NATIONAL BUREAU OF STANDARDS REPORT

9004

PROGRESS REPORT

July 1 through September 30, 1965

Development of Methods of Test
For Quality Control of Porcelain Enamels

PORCELAIN ENAMEL INSTITUTE RESEARCH ASSOCIATESHIP
NATIONAL BUREAU OF STANDARDS
WASHINGTON, D.C.

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards is a principal focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. Its responsibilities include development and maintenance of the national standards of measurement, and the provisions of means for making measurements consistent with those standards; determination of physical constants and properties of materials; development of methods for testing materials, mechanisms, and structures, and making such tests as may be necessary, particularly for government agencies; cooperation in the establishment of standard practices for incorporation in codes and specifications; advisory service to government agencies on scientific and technical problems; invention and development of devices to serve special needs of the Government; assistance to industry, business, and consumers in the development and acceptance of commercial standards and simplified trade practice recommendations; administration of programs in cooperation with United States business groups and standards organizations for the development of international standards of practice; and maintenance of a clearinghouse for the collection and dissemination of scientific, technical, and engineering information. The scope of the Bureau's activities is suggested in the following listing of its four Institutes and their organizational units.

Institute for Basic Standards. Applied Mathematics. Electricity. Metrology. Mechanics. Heat. Atomic Physics. Physical Chemistry. Laboratory Astrophysics.* Radiation Physics. Radio Standards Laboratory:* Radio Standards Physics; Radio Standards Engineering. Office of Standard Reference Data.

Institute for Materials Research. Analytical Chemistry. Polymers. Metallurgy. Inorganic Materials. Reactor Radiations. Cryogenics.* Materials Evaluation Laboratory. Office of Standard Reference Materials.

Institute for Applied Technology. Building Research. Information Technology. Performance Test Development. Electronic Instrumentation. Textile and Apparel Technology Center. Technical Analysis. Office of Weights and Measures. Office of Engineering Standards. Office of Invention and Innovation. Office of Technical Resources. Clearinghouse for Federal Scientific and Technical Information.**

Central Radio Propagation Laboratory.* Ionospheric Telecommunications. Tropospheric Telecommunications. Space Environment Forecasting. Aeronomy.

---

* Located at Boulder, Colorado 80301

** Located at 5285 Port Royal Road, Springfield, Virginia 22171.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT 42104-12-4212240

NBS REPORT 9004

PROGRESS REPORT
July 1 through September 30, 1965

Development of Methods of Test For Quality Control of Porcelain Enamels

PORCELAIN ENAMEL INSTITUTE RESEARCH ASSOCIATESHIP
NATIONAL BUREAU OF STANDARDS
WASHINGTON, D.C.

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

PROGRESS REPORT

July 1 through September 30, 1965

PORCELAIN ENAMEL INSTITUTE RESEARCH ASSOCIATESHIP

NATIONAL BUREAU OF STANDARDS

WASHINGTON, D.C.

SUMMARY

Work was continued on the development of a cleanability test for porcelain enamel finishes. Because of its increased stability, BBOT was used instead of anthracene as the fluorescent ingredient of the soil. The BBOT was combined with powered graphite and mineral oil to give a composite soil that was mechanically applied to specimens. These specimens were then subjected to two slightly different cleaning treatments. After cleaning, the residual fluorescent material was removed from the test surface with toluene. Evaluation of the amount of soil extracted, which was done with a commercial fluorometer, showed an improved reproducibility when the second cleaning treatment was used (longer cleaning time).

The storage panels of porcelain enameled aluminum as well as those exposed for six months' at Washington were washed and measured for changes in both gloss and color. These data, however, have not been reduced into percent gloss retained and color retention.

The Nature-Tone enamels exposed for six months' at Kure Beach-80 were also washed and measured for changes in gloss and color. The reduced data indicated that the Nature-Tone enamels have gloss and color retentions comparable to the regular acid-resistant enamels on steel exposed for six months' at this site in an earlier exposure test. However, 25% of these Nature-Tone enamels showed rust stains at pinhole type defects in the enamel coating.

Work on a continuity of coating test was continued with the major emphasis on the development of direct current, high-voltage test procedures. Test results indicated that 1) the high-voltage test equipment was capable of indicating weak areas in the coating that were not detectable by an electrolytic cell test, 2) a test voltage of either 1.5 and 2.5 KV could be used for testing architectural enamels depending on the severity of test desired, and 3) good reproducibility of results was obtained when using the selected test procedure.

I. CLEANABILITY

INTRODUCTION

Previous reports in this series have pointed to the need for a cleanability test that would differentiate between different porcelain enamels as well as between porcelain enamels and the various competetive finishes.

One approach to the evaluation of cleanability is to measure the effort required to completely remove a uniformly applied standard soil. Either the energy or the time required for complete removal of soil could then be used as a measure of the cleanability of the surface. If this approach was used, considerable difficulty could be anticipated in determining when complete cleaning had been achieved, and in addition, the precision of a well-defined visual end-point in the cleaning operation would certainly depend strongly on the color of the finish.

In order to circumvent these difficulties associated with complete removal of soil, a second method is being investigated in which (a) soil is applied to the specimen, (b) a standard cleaning treatment is used, and (c) the amount of soil remaining on the surface after the cleaning treatment is evaluated. Because measurements of a fluorescent soiling agent, in situ, were unsuccessful in earlier trials, emphasis during this report period was placed on extracting the fluorescent soil from the partially cleaned surface with a solvent and then determining the amount that was present by measuring the fluorescence of the resulting solution.

RESULTS AND DISCUSSION

1. Fluorescent Tracer Selection.

Anthracene was used as the fluorescent material in the earlier work. It was found, however, that this material was not stable when dissolved in benzene, or other organic solvents. A photosensitive reaction occured during aging of the solutions that transformed the anthracene to dianthracene which is not fluorescent.

For this reason, another material was substituted for anthracene. It is 2,5-bis-[5'-tert-butylbenzoxazolyl(2')]-thiophene which will be referred to as BBOT. This material has a maximum fluorescence at a wave length (435 mμ) that matches the optimum response of the commonly used photo-multiplier detectors. BBOT is chemically stable and its solutions are not altered by light exposure under normal conditions. The solubility of BBOT in toluene is 53 g/l.

BBOT was used as a soiling agent both by itself, and in mixtures with powdered graphite and mineral oil. A soil of the latter type which was used with promising results consisted of:

Ingredient	Amount in Percent by weight
BBOT	3.3
Powdered graphite	32.3
Mineral oil	63.9

The BBOT and powdered graphite were mixed by dry ball-milling for five hours after which the mineral oil was added to form the composite soil.

