90 days with few bare spots
49,50	Cut-back	Commercial curing agent B	In both panels, adhesion was fair to poor after 10 days and good after 30 days. After 90 days there were some bare spots but these appeared to be due to concrete being torn from the surface by removal of tile, indicating a good bond
52,53	Emulsion	Commercial curing agent B	Fair adhesion after 10 days with some bare spots. Good adhesion after 30, 90 days with a few bare spots
55	Emulsion	Water cured	Adhesion excellent
57	Emulsion	Commercial curing agent D	Poor adhesion, especially after 10 days. Somewhat better adhesion with less bare concrete after 90 days
59	Emulsion	Commercial curing agent O	Very poor adhesion. Panel practically bare.
61	Emulsion	Commercial curing agent P	Adhesion excellent. A few bare spots after 10 days

Table 2. (Cont'd) - 4

Panel No.	Adhesive Asphalt	Concrete Treatment	Remarks
63	Emulsion	Commercial curing agent Q	Adhesion fair after 10 days; considerable bare area. Adhesion good after 30, 90 days; a few small bare spots
65	Emulsion	Commercial curing agent J	Adhesion very good with only a few scattered bare spots
67	Emulsion	Commercial curing agent R	Very good adhesion after 10, 30 days. Better after 90 days but large bare spot
69	Emulsion	Commercial curing agent L	Adhesion poor especially after 10 days
71	Emulsion	Piccopale 100	Adhesion poor after 10, 30 days and fair after 90 days
73	Emulsion	Water cured	Adhesion good after 3 days; fair after 30 days; poor after 90 days
77	Emulsion	Pliolite S-5	Adhesion good after 3 days; poor after 30 days; very poor after 90 days, with large area of exposed concrete

Table 3. Adhesion of Asphalt Tiles Using Asphalt Adhesives

3a. Comparison of Three Brands Each of Asphalt Cut-Back and Emulsion Adhesive

	Brand A	Brand B	Brand C
Asphalt Cut-back Adhesive			
Number of Tests	11	11	11
Average maximum load, pounds	26.7	25.0	29.0
Standard Deviation	21.82	20.44	17.48
Asphalt Emulsion Adhesive			
Number of Tests	11	12	11
Average maximum load, pounds	19.5	19.7	20.1
Standard Deviation	8.51	11.60	8.32

Analysis of variance showed no significant difference between brands.

3b. Comparison of Adhesives, Curing Time of Adhesives, and Pressures Applied to Tiles After Laying

Pressure on Tile	Adhesive	Curing Time of Adhesive	No. of Tests	Average Max. Load	Std. Dev.
psi	asphalt	days		lbs.	
625	Emulsion	30	12	163.2	64.51
625	Emulsion	14	12	112.3	53.61
625	Emulsion	1	12	71.8	32.05
625	Cut-back	30	12	65.5	38.47
0.2	Cut-back	14	6	63.8	34.48
0.2	Cut-back	30	12	62.2	33.81
625	Cut-back	14	12	57.8	31.37
0.2	Emulsion	30	12	27.6	11.52
0.2	Emulsion	14	6	14.3	10.01
0.2	Emulsion	1	12	11.2	7.65
625	Cut-back	1	12	7.5	9.67
0.2	Cut-back	1	11	4.0	6.47

NOTE: There are no significant differences between values opposite the same bar. Values outside a bar are significantly different at 5% level from values inside the bar.

Table 3. (Cont'd) - 2

3c. Effect of Membrane Curing Agents on Adhesion of Asphalt Tile to Concrete

Adhesive	Treatment	No. of Tests	Average Max. Load	Standard Deviation
asphalt			lbs.	
Emulsion	Control	12	163.2	64.57
Emulsion	N	6	158.0	48.91
Emulsion	M	6	78.2	53.81
Cut-back	B	9	72.8	25.56
Cut-back	Control	12	65.5	38.47
Cut-back	C	9	44.9	26.15
Cut-back	N	6	42.0	30.56
Cut-back	M	6	37.2	27.24
Cut-back	D	9	30.8	17.30
Cut-back	E	9	30.7	13.74
Cut-back	A	9	30.7	17.21
Cut-back	F	9	27.2	23.73
Emulsion	F	9	21.8	19.46
Emulsion	D	9	14.3	14.11
Emulsion	C	9	11.4	16.79
Emulsion	E	9	10.3	9.25
Emulsion	B	9	9.8	6.22
Emulsion	A	9	7.9	7.51

