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NATIONAL BUREAU OF STANDARDS REPORT

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Progress Report

July 1, 1968 Through September 30, 1968

DEVELOPMENT OF METHODS OF TEST FOR QUALITY CONTROL OF PORCELAIN ENAMELS

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS

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DEVELOPMENT OF METHODS OF TEST FOR QUALITY CONTROL OF PORCELAIN ENAMELS

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U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

SUMMARY

The effect of storing specimens and cleaning tissues, used in the cleanability test, at controlled relative humidity prior to testing was investigated. The results obtained indicate that a conditioning treatment may be an important factor in day-to-day reproducibility.

The one-year inspection of nature-tone enamels on steel was completed with the inspection of the enamels on steel exposed at Gaithersburg.

Four hundred grit silicon carbide paper and fifteen seconds abrasion time were selected for the scratch-abrasion test procedure.

I. CLEANABILITY

INTRODUCTION

Previous reports in this series have recorded the development of a method to measure the cleanability of porcelain enamels and other materials used in similar situations. The test involves the application of a formulated fluorescent, water soluble soiling agent. The soil is mechanically spread and later partially removed through the use of cleaning tissues and the uniform motion provided by automatic metallurgical polishing equipment. The amount of soil remaining on a known area after the cleaning operation, is extracted with a water solvent, determined by a fluorometric analysis and used as an indicator of the cleanability of the tested surface. It should be emphasized that many of the manipulative steps in the procedure must be carefully defined and controlled in order to obtain reproducible results. A time consuming quest has yielded many of the procedural steps which influence the uniformity of the results obtained. The occasional occurrence of out-of-line results prompted the search for yet another variable which, if controlled, would make more certain the ability of a single operator to reproduce previous determinations.

RESULTS AND DISCUSSION

Several experiments were made to determine if a relationship existed between the amount of soil originally applied to a specimen and the amount of soil retained after the uniform cleaning steps. The amount of soil used was one drop from a hypodermic syringe held vertically above a specimen. In these experiments the weight of each drop of soil, dispensed as above, was determined by before-and-after weighing of the specimens. In a series of twenty-four determinations on the same six specimens, the size of the drops varied from 0.029 to 0.037 gram. Table 1 summarizes the results obtained in these tests. No way has been found to establish or show any dependence of the amount of soil retained on the amount of soil originally used (within the narrow range of these experiments). If there is a relationship between these parameters it appears to be entirely masked by the influence of some other variable step in the procedure. The lower part of Table 1 gives results obtained in an earlier period of testing the same test pieces.

Another experiment was performed in an attempt to assign a cause for occasional divergent results. The method of wet cleaning in the proposed test appears to be markedly dependent upon the amount of moisture added to the tissue used for cleaning. It was hypothesized

that the residual moisture content of the extremely absorbant tissues might vary from day to day if there were significant changes in the moisture content of the laboratory atmosphere. It was also considered possible that the surface of the test specimens might vary in the amount of adsorbed moisture. Observations on four different days given in the top part of Table 2 show that there was indeed a difference of five or six milligrams, from day to day, in the amount of residual moisture contained in that part of the tissue which was effective in the cleaning process. The amount of water added to the tissues prior to the cleaning was 21 microliters or approximately 21 milligrams. Thus it appeared that better control of the residual moisture in the tissue might result in values with improved day-to-day repeatability.

Two desiccators, charged with saturated solutions of K_2CO_3 and of NaCl were used to maintain conditioning atmospheres of about 45 and 75 percent relative humidity at room temperature. The lower part of Table 2 shows that the moisture contents of tissues conditioned in these atmospheres were moderately uniform and varied significantly between 45 and 75 percent relative humidity.

Three of the surfaces tested during a previous period showed an unfortunate scatter in the day-to-day values of soil retained. Values previously reported for these surfaces are given in the first column of Table 3. No special conditioning treatment was provided for the specimens or the tissues used in the cleaning and no record was made of the relative humidity of the laboratory atmosphere during the previous tests. The second and third columns of Table 3 give the results obtained over a period of days for the same specimens. The tissues used had been conditioned at the indicated relative humidity through the use of the desiccator hygrostats. The range of the values obtained on three different days may be used as an estimation of the degree of repeatability. The first porcelain enamel and the Ni-Cr plated steel gave some indication of improved repeatability when the tissues used were conditioned at 45 or 75 percent relative humidity. No improvement was noted for the second porcelain enamel. Limited results given in the last column of Table 3 were either fortunate or indicated that it might be important to condition <u>both</u> the tissues and the specimens prior to test.