To determine if uniform mixing was achieved by this ball-milling treatment, weighed amounts of a nominal 10 percent by weight BBOT and 90 percent powdered graphite mixture were treated with known amounts of toluene. The BBOT dissolved in the solvent and the graphite dropped out. After sufficient dilution, the fluorescence of the solution was measured and its BBOT content was calculated. Table 1 indicates that good mixing uniformity was obtained. The average determined BBOT content of the dry powder mixture of 9.17 percent suggests that a disproportionate amount of the fluorescent tracer adhered to the ball mill and balls, slightly changing the intended BBOT content. The results for the samples analyzed was used as a basis for the soil composition given above.

2. Soil Application and Cleaning Treatment.

The mechanical equipment used for soiling and cleaning porcelain enameled specimens is shown in Figure 1. The main part of the equipment is a conventional polishing lap. The specimen is attached to the lapping plate in a face-up position. A plastic covered brass head, one inch in diameter then moves in a reciprocating motion across the rotating specimen. The combined action of the specimen rotation and the back-and-forth motion of the head uniformly distributes a weighed amount of soiling agent over a controlled circular area at the specimen center.

The cleaning treatment employed a heavier brass head of larger diameter than was used for soil application. This 2 1/4-inch diameter head was covered with four thicknesses of an industrial wiping tissue. The extent of the back-and-forth stroke was increased for the cleaning operation. This meant that the soil was first smeared over the entire specimen surface and then partially absorbed by the tissue. The degree of cleaning could be controlled both by varying the time of operation and by varying the number of tissue renewals during the course of the cleaning.

3. Extraction of Soil and Fluorescence Measurement.

Figure 2 shows the fluorometer used for measuring the extracted solutions and also the device used for the extraction of the BBOT from the soiled and cleaned surfaces. The extraction cell was a 150 ml reagent bottle from which the bottom had been removed and the sawed edge ground smooth. The bottle and a fitted teflon gasket were clamped against the specimen. In this way a reproducible test area was defined when toluene was introduced into the reagent bottle. Several rinse portions of toluene were introduced until a point was reached where the last rinse portion showed a fluorescence that did not exceed the fluorescence of the toluene blank.
The various extracts were combined in a flask, diluted if necessary, to bring them onto the fluorometer scale, and a five ml portion used for the fluorometric intensity measurement.

4. Calibration of the Fluorometer.

The fluorometer was calibrated with solutions of known BBOT content, prepared by careful dilution techniques. Table 2 and figure 3 give the results of the calibration.

5. Effect of Cleaning Time.

It is obvious that the amount of soil that remains on a surface will be dependent not only on the cleaning method that is used but also on the time of cleaning. In an attempt to arrive at a value for the reproducibility of one possible cleaning method and also to determine the effect of cleaning time, the following experiment was performed: First a glossy, buff colored enamel with a reflectance of about 50 percent was selected for test. A total of 16 specimens of this enamel were then tested, eight by Method No. 1 and the remainder by Method No. 2.

In Method No. 1, 0.097 grams of the graphite-BBOT-mineral oil soil, described in section 1 was placed on a specimen. The specimen was then placed on the lap and the one-inch diameter head was operated for one minute with the stroke set at 1.5 inches. This served to distribute the soil over the test surface. Next, the small head was removed and replaced with the 2 1/4-inch head that had been covered with four thicknesses of a commercial wiping tissue. After 15 seconds, with a stroke of 3.0 inches, the equipment was stopped and the soiled tissues replaced with clean ones. After three cycles of this treatment (total time 45 seconds) the specimen was removed and the BBOT remaining on the surface was extracted with toluene, and the fluorescence of the resulting solution evaluated.

The soil application, cleaning procedure, and measurement technique were the same for Method No. 2 as for Method No. 1 except that the final cleaning treatment was continued for 60 seconds rather than for 15 seconds.

Table 3 gives the results of the measurements. The calculation of the total soil retained on the specimen is based on the BBOT content of the soil, the BBOT found, and the area from which the extraction was made:

$$\text{BBOT} = 3.3 \text{ percent of soil}$$

$$\text{Total soil retained, } \mu g/cm^2 = \frac{\text{BBOT, } \mu g \text{ found}}{\text{BBOT content x Extraction Area}}$$

$$= \frac{\text{BBOT, } \mu g \text{ found}}{0.033 \times 14.3 \text{ cm}^2}$$

$$= \text{BBOT, } \mu g \text{ found} \times 2.12$$

As would be expected the amount of soil retained on the treated specimens was less with the second method than with the first. This was true whether the indicator of retained soil was by change in reflectance or by measurement of fluorescence.

The variability of Method No. 2 was smaller than that of Method No. 1 by a factor of three when the fluorometric determinations were considered, and by a factor of two for the reflectance measurements.

Separate analyses of variance were made on the fluorometric and on the reflectance data. These indicated a statistically significant difference existed between the two methods with respect to the fluorometric measurements but no significant difference was indicated for the reflectance data. The results of the tests showed that Method No. 2 with its longer cleaning treatment was superior to Method No. 1

in that much more reproducible and precise measurements were obtained. The results also indicated that the fluorescence measurements gave better precision than the reflectance approach.

PLANS FOR NEXT REPORT PERIOD

Work during the next report period will be concerned with (a) development of a cleanability testing procedure, (b) testing of various porcelain enamels to determine if small differences in cleanability can be detected and (c) testing of other types of finishes for which values of cleanability might be of interest.

Brief progress reports on the development of a cleanability test method will be presented at the PEI Shop Practice Forum and at the November meeting of the Eastern Enamelers Club in Philadelphia.

II. EXPOSURE TEST OF PORCELAIN ENAMELS ON ALUMINUM

INTRODUCTION

Early in 1964, the Aluminum Council of the Porcelain Enamel Institute authorized an exposure test of Porcelain Enamels on aluminum to be conducted jointly by the Porcelain Enamel Institute and the National Bureau of Standards. The 53 enamels included in the test were applied by nine fabricators to a single lot of 6061 aluminum alloy. The first specimens were installed in March of 1965 and have been exposed for six months.

RESULTS AND DISCUSSION

The aluminum specimens that had been exposed for six months at Washington were removed from the racks at the National Bureau of Standards at Washington. Similar specimens have been removed from the exposure racks in Montreal and Kure Beach. However, these specimens did not arrive at the laboratory in time for inspection during this report period.

The storage specimens and the Washington specimens were cleaned by (1) scouring 30 strokes with a sponge that had calcium carbonate sprinkled on it and was moistened with a one percent solution of trisodium phosphate, (2) rinsing with tap water, (3) rinsing with distilled water, and (4) rinsing with alcohol. The 45° specular gloss and the change in color were measured on a Hunter Precision Glossmeter and a Gardner Automatic Color Difference Meter, respectively. The data for the storage specimens were reduced with a desk calculator. These calculations showed that the average percentage gloss retained was 99.0, and that the average color change was 0.3 NBS units.

