NATIONAL BUREAU OF STANDARDS REPORT

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PROGRESS REPORT

January 1 through March 31, 1966

Development of Methods of Test
For Quality Control of Porcelain Enamels

by

M. D. Burdick and M. A. Rushmer

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

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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SUMMARY

The essence of one approach to the determination of the cleanability of surfaces is the use of a soiling agent which is stable, which characterizes a typical use condition, and at the same time includes a fluorescent tracer to permit a sensitive determination of small residual amounts. Progress has been made in the development of such a soiling agent.

Mechanical and analytical equipment have been selected and a procedure for use demonstrated.

The non-destructive nature of this type of cleanability procedure was demonstrated for porcelain enamels.

Preparations for a weathering test of 25 Nature-Tone (low gloss) enamels on steel are described. A novel feature of this exposure test is the use of a continuity of coating criteria in the selection of a part of the specimens to be exposed.

The status of the current study of the weathering stability of porcelain enamels on aluminum is described and includes the addition of 8 Nature-Tone (low gloss) enamels at the end of the first year of the test.

I. CLEANABILITY

INTRODUCTION

The ease with which porcelain enamel can be cleaned is one of its important performance characteristics, whether the finish is employed on domestic appliances or for exterior architectural applications. In spite of this importance it is a characteristic for which no single, objective and non-destructive evaluation procedure is available.

A recent article [1] comparing porcelain enamel with other competetive finishes for domestic appliances pointed out that three important performance characteristics were: "durability, cleanability and soilability". Thirty-six properties were then enumerated which were thought to contribute to durability and cleanability.

A British Standard for domestic appliances requires that "the external finish shall be easily cleanable". This requirement is an important one but imposes a real question regarding compliance of a given finish.

It is felt that there is a need for a test procedure which will permit a numerical determination of cleanability. Such a test should assist in comparing various finishes for products of different materials intended for the same service. A cleanability test could also make a contribution in quality control and in the development of porcelain enamels of superior cleanability characteristics.

It is well know that oil films on a surface are difficult to remove. Oil is a soiling agent common to many areas of porcelain enamel use. For this reason the soil used in the development of a cleanability test was an oil, colored black for psychological effect, to which a tracer amount of an intense fluorescent material was added to aid in the determination of small amounts of soil retained on a soiled and cleaned surface.

This concept has, in the recent past, proved to have some merit.

TENTATIVE TEST PROCEDURE

A tentative test procedure for the determination of cleanability was outlined in the previous quarterly report. Several modifications have been developed, but in general the procedure was the same as previously described. The modifications involved: different soiling agents, soil dispensing methods, and somewhat different equipment for the mechanical soiling and cleaning operations. The rather elaborate lapping equipment originally used was replaced with more readily available laboratory equipment.

RESULTS AND DISCUSSION

At the start of this investigation the mechanical soiling and cleaning methods were explored with an equipment which was at hand. This device with its many adjustments served well in the early stages but was quite expensive and more elaborate than was required. The mechanical motion, which worked well, was duplicated using a Buehler lap and an Olsen S.M. Polisher.

The Buehler-Olsen lapping equipment was adapted for use in the cleanability work. Minor adjustments in cleaning time and rubbing pressure were made, see Table 1, to achieve comparability with previous results. Table 2 gives results obtained on a glossy brown porcelain enamel. The values for specimens 4 through 11, obtained with the old lapping equipment using soil A and cleaning method 7, may be compared with values for specimens 13 through 19 using different equipment and a different soil composition. Given in this