It is felt that the use of a hygrostat for conditioning specimens and tissues is more convenient and economical than the elaborate control of laboratory humidity. Further work must be done to confirm or deny the contribution of these conditioning treatment to reliable results.

II. WEATHERING TESTS OF PORCELAIN ENAMELS

INTRODUCTION

One of the continuing research efforts of the research associates has been the weathering tests of various porcelain enamels. During this report period the one-year inspection of the 1966 Exposure Test of Nature-Tone Enamels on Steel was completed with the inspection of the enamels exposed at Gaithersburg and the 3-year inspection of the enamels in the 1964 Exposure Test of Porcelain Enamels on Aluminum was begun with the enamels exposed at New York City.

INSPECTION PROCEDURE

1. Cleaning the Specimens

Before meaningful gloss and color measurements can be made on specimens that have been exposed it is necessary to remove accumulated dirt, fingerprints, etc. from the enamel surface. The procedure selected for cleaning the enamels in the above tests was to 1) scour 30 strokes with a sponge that had been moistened with a solution containing one weight percent trisodium phosphate and sprinkled with calcium carbonate, 2) rinse with tap water, and 3) rinse with distilled water and 4) rinse with alcohol.

2. Gloss and Color

The 45° specular gloss of the specimens was measured at four orientations near the center of the specimen. The gloss is reported as the percentage of initial gloss retained after exposure.

The change in color was measured with a color difference meter. One of the three storage specimens of each enamel was used as the standard in measuring the color difference. This was done to obtain maximum efficiency with this type of instrument. The storage specimens were, in turn, measured against NBS Color standards to determine whether the enamels have changed color during cleaning and storage.

3. Visual Examination

The enamels in the 1966 Exposure Test of Nature-Tone Enamels on Steel were examined visually to see if there was any evidence of rust occurring on the specimens.

RESULTS AND DISCUSSION

A. 1964 Exposure Test of Porcelain Enamels on Aluminum

Since the enamels exposed at the other four sites will be inspected during the next report period, the results for the enamels exposed at New York City will be reported at the end of the Next Report Period.

B. 1966 Exposure Test of Nature-Tone Enamels on Steel

1. Cleaning of Specimens

The enamels exposed at Gaithersburg were covered with a loosely adherent layer of dirt which was readily removed by the standard cleaning procedure.

2. Gloss and Color

The gloss and color were measured as previously described. These data have been reduced to the percentage gloss retained and color retention and are presented in Table 4. Both the gloss and color retention of these enamels appear to be very good at this time.

3. Continuity of Coating

The visual inspection of the enamels exposed at Gaithersburg revealed only one specimen (enamel 105) that had rusted and this specimen was one that had not been tested for continuity of coating before being exposed.

PLANS FOR NEXT REPORT PERIOD

A. 1964 Exposure Test of Porcelain Enamels on Aluminum

During the next report period the enamels exposed at Montreal, Kure Beach, Washington, and Los Angeles will be inspected after three-years' exposure. Following this inspection, a report will be written for publication summarizing the findings of the 6-month's, one-year, and three-year inspections.

B. 1966 Exposure Test of Nature-Tone Enamels on Steel

A separate report on the six-months' and one-year inspections will be prepared. The next inspection is scheduled after three-years' exposure.

III. SCRATCH-ABRASION

INTRODUCTION

After a period of service many domestic appliances show a random pattern of small scratches resulting from abrasion of various sorts. These scratches tend to collect dirt and make its removal difficult. It was thought that an appropriate test for abrasion resistance might impose a random scratch damage similar to that often found in service. The change in cleanability of a surface before and after abrasion might serve as a sensitive indicator of abrasion damage.

RESULTS AND DISCUSSION

The specimens were abraded on the same metallurgical polishing apparatus that is used in the cleanability test except the head used in the cleanability test was replaced with one covered with an abrasive paper. During this report period, the effect of different abrasive papers and abrasion times was studied.

A. Effect of Abrasive Paper

During the last report period, several abrasive papers had been investigated and it was found that somewhere between 240 and 400 grit the enamel was scratched without causing spalling to occur. During this report period, 360 and 400 grit papers were investigated. Neither of these grits caused the enamels to spall around the scratch. The change in soil retained for the different surfaces that were abraded are given below:

Surface	Change in Soil Retained - $\mu g/cm^2$	
	360 SiC	400 SiC
Enamel "0"	0.40	0.12
Enamel "F"	2.21	2.98
Acrylic "15"	21.97	19.82

Since there was only a slight difference in the change in soil retained for these two papers, the 400 grit paper was chosen because it is more widely available.

