Fifteen-Year Exposure Test of Porcelain Enamels

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Building Materials and Structures Report 148

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Fifteen-Year Exposure Test of Porcelain Enamels

Dwight G. Moore and William N. Harrison

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The weather resistance of 768 panels representing 14 types of enamel was evaluated after 15 years of exposure at Washington, D. C., St. Louis, Mo., Lakeland, Fla., and Atlantic City, N. J. Changes in gloss and color were taken as criteria of weather resistance. A direct relation existed between acid resistance and weather resistance, except that a few of the red enamels of good acid resistance showed a pronounced fading after exposure. A modified acid-resistance test was devised that separated red enamels that showed pronounced fading from those that were highly resistant to color change.

The ease of cleaning was related to the weather resistance, the enamels that lost most of their initial gloss on weathering being more difficult to clean than those that showed high gloss retention. All of the enamels protected the steel from corrosion when initial coverage was complete.

Except for a relatively few enamels, variations in climate at the four exposure sites had only a minor effect on their weathering behavior.

1. Introduction

Porcelain enamel is being used in increasing volume as an exterior finish for such structures as office buildings, store fronts, and gasoline filling stations [1,2].* Curtain-wall construction methods together with the emphasis on color in modern architecture have helped to popularize this particular finish. In fact, probably because of this trend toward color, porcelain enamel has been used to a greater extent than either stainless steel or aluminum as an exterior facing for buildings [2]. The relative ease of decontaminating an enamel surface in case of atomic attack is an additional consideration in some types of structures [1].

Another reason for the increasing use of porcelain enamel is the excellent resistance to weathering shown by some of the early installations. For example, there are reliable reports of street and advertising signs and of building fronts, that have been installed for 25 yr and longer without noticeable deterioration. On the other hand, there have been occasional installations where both the gloss and color of the finish have changed appreciably in as short a time as 10 yr. To prevent future installations of enamels of poor resistance, it is desirable to have data on the weather resistance of various enamel types and it is also important to devise a test that will indicate weather resistance.

A test to obtain such information was begun by the National Bureau of Standards in 1940. A total of 864 panels, 1 ft square, and an equal number of 4-by 6-in. laboratory specimens, were prepared by 12 cooperating manufacturers. Of the 864 large panels, 768 were exposed and 96 were placed in storage for use as reference standards. The base metal in each case was enameling iron. The exposure sites selected were Washington, D. C., St. Louis, Mo., Lakeland, Fla., and Atlantic City, N. J.

Fourteen types of enamel were included. These types represented enamels that were in common use from 1930 to 1940. Shortly after World War II, enamels opacified with titanium dioxide came into widespread use for white and pastel finishes. These enamels are considerably different from the acid-resistant white and buff compositions included in the present investigation. However, because one of the principal objectives of study was to correlate weather resistance with some more easily measured property of the enamel finish, the obsolescence of some of the compositions does not necessarily detract from the value of the data.

The present paper constitutes the third progress report of the investigation. The first report was published in 1942 [3] and the second in 1949 [4]. It was originally intended that the test would be terminated after 15 yr, but, because many of the enamels showed only a minor deterioration after this testing period, it was decided to expose the Washington panels until a testing time of possibly 25 or 30 yr had been accumulated. However, mostly because of difficulties in providing proper maintenance, the panels from Lakeland, St. Louis, and Atlantic City were returned to Washington and the testing at these locations was terminated.

2. Types and Sources of Enamel

The enameled panels were supplied by the 12 cooperating companies who were active in the field of architectural enamels in 1939. The frits[1] and various mill additions[2] for preparing the enamels were supplied by 4 frit companies. Each of these 4 companies furnished the materials for each of the 14 enamel types to 1 or 2 of the 12 different fabricators, who then applied the enamels to the specimens. By this arrangement, it was possible to introduce the two variables of frit source and fabricator. The frits were all proprietary products for which no chemical analyses were available. It is probable that there were at least minor variations in composition for any 1 enamel type as supplied by the 4 frit companies.

*Figures in brackets indicate the literature references at the end of this report.

[1] Frit is the principal ingredient used in preparing porcelain enamels. It is formed by melting suitable raw materials and then quenching the molten mass, usually by pouring into cold water.

[2] In the preparation of an enamel for application to sheet iron, the enamel frit is ball-milled together with such ingredients as clay, opacifier, color oxide, electrolyte, and water. The materials added at the mill constitute the mill additions.

Likewise, each fabricator produced an enamel of slightly different properties from the same frit because of minor variations in the mill batch, the milling procedure, and the firing conditions. Thus, although the investigation included nominally only 14 types, there were, in effect, 96 enamels under study.

3. Description of Panels, Method of Mounting, and Weather Conditions at Exposure Sites

The panels, which were 1 ft square, were fabricated of 16-gage iron and had 1-in. flanged edges. The flange of the lower side had a ½-in. outward extension parallel to the face of the panel. Two clips made of 1-in. strap iron were welded to the top flange so as to extend downward (see fig. 1). The clips and the lower flange extension fitted into 18-gage galvanized-iron channels, which in turn were firmly attached to the supporting racks. The crevices between the specimens were not calked but were left open to facilitate removal of the panels during periods of inspection.

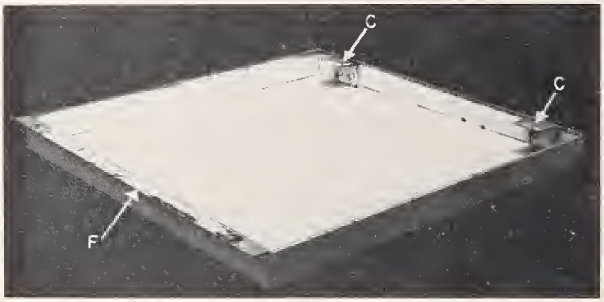

FIGURE 1. *Reverse side of an exposure panel.*

C, Attachment clips; F, lower flange extension used for fastening panels to racks.

The supporting racks were constructed of 3/16-in. angle iron and, after priming, were painted periodically with aluminum paint. Each rack was constructed to support 28 of the 1-ft-square panels. Seven racks were required for each location. At those locations where the panels were exposed on flat roofs (see table 1), the racks were anchored with weights, and at the ground locations the racks were anchored to piers of concrete blocks. Figure 2 shows the installation at Washington, D. C.

TABLE 1. *Exposure-test locations*

City	Exposure site	Exposure conditions represented
Washington, D. C.	Roof, Industrial Bldg., National Bureau of Standards.	Temperate, residential.
St. Louis, Mo.	Roof, Union Electric Co. Warehouse.	Temperate, industrial.
Lakeland, Fla.	Ground, Municipal Airport	Semitropical, residential.
Atlantic City, N. J.	Ground, U. S. Coast Guard Station.	Temperate, "salt air."

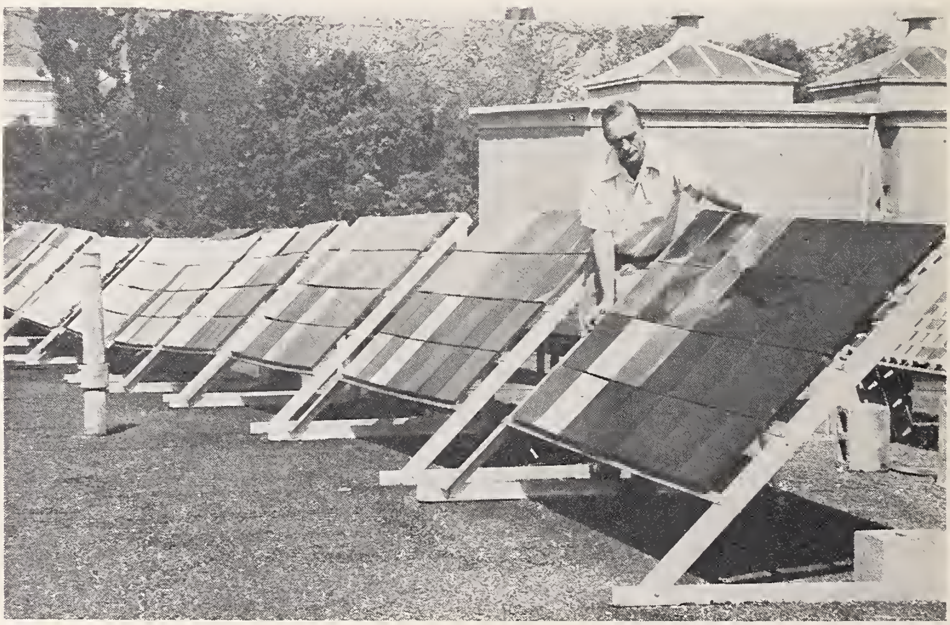

FIGURE 2. *Exposure-test installation on the roof of the Industrial Building at the National Bureau of Standards.*

Table 1 lists the locations and general conditions of exposure. Table 2 gives weather data for the actual period of exposure. At all four locations the racks faced south, the panels being exposed at 45° to the horizontal.

