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# Three-Year Inspection of Nature-Tone Porcelain Enamels on Steel

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## Three-Year Inspection of Nature-Tone Porcelain Enamels on Steel

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#### M. A. Baker\*

A weather exposure test of nature-tone porcelain enamels on steel was initiated by the National Bureau of Standards and the Porcelain Enamel Institute in 1966. Laboratory measurements for changes in gloss and color were made after the enamels had been exposed for 0.5, 1 and 3 years at Kure Beach, North Carolina, Miami, Florida, and Gaithersburg, Maryland. The average gloss and color retained for all 450 specimens exposed at 3 sites for 3 years were 90.5 and 99.1 respectively. There was a tendency for the enamels exposed at the marine sites to rust around pinhole-type defects. Scanning electron microscope studies of these defects showed a layer of readily corrodible enamel on the bottom of the defects. A high-voltage test for continuity of coating was effective in detecting specimens that were apt to rust during the first year's exposure.

Key words: Acid resistance; color; continuity of coating; porcelain enamel; weather resistance.

#### 1. Introduction

Porcelain enamel has been used as an exterior finish for buildings ranging from gasoline filling stations to high rise office buildings. The wide variety of colors available, the ease of cleaning and the relative ease of installation are factors contributing to the use of porcelain enamel in architectural applications. The weatherability of porcelain enamels has been evaluated by exposure testing  $[1,2,3]^1$  and these exposure test data have been correlated with accelerated laboratory test data. These data provide manufacturers of porcelain enamels with laboratory tests to estimate the weatherability of their products.

As new enamel types are developed, it is necessary to subject them to exposure testing to determine whether the accelerated tests used in the past are still accurate estimators of the enamels weatherability. This report is a summary of the first three inspections of a new series of enamels called nature-tones which are characterized by low gloss, muted, and earthy colors.

#### 1.1 Description of enamels and specimens

There were twenty-five different enamels included in this program. Twenty of these enamels are the standard nature-tone colors 101-120 [4] while the five other enamels 1,3,4,6, and 7 in table 1 are a yellow, tan, orange-beige, green and grey-beige color respectively. Each enamel was commercially applied to twenty 4 x 6 inch and twenty-eight 4-7/16 inch square panels of enameling grade steel.

\*Research Associate from the Porcelain Enamel Institute at the National Bureau of Standards, Washington, D.C., 1964 to present.

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

Enamel	45°	Acid	Acid	Thickness	Continuity of	Coating		
	Specular	Spot	Solubility	(mils)	Probe Voltage	Overvoltage		
	01033	Nacing						
101	13.8	В	14.4	8.8	1.5	0.6		
102	13.5	В	9.5	8.0	2.0	1.2		
103	13.3	A	8.3	7.5	1.5	0.7		
104	17.4	В	7.4	9.2	2.0	1.1		
105	14.9	A	1.6	9.6	1.0	0.1		
106	27.3	А	7.8	9.1	2.0	1.1		
107	14.4	A	1.8	9.9	1.5	0.5		
108	19.2	A	1.2	9.4	2.5	1.6		
109	16.6	A	0.7	9.2	2.5	1.6		
110	20.7	AA	0.8	9.0	2.0	1.1		
111	18.5	A	1.0	10.8	2.5	1.5		
112	13.5	A	0.9	10.7	2.0	1.0		
113	10.1	А	1.0	10.8	2.5	1.5		
114	15.0	А	1.0	8.8	2.0	1.1		
115	9.3	A	0.9	10.6	2.5	1.5		
116	16.8	А	0.9	9.9	2.5	1.5		
117	20.7	A	2.4	8.7	2.0	1.1		
118	14.7	A	2.4	10.2	2.5	1.6		
119	27.7	A	3.9	9.4	2.5	1.6		
120	21.2	A	0.7	9.2	2.0	1.1		
1	24.4	Å	2.6	9.0	2.0	1.1		
3	20.5	A	3.0	9.6	2.5*	1.6		
4	11.3	А	2.8	8.8	2.0	1.1		
6	2.2	В	8.4	9.1	2.0	1.1		
7	3.4	В	6.9	9.4	2.5	1.6		

Table 1. Summary of Pre-Exposure Data for Enamels in the 1966 Exposure Test of Nature-Tone Enamels on Steel.