The specimens exposed at Washington had a slight dirt film on them which was easy to remove by the aforementioned cleaning procedure. However, tan stains were present on two of the white enamels (AA-D and AD-D) and these stains were still present after the cleaning treatment. Microscopic examination of the surfaces did not reveal any difference between the stained and unstained areas. At the present time the cause of the staining is not known. However, two additional specimens of enamel AD-D, which showed the worst stains, have been installed on the Washington, D.C. racks. After six months' these two specimens will be removed and studied in considerable detail, including sectioning, in an attempt to arrive at a logical explanation.

The only enamels that showed a visually detectable color change after six months at the Washington, D.C. site were the red enamels. The worst of these, AH-D showed a measured change of 11.1 NBS units.

It should be mentioned in this connection that all four of these red enamels failed the 15-second nitric acid spot test that is currently included in the aluminum architectural specification (PEI Specification No. ALS-105). In addition, they also failed the cupric sulfate test which is a part of the architectural specification for steel enamels (PEI Specification No. S-100).

A second observation of interest was that no spalling on any of the enamels at Washington after six months' exposure.

PLANS FOR NEXT REPORT PERIOD

The specimens from Kure Beach, Montreal, New York and Los Angeles will be returned to the National Bureau of Standards in Washington for measurement early in the next report period. The changes in both gloss and color will be measured, these data will be reduced and presented at a meeting of the Aluminum Council on November 9. All specimens will be returned to the exposure racks after examination.

III. NATURE TONE ENAMELS

INTRODUCTION

Nature-Tone enamels were introduced recently as a new concept for architectural porcelain enamels. These particular enamels, which have been well received by architects, are characterized by both (1) very low gloss and (2) neutral or "earthy" colors. However, data is needed on their weather resistance. To fill this need, duplicate specimens of thirty of the new Nature-Tone enamels on steel were exposed at Kure Beach-80, a site known from previous testing to cause fairly rapid deterioration of some porcelain enamels. Late in September, the specimens were removed from the racks and returned to the laboratory at the National Bureau of Standards for evaluation of changes that occurred after the first six months of exposure.

RESULTS AND DISCUSSION

Upon arrival at the laboratory the specimens were cleaned by: (1) scouring for 30 strokes with a sponge that had been dampened with a one-percent solution of trisodium phosphate and then sprinkled with calcium carbonate, (2) rinsing with tap water, (3) rinsing with distilled water and finally, (4) rinsing with alcohol. The cleaned specimens were then measured for changes in both gloss and color.

The results of the measurements are listed in Table 4. Since all of the Nature Tone enamels have either satin or mat finishes, their initial gloss readings were quite low. These low values mean that small changes in gloss from exposure to weathering will be reflected by small values for percentage gloss retained. For this reason the gloss retained values in Table 4 are of only limited usefulness in gaging the weather resistance of the enamels.

Color change, on the other hand, is a very good criterion. Table 4 shows that the average color change of the 30 enamels was 0.80 NBS unit with the individual values varying from a low of 0.17 (brown enamel, ME-11 and Light gray enamel, MC-5) to a high of 2.02 (blue enamel, MC-4). This is comparable to the color changes observed in the currently operating PEI-NBS test for the acid resisting enamels at the end of the first six months of exposure. Thus, from the standpoint of color stability, the Nature-Tone enamels appear to have a stability equal to the earlier enamel types. The only exception is the blue enamel MC-4 which changed 2.02 NBS units in only six months. This particular enamel will be carefully monitored in future inspections. If it continues to change at this high rate, either its composition should be modified or it should be eliminated from consideration as one of the Nature-Tone series of enamels.

The next to the last column in Table 4 lists the measured coating thicknesses of the Nature-Tone specimens. Good coverage of the steel would normally be expected at this thickness of application; yet the last column shows that rust spots were observed on the face surfaces of 15 of the 60 specimens (25%). Admittedly, the Kure Beach-80 site greatly accellerates corrosive attack over that which would occur if the enamels were exposed in commercial or industrual locations. At the same time, however, past experience has shown that those enamels that permit rusting of the metal in six months at Kure Beach-80 will also permit rusting to occur after longer periods at the other exposure sites. Therefore, the appearance of the rust spots at Kure Beach-80 in such a short time is a cause of serious concern.

Examination of the specimens indicated that the observed rusting occured at very small pinholes of a type that would not normally be detected by a visual inspection of the enamel surface. The rusting was evidenced by iron stains on the enamel surface which would be removed by scouring.

The tentative conclusion that might be made from this first inspection is that the Nature-Tone enamels are susceptible to pinhole defects and that careful inspection by a coating continuity test would be highly desirable on all architectural parts to which the Nature-Tone enamels a are applied. The continuity test discussed in the next section would probably be satisfactory for this purpose.

PLANS FOR NEXT REPORT PERIOD

The 60 specimens will be returned to the exposure racks at Kure B each-80 during the next quarter. The next inspection of these enamels is tentatively scheduled after one years' exposure.

IV. CONTINUITY OF COATING

INTRODUCTION

Since porcelain enamels are applied to many products to prevent corrosion of the base metal, it follows that a method of test to evaluate the protection offered to the base metal or the degree of continuity of would prove useful in avoiding defective ware. The development of such a test is one of the current goals of the Research Associates.

Previous reports have outlined the various defects that should be detected with a continuity of coating test. A review of the literature showed that a number of different tests had been used for this purpose. Preliminary work indicated that the direct current, high-voltage test showed considerable promise, although a low-voltage electrolytic cell test also might have merit.

RESULTS AND DISCUSSION

1. Comparison of Electrolytic Cell Test with D.C. High-Voltage Test

The electrolytic cell test was outlined in the previous report.

Briefly, it consists of first forming a reservoir on the enameled surface with any good calking compound, and then filling the reservoir with an aqueous solution consisting of 1% by weight sodium chloride and 1/2% sodium carbonate. Next, a DC potential of nine volts is applied for 15 minutes between the solution and the metal backing of the specimen after which the enameled surface is washed and the number of rust spots counted. This test was performed on duplicate specimens of several enamels that had rusted badly after only short-time exposure at Kure Beach-80. No rust spots were indicated after the 15-minute treatment. The same specimens were then probed with the direct current, high-voltage tester after which they were again subjected to the electrolytic cell treatment. Rust spots appeared during this second treatment at the points where the high voltage punctures had occurred. These results indicate that the electrolytic cell test detects only those defects that are open to both the surface of the enamel and the base metal. On the other hand, the high-voltage discharge method, operated at properly selected voltages, will also detect weak areas in the coating of a type that permit rusting to occur during actual weathering.