3d. Comparison of Rough and Smooth Surfaces of Asphalt Tile

	Asphalt Cut-back Adhesive: Surface of tile in contact with adhesive		Asphalt Emulsion Adhesive: Surface of tile in contact with adhesive	
	Smooth	Rough	Smooth	Rough
Number of Tests	6	5	6	6
Average maximum load, pounds	119.0	143.2	183.3	176.3
Standard Deviation	15.06	19.52	28.26	20.58

Analysis of variance showed significant difference at 1% level between asphalt cut-back and emulsion adhesives but no significant difference between rough and smooth sides of asphalt tile.

In Table 3d are recorded results of experiments in which all of the specimens were subjected to 625 psi for 30 seconds. In one set of tests the smooth sides of the tiles were in contact with the asphalt adhesive and in the other set of tests, the rough sides of the tiles. Statistical analysis showed a significant difference between asphalt cut-back and emulsion adhesives but not between the smooth and rough sides of the asphalt tile.

4.2.3 Quantitative Stripping-pull Tests with Flexible Resilient Tile and Cement Mortar Prisms (See 3.2.2)

In the stripping-pull tests, ten replicates were run at a crosshead speed of 0.5 inch per minute on specimens in which asphalt cut-back adhesive was used. Another ten replicates were run at the same crosshead speed with asphalt emulsion adhesive. Eight replicates were performed at a crosshead speed of 2.0 inches per minute on asphalt cut-back and eight on asphalt emulsion adhesive. All specimens showed good adhesion from qualitative or visual inspection. Both tile and concrete surfaces remained coated with adhesive except for portions where the concrete surface was removed by pulling away the strip. This was shown by a whitish or grayish deposit on the underside of the tile, corresponding to bare portions on the concrete surface. There was very little difference in maximum load on any of the specimens. The load was about 1-2 pounds in each case, never zero and never over 2 pounds.

5. SUMMARY AND COMMENTS

A number of tests have been performed under this program on adhesion of organic coatings and resilient flooring to test panels and blocks, which were made to simulate actual concrete floors as closely as possible. The panels and blocks were made of a cement mortar mix and were not concrete in the strict sense of the word. The physical dimensions of the specimens were too small to permit the addition of aggregate. However, concrete sand was used and the specimens were hard troweled, so that the top surfaces should be similar to those of actual concrete floors.

Our tests have established that most concrete curing agents can be used safely under asphalt or vinyl asbestos tile where asphalt cut-back adhesive is used. When an asphalt emulsion adhesive is used, on the other hand, oily or waxy materials may cause trouble unless an asphalt primer is used. The Rubber Manufacturers Association, as well as prominent flooring manufacturers, are of the opinion that other types of resilient flooring, such as flexible vinyl and rubber tile, behave similarly.

Adhesion of cement-water paints was poor to concrete treated with curing agents, as these paints require a somewhat porous hydrophilic surface. Adhesion of other types of paints was excellent to curing agents formulated with butadiene-styrene type copolymer or chlorinated rubber-chlorinated paraffin type of formulation. Other types of curing agents varied regarding their compatibility with various paints. In general, emulsion or latex paints did not adhere well to concrete floors treated with oily or waxy materials. Organic solvent based paints were generally satisfactory when applied over oils and waxes. Paints formulated with butadiene-styrene copolymer or chlorinated rubber in organic solvents (such as xylene, mineral spirits or petroleum distillate) appeared to have the best adherence.

The most practical laboratory bond or adhesion test for resilient flooring is qualitative or visual such as the one recommended by the Armstrong Cork Company. Quantitative bond tests require expensive equipment, are slow, and require a relatively large number of replicates. Even with the most careful quantitative tests, it is doubtful whether any more information can be gained than from a qualitative bond test. The qualitative test for adhesion of floor coverings reported here is to apply pieces of floor covering to cement mortar panels which have been water cured or cured with membrane curing agents. The floor covering material is cut into strips, using a special tip inserted in an electric soldering gun. The test for adhesion consists in pulling back a strip of material and observing whether the concrete surface remains coated with adhesive. Strips are removed at various intervals of time and then the surface under the removed strips is photographed. More reliable results are obtained with large panels in which nine 9- by 9-inch tiles are applied to a cement mortar panel. This type of test was described in our Second Progress Report, NBS Report No. 8009, 30 June 1963.