\* All of the specimens exposed at Miami and Gaithersburg passed the highvoltage probe at 2.5 kV, but the specimens exposed at Kure Beach passed the high-voltage probe at 1.5 kV.

#### 1.2 Exposure Sites

Six specimens of each enamel were exposed at each of three exposure locations: the exposure site of the National Bureau of Standards, Gaithersburg, Maryland; the tidewater exposure site of the South Florida Test Service, Miami, Florida; and the 80-foot exposure site of the International Nickel Company, Kure Beach, North Carolina. The 4-7/16 inch square specimens were exposed at Miami and Gaithersburg while the 4 x 6 inch specimens were exposed at Kure Beach. The specimens at Miami and Gaithersburg were exposed at an angle of 45° to the horizontal and they faced south. These exposure parameters were modified for the enamels exposed at Kure Beach where the enamels faced the ocean at east-southeast and were exposed on the racks of the International Nickel Company which were at an angle of 30° to the horizontal. In addition to the exposed specimens, three specimens of each enamel were kept in indoor storage and were used as reference panels. The remaining specimens of each enamel were kept as laboratory specimens to be used for routine testing and new test development.

#### 1.3 Cleaning of Specimens

The specimens were cleaned both before and after exposure by scouring 30 strokes with a sponge that had been sprinkled with calcium carbonate and moistened with a one percent solution of trisodium phosphate. Following scouring, the specimens were rinsed successively with tap water, distilled water and alcohol. Before this method of scouring was selected for use, a glossy and a matte enamel were scoured for 660 strokesby hand with the calcium carbonate abrasive on a cellulose sponge. The gloss was measured after scouring for given intervals. The glossy enamel was not affected by scouring 660 strokes, while the matte enamel showed a 3 percent increase in gloss after scouring 260 strokes and an 18 percent increase after scouring 660 strokes. It, therefore, appeared as though scouring might have an effect on the enamel's gloss but this effect would be minimal through the first eight inspections if the enamels are scoured only 30 strokes per inspection as specified.

#### 1.4 Enamel Thickness

The enamel thickness was measured, at one area on each of seven laboratory specimens of each of the two sizes. These 14 measurements were averaged to give the thickness values reported in table 1. The maximum standard deviation of the fourteen measured values was 1.0 mil among the 25 different enamels.

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#### 1.5 Continuity of Coating

The high viscosity inherent with matte enamels makes it difficult for bubbles formed during the fusing of the enamel to travel through the enamel and for the enamel to flow back together once the bubble has burst. This may result in some discontinuities in the finished nature-tone specimens. In order to insure that part of the porcelain enameled specimens placed on exposure were free from any apparent discontinuities, one half of the specimens were placed on exposure on the condition that they pass the high-voltage continuity of coating test [5] without any discontinuities being located. The other half of the exposure specimens were selected at random from the original lots of 48 specimens of each enamel.

The probed specimens were tested at arbitrarily selected voltages of 2 or 2.5 kV dc. When too many specimens failed at these voltages, and the possibility of running out of specimens before the required number passed the continuity of coating test existed, the probe voltages were lowered to 1 or 1.5 kV dc. The actual probe voltages and overvoltages<sup>2</sup> are presented in table 1.

#### 1.6 Color

The three tristimulus color values [6] necessary to evaluate color change were measured with a color difference meter both before and after exposure. One of the three storage specimens was used as the color standard. The color change after exposure is reported as color retention which is 100 minus the color change in NBS units<sup>3</sup>.

#### 1.7 Gloss

The 45° specular gloss was measured both before and after exposure at four orientations at 90° to each other near the center of the specimen. Since these enamels have low gloss values, the gloss meter was calibrated with a glazed ceramic tile having a specular gloss of 15. The initial specular gloss values are presented in table 1. The gloss retained after exposure is reported as the percentage of the initial gloss retained.