Table 2. *General weather data for the 15-yr exposure period (from U. S. Weather Bureau Records)*

City	Exposure Period	Annual rainfall [a]	Annual sunshine [a]	Average temperature [a]
		in.	*hr*	° *F*
Washington, D. C.	Dec. 1939 through Sept. 1955.	42.0	2,584	57.6
St. Louis, Mo.	Apr. 1940 through Oct. 1955.	36.3	2,718	58.2
Lakeland, Fla.	July 1940 through Nov. 1955.	47.8	[b] 2,878	72.8
Atlantic City, N. J.	Aug. 1940 through Nov. 1955.	40.0	2,675	55.6

[a] Average computed for actual period of exposure.
[b] Taken from Tampa, Fla., records. Average annual sunshine for Lakeland not available.

4. Results

4.1. Ease of Cleaning

At the beginning of the investigation it was planned to clean the panels prior to making gloss and color measurements by washing the test surface with a 1-percent-by-weight solution of trisodium phosphate. This cleaning procedure was satisfactory for the first year's inspection [3], and at all locations except St. Louis for the inspection after 7 yr [4]. However, it was not practicable to clean the panels between inspections, and, as a result, both the Lakeland and Atlantic City panels, in addition to those from St. Louis, had accumulated surface films after the 15-yr exposure that could not be removed by washing with the trisodium phosphate solution. The film on some of the Lakeland panels was chalky in nature, whereas rust stains were present on many of the Atlantic City panels. The film on the St. Louis panels consisted of a fairly heavy deposit of fly ash and soot bonded with a tar-like substance. Gloss- and color-difference measurements made on such surfaces would be meaningless; therefore, all panels, including those at Washington, were cleaned prior to the 15-yr measurements by scrubbing with a commercial scouring powder. It was believed that this cleaning procedure was justified inasmuch as it followed closely the practice of commercial sign-cleaning services. The procedure followed in the cleaning was to continue the scouring until further treatment caused no appreciable change in the appearance of the enamel surface. In most cases, it was found that the surfaces reached substantially constant values of gloss and color by the time the cleaning was adjudged satisfactory by visual examination.

The ease of cleaning varied with the acid resistance of the enamel, the surfaces of high acid resistance being easier to clean than those of poor acid resistance. The semimat or satin-textured enamels showed about the same cleaning behavior as the glossy surfaces after weathering. On the other hand, none of the full-mat enamels could be cleaned satisfactorily even by a vigorous and prolonged scouring treatment.[3]

4.2. Corrosion Protection

All of the panels were inspected for evidence of corrosion. Where the initial coverage was complete on all parts of the panel no corrosion was noted, irrespective of the type of enamel applied. However, on many panels the under side was protected with only a single groundcoat application, and good coverage of the metal was not always achieved. Corrosion started at these areas of poor coverage, and spalling of the enamel on the face side occurred after the corrosion had progressed only part way through the thickness of steel.

Hydrogen originating from the action of condensed moisture on the unprotected steel is believed responsible for the spalling. Norton [5] has shown by a tracer technique that the hydrogen generated from such a reaction will permeate the steel structure at room temperature, and numerous investigators, including Zappfe and Sims [6], have demonstrated that whenever hydrogen diffuses through steel, sufficient pressure can be generated to rupture the enamel on the opposite face. Thus, it appears entirely reasonable that the observed spalling behavior could be caused by hydrogen diffusion.

The surface spalls, which resembled large fish-scales, were noted on only a few of the panels at Atlantic City at the 7-yr inspection. However, after 15-yr, the majority of the panels at that location showed one or more of these surface spalls.

Figure 3 is a comparison of panels having good coverage on the under side (two or more enamel applications) with similar panels that had poor coverage (a single ground coat with local breaks in the coating). All four of these panels were from the Atlantic City site, where corrosion on the unprotected metal was especially severe because of the "salt-air" conditions. Fortunately, the center area of the panels was free of spalls in almost all cases, and therefore they did not interfere with the gloss and color measurements.

Surface spalls from poor coverage on the under side were noted also on a few of the panels at St. Louis and Lakeland. The following tabulation

[3] Enamels with 45° specular-gloss readings of 4.5 or greater are referred to as glossy, those with readings between 2.0 and 4.5 as semimat or satin-textured and those with readings of less than 2.0 as full mat. The designations of glossy and semimat used in table 3 are those assigned by the manufacturers. Many of the specular-gloss readings do not conform with these designations.

gives the prevalence of surface spalls at each of the four locations:

Exposure site	Percentage of penels showing one or more surface spalls
Atlantic City	77
St. Louis	5
Lakeland	2
Washington	0

Noticeable edge corrosion [4] was observed only on the panels exposed to the salt-air conditions at Atlantic City. In no case, however, was this edge corrosion considered to be serious. Numerous observations made during the 15-yr inspection indicated that whenever corrosion started at an edge or at any area of poor coverage, there was very little spreading of the corrosion under the adjacent enamel.

Some of the top attachment clips (see fig. 1) showed serious corrosion in the salt air at Atlantic City. These clips had been spotwelded to the panels prior to enameling and, in some cases, the enamel coverage was poor where the clip joined the panel. Corrosion occurred at these points, the clips came loose from the panels, and, in a few instances, the affected panels were blown away from the rack during the periods of high wind velocity. As a result, some of the data for Atlantic City are missing from table 3.

[4] Good coverage is difficult to achieve on the edge of a thin sheet and, hence, many of the panel edges were not properly covered with enamel.

Figure 3. *Four panels after 15 years of exposure at Atlantic City.*

Specimens on left had good enamel coverage, whereas the two on the right show holes in the panel caused by rusting through from areas of incomplete coverage on the back side.