We have attempted to develop a field test for adhesion, but such a test does not appear to be feasible. The only practical field test is to apply the floor covering or paint at various areas on the surface to be covered and examine visually. Inasmuch as our investigation has shown that asphalt cut-back adhesive can be used successfully over most types of concrete treatment the need for a field test is not very important. Further, if the use of an emulsion adhesive is desired, an asphalt primer can be used as a base over oily or waxy curing agents to provide good adhesion.

Parting agents are oily materials used to separate slabs in lift-slab and tilt slab construction. These are similar to the oily type of curing agents, which are commonly advertised for both curing and parting. Since there is nothing unique about parting agents, they were not studied extensively. The only widely used parting agent of which we have any knowledge is code K.

So-called concrete sealers and hardeners are generally similar to the resin type of curing agents and the same product is generally advertised to be a combination curing agent, sealer, and hardener. An organic coating may be used as a sealer in the sense that an improperly cured concrete surface is characterized by "laitance" or dust and the organic coating or sealer will cement this laitance to the surface until the coating is worn off or tile or paint applied. Some alkaline "hardeners" are used, such as sodium silicate, code M, which is worthless as a curing agent because it is ineffective as a water barrier.

6. ACKNOWLEDGMENT

The author wishes to acknowledge the assistance of Dr. David Hogben in the statistical analysis of the data on straight-pull tests, using the Instron tensile testing instrument.

APPENDIX 1. APPLICABLE TEST METHODS

1.1 Tests for Oily and Waxy Curing Agents on Concrete Floors

We have tried to develop several methods of test to detect the presence of oily and waxy materials on concrete floors simply and rapidly in the field. None of these methods were entirely satisfactory. The only one which shows any promise is as follows:

> Scrape an area about 3- by 5-inches with a pen knife and brush the scrapings into a small beaker or test tube. Add three medicine droppers full of reagent diethyl ether and swirl to contact the scrapings and ether. Filter through qualitative (Whatman No. 1) filter paper in a small funnel onto a carefully cleaned watch glass about 100 mm. in diameter. Repeat this procedure with the same amount of ether from the same bottle. Add six medicine droppers of ether from the same bottle to another clean watch glass. Allow the ether to evaporate overnight in a place as dust free as possible. Examine the watch glasses for oily or waxy residue.

This procedure was tried on 3- by 5-inch cement mortar panels. Both panels had been treated with paraffin wax in kerosene mixture. One of the panels had also been given three coats of paste wax. In both cases, a waxy residue was visible on the watch glass, more in the one from the waxed panel. The watch glass in which ether alone had been evaporated was fairly clean.

Another simple test was tried on two similar panels, both treated with paraffin wax and kerosene mixture and one also waxed with paste wax. This consisted in placing a piece of qualitative filter paper on each panel and pressing with a household iron, set at maximum temperature, to see whether the oil and wax would change the appearance of the paper. However, there was no detectable change in appearance.

Other tests which have been tried include measuring contact angles or observing the wettability of concrete surfaces, treated and untreated, and drilling into cement mortar panels with a masonry drill and extracting with solvent. These methods were unsuccessful.

1.2 Water Retention of Concrete Curing Agents

Although this subject is outside the scope of the project, it cannot be ignored when we are asked to make recommendations concerning concrete curing agents. There are three known tests and requirements for moisture retention of concrete curing agents. According to ASTM C-309, a curing agent should restrict the loss of water to not more than 0.055 g/cm^2 of surface, when tested according to ASTM C-156. According to U. S. Army Engineers CRD-C 300-59, the unit moisture loss shall not be more than 0.031 g/cm^2 under conditions set forth in this specification. The Concrete Manual of the U. S. Department of the Interior, Bureau of Reclamation, Denver, Colorado, does not specify moisture retention required of concrete curing agents. However, Mr. G. E. Burnett, Chief Research Scientist of the Bureau of Reclamation at Denver, has given NBS some information regarding their work on moisture retention of concrete curing agents and a copy of their "Specifications for Sealing Compound for Curing Concrete", dated June 1, 1961. Details of the test method and moisture retention requirement are given in this specification. The moisture loss specified is 30 grams per 72 sq. in. or 0.064 g/cm^2. The Bureau of Reclamation has tested about 40 different commercial concrete curing agents, none of which passed their moisture retention requirement. However, chlorinated rubber formulations were close to the requirement.