Color Change ( $\Delta E$ ) =  $\sqrt{\Delta (10 \sqrt{Rd})^2 + \Delta a^2 + \Delta b^2}$ 

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<sup>&</sup>lt;sup>2</sup>The term overvoltage is used to designate the difference between the applied test voltage and the air gap voltage for an air gap whose length is estimated by the enamel's thickness.

<sup>&</sup>lt;sup>3</sup>The color difference parameters, Rd (the 45°, 0° luminous daylight reflectance), a (red-green), and b (yellow-blue) were measured with a color difference meter. The color change in NBS units was calculated by the following formula:

The acid resistance of porcelain enamels has been used as an indicator of the enamels' weatherability [2,3]. The standard test used to determine the acid resistance is the citric acid spot test [7]. This test was performed on triplicate laboratory specimens of each enamel and these acid spot test ratings are presented in table 1. All the enamels had acid spot test ratings of AA, A or B which is indicative of good weather resistance even though enamels with AA and A acid spot test ratings are the only ones recommended for architectural applications [8].

Since the citric acid spot test is difficult to evaluate on matte enamels, triplicate specimens of these enamels were also subjected to the boiling citric acid solubility test [9]. The results of this test are presented in table 1 where it can again be seen that the enamels in this test have good acid resistance as indicated by their low boiling-acid solubilities.

#### 2. Results and Discussion

#### 2.1 Color Retention

The color retention after 0.5, 1, and 3 years' exposure for these enamels is presented in table 2. These data indicate that the 450 enamels had an average color retention of 99.1, after 3 years' exposure.

The color retention data are also plotted as a function of exposure time in figure 1. These data indicate that the color change is greatest during the first six months' exposure after which it tends to reach a constant rate of change. If the color continues to change at the current rate, these enamels will change color on the average of 0.2 NBS units per year.

#### 2.2 Gloss Retention

The gloss retained after 0.5, 1 and 3 years' exposure is presented in table 2. These data indicate that enamels with higher initial gloss tend to decrease in gloss during exposure while the enamels with the lower initial glosses tend to increase in gloss. This apparent increase in gloss is probably caused by a combination of three effects: 1) the natural degradation of the enamel caused by exposure to the weather, 2) the polishing action of the scouring given these specimens during cleaning and 3) slight errors in measuring the gloss of enamels with extremely low gloss values. Therefore, it should be kept in mind that these gloss data are probably less representative of the effect of weathering than the color data.

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Summary of Color and Gloss Retention for the Enamels Exposed in the 1966 Exposure Test of Nature-Tone Enamels on Steel. Table 2.