Table 3. *Percentage of initial specular gloss retained, color-difference values, weather-resistance ratings, and acid resistance of enamels of 14 types after 15 years of exposure at 4 sites*

Specimen identification[a]	Fabricator of specimens	Frit supplier	Average initial specular gloss,[b] G_S	Initial gloss retained (G_R) and change in color (ΔE) at—[c] Washington		Lakeland		St. Louis		Atlantic City		Acid resistance (PEI test)[d]	Weather resistance rating[e]
				G_R	ΔE	G_R	ΔE	G_R	ΔE	G_R	ΔE		
WHITE, GLOSSY, ACID-RESISTANT ENAMEL													
			%	%		%		%		%			
A-1 to 8	a	1	5.70	83.1	0.7	68.9	0.8	76.6	0.8	66.7	1.1	AA	E
A-11 to 18	b	1	5.74	86.9	1.0	88.7	1.0	75.5	.9	89.6	2.2	AA	E
A-21 to 28	a	2	6.04	84.2	0.6	68.4	0.3	77.5	1.4	88.3	0.9	AA	E
A-31 to 38	b	2	6.01	76.6	.5	66.6	.3	85.8	0.6	84.4	.2	AA	E
A-41 to 48	a	3	5.85	85.0	3.0	62.2	2.9	82.7	2.1	81.7	1.6	AA	G
A-51 to 58	b	3	5.81	85.4	1.2	48.8	2.4	85.6	1.0	46.8	2.2	AA	F
A-61 to 68	a	4	6.20	83.3	1.1	66.0	3.5	73.6	1.6	72.1	3.6	AA	G
A-71 to 78	b	4	6.01	73.4	1.7	86.3	1.7	79.9	2.3	77.8	3.1	AA	G
Average	------	----	------	82.2	1.2	69.5	1.6	79.6	1.3	75.9	1.9	------	------
WHITE, GLOSSY, NONACID-RESISTANT ENAMEL													
B-1 to 8	b	1	5.26	18.0	2.9	62.8	1.1	46.1	8.6	(f)	(f)	C	P
B-21 to 28	b	2	5.41	15.0	4.8	32.6	4.6	41.3	6.3	19.0	2.7	C	P
B-41 to 48	b	3	5.16	15.0	2.7	38.5	1.9	38.6	5.9	15.0	3.0	D	P
B-61 to 68	b	4	5.32	16.4	3.1	56.0	0.5	35.5	6.9	18.7	1.3	C	P
Average	------	----	------	16.1	3.4	47.5	2.0	40.4	6.9	17.6	2.3	------	------
WHITE, SEMIMAT, ACID-RESISTANT ENAMEL													
C-11 to 18	e	1	4.06	30.3	1.5	54.4	1.1	82.6	5.2	43.5	1.6	C	P
C-31 to 38	c	2	5.24	42.1	2.3	40.8	1.7	92.3	0.9	46.6	1.5	A	F
C-51 to 58	e	3	5.16	76.7	2.2	63.9	1.9	80.7	3.8	55.6	2.2	A	G
C-71 to 78	e	4	5.32	54.8	1.3	33.0	1.9	86.7	0.6	46.1	0.8	A	P
Average	------	----	------	51.0	1.8	48.0	1.6	85.6	2.3	47.9	1.5	------	------

TABLE 3. *Percentage of initial specular gloss retained, color-difference values, weather-resistance ratings, and acid resistance of enamels of 14 types after 15 years of exposure at 4 sites*—Continued

Specimen identification[a]	Fabricator of specimens	Frit supplier	Average initial specular gloss,[b] G_S	Initial gloss retained (G_R) and change in color (ΔE) at—[c] Washington		Lakeland		St. Louis		Atlantic City		Acid resistance (PEI test)[d]	Weather resistance rating[e]
				G_R	ΔE	G_R	ΔE	G_R	ΔE	G_R	ΔE		
WHITE, SEMIMAT, NONACID-RESISTANT ENAMEL													
D-1 to 8	c	1	------	------	1.0	----	3.5	----	9.2	----	2.6	------	------
D-21 to 28	c	2	5.69	15.0	3.3	42.9	2.9	53.9	2.4	17.4	2.9	D	P
D-41 to 48	c	3	5.51	15.0	3.3	29.4	0.9	45.0	5.2	17.7	3.9	D	P
D-61 to 68	c	4	------	------	3.6	----	3.0	-	16.5	----	4.4	------	----
Average	------	----	------	15.0	3.4	36.1	2.6	49.4	8.3	17.5	3.4	------	----
BUFF, GLOSSY, ACID-RESISTANT ENAMEL													
E-11 to 18	d	1	5.09	60.2	1.4	61.4	1.2	84.3	0.1	48.6	2.9	B	F
E-31 to 38	d	2	5.43	54.8	1.8	76.3	1.1	87.6	1.7	76.7	1.0	AA	G
E-51 to 58	d	3	5.44	90.0	0.4	63.6	0.8	84.8	0.9	91.0	0.9	AA	G
E-71 to 78	d	4	5.35	76.0	1.6	50.8	2.6	78.6	1.2	82.5	1.7	AA	G
Average	------	----	------	70.2	1.3	63.0	1.4	83.8	1.0	74.7	1.6	------	------
BUFF, GLOSSY, NONACID-RESISTANT ENAMEL													
F-1 to 8	d	1	5.15	24.3	7.8	22.4	3.3	58.9	3.0	18.4	8.4	D	P
F-11 to 18	e	1	4.87	35.6	4.4	21.5	4.1	49.0	3.0	19.8	6.4	D	P
F-21 to 28	d	2	5.02	33.2	3.8	67.9	2.8	67.9	1.6	25.3	4.9	C	P
F-31 to 38	e	2	5.56	44.3	2.9	33.1	6.9	65.0	2.3	18.5	6.4	D	P
F-41 to 48	d	3	4.31	26.2	13.4	28.6	4.8	45.9	8.3	34.1	19.7	D	P
F-51 to 58	e	3	5.66	27.6	5.3	47.5	3.1	29.4	3.4	17.6	6.7	D	P
F-61 to 68	d	4	4.64	24.4	12.0	20.5	6.6	37.0	6.9	24.5	20.6	D	P
F-71 to 78	e	4	5.26	23.7	12.1	22.1	4.8	31.0	9.2	34.3	16.4	D	P
Average	------	----	------	29.9	7.7	32.9	4.5	48.0	4.7	24.1	11.2	------	------
BUFF, SEMIMAT, ACID-RESISTANT ENAMEL													
H-1 to 8	e	1	3.96	64.3	2.0	61.4	2.1	62.5	2.1	76.6	1.8	A	G
H-11 to 18	f	1	4.81	52.1	0.9	63.0	2.7	71.3	1.3	72.8	1.4	A	G
H-21 to 28	e	2	5.65	77.0	.8	58.2	1.6	84.8	0.8	(f)	(f)	A	G
H-31 to 38	f	2	5.45	74.3	.5	62.0	1.7	88.6	.3	81.6	1.7	A	G
H-41 to 48	e	3	4.74	87.5	.6	95.1	1.4	92.0	.3	98.2	0.4	AA	E
H-51 to 58	f	3	4.85	76.8	1.2	85.9	1.1	82.9	1.5	87.1	3.1	AA	G
H-61 to 68	e	4	5.51	83.9	4.3	68.2	1.2	95.8	0.8	87.5	0.3	AA	G
H-71 to 78	f	4	5.54	83.5	2.0	76.7	4.3	88.8	2.7	90.2	1.7	AA	G
Average	------	----	------	74.9	1.5	71.3	2.0	83.3	1.2	84.9	1.5	------	------
BUFF, SEMIMAT, NONACID-RESISTANT ENAMEL													
K-1 to 8	f	1	------	------	7.4	----	2.0	----	3.7	----	----	------	----
K-11 to 18	g	1	------	------	7.2	----	1.5	----	6.9	----	8.0	------	----
K-21 to 28	f	2	5.41	21.3	4.1	38.5	1.1	52.9	2.4	19.5	6.0	D	P
K-31 to 38	g	2	5.37	18.8	7.2	34.2	4.5	58.6	2.5	18.4	6.9	D	P
K-41 to 48	f	3	5.20	63.8	1.7	23.2	2.1	69.7	0.8	23.7	3.8	D	P
K-51 to 58	g	3	5.35	16.2	9.2	19.4	4.6	36.1	2.9	19.2	6.5	D	P
K-61 to 68	f	4	------	------	6.9	----	12.2	----	11.1	----	10.0	------	----
K-71 to 78	g	4	------	------	14.4	----	6.0	----	3.3	----	4.9	------	----
Average	------	----	------	30.0	7.3	28.8	4.2	54.3	4.2	20.2	6.6	------	
RED, GLOSSY, ACID-RESISTANT ENAMEL													
L-1 to 8	g	1	5.58	85.5	3.8	91.2	3.5	67.3	2.1	84.1	0.4	AA	G
L-11 to 18	h	1	5.61	80.4	0.8	85.9	2.3	84.8	1.5	(f)	(f)	AA	E
L-21 to 28	g	2	5.42	82.1	2.2	73.5	10.6	80.8	0.5	(f)	(f)	B	P
L-31 to 38	h	2	5.64	82.0	3.1	62.5	12.1	60.7	4.5	84.7	1.8	A	P
L-41 to 48	g	3	5.58	77.0	3.0	88.6	1.0	90.5	1.6	81.1	0.4	AA	G
L-51 to 58	h	3	5.23	68.5	0.9	85.8	1.0	90.7	2.7	90.2	1.9	A	G
L-61 to 68	g	4	5.30	80.8	2.3	93.2	1.6	78.1	2.2	91.0	5.3	A	F
L-71 to 78	h	4	4.18	91.9	1.3	83.4	4.3	90.3	2.8	87.9	4.4	AA	G
Average	------	----	------	81.0	2.2	83.0	4.5	80.3	2.2	86.5	2.4		