2. Comparison of High-Voltage Test Results with Service Data for Architectural Enamels.

The previous report indicated that storage specimens of many of the architectural enamel specimens in the PEI-NBS Exposure Test[7/] that corroded after 7-years exposure had defects that could be located at a probe voltage of 2.5 KV. During the present report period, additional test were made. Triplicate specimens of 59 of the storage enamels were probed.

When making the tests, the voltage was increased from 1 to 5 KV in increments of 0.5 KV. As an aid in analysing the data, the enamels were arbitrarily separated into two classes: good and fair. Exposed specimens of the enamels designated good, showed no rusting at any of the seven exposure sites after 7-years exposure, while the fair enamels showed rusting at Kure Beach-80 after this same time interval. The results of the high-voltage tests made on storage specimens of these same enamels are illustrated in Figure 4. Examination of this figure shows that when the storage specimens were probed at 2.5 KV approximately 90% of the enamels that rusted at Kure Beach-80 in 7-years would have failed the test as compared to only 30% for the enamels that did not rust under this same exposure. Kure Beach-80 is, of course, a very severe site with respect to corrosion. Therefore, the fair enamels were further subdivided. Those enamels that rusted at the less severe sites, either Kure Beach-800 orNew Orleans, in addition to Kure Beach-80 were separated from the fair enamels and reclassified as poor enamels. Figure 4 shows that a test voltage as low as 1.5 KV would eliminate 90% of the enamels that fell into the poor catagory.

3. Reproducibility of High-Voltage Test Results

Triplicate specimens of two hot water tank enamels as well as 59 architectural enamel were probed with the high-voltage test equipment. The test results were then analyzed to determine the reproducibility of test procedure. Results that are typical of the scatter are plotted in Figure 5. In each case the points represent the average values and horizontal lines the maximum spread. It can be seen that the good enamels gave fairly reproducible responses from specimen to specimen. However, the enamels that were designated poor showed fairly high scatter between specimens. In many cases, one of the three specimens would show only a very few defects or none at all while the remaining specimens a large number of defects would be detected. This trend of large variability was also noted in the specimens that were exposed to weathering.

4. Results of Questionnaire on Testing for Coating Continuity

It was mentioned in the preceding report that a questionnaire had been prepared and sent to porcelain enamel fabricators, who are members of the PEI. To date 12 replies have been received. The following is a summary of the findings:

1. Approximately 90% of the fabricators agree that a "discontinuity is any defect which leads to early corrosion of the base metal."

2. While all fabricators inspect the ware for discontinuities, most of the inspections are visual.

3. The defects looked for are primarily: pinholes, blisters, cracks, fishscale, and copperheads. (It is significant that the high-voltage test equipment is capable of locating all five of these defects.)

4. Most of the fabricators thought of a test for continuity of coating as a Quality Control test.

5. Everyone who answered the questionnaire thinks that continuity of coating is important for at least one of the products he produced.

PLANS FOR NEXT REPORT PERIOD

During the next report period it is planned to test current production enamels that are used for specific applications with a goal of selecting probe voltages that will differentiate between satisfactory and unsatisfactory enamel application. The actual performance of this task is of course dependent on receiving suitable specimens from the fabricators. Also, a paper will be prepared for presentation at the PEI Forum at Ohio State.

STANDARD REFERENCE MATERIALS

The following stock of standards was on hand October 1, 1965:

Corundum abrasive, March 1960 issue,
for subsurface abrasion . . . . 261 lbs., 65 jars.

Standard Pennsylvania Glass Sand, July 1963 issue,
for surface abrasion . . . . 285 lbs., 94 jars.

Distinctness of image gloss standards . . . . 20 sets.

Calibrated glass paltes for abrasion testing . . . . 27.5 doz.

Respectfully submitted,

M. D. Burdick

M. A. Rushmer

M. D. Burdick
M. A. Rushmer
Research Associates, PEI

REFERENCE

1. Rushmer, M.A. and M.D. Burdick, Weather Resistance of Porcelain Enamels Exposed for Seven Years at Various Sites, Proceedings of of the Porcelain Enamel Institute Forum, Vol. 26, 1964.

TABLE 1. Results of Measurements to Test Uniformity of Mixing of a Graphite-BBOT Soil

Specimen No.	BBOT Content[a/]
	percent
1	9.14
2	9.26
3	9.04
4	9.41
5	9.06
6	9.07
7	9.16
8	9.20
Average	9.17
Coef. Variation	1.3

a/ Applied soil consisted of 10% by wt. BBOT and 90% powdered graphite.

TABLE 2. The Fluorescence of Solutions of Known Concentration of BBOT[a/] in Toluene.

Solution	Concentration	Fluorescence Readings			
	µg/ml	No Filter	Filter A	Filter B	Filter C[b/]
CC	0.0699	--	100+	100+	79.0
DD	0.0599	--	100+	100+	69.0
EE	.0498	--	100+	100+	57.0
FF	.0399	--	100+	79.0	46.0
GG	.0299	--	90.5	60.0	35.0
HH	.0200	--	60.5	41.0	24.0
II	.0100	100+	31.0	22.0	12.5
C	.0042	47.0	16.0	10.0	6.5
E	.00312	36.0	--	--	--
F	.00259	31.5	--	--	--
G	.00207	24.0	--	--	--
H	.00155	18.0	--	--	--
I	.00104	11.0	--	--	--
J	.00052	5.0	--	--	--
BLANK	.00000	0.0	0.0	0.0	0.0

a/ 2,5-bis[2-(5-tert-Butylbenzoxazolyl)]-Thiophene (Scintillation Grade): M.W. 430.58, M.P. 201-202°C, Fluor. Max 4350 Å, and Solubility in Toluene: 53 g/l.

b/ The neutral density filters used had the following percentage transmission:

Filter Designation	Neutral Density Designation	Transmission percent
A	0.5	31.6
B	0.7	20.0
C	0.9	12.5

TABLE 3. Comparison of Two Soiling and Cleaning Methods in a Cleanability Test

	Fluorometric Determination of Total Soil Retained		Reflectance Change	
	Method 1 [a]	Method 2 [a]	Method 1	Method 2
	μg/cm²	μg/cm²	percent	percent
	1.97	1.46	3.9	5.5
	3.09	1.63	5.1	4.5
	1.82	1.40	2.8	2.2
	1.25	1.61	2.2	5.7
	3.69	1.31	10.4	4.0
	2.80	1.72	5.6	5.6
	2.37	1.65	7.6	4.6
	1.78	1.80	5.0	6.3
Average	2.35±0.67 [b]	1.57±0.14	5.32±2.2	4.80±1.08
Coef. of Variation	34%	11. %	50. %	27. %

a/ Pressure applied for application was 1.1 psi (79. g/cm^2)
Pressure applied for cleaning was 0.5 psi (32 g/cm^2)

b/ 95 percent confidence limits.