	2	3-Yr	76.0	1 1 1	5 VL	73.1	74.6	,	04./ 73 8	80.8	90.2	88.6	0	80.9	98.6	9.7.4	76.3	85.2	78.1	72.7	80.3	67.8	72.5	71.2	5.9.2	79.8	192.2	142.4	84.9
	ersbur	1-Yr	α ο	0.77 26 27	0.07	89.1	85.0	,	81.9 83.83	95.2	1.66	98.0		90.4	101.4	99.8	87.0	37.8	87.9	83.4	86.5	85.3	86.6	84.9	82.5	80.6	139.8	112.5	91.0
t	Gaith	0.5-Yr	2 7 X		4 - 10 4 - 1	98.5	90.7		1.06	94.1	103.6	102.1	0	94.3	101.7	C•201	91.4	95.0	92.5	87.4	95.0	92.5	92.5	91.3	90.5	87.5	133.7	110.0	95.6
percen		3-Yr	7 08	a 18		2.06	89.2	1	1°02	91.2	87.1	86.5	5	0.14	95.0	T 03 • T	91.5	97.6	78.1	84.1	85.3	85.1	90.5	83.8	84.3	92.6	177.7	149.8	95.5
ained,	Miani	1-Yr	7 C0	0 t 0 t	90.6	99.3	89.2	000	9.06	99.6	97.8	93.3	1	1.99.1	102.6	105.7	92.1	93.6	94.0	87.1	93.7	93.1	96 L	88.0	88.3	89.2	144.7	130.0	96.7
oss Reta		0.5-Yr	03 7	1.0%	0.6.2	98.1	93.7		93.6	101.6	101.7	102.4		T 02.2	104.6	107.3	94.9	97.5	97.4	90.2	94.7	96.8	99.5	93.0	94.2	92.3	151.7	132.5	9.001
G1		3-Yr	78 1	13 0	72.5	7.49	79.9	0	78.0	92.5	97.2	101.2	0.00	32.9	94.3	93.9	76.2	86.5	79.8	74.3	72.4	70.5	74.8	74.8	70.1	71.6	158.6	139.9	84.9
	Beack	1-Yr	84.6	76.6	88.9	83.5	87.2	, , ,	85.0	97.8	100.5	101.9	( L	0.06	102.7	104.9	89.1	89.9	91.0	84.4	92.8	86.6	53.8	82.4	85.0	85.9	142.6	125.2	93.4
	Kure	0.5-Yr	ر م	6,08	90.8	89.9	87.5		89. D	102.9	L04.8	103.1	0	8.66	101.5	105.3	89.2	91.6	93.1	87.1	93.9	90.0	93.4	86.4	88.0	89.2	145.2	127.3	96.2
	60	3-Yr	98 5	5 20	99.3	0.66	99.1	r	99.1	99.5	99.7	9.66	1	77.L	99.5	93.4	99°2	99.6	99.4	98.6	99.4	99.9	0.66	99.2	99.4	98.7	9.66	99.5	1.66
	angsua	1-Yr	99 1	1.20	99.5	9.66	99.5	000	99.6	99.5	99.2	7.96		0.66	9.6	99.8	99.3	7.96	99.3	99.2	99.3	99.6	93.6	7.66	99.1	99.3	99.7	99.5	99.5
	Gaith	0,5-Yr	6 00	1 8 80	9.66	9.66	9.66	·	99.4	99.5	9.66	99.8		9.9.0	99.5	99.6	99.5	99.6	99.5	99.4	99.7	99.8	99.4	99.6	99.3	99.6	99.8	99.6	99.5
on a/		3-Yr	۵۵ ع	00	99.3	9.66	99.4	L C	00°.066	99.2	99.3	99.7		7.6A	99.6	99.3	99.7	99.4	99.5	99.4	93.8	9.66	99.6	69.7	98.6	99.3	99.66	99.4	99.3
etenti	iani	1-Yr	5 00	0 00	0.99	9.9.5	9.66		99°.4	99.5	99.5	7.66		1.66	99.6	99.5	99.5	99.4	99.66	99.5	99.5	99.8	99.6	7.66	99.3	99.5	99.8	99.3	99.5
Color R	Σ	0.5-Yr	99 5	03.0	9.86	9.9.8	99°7	r C	99.5	7.66	5.69	9.66	г (	1.66	66°	99.4	9.66	99.6	7.99	99.7	99.1	99.7	9.66	99.8	99.5	39.5	99.8	99.5	99.6
		3-Yr	98 5	0. 22. 25.	98.9	0.66	98.9	L CO	2.06	99.5	9.66	7.06	: 	93.2	99.4	199.7	0.66	99.3	98.4	97.9	39.2	0.66	98.7	98.9	98.8	97.9	99.4	99.8	98.9
	each	1-Yr	99.7	10.66	99.5	5.66	99.66	000	5.99	99.5	99.5	7.66		7.60	9.66	99.4	99.5	99.5	99.8	99.2	99.5	9.6	99.6	99.5	99.3	99.3	99.8	99.7	99.4
	Kure Bu	0.5-Yr	1.00	4 X X 6	9.96	9.66	9°66	00	99.6	99.5	9.9.8	99.8		99.4	99.7	99.6	9.9.8	99.6	99.5	99.3	99.5	99.6	7.96	99.5	99.4	99.4	99.8	99.5	99.5
Enamel			101	102	103	104	105	, ( , r	107	108	109	110	Ţ		112	113	114	115	116	117	118	119	120	1	e	7	9	7	Average

 $\underline{a}/_{\rm Color}$  retention is 100 minus the color change in NBS units.