Table 3. *Percentage of initial specular gloss retained, color-difference values, weather-resistance ratings, and acid resistance of enamels of 14 types after 15 years of exposure at 4 sites*—Continued

Specimen identification[a]	Fabricator of specimens	Frit supplier	Average initial specular gloss,[b] G_S	Initial gloss retained (G_R) and change in color (ΔE) at—[c] Washington		Lakeland		St. Louis		Atlantic City		Acid resistance (PEI test)[d]	Weather resistance rating[e]
				G_R	ΔE	G_R	ΔE	G_R	ΔE	G_R	ΔE		
RED, GLOSSY, NONACID-RESISTANT ENAMEL													
N-1 to 8	h	1	5.17	65.8	2.5	37.2	6.3	65.4	2.1	38.4	8.6	C	P
N-11 to 18	k	1	5.05	63.8	2.1	38.9	4.3	80.2	3.3	19.0	8.8	C	P
N-21 to 28	h	2	5.12	67.3	1.9	19.6	10.3	54.4	3.2	38.4	12.3	D	P
N-31 to 38	k	2	4.55	62.3	2.7	40.0	6.9	80.7	2.1	19.0	8.8	D	P
N-41 to 48	h	3	5.17	63.2	3.3	39.2	7.4	57.6	8.2	([f])	([f])	C	P
N-51 to 58	k	3	4.68	49.7	2.7	48.4	3.6	80.0	3.6	38.5	9.3	C	P
N-61 to 68	h	4	4.71	33.8	4.3	21.2	4.9	31.6	24.5	23.7	15.1	D	P
N-71 to 78	k	4	5.44	28.7	22.0	46.0	4.4	29.7	22.5	19.6	13.1	D	P
Average	------	----	------	54.3	5.2	36.3	6.0	59.9	8.7	28.1	10.8	------	------
RED, SEMIMAT, ACID-RESISTANT ENAMEL													
P-1 to 8	k	1	5.54	86.3	3.8	84.3	3.5	90.3	3.4	68.0	2.8	AA	G
P-11 to 18	l	1	------	------	24.7	----	17.1	----	26.0	----	35.3	------	------
P-21 to 28	k	2	------	------	38.0	----	29.4	----	46.2	----	41.2	------	------
P-31 to 38	l	2	3.99	48.6	6.2	26.9	10.8	73.9	1.1	55.6	8.7	C	P
P-41 to 48	k	3	4.95	70.6	2.6	91.1	4.2	71.1	2.9	85.8	4.4	A	G
P-51 to 58	l	3	3.34	84.2	5.9	79.0	12.9	94.4	10.1	94.2	10.9	A	P
P-61 to 68	k	4	4.97	83.5	2.7	89.8	2.4	72.6	4.6	94.4	4.0	AA	G
P-71 to 78	l	4	5.54	89.7	5.3	85.1	6.1	93.8	6.3	87.8	1.4	AA	F
Average	------	----	------	77.1	11.1	76.0	10.8	82.7	13.2	81.0	14.3	------	------
RED, SEMIMAT, NONACID-RESISTANT ENAMEL													
S-1 to 8	l	1	------	------	24.3	----	14.6	----	28.0	----	40.6	------	------
S-11 to 18	m	1	------	------	15.6	----	4.1	----	8.8	----	22.6	------	------
S-21 to 28	l	2	4.51	76.9	2.9	30.6	5.6	80.3	1.0	76.9	4.3	B	P
S-31 to 38	m	2	4.51	63.8	6.7	36.3	12.4	63.5	5.4	43.6	9.7	C	P
S-41 to 48	l	3	3.50	61.9	2.6	29.3	5.6	70.5	3.5	41.9	7.4	A	P
S-51 to 58	m	3	3.95	89.4	1.0	63.0	6.0	90.0	1.8	82.2	1.9	A	F
S-61 to 68	l	4	4.50	38.8	14.3	23.1	7.9	48.7	12.2	24.3	21.4	C	P
S-71 to 78	m	4	3.57	50.0	18.6	35.7	7.9	61.0	12.7	27.9	9.9	C	P
Average	------	----	------	63.5	10.7	36.3	8.0	69.0	9.2	49.5	14.7	------	------
BLACK, GLOSSY, ACID-RESISTANT ENAMEL													
T-1 to 8	m	1	7.30	57.9	0.8	71.8	0	78.7	0.8	69.0	0.8	AA	G
T-11 to 18	n	1	6.60	73.4	0.4	88.9	.8	82.5	.8	80.4	0	AA	E
T-21 to 28	m	2	5.38	87.8	1.4	97.8	.7	95.6	.6	86.4	.7	AA	E
T-31 to 38	n	2	5.55	79.1	0.8	93.9	0	89.3	.8	([f])	([f])	AA	E
T-41 to 48	m	3	6.09	64.2	0	92.6	0	88.1	.6	79.4	0	AA	E
T-51 to 58	n	3	5.90	85.4	1.5	93.6	0	81.6	1.6	80.6	0	A	E
T-61 to 68	m	4	6.55	66.7	1.1	68.2	.6	86.8	2.0	69.0	.1	AA	E
T-71 to 78	n	4	6.56	57.6	0.8	65.7	.6	86.2	1.4	67.9	.6	AA	G
Average	------	----	------	71.5	0.8	84.1	.3	86.1	1.1	76.1	.3	------	------
BLACK, GLOSSY, NONACID-RESISTANT ENAMEL													
V-1 to 8	n	1	5.52	61.5	19.9	69.0	11.5	74.2	15.8	53.3	20.5	C	P
V-11 to 18	a	1	5.62	34.5	41.3	46.7	13.2	17.8	40.5	23.8	32.6	C	P
V-21 to 28	n	2	5.76	64.0	1.4	53.7	4.6	86.7	2.6	34.4	7.9	C	P
V-31 to 38	a	2	5.60	41.1	4.0	42.5	6.4	58.5	5.8	55.3	5.6	C	F
V-41 to 48	n	3	5.30	66.3	4.6	76.8	5.2	81.8	4.6	([f])	([f])	C	F
V-51 to 58	a	3	5.49	58.2	12.3	75.6	6.9	82.3	9.0	60.9	7.2	C	P
V-61 to 68	n	4	4.67	54.1	13.4	59.0	13.1	74.7	5.1	29.9	17.4	C	P
V-71 to 78	a	4	5.45	61.7	6.4	62.5	16.3	56.1	4.5	65.0	6.4	C	P
Average	------	----	------	55.2	12.9	60.7	9.6	66.5	11.0	46.1	13.9	------	------

[a] Groups of 8 panels exposed, 2 at each location. A ninth panel was kept in storage.

[b] Figures reported are for percentage of light incident at 45° that was specularly reflected. Enamels with 45° specular-gloss readings of 4.5 or greater are referred to as glossy, those with readings between 2.0 and 4.5 as semimat or satin-textured, and those with readings of less than 2.0 as full mat. The designations of glossy and semimat used in the headings are those assigned by the manufacturers. Many of the initial specular-gloss readings do not conform with these designations. The full-mat enamels having initial gloss too low for measurement are represented by blank spaces in columns.