APPENDIX 2. REPORT ON CONCRETE DURING AGENTS FROM THE RUBBER MANUFACTURERS ASSOCIATION

A comprehensive study of concrete curing agents has been made by the Technical Committee, Vinyl and Rubber Flooring Division, Rubber Manufacturers Association, Inc., 444 Madison Avenue, New York, N. Y. 10022 (RMA). The basis of the study was NAVDOCKS Specification 13Yf, Section 2.13.7, previously mentioned in this report. The test method specified in Specification 13Yf was used in the RMA laboratory investigations. The Portland Cement Association prepared test concrete blocks in their laboratory in Skokie, Illinois, and forwarded these blocks to six manufacturers of vinyl and rubber flooring, who conducted tests in their own laboratories. Some of the blocks were water cured and some membrane cured. The membrane cured blocks were wrapped in polyethylene bags during the curing period, as the membrane coating was applied only to the top surface and excessive moisture would otherwise be lost from relatively small specimens.

In addition, the RMA cooperated with other interests in a field study in which a new building was used as a test site. The test building is the Cerebral Palsy Treatment Center in Edison, New Jersey. Of 12,000 square feet of concrete flooring in this building, half was water cured and half was cured with commercial curing agent B. The floors were finished and cured during the summer of 1961. Vinyl and rubber flooring was installed beginning in September, 1961 and completed during the summer of 1962. Recent inspection, about two years after all of the flooring had been installed, showed all of the flooring to be in perfect condition. There has never been any loss of adhesion or any other flooring problem in the entire installation.

APPENDIX 3. IDENTIFICATION OF COMMERCIAL PRODUCTS

3.1 Curing Agents, Etc. - Information obtained by analysis or from manufacturers

B. CURING AGENT - Xylol solution containing about 18% of butadiene-styrene copolymer resin. (Resin similar to "Pliolite S-5", product of Goodyear Tire and Rubber Co.)

C. CURING AGENT - Xylol solution containing about 28% of a petroleum hydrocarbon resin. (Resin similar to "Piccopale 100", product of Pennsylvania Industrial Chemical Corporation.)

D. CURING AGENT - Composition unknown.

E. CURING AGENT - Composition unknown.

(Materials coded F, G, H, and I were prepared at the Bureau, but are included here for completeness.)

F.

Material	Parts by Weight
Butadiene-styrene copolymer resin (Pliolite S-5, Goodyear)	18
Xylene, reagent grade	82
	100

G.

Material	Parts by Weight
Petroleum hydrocarbon resin (Piccopale 100, Pennsylvania Industrial Chemical Corp.)	30
Mineral spirits	70
	100

H.

Material	Parts by Weight
Chlorinated rubber (Parlon S-10, Hercules)	12.5
Plasticizer (Arochlor 5460, Monsanto)	10.0
Chlorinated paraffin (Chlorafin 40, Hercules)	2.5
Xylene, reagent grade	75.0
	100.0

I. Material — Parts by Weight

Material	Parts by Weight
Paraffin, U.S.P., melting point 52-4°C	17
Kerosene, commercial grade	83
	100

J. CURING AGENT - Composition unknown.

K. CURING AND PARTING AGENT - Kerosene solution containing about 13% paraffin wax.

L. CURING AGENT - Composition unknown.

M. CURING AGENT - Sodium silicate solution.

N. 40% solution of sodium silicate prepared at the Bureau.

O. PARTING AGENT - Mixture of heavy oils, mineral filler, and a small amount of a medium boiling solvent.

P. EXPERIMENTAL PARTING AGENT - Mainly heavy oils, somewhat similar to O, but without mineral filler.

Q. CURING AGENT - Composition unknown.

R. CURING AGENT - Composition unknown.

S. EXPERIMENTAL CURING AGENT -

Material	Parts by Weight
Polyvinyl toluene-butadiene copolymer resin	15.0
Paraffin wax	1.5
Aluminum stearate	1.5
Naptha	82.0
	100.0