Figure 1. Effect of exposure time on the color retention of nature-tone enamels on steel.



Figure 2. Effect of exposure time on the gloss retention of nature-tone ename on steel.

#### 2.3 Corrosion of the Base Metal

One of the major reasons for applying porcelain enamel is to protect the base metal from corrosion. Therefore, it is of interest to determine whether newly developed enamels also provide this protection. After cleaning, the enamels were visually inspected for signs of corrosion of the base metal. This corrosion was noted by rust around pinhole-type defects or by pinhead-sized iridescent spots. The iridescent spots were thought to be forerunners of the rust occurring around pinhole-type defects. The total number of specimens with these two types of defects is presented in table 3. The data in table 3 indicate that the high-voltage continuity of coating test was fairly efficient in predicting the performance of enamels with discontinuities that became evident after 6-months' and 1-year's exposure. However, this difference between selected and unselected enamels did not continue after 3 years' exposure. It was hypothesized that the apparent failure of the test to detect enamels that are apt to rust after longer periods of exposure was caused by the existence, at the bottom of the pinhole-type defects, of a thin layer of glass that is less corrosion resistant than the bulk of the enamels. This glass layer would protect the base metal for a short period but would corrode itself after longer exposure periods. The existence of a thin layer of glass would increase the voltage required to locate a discontinuity.

Figure 3,4, and 5 are scanning electron micrographs of three of these pinhole-type defects. Figure 3 confirms the existence of a thin layer of apparently less corrosion-resistant glass on the bottom of a pinhole-type defect. The glass is beginning to crack, and bits of corrosion products can be seen collecting on the cracked glass surface. Once the glass has cracked, the corrosion continues as evidenced in figure 4 which shows the bottom of another pinhole-type defect completely covered with the corrosion product. Figure 5 shows another defect where the corrosion has evidently progressed further. The corrosion products apparently collected and built up enough pressure to crack the enamel surface and open up the defect thus allowing much of the corrosion product to be removed.

These figures verify the existence of glass at the bottom of pinhole-type defects that rust after exposure. This indicates that higher overvoltages may be needed when testing for continuity of coating to insure that the enamels will not rust when placed on exposure.

No evidence has been found as yet to verify that the iridescent areas are indeed forerunners of rust. Therefore, the specimens with rust spots appearing on the enamel's surface after three years' exposure are presented in table 4. Again there is no significant difference between the enamels that passed the continuity of coating test and those that did not in regards to rust after three years' exposure.

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Summary of enamels with rust appearing on the surface after three years' exposure. Table 3.

·		-	-		-	_	-	_	-	_	_			-	-	_	-	_	_	_		-	_	-	-	_	_	-		-			-	_	1
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Specimens in this column passed the high-voltage continuity of coating test prior to exposure. Specimens in this column were not tested for continuity of coating prior to exposure.

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Figure 3. Scanning electronmicrograph of the bottom of a pinhole-type defect that rusted after being exposed for three years at Kure Beach. Note the cracks and corrosion products at the bottom of the defect. Original magnification 1150 times.



Figure 4. Scanning electronmicrograph of the bottom of a pinhole-type defect that rusted after after being exposed for three years at Kure Beach. Note the build up of corrosion products on the bottom of the defect. Original magnification 700 times.



Figure 5. Scanning electronmicrograph of the bottom of a pinhole-type defect that rusted after being exposed for three years at Kure Beach. Note the cracks in the enamel near the pinhole opening and the relative lack of corrosion products inside the pinhole. Original magnification 560 times. Table 4. Summary of enamels with obviuosly rusted surfaces after three years' exposure.