[c] Percentage of gloss retained, G_R, was computed from 45° specular-gloss readings. Color change, ΔE, is in NBS units as computed from color-difference measurements. Values in both cases are average of 2 panels with 2 readings on each panel.

[d] Test made on storage panel in accordance with Test for Acid Resistance of Porcelain Enamels, part I, Flatware. Issued by the Procelain Enamel Institute, 1145 19th St., N. W., Washington 6, D. C. In this test the degree of attack is evaluated after treatment with citric acid. Class AA shows no visible effect from the treatment and is the most resistant, with class A, class B, class C, and class D following in that order. Enamels falling in the latter two classes are not considered acid resistant.

[e] E, excellent; G, good; F, fair; and P, poor. See text for procedure used in assigning ratings.

[f] Panels lost to study because of corrosion of attachment lugs.

4.3. Gloss Measurements

Specular-gloss measurements were made on each panel with the Hunter Multipurpose Reflectometer [7] adjusted for a 45° angle of incidence. The gloss scale of the instrument was calibrated against liquid films [8]. Measurements were made at two fixed locations at the center of each panel and compared with similar measurements made at the start of the investigation. The data were expressed as the percentage of initial specular gloss retained.

Table 3 includes the gloss data for each enamel at the four exposure sites.[5] Comparison with the results of the 7-yr inspection [4] shows that the percentage of gloss retained in many cases was higher after 15 yr than it was after 7 yr. This effect is due to the difference in cleaning treatment, the panels at 7 yr having been washed with trisodium phosphate solution, whereas the scouring treatment was required to clean the specimens for the 15-yr measurements.

Except for a few isolated cases, the percentage of gloss retained was considerably higher for enamels of high acid resistance than for the nonacid-resistant types. The enamel showing the least change in gloss for all four locations after 15 yr was an acid-resistant black (T–21 to 28), which retained an average of 91.9 percent of its initial gloss. Among the poorest was a nonacid-resistant composition (K–51 to 58), which had an average percentage of gloss retained for the four locations of only 22.7.

In general it was noted that the specular gloss changed at a faster rate in the earlier stages of exposure than later. Figure 4 illustrates this effect for five panels at Washington, D. C., whose surfaces had been washed with trisodium phosphate solution. These same panels, along with all others from Washington, were later cleaned by scouring so as to conform to the cleaning procedure used at the other three locations. The effect of the scouring treatment in raising the percentage of gloss retained by the five panels is shown by the following tabulation:

Panel	Acid resistance (PEI test)	Percentage of gloss retained	
		Washed with Na_3PO_4	Cleaned with scouring powder
E–71	AA	64. 3	76. 0
P–41	A	56. 3	71. 7
E–12	B	48. 0	53. 1
C–11	C	27. 2	28. 6
B–41	D	16. 1	15. 0

[5] The initial gloss values, G_s, are expressed as the percentage of incident light that is specularly reflected. This conforms to the practice used in the earlier papers. Recent practice in the industry, however, is to express the values in "gloss units," defined as $10 \times G_s$.

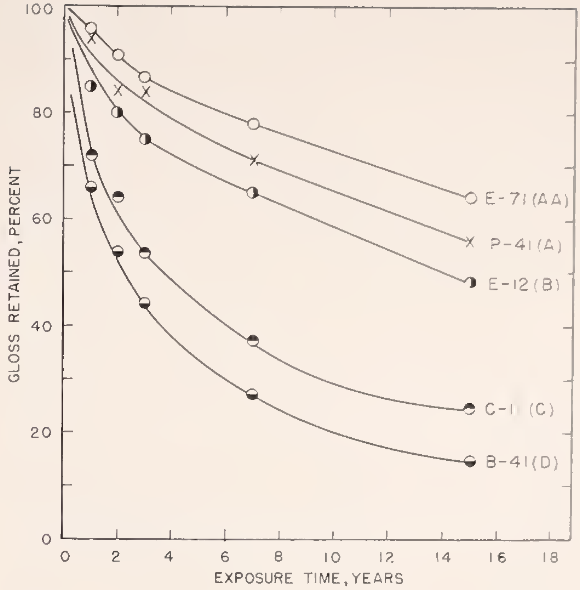

Figure 4. *Selected data showing change in gloss with exposure time for five panels at the Washington, D. C., site.*

Letters following panel numbers indicate acid-resistance ratings by citric acid spot test.

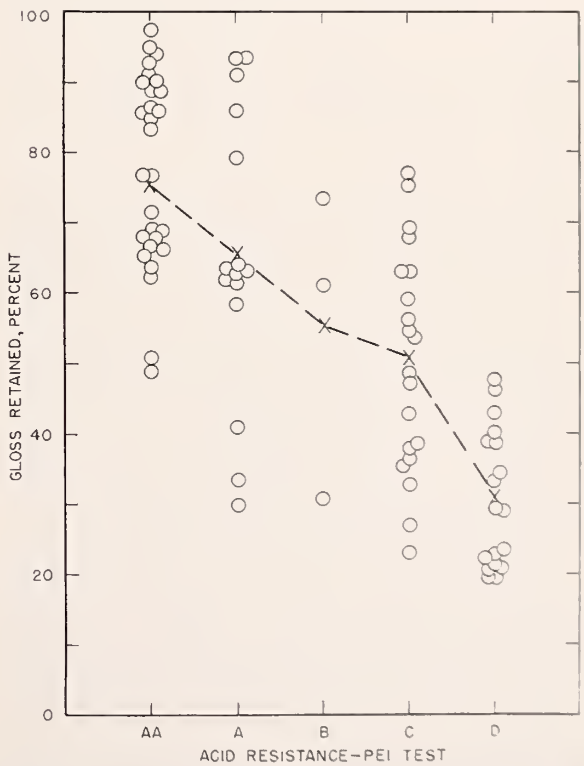

Figure 5. *Percentage of gloss retained for enamels of five classes of acid resistance after exposure for 15 years at Lakeland, Fla.*

Dashed line is drawn through average value for each class of acid resistance.

The curves plotted in figure 4 represent selected data, in that these particular five panels were the ones that best illustrated the usual weathering behavior of enamels having different degrees of acid resistance when measured by the citric acid spot test of the Porcelain Enamel Institute.[6] Actually, table 3 shows appreciable differences in the percentage of gloss retained for enamels of the same acid resistance. This high scatter for the Lakeland panels is illustrated in figure 5.

4.4. Color-Difference Measurements

The color changes that occurred during the 15-yr exposure period were measured with a Hunter Color-Difference Meter [9]. In each case, the exposed panel was compared with the storage panel, and the difference between the two expressed in NBS units [10]. Diffuse reflectance measurements were made at the beginning of the investigation, and these indicated excellent color match between the storage panels and the ones selected for exposure. One NBS unit is about five times the smallest difference perceptible to the eye under the best experimental conditions [11]. It is the opinion of the authors that five NBS units might be considered objectionable by a user, especially if two panels showing such a color difference were placed side by side in a structure. A change of 10 units due to weathering would probably be objectionable in most installations where retention of a specified color is important, for example in signs and structures where the color has been specially selected as an identifying characteristic.

Table 3 shows that color changes were minor for the white enamels, except for the St. Louis panels of poor acid resistance. These are the same enamels that became etched during weathering. Dirt and grime in the atmosphere at the St. Louis site entered the pores created by the etching action, and these dirt particles could not be removed completely by the cleaning treatment. The presence of the dirt particles retained after cleaning made the panels appear off-white to the eye and darker to the color-difference meter. At the other sites, there was less pollution of the atmosphere, and dirt retention after cleaning of the nonacid-resistant enamels had a much smaller effect on the color-difference measurements.