Table 4. Summary of Measurements on Nature-Tone Enamels after Six Months' Exposure at the 80-ft. Site at Kure Beach, North Carolina.

Specimen	Visual Color	Color Change (NBS Units)	Initial Specular Gloss	Specular Gloss Retained (%)	Acid Spot Test Ratings	Acid Solubility (mg/in^2)	Thickness (mils)	Number of Specimens Rusted
MA-4	Light Gray	0.82	1.8	0.0	A	0.9	9.1	0
MC-5 *	Light Gray	0.17	25.7	93.0	A	1.1	8.7	0
MA-5 *	Medium Gray	1.57	10.4	71.8	A	1.8	9.3	0
ME-3	Medium Gray	0.25	18.7	95.2	A	0.8	9.9	0
MB-5 *	Gray	0.66	5.3	45.5	A	7.0	14.3	0
MC-6	Dark Gray	1.04	30.2	94.5	A	1.6	8.7	1
MA-12 *	Dark Gray	1.51	20.4	74.0	A	1.2	9.8	0
MC-10	Black	0.34	22.4	89.5	A	1.1	8.1	0
MD-5	Black	0.44	29.6	108.9	A	1.1	7.6	0
ME-8	Black	0.83	25.6	82.4	A	1.9	8.6	1
ME-12*	Black	0.57	17.1	79.4	A	1.3	8.9	0
MA-3*	Yellow-Green	0.59	6.5	55.5	A	2.1	9.1	0
MB-4	Yellow-Green	0.63	8.5	82.5	B	9.0	11.6	0
ME-2	Chartruse	0.39	17.6	79.9	A	1.7	10.8	0
MD-6	Medium Green	0.65	48.4	99.7	A	0.6	7.1	0
ME-6	Green	0.32	7.2	62.3	A	0.9	9.3	2
ME-7	Dark Gray-Green	1.01	12.7	74.0	A	1.2	6.6	2
MC-2	Light Blue	1.32	22.0	87.2	A	1.2	9.3	0
MC-12	Light Blue	1.58	5.5	62.6	A	3.2	8.1	1
MC-4*	Blue	2.02	22.0	89.5	A	1.3	8.7	0
MD-1	Peacock Blue	0.55	20.5	94.9	A	1.2	7.2	1
ME-4*	Slate Blue	0.84	36.0	81.7	A	1.0	9.5	1
ME-9*	Steel Blue	0.30	20.2	90.6	A	0.7	7.4	1
MA-6	Red Brown	0.73	8.8	57.3	A	1.7	9.1	2
MC-9*	Red Brown	1.03	30.1	92.6	A	1.0	7.8	1
MD-4	Red Brown	0.79	26.6	88.6	A	0.9	7.5	0
ME-5*	Red Brown	1.15	21.7	82.7	A	0.7	11.0	1
ME-11*	Brown	0.17	1.2	0.0	A	0.9	8.7	0
MD-3	Chocolate Brown	0.73	25.4	94.4	A	0.9	8.1	0
ME-10*	Chocolate Brown	0.85	19.1	81.7	A	2.6	7.9	1
	Average	0.80		76.4			8.9	

* Enamels selected by the architects.

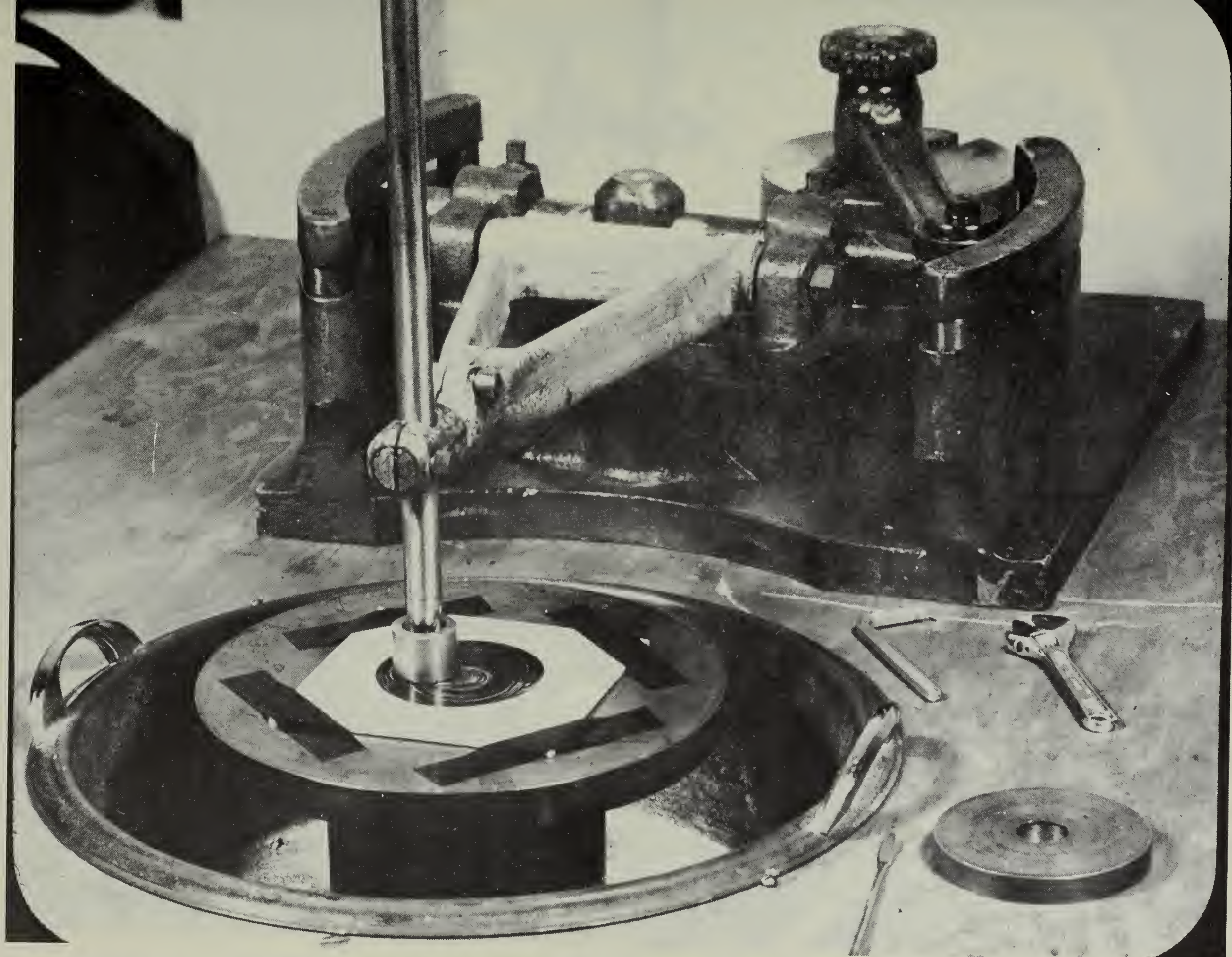


Figure 1. Lapping Equipment Used for Soiling and Cleaning Specimens.