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Specimens in this column were not tested for continuity of coating prior to exposure.

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The three-year color and gloss data in table 2 were subjected to a two sided sign test [10] at the 95 percent confidence level to determine whether the atmospheric conditions at the three sites caused the enamels to change color and gloss at significantly different rates. These analyses indicated that, even though the color changes are extremely small at all sites, the color changed significantly more at Kure Beach than at either Miami or Gaithersburg, while it did not differ significantly at the latter two sites.

The gloss data for the enamels exposed at these three sites indicated that the greatest gloss changes occurred at Gaithersburg, followed by those exposed at Kure Beach and Miami.

These data indicate that the color and gloss change at different rates. This apparent difference is probably caused by the increase in gloss of many of the enamels with very low initial glosses. It is probable that the ranking of sites based on color change is more reliable than the rankings based on gloss retention.

The two marine exposure sites, Kure Beach and Miami, were included in this test to determine whether the atmosphere at Miami, an exposure site operated by a commercial testing laboratory, was as severe as that at Kure Beach, a privately owned exposure site not normally available to the general public. There are three indicators of an exposure site's severity: changes in color; gloss retention; and the number of specimens placed on exposure which had rust occurring on the enamel surface. Since it was shown above that color change is a better indicator of the site severity than gloss retention, only the color change and the number of specimens with rust on the surface will be considered when determining whether Miami could replace Kure Beach as a severe marine environment. These two parameters indicate that the environment at Miami is not quite as severe as that at Kure Beach. However, it appears to be much more severe than Gaithersburg when the corrosion of the base metal is considered. The Miami site could, therefore, be used by frit companies or enamelers for the relatively rapid (2-3 yr) evaluation of new enamels as they are developed. Once it was determined that the Miami site was suitable for the rapid evaluation of new enamels, exposure testing at Miami was discontinued.

#### 2.5 Comparison with Enamels in the 1956 Exposure Test

The color retention data for the enamels exposed three years at Kure Beach and Gaithersburg are presented in table 5 together with the color retention data for glossy acid resistant enamels exposed at Kure Beach and Washington in the 1956 exposure test [11]. (The Washington and Gaithersburg exposure sites are only 20 miles apart and are thought to be similar enough exposure conditions for direct comparison. These data indicate that the nature-tone enamels have better color retentions than the glossy acid-resistant enamels on steel tested previously [11]. Therefore, if the rusting tendency of the nature-tone enamels can be overcome, they may have better weather resistance than conventional glossy porcelain enamels on steel.

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Table 5. Comparison of nature-tone enamels with the acid-resistant enamels in the 1956 exposure test after three years' exposure.

Exposure	Enamel Type	Average Color Retention for Enamels Exposed										
Test		Kure Beach	Washington-Gaithersburg									
1956	Acid-Resistant on Steel	97.9	98.2									
1966	Nature-tone on Steel	98.9	99.1									

#### 3. Plans for Future Exposure Testing

The enamels at Kure Beach and Gaithersburg were returned to the exposure racks for additional weathering. The next inspection of these enamels is tentatively scheduled for 1974 after seven years' exposure.

#### 4. Summary

An examination of 450 nature-tone porcelain enamels on steel was made after 0.5, 1 and 3 years' exposure at Kure Beach, Miami, and Gaithersburg. A summary of the findings follows:

- 1. Enamels with good acid resistance hadaverage color retentions of 98.9, 99.3, and 99.1 at Kure Beach, Miami, and Gaithersburg respectively.
- 2. Enamels with good acid resistance had average gloss retentions of 84.9, 95.5 and 84.9 percent at Kure Beach, Miami, and Gaithersburg respectively.
- 3. Some enamels may have pinhole-type defects which permit early corrosion of the base metal, particularly at the severe exposure sites such as Kure Beach and Miami.

4. Many of the enamels with pinhole-type discontinuities that rusted after 0.5,to 1 year's exposure could be detected with the high voltage continuity of coating test at overvoltages ranging form 0.1 to 1.6 kV dc.

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