The colored enamels of poor weather resistance became lighter and weaker in color with exposure, which gave them a faded appearance. The greatest color change was observed for the full-mat enamels, P–11 to 18, P–21 to 28, S–1 to 8, and S–11 to 18 in table 3; in addition, these were the compositions that were the most difficult to clean.

[6] Test for Acid Resistance of Porcelain Enamels, part 1, Flatware. Issued by the Porcelain Enamel Institute, 1145 19th St., N. W., Washington 6, D. C. In the commercial test, which separates enamels according to classes, a small pool of 10-percent citric acid is placed on the specimen for 15 min at 80° F. The degree of attack is then evaluated visually by observing such characteristics as staining, blurring of image, and ease of removal of a pencil mark. Class AA shows no visible effect from the treatment and is most resistant, with Class A, Class B, Class C, and Class D following in that order. Enamels falling in the latter two classes are not considered acid resistant.

4.5. Microstructure of Surfaces

The surfaces of the enamels that were examined with the metallographic microscope at the 7-yr inspection were reexamined after 15 yr of exposure. In general, the appearances of the surfaces were unchanged. The enamels showing little or no change in surface microstructure at 7 yr still showed little or no change at 15 yr. The photomicrographs shown in the earlier report [4] of enamels of poor acid resistance are representative also of the 15-yr appearance. A gel-type layer was present on enamels of this type, and the thickness of the layer was found to increase with exposure time, but this increase occurred without much change in surface appearance.[7] The thickness of the layer varied with enamel composition. On a black nonacid-resistant enamel, represented by panels V–11 to V–18, the thickness had increased from an average of 0.001 in. at 7 yr to a thickness of about 0.0025 in. at 15 yr. The film still maintained a glossy surface appearance, but was soft enough to be scraped away with a knife blade. Table 3 shows the average color change of this enamel at the four locations to be 31.9 NBS units.

Similar gel-like films of measurable thickness had formed on some of the other enamels of poor acid resistance after the 15-yr exposure period. For example, panel P–27 (Atlantic City) showed a film thickness of 0.0005 in., V–63 (Lakeland) a thickness of 0.0004 in., and V–67 (Atlantic City) a thickness of 0.001 in.

It should be pointed out that only those enamels of very poor weather resistance showed layers of this type. The acid-resistant compositions may have had very thin films of a gel-like nature present on their surfaces, but, if so, the films were too thin to have any marked effect on gloss or color.

5. Effect of Variation in Weather Conditions at Four Exposure Sites

Tables 4 and 5 are summaries of the data on gloss and color difference, respectively, at the four exposure sites. For convenience, the enamels have been grouped according to their acid resistance by the Porcelain Enamel Institute Spot Test for the Acid Resistance of Flatware. In the table giving the gloss summaries (table 4) the grouping is made without considering color. The initial color, however, had a definite effect on the magnitude of the color difference after exposure. Therefore the enamels in table 5 are grouped according to both color and acid resistance.

Standard deviations are listed in both tables. In practically all cases these values are high, indicating poor agreement among panels in any one group. Nevertheless a trend appears to be

[7] The scouring treatment used for cleaning removed the thinner gel-like layers, but on panels having layers 0.001 in. or heavier, some of the film remained even after prolonged scouring.

TABLE 4. *Average percentages of initial specular gloss retained for enamels of various classes of acid resistance after 15 yr of weathering at 4 exposure sites*

Number of enamels averaged [a]	Acid-resistance class [b]	Percentage of initial specular gloss retained [c]									
		Washington		Lakeland		St. Louis		Atlantic City		All sites	
		Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD
29	AA	79.0	9.9	77.7	13.3	84.5	7.2	80.6	10.7	80.4	10.6
15	A	71.1	13.3	65.3	19.8	80.3	10.4	73.6	12.4	72.8	14.4
3	B	73.1	9.3	55.2	18.1	81.8	1.8	43.1	5.5	63.3	10.6
21	C	47.5	16.9	50.3	15.5	63.6	17.6	37.6	14.9	49.7	16.3
18	D	31.1	12.0	30.5	9.5	48.2	8.9	22.6	6.4	33.1	11.1

[a] Each enamel represented by two panels at each location.
[b] From spot tests made on 12- by 12-in. storage panels, using the standard test of the Porcelain Enamel Institute.
[c] Measurements made on panel surfaces cleaned with a commercial scouring powder; SD denotes the standard deviation of the individual readings about the average.

TABLE 5. *Average color differences between exposed panels and storage panels for enamels of two classes of acid resistance after 15 yr of weathering at 4 exposure sites*

Enamel color	Number of enamels averaged [a]	Acid resistance [b]	Color difference in NBS units [c] at—									
			Washington		Lakeland		St. Louis		Atlantic City		All sites	
			Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD
White	11	AR	1.4	0.8	1.7	1.0	1.5	0.9	1.8	1.0	1.6	0.9
	7	Non-AR	2.7	1.4	1.9	1.3	5.7	1.8	2.6	0.9	3.2	1.2
Buff	12	AR	1.4	1.2	1.8	0.9	1.1	0.7	1.2	.8	1.4	0.9
	12	Non-AR	7.0	3.8	4.0	1.6	3.8	2.6	9.4	5.7	6.1	3.8
Red	16	AR	2.8	2.3	5.2	3.6	3.5	2.6	4.1	3.0	3.9	2.9
	12	Non-AR	7.3	6.7	7.2	2.8	8.4	7.7	11.4	3.8	8.6	5.6
Black	8	AR	0.9	0.4	0.7	0.1	1.1	0.5	0.5	0.3	0.8	0.4
	8	Non-AR	12.9	12.2	9.7	4.1	11.0	11.8	14.0	9.4	11.9	9.9
White [d]	2	----------	2.3	0.8	3.2	0.3	12.8	3.4	3.5	0.9	5.5	1.8
Colored [d]	8	----------	18.3	10.4	10.0	9.2	16.8	14.2	23.2	14.7	16.8	12.4

[a] Each enamel represented by two panels at each location.
[b] Grouped according to spot test of Porcelain Enamel Institute; AR includes class AA, A, and B enamels, Non-AR includes class C and D.
[c] Measurements made on panels cleaned with a commercial scouring powder; SD denotes the standard deviation of the individual readings about the average.
[d] Full-mat enamels; all others are glossy or semimat.

present, the conditions at Lakeland being more severe on the acid-resistant panels (class AA, A, and B) and the conditions at Atlantic City giving the most attack on the nonacid-resistant (class C and D) compositions. This order of severity is a reversal of the trend noted in the 7-yr inspection [4]. The cause of the reversal is believed to be the difference in the cleaning procedures. The scouring treatment used at the 15-yr inspection removed surface films more effectively than the washing treatment used at the 7-yr inspection, and, in addition, scouring corresponds to commercial cleaning practice. Therefore, it is suggested that the 15-yr results give a more realistic picture of the relative severity of the exposure conditions at the four sites than do the 7-yr data.

The greater severity of conditions at Lakeland on acid-resistant compositions is especially noticeable for some of the red enamels. For example, table 3 shows a color change at Lakeland of 10.6 NBS units for the enamel represented by panels L–21 to L–28, and 12.1 units for L–31 to L–38, yet the greatest color change for these same two compositions at any of the other sites was only 4.5 units. The cause of the greater changes in color and gloss for acid-resistant enamels at Lakeland has not been determined, but it could be associated with (a) the higher values for annual sunshine, rainfall, and temperature, as given in table 2, or (b) the attack by organic acids produced by algae and fungi that were found to be attached on the surfaces of all panels at the Lakeland exposure site, and that were described in the earlier paper [4]. Similar growths have been found by Jones [12] to cause etching of certain optical glasses used in the tropics.