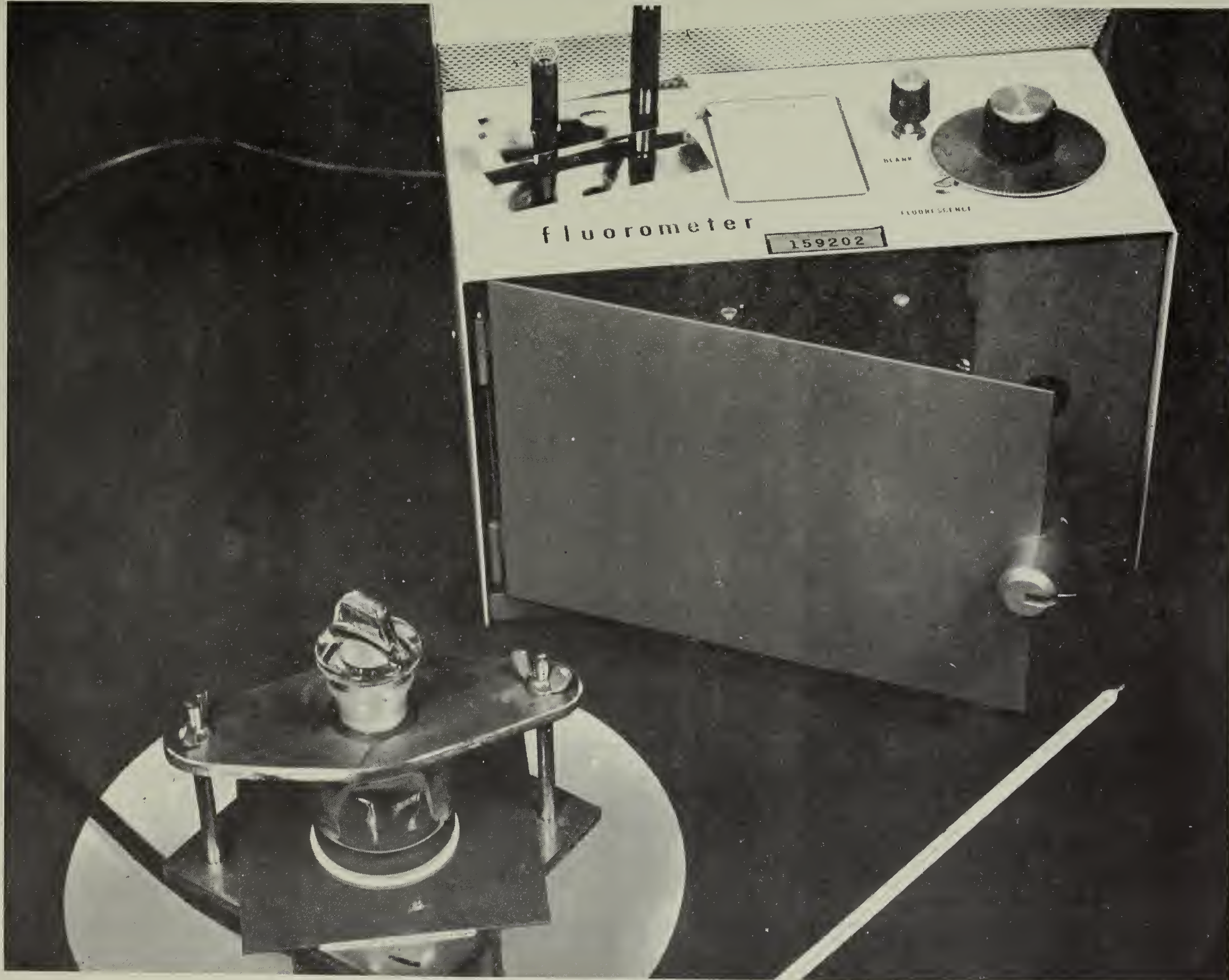


Figure 2. Extraction Device and Fluorometer for Fluorescence Analysis.