T. CURING AGENT - Composition unknown.

3.2 Concrete Paints - Information obtained from label

I. CEMENT WATER TYPE

Material	By Weight
Hydraulic calcium silicates and aluminates	70.0%
Calcium hydroxide	25.0%
Sodium chloride	5.0%
	100.0%

Tinting pigments not over 5%; T-V-26-R-3

II. CEMENT WATER TYPE

Material	By Weight
Silicon oxide	22.0%
Aluminum oxide	4.0%
Calcium oxide	64.0%
Magnesium oxide	2.0%
Calcium stearate	1.0%
Sodium chloride	4.0%
Calcium sulfate	3.0%
	100.0%

Tinting materials less than 1%

III. CHLORINATED RUBBER IN SOLVENT

Pigment (22% by weight)	
Titanium dioxide	100.0%
Vehicle (78% by weight)	
Chlorinated rubber	23.0%
Plasticizer	10.0%
Esters	18.0%
Aliphatic, aromatic hydrocarbons	49.0%
	100.0%

IV. BURADIENE-STYRENE COPOLYMER IN SOLVENT

Pigment (48% by weight)	
Silicates	58.0%
Titanium dioxide	42.0%
	100.0%
Vehicle (52% by weight)	
Non-volatile	
Butadiene-styrene resin	28.0%
Volatile	
Aromatic solvents	40.0%
Mineral spirits	32.0%
	100.0%

V. BUTADIENE-STYRENE AND ACRYLIC RESIN EMULSION

Pigment (23.3% by weight)

Titanium dioxide	90.0%
Aluminum silicates	10.0%
	100.0%

Vehicle (76.7% by weight) (blend of butadiene-styrene and acrylic type copolymers)

Non-volatile (synthetic rubber)	24.7%
Volatile (water)	75.3%
	100.0%

VI. BUTADIENE-STYRENE COPOLYMER EMULSION

	By Weight
Titanium dioxide	18.0%
Barytes	5.0%
Silica and silicates	8.0%
Mineral spirits	7.0%
Synthetic latex solids	22.0%
Volatile thinner (water)	40.0%
	100.0%

VII. POLYVINYL ACETATE EMULSION

	By Weight
Titanium dioxide	14.60%
Silica	7.36%
Calcium carbonate	15.40%
*Polyvinyl acetate emulsion	29.00%
Water	33.64%
	100.00%

*Non-volatile (Polyvinyl acetate)	42.0%
Volatile (water)	58.0%
	100.0%

Tinting color less than 5%

VIII. ALKYD MODIFIED VINYL RESIN EMULSION

Pigment (45.1% by weight)

Titanium dioxide	43.9%
Silicates	56.1%
	100.0%

Vehicle (54.9% by weight) (*Alkyd modified vinyl resin emulsion)

*Non-volatile - Soya alkyd modified vinyl resin	20.8%
Volatile (water)	79.2%
	100.0%

IX. OIL-ALKYD MODIFIED LATEX PAINT

Pigment (37% by weight)

Titanium dioxide	50.0%
Magnesium and aluminum silicates	43.0%
Silica	7.0%
	100.0%

Vehicle (63% by weight)

(Pentaerythritol-Soya Ester-Resin Latex Emulsion)

Non-Volatile resin	9.5%
Non-Volatile synthetic latex	12.5%
Volatile (water)	78.0%
	100.0%

X. ACRYLIC RESIN EMULSION

Pigment (32.9% by weight)

Titanium dioxide	70.4%
Silica and silicates	29.6%
	100.0%

Vehicle (67.1% by weight)

Non-volatile (acrylic resin emulsion)	23.5%
Volatile (water)	76.5%
	100.0%

XI. ALKYD ENAMEL

Pigment (45% by weight)

Titanium calcium	63.0%
Titanium dioxide	20.0%
Silicates	17.0%
	100.0%

Vehicle (55% by weight)

*Varnish	85.0%
Mineral spirits	15.0%
	100.0%

*Non-Volatile (Linseed Maleic Alkyd Resin)	58.0%
Volatile (mineral spirits)	42.0%
	100.0%

XII. ALKYD ENAMEL

Pigment (27.6% by weight)

Titanium dioxide	53.3%
Calcium carbonate	46.5%
Carbon black	0.2%
	100.0%

Vehicle (72.4% by weight)

Soya alkyd resin, Tung oil, Linseed oil, Ester gum	52.8%
Mineral spirits and driers	47.2%
	100.0%

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