The sulfur dioxide content of the atmosphere at the St. Louis site was found to be about 45 times as great as in Washington [3], yet table 3 shows that, on the average, the Washington panels were affected more by the 15-yr exposure than those at St. Louis. This could be caused by the heavier rainfall in Washington (see table 2), but a more likely explanation is that the heavy accumulation of dirt and grime on the panel surfaces at St. Louis acted as a protective layer. Had the St. Louis panels been thoroughly cleaned at periodic intervals, the attack at St. Louis might well have been greatly increased.

A statistical analysis, performed by W. J. Youden of the Applied Mathematics Division at the National Bureau of Standards, showed that the observed differences in behavior at the four sites were statistically significant. The same analysis showed the presence of small but real differences in frits of the same type made by different manufacturers; however, there was no evidence of significant differences between enamels made with the same frit by two fabricators.

6. Laboratory Tests for Predicting Weather Resistance

An acceptable test for predicting weather resistance should give a definite separation between the enamels that showed only minor deterioration after 15 yr of exposure and those that showed objectionable fading and loss of gloss. To determine whether or not the citric acid spot test would give such a separation, all of the enamels except the full mats were first given ratings of excellent, good, fair, or poor, depending on a combination of their measured gloss and color-change values. These ratings were then compared with the results of the citric acid spot test.

The adjective ratings for the comparison were assigned in accordance with the following arbitrarily chosen criteria:

Rating	Percentage of gloss retained	Color change in NBS units
Excellent	65 to 100	0 to 2.5
Good	50 to 65	2.5 to 5.0
Fair	40 to 50	5.0 to 7.5
Poor	15 to 40	>7.5

Enamels with values of gloss retained between 65 and 100 percent at all four sites were rated excellent only if the maximum color change did not exceed 2.5 NBS units at any of the locations. If the values for either gloss or color change fell outside of these limits at any one of the four sites, the enamel was given the lowest applicable rating. For example, in table 3 the enamel represented by panels P–51 to 58 would be rated excellent from the standpoint of gloss retained (lowest value 79.0), but the maximum color change at one of the test sites was 12.9 units. On the basis of color change alone this particular enamel is rated poor, in spite of its excellent gloss retention. Conversely, the white enamel represented by panels C–71 to 78 shows a maximum color change of only 1.9 units; yet, because the percentage of gloss retained at Lakeland was as low as 33.0, the enamel received a rating of poor.

The effectiveness of the spot test in separating enamels of varying weather resistance when rated in accordance with the aforementioned procedure is given in table 6. The figures in parentheses give the number of red enamels included in the total. From these figures it can be seen that if the red enamels are eliminated from consideration, there are only three remaining cases in which an acid-resistance rating of A or AA did not correctly indicate a weather-resistance rating of good or better. Two of these three had fair weather resistance.

Table 6. *Number of enamels having weather-resistance ratings of excellent, good, fair, and poor when grouped according to the citric acid test*

Acid resistance (PEI test)	Number of enamels tested	Number of enamels with weather-resistance rating of—			
		Excellent [a]	Good [a]	Fair [a]	Poor [a]
AA	29	11(1)	16(5)	2(1)	0
A	15	1	7(2)	3(2)	4(3)
B	3	0	0	1	2(2)
C	21	0	0	2	19(8)
D	18	0	0	0	18(4)

[a] Figures in parentheses are number of red enamels included in the total. See text for procedure used in assigning ratings.

The poor ratings of most of the red enamels of class AA, A, and B acid resistance is a result of excessive color change. It should be pointed out that red enamels are pigmented with the so-called cadmium sulfo-selenide complexes. When such pigments are subjected to strongly oxidizing conditions, they are known to change color. Thus, if the surface of a red enamel were to become slightly etched during weathering, the exposed pigment particles would be expected to change in color if oxidizing influences were present. When the vitreous phase of the enamel initially surrounds and covers the particles of pigment completely, and is not subsequently etched away, the pigment is protected against the color change associated with oxidation. Correspondingly, a laboratory test, to correlate well with performance data, must provide a commensurate degree of etching, and for pigments that are highly sensitive to oxidation, also a commensurate degree of oxidizing potential. In this connection, McDonald [13] found that certain red screening enamels faded noticeably in 1 to 4 years, although they showed class AA acid resistance by the citric acid spot test. He also found that a spot test with concentrated nitric acid, which is more corrosive and highly oxidizing, would distinguish the red enamels showing poor weather resistance from those that were resistant to fading.

Sweo [14] noted that a red semimat enamel of class A acid resistance showed poor weatherability when exposed for 10 months. This same enamel gave a weight loss from 40 to 200 times greater than class A enamels of good weather resistance when the specimens were subjected to a boiling solution of 6-percent (by weight) citric acid for 2½ hr. Therefore, Sweo suggested that the boiling citric acid test might be useful in predicting the suitability of enamels for exposure applications.

Both the nitric acid spot test [13] and the boiling citric acid test [14] were used in the present investigation on storage panels of several of the red enamels. The correlation was somewhat better than the citric acid spot test, but neither test gave results that would predict the rather large color change found for enamels L–21 to 28 and L–31 to 38 at Lakeland. On the other hand, a treatment for 2½ hr in a boiling aqueous solution of 10-percent (by weight) nitric acid did distinguish these two enamels from the acid-resistant reds that gave satisfactory weather resistance at all sites. Therefore, because of the promise shown by the boiling nitric acid test, all of the red enamels were subjected to this treatment.

The test was made on the 4- by 6-in. laboratory specimens. Each specimen was weighed to the closest milligram and then clamped to the ground face of a Pyrex-brand glass tube of 2¼-in. inside diameter, a rubber gasket being used to prevent leakage. A reflux condenser was attached to the opposite end of the tube, and 70 ml of the acid solution was added. The assembly was next placed on a hotplate with the back of the specimen in contact with the hot surface and the solution allowed to boil for 2½ hr. After removal from the hotplate, the specimen was cleaned by rubbing with a sponge wetted with a 1-percent (by weight) solution of trisodium phosphate, rinsed in ethyl alcohol, air dried, and then reweighed, the change in weight being expressed as milligrams per square centimeter of exposed surface. In addition, the difference in color between the exposed and unexposed areas was measured, and the results expressed in NBS units.

Table 7 lists the results of the boiling nitric acid test for the 28 glossy and semimat red enamels included in the study. The enamels are arranged in groups according to their weather resistance rating. The acid-resistance class by the citric acid spot test is given for purposes of comparison. It can be seen from this table that there are 5 enamels of class A and B acid resistance by the citric acid spot test that show unsatisfactory weathering behavior at 1 or more of the 4 exposure sites. These 5 enamels all gave weight losses greater than 1.0 mg/cm². The 4 class A finishes with passable weather resistance (excellent, good, or fair) and all of the class AA enamels show weight losses of less than 1.0 mg/cm². Thus, for the red enamels included in this investigation, the weight change after 2½ hr of boiling in 10-percent-by-weight nitric acid gave the desired separation between enamels of poor weather resistance and those that were considered passable.

The data in table 7 show that the measured color change after test is not as reliable a criterion as weight loss, because one enamel of good weather resistance (P–1 to 8) shows a fairly high color change (5.3 NBS units) after the nitric acid treatment, whereas one of the enamels of poor weather resistance (P–51 to 58) shows a color change of only 3.6 NBS units.