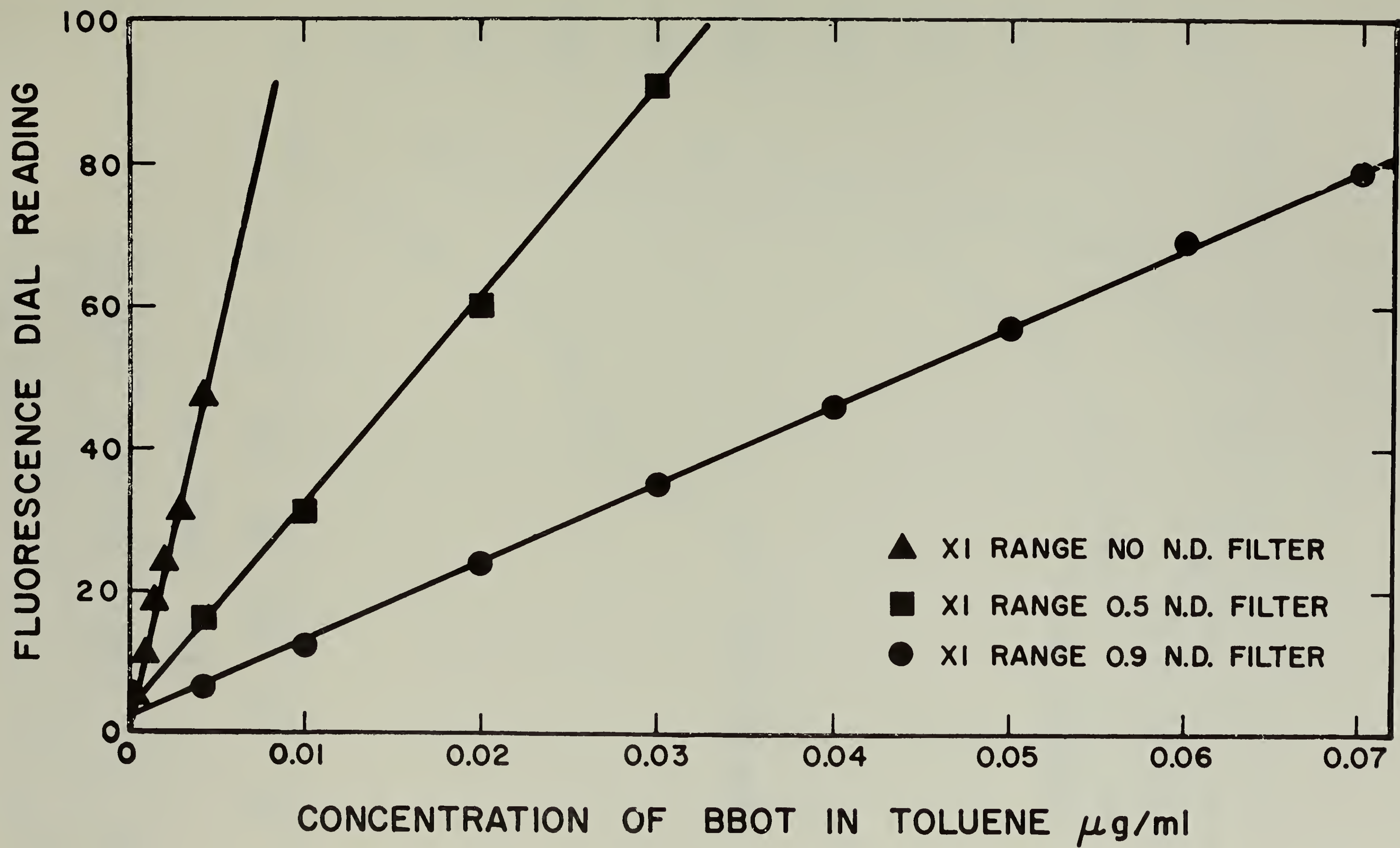
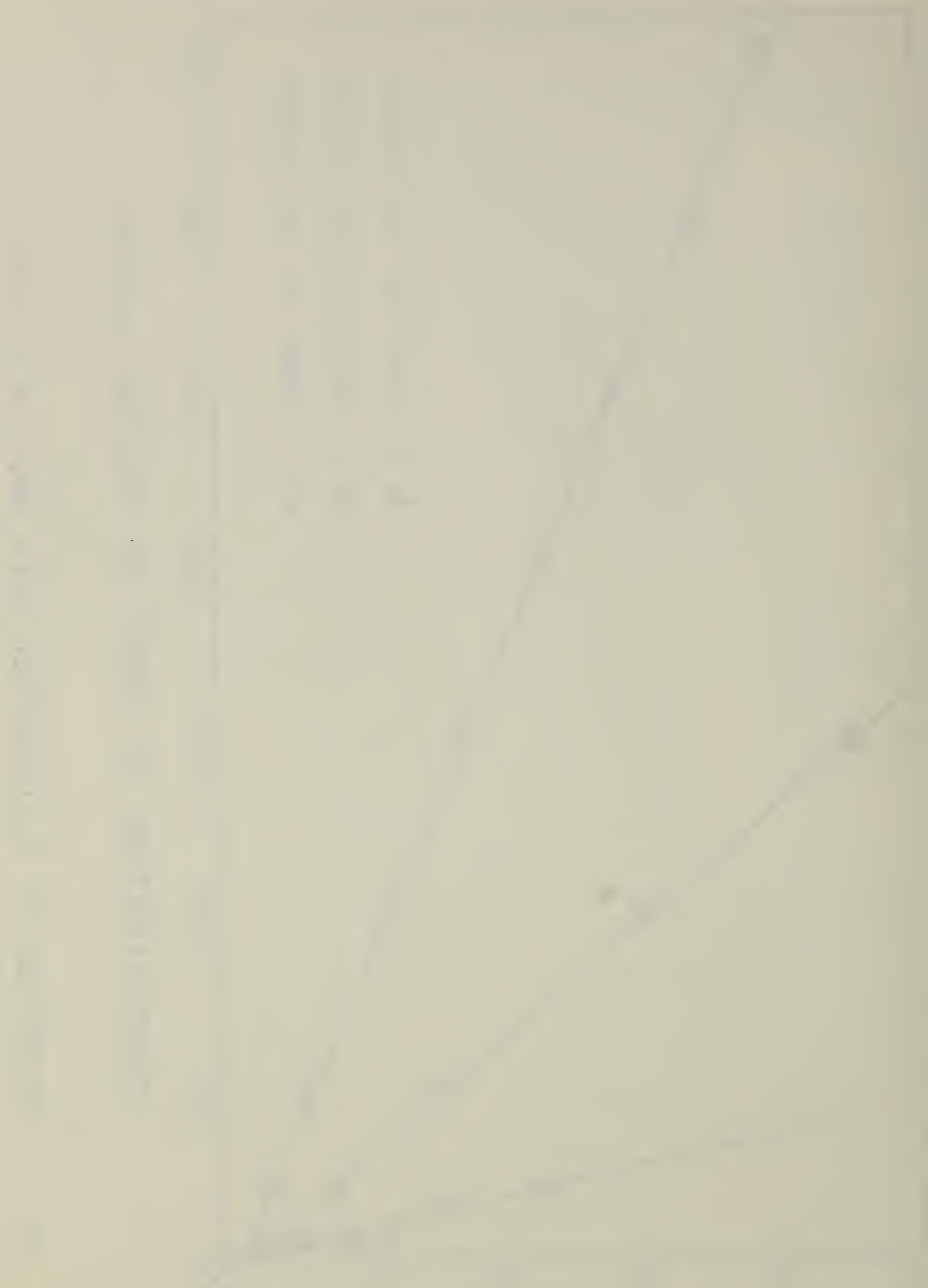


FIG. 3 CALIBRATION OF FLUOROMETER WITH BBOT IN TOLUENE.