B. Effect of Abrasion Time

Five different surface finishes (3 organic and 2 porcelain enamel) were abraded for times varying from 15 to 150 seconds. The changes in soil retained for the different abrasion times are presented in Figure 1. The decrease in soil retained at the longer abrasion times for the organic finishes (18 and 19) is probably caused by the abrasive paper becoming clogged with the organic material. Two ways to overcome this are to change the abrasive paper at frequent intervals so it doesn't become clogged or to choose an abrasion time short enough so the paper would not become clogged. The latter alternative, a 15-second abrasion time, was selected.

PLANS FOR NEXT REPORT PERIOD

During the next report period it is planned to abrade several additional surfaces that were submitted for use in the cleanability test procedure.

IV. CONTINUITY OF COATING

During the current report period, a test procedure for continuity of coating of porcelain enamels and a paper describing the development of this test procedure have been written and submitted for editorial review.

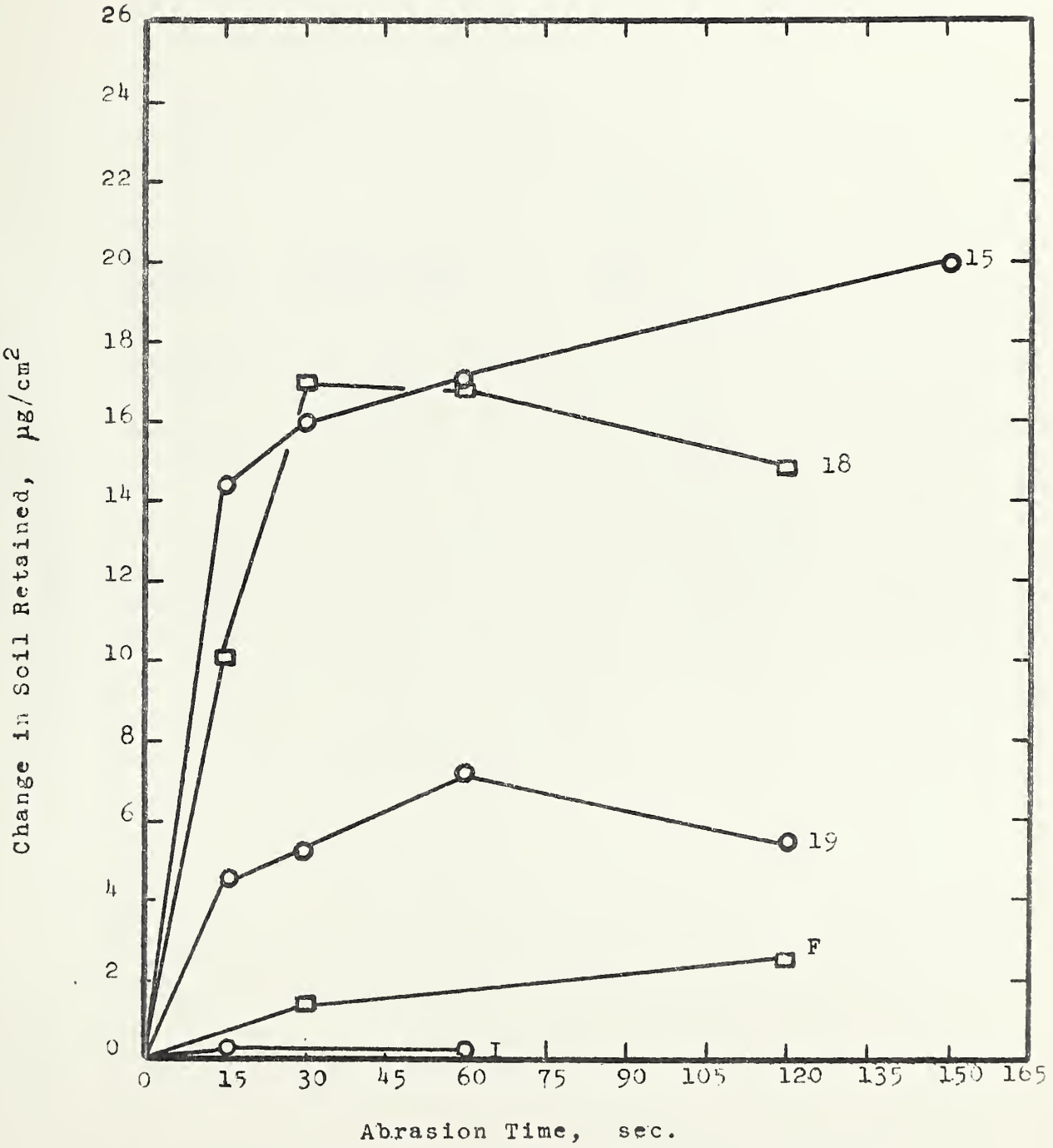

Figure 1. The effect of abrasion time on the change in soil retained.