Table 7. *Citric acid spot test and boiling nitric acid test results for red enamels having excellent, good, fair, and poor weather resistance*

Specimen identification	Weather resistance rating	Acid resistance (PEI test)	Boiling nitric acid test[a]: Weight loss	Color change
			mg/cm²	*NBS units*
L–11 to 18	Excellent	AA	0.29	2.3
L–1 to 8	Good	AA	26	1.6
L–41 to 48		AA	.30	1.8
P–1 to 8		AA	.44	5.3
L–71 to 78		AA	.58	2.5
P–61 to 68		AA	.88	2.6
L–51 to 58		A	.63	1.9
P–41 to 48		A	.30	0.4
P–71 to 78	Fair	AA	.53	2.5
L–61 to 68		A	.30	1.1
S–51 to 58		A	.93	9.3
S–41 to 48	Poor[b]	A[b]	1.30	7.7
P–51 to 58		A	1.37	3.6
L–31 to 38		A	1.62	10.9
L–21 to 28		B	2.35	9.6
S–21 to 28		B	3.50	23.7
S–31 to 38		C	4.98	33.5
P–31 to 38		C	1.89	6.8
N–41 to 48		C	2.60	17.1
N–51 to 58		C	3.22	14.1
N–1 to 8		C	17.73	51.9
N–11 to 18		C	5.46	50.1
S–61 to 68		C	34.52	50.0
S–71 to 78		C	12.84	46.0
N–21 to 28		D	24.38	56.7
N–31 to 38		D	23.00	60.0
N–61 to 68		D	89.59	37.5
N–71 to 78		D	22.59	39.7

[a] Measurements made on one specimen from each group.
[b] Poor ratings of two of the three A and one of the two B enamels were assigned because of the large color changes noted at the Lakeland site.

7. Discussion

The four most important requirements of a porcelain enamel finish that is to be used for outside exposure are (1) corrosion protection, (2) color stability, (3) ease of cleaning, and (4) absence of major changes in initial gloss.

The results of the 15-yr study show that corrosion protection on a steel base can be obtained with any of the enamels included if proper attention is given to initial coverage. Two coats of enamel should be applied to the back surfaces of all panels and to attachment lugs, except possibly for those installations where absence of moisture on the back surface of the panels can be assured for the life of the installation.

From observations made during the test, and from a study of the literature, the change in color with exposure is believed caused by three effects. The first and most important effect is the slow leaching of soluble constituents from the structural network of the enamel glass, leaving behind a gel layer rich in silica. The hydrated layer usually assumes a lighter color than the original surface and gives a faded appearance to the panel. The layer is probably present on all enamels after long exposure, but only on the enamels of poor acid resistance does it become sufficiently thick after 15 yr to give objectionable changes in color and gloss. It is significant that glass on long exposure shows this same type of gel formation at the surface [15, 16].

The second effect that can change the color of enamel glasses is solar radiation. Parmalee and Badger [17] found a distinct darkening of a certain whiteware glaze when exposed for 2 months to sunlight through a window, or when exposed for a few hours to radiation from either a quartz mercury arc or a carbon arc. Tests made early in the present study [3] showed that there was no detectable change in color for any of the 14 types of enamel after 500 hr of exposure to carbon arc radiation. Nevertheless, solar radiation striking the panels over a 15-yr period could conceivably have an unfavorable effect. In fact, the greater color change for the acid-resistant red enamels at Lakeland, which had the highest annual sunshine, might have been caused, in part, by the effects of solar radiation.

The third factor that might affect color change is the oxidation of the pigment used for coloration. Most ceramic pigments are mixtures of oxides that are known to be chemically stable. The red and orange enamels, on the other hand, are normally pigmented with complexes formed from mixtures of cadmium, selenium, and sulfur, whereas yellow enamels normally contain cadmium sulfide. Such compounds are sensitive to strong oxidizing agents. It is conceivable that a slow oxidation of these compounds could occur during exposure and thereby cause a color change. This effect would be most noticeable in enamels with high pigment concentrations, and especially in those cases where the particles of pigment at the surface were not properly covered by the enamel glass.

The ease of cleaning of enamel surfaces after long exposure is one feature of an enamel finish that helps to make it popular both for signs and for architectural installations. All enamels, with the exception of those with a full-mat finish (see footnote 3), clean easily when first installed. As exposure time increases, however, the finishes of poor acid resistance become etched and cleaning becomes more difficult. After 15 yr of exposure, wide differences in cleanability were noted between acid-resistant and nonacid-resistant compositions, the surfaces with poor acid resistance being more difficult to clean. At the two sites where there was little atmospheric contamination by soot particles (Washington and Lakeland), cleaning was not a problem even on those finishes that had become etched during weathering.

The four exposure sites selected for the tests have fairly high annual rainfalls (see table 2). Moisture is essential to the formation of the gel layer mentioned earlier; therefore, in a dry climate, weathering would be expected to proceed at a slower rate than at Washington, Lakeland, St. Louis, or Atlantic City. Likewise, in an area of extremely heavy rainfall the weathering action would undoubtedly be accelerated.

The direct relation between the acid resistance of an enamel finish and its resistance to weathering is evident in tables 3, 4, and 5. These results are in agreement with the findings of the earlier inspections on the same panels [3, 4]; also with conclusions of other investigators [14, 18, 19, 20, 21, 22].

The citric acid spot test might be considered as a reasonably satisfactory laboratory test for gaging weather resistance if the correlation was as good for the red enamels as for the other types. However, the fading of the red finishes cannot be predicted with certainty by the citric acid spot test; hence for red enamels it should be supplemented with a second treatment that provides strongly oxidizing conditions. The boiling 10-percent nitric acid test appears to fulfill this requirement.

In the present study, all panels were exposed at a 45° angle. This type of exposure would be expected to give a more rapid weathering of the enamel than if the same enamel were exposed on a sheltered vertical wall. It is significant that even under the severe conditions of 45° exposure, the enamels with class AA acid resistance by the PEI test showed only small changes in gloss and color after the 15-yr exposure period.

8. Recommendations

On the basis of the 15-yr data the authors feel justified in making the following recommendations:

1. When the enamel is applied to an iron or low-carbon-steel base, at least two coats of enamel should be applied to the back surface so as to insure good coverage of the metal and prevent damage from corrosion. Good coverage on the face side is an obvious requirement.

2. Only enamels of class A and class AA acid resistance (PEI test) should be used in any architectural installation where general appearance, absence of fading, and ease of cleaning are important. In addition, when the color of the enamel is red, orange, or yellow, only those enamels should be selected that will give a weight loss of less than 1.0 mg/cm^2 when subjected to a solution of boiling 10-percent nitric acid for 2½ hr.

3. Where appearance is an important factor, full-mat enamels of the type included in this investigation should not be used for outside installations, as they tend to accumulate and retain a dirt film. In addition, the colored full-mat enamels fade after short exposure periods.

4. New enamel types, and especially those prepared from screening pastes, should be tested for weather resistance as they are developed to determine whether or not they show the same correlation between acid resistance and weather resistance that was shown by the enamels included in this study. This correlation could be established in periods of considerably less than 15 yr.

9. Summary

An examination of 784 1-ft-square porcelain enameled panels of varying types was made after approximately 15 yr of exposure at Washington, D. C., Lakeland, Fla., St. Louis, Mo., and Atlantic City, N. J. The observations made after this examination may be summarized as follows.

1. Ease of cleaning was related to weather resistance, the enamels showing large losses in gloss being more difficult to clean than those that showed only minor losses.

2. All enamels successfully protected the steel base from corrosion when initial coverage was complete.

3. Poor coverage on the backs of panels resulted in spalls on the face surface at 3 of the 4 locations after 15 yr of exposure.

4. Based on gloss and color-difference measurements on panels cleaned by scouring, the exposure conditions at Lakeland were found to be somewhat more severe on the enamels of high acid resistance whereas the conditions at Atlantic City were slightly more severe on the nonacid-resistant compositions.

5. A direct relationship was found between acid resistance as measured by the citric acid spot test and weather resistance as measured by changes in gloss and color, except that some of the red enamels of good acid resistance showed excessive fading after 15 yr of exposure at Lakeland.

6. A test that involved exposure of red enamels to a boiling solution of 10-percent nitric acid for 2½ hr was found to give a separation of the compositions that faded from those that had superior resistance to fading.

7. The best of the enamels showed no objectionable changes in either color or gloss at any of the four exposure sites.

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General Porcelain Enameling & Mfg. Co.
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Seaporcel Corp.
J. M. Seasholtz & Sons
Texlite, Inc.
Toledo Porcelain Enamel Products Co.
Wolverine Porcelain Enameling Co.

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