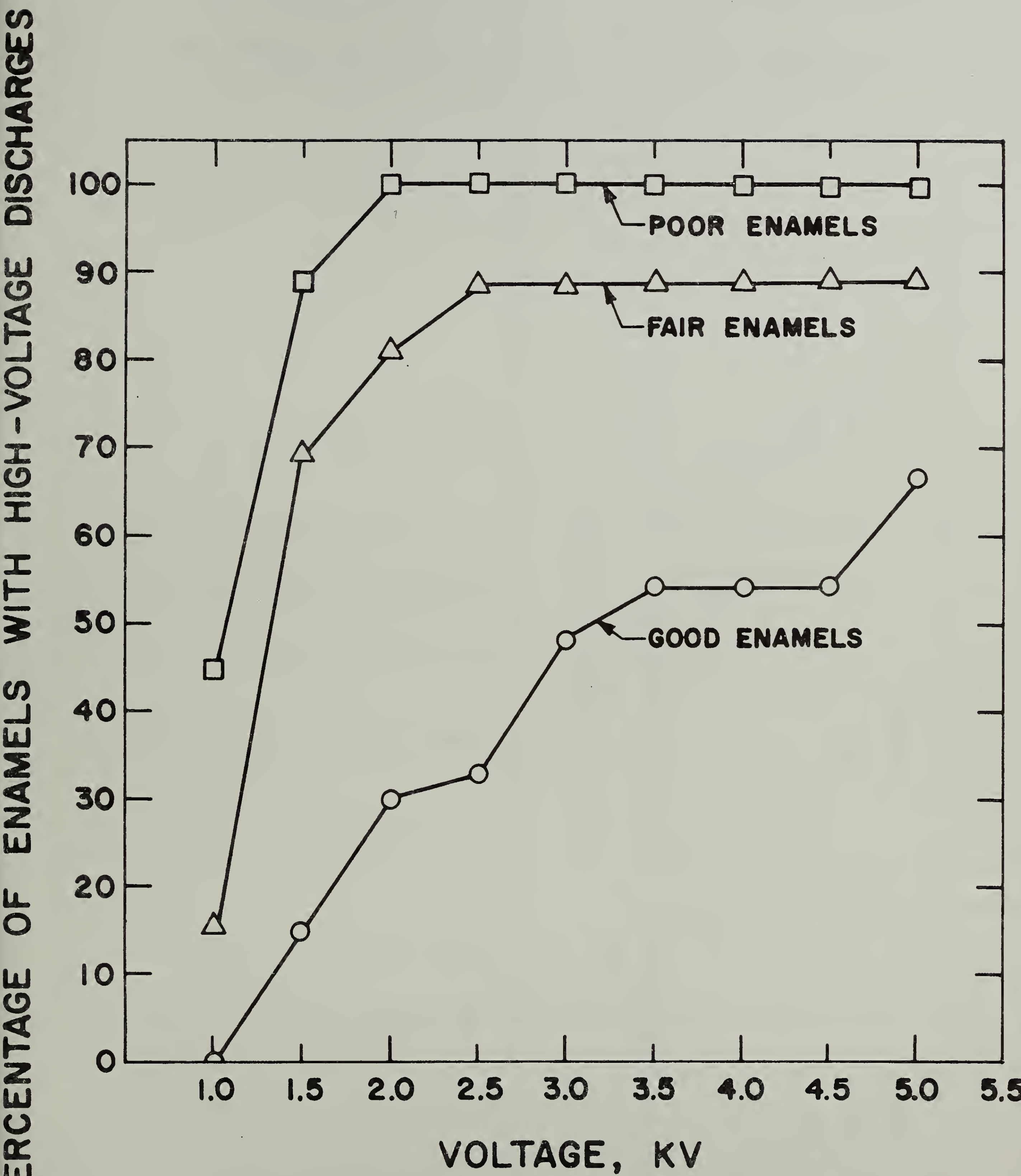


Figure 4. High-Voltage test results for storage specimens of enamels in PEI-NBS exposure test. The good enamels did not rust at any site after 7-yrs exposure, the fair enamels rusted at Kure Beach-80 in 7-yrs, while the poor enamels rusted at both Kure Beach-80 and either Kure Beach-800 or New Orleans.

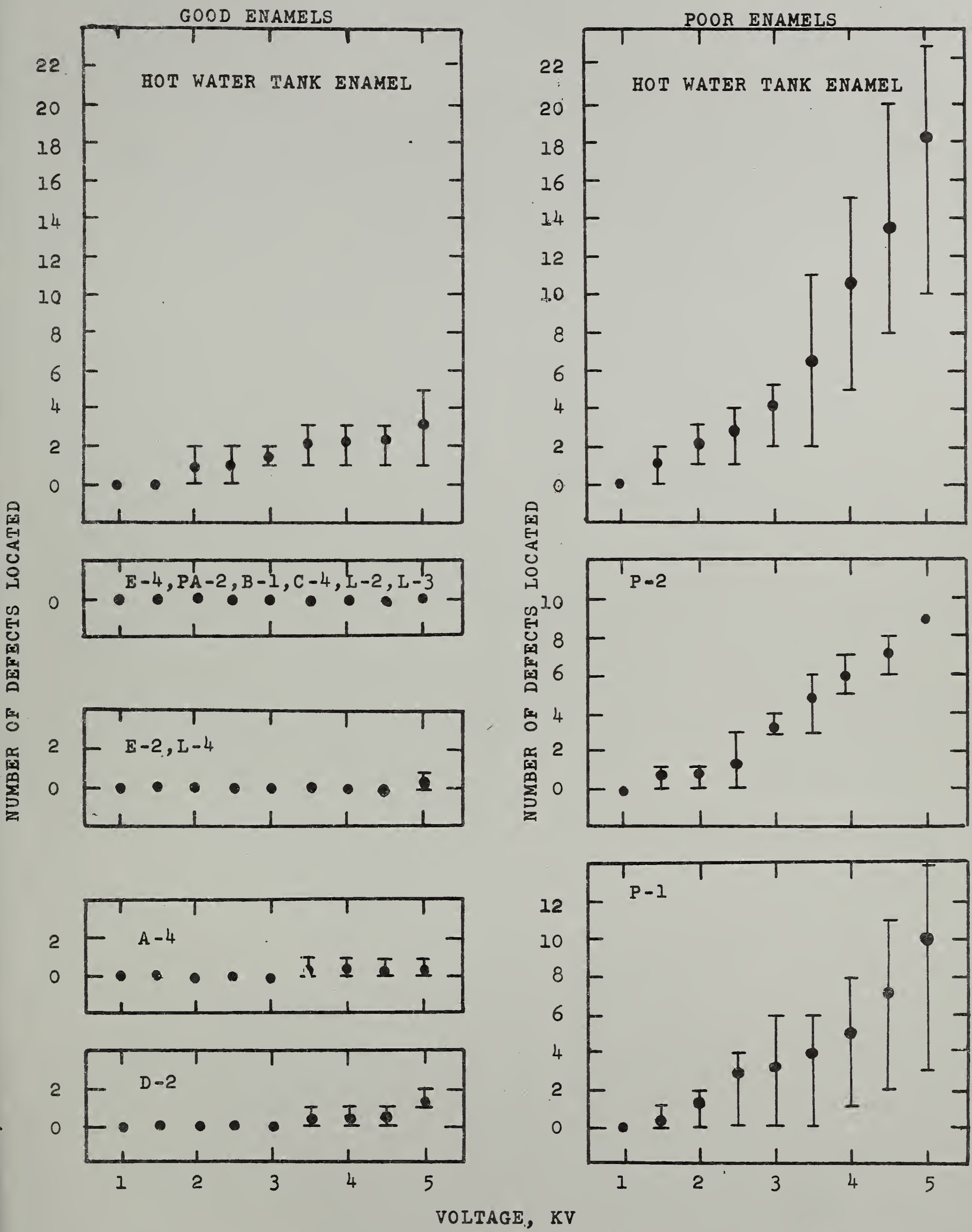


Figure 5. Reproducibility of testing good and poor hot water tank and architectural enamels. The architectural enamels are designated by their code letters. 1/

DEPARTMENT OF COMMERCE ★ NATIONAL BUREAU OF STANDARDS ★