Table 1. The Agreement of Repeated Determinations of the Soil Retained on an Anodized Aluminum.

Date of Testing	Average Size of Soil Drops	Mean Value of Soil Retained for Six Specimens Measured as a Group
	g.	$\mu g/cm^2$
8-30-68	0.0346	7.5
"	.0342	7.6
"	.0308	7.6
9-3-68	.0353	6.2
5-24-68	---	7.1
5-27-68	---	7.9
5-28-68	---	7.7
6-21-68	---	7.5

Table 2. Adsorbed Moisture on Cleaning Tissues Conditioned in Atmospheres of Various Relative Humidities.

Conditioning Atmosphere	Time in Atmosphere Prior to Weighing	Moisture Found in 25.6 cm^2 of Tissue a/	Range of Values
	hrs	mg	mg
Ambient room atmosphere	24	4.9	
	"	11.3	6.4
	"	9.7	
	"	10.5	
45 percent R.H. hygrostat (K_2CO_3)	3	10.0	
	15	10.6	1.5
	24	9.3	
	72	9.1	
	Mean	9.8	
75 percent R.H. hygrostat (NaCl)	24	16.6	
	"	16.2	
	"	13.6	3.0
	"	15.4	
	"	14.1	
	"	14.2	
	Mean	15.0	

a/ Conditioned tissues were weighed, oven dried and re-weighed to determine the absorbed moisture. Each value given is the mean of the moisture found in six individual tissues on a given day or time. The area of the cleaning head in contact with the specimen was 25.6 cm^2.

Table 3. The Effect of Relative Humidity on the Repeatability of the Cleanability Index.

Specimen Type	For Tissues: For Specimens:	Ambient R.H. Ambient R.H.	45% R.H. a/ Ambient R.H.	75% R.H. a/ Ambient R.H.	45% R.H. b/ 45% R.H. b/
		Ave. Soil Retained c/ $\mu g/cm^2$	Ave. Soil Retained c/ ug/cm^2	Ave. Soil Retained c/ ug/cm^2	Ave. Soil Retained c/
Porcelain Enamel		1.66	1.76	1.78	1.70
		1.31	1.62	1.74	1.43
		2.06	1.57	1.86	1.51
	Mean	1.68	1.65	1.79	1.55
	Range	0.75	0.19	0.12	0.27
Ni-Cr Plated Steel		4.28	5.69	6.40	
		3.19	5.17	6.09	
		4.73	5.87	5.74	
	Mean	4.07	5.58	6.08	
	Range	1.54	0.70	0.66	
Porcelain Enamel		4.06	5.35	4.38	4.87
		3.74	4.04	5.79	4.82
		4.65	3.99	----	4.87
	Mean	4.15	4.46	4.86	4.85
	Range	0.91	1.36	1.41	0.05

a/ Cleaning tissues were conditioned overnight, prior to the test, in a hygrostat over a saturated solution of K_2CO_3 (45% R.H.) or NaCl (75% R.H.). A glass desiccator charged with the appropriate solution was used.
b/ Both specimens and cleaning tissues were conditioned overnight in the 45% R.H. hygrostat prior to test.
c/ Each value given is the mean of six individual specimens on a single day. The spread or range of the values on three different days was used as a measure of the repeatability.

Table 4. Summary of Data for Nature-Tone Enamels on Steel Exposed at Gaithersburg

Enamel	Color Retention		Percentage Gloss Retained	
	Six Months	One Year	Six Months	One Year
101	99.20	99.13	87.46	82.78
102	98.76	98.73	82.15	76.77
103	99.60	99.45	91.49	84.92
104	99.58	99.63	98.48	89.07
105	99.58	99.54	90.70	84.99
106	99.36	99.30	90.11	81.86
107	99.70	99.58	90.11	83.83
108	99.50	99.48	94.05	96.17
109	99.63	99.20	103.62	99.10
110	99.82	99.69	102.11	98.04
111	99.56	99.46	92.29	90.36
112	99.47	99.62	101.68	101.39
113	99.61	99.75	102.45	99.82
114	99.54	99.31	91.37	87.04
115	99.57	99.69	94.96	87.76
116	99.45	99.70	92.49	87.91
117	99.44	99.20	87.42	83.39
118	99.65	99.31	95.04	86.48
119	99.75	99.55	92.45	85.31
120	99.40	99.64	92.46	86.58
1	99.63	99.69	91.33	84.89
3	99.31	99.09	90.48	82.48
4	99.57	99.25	87.47	80.63
6	99.84	99.73	133.73	139.82
7	99.56	99.50	109.95	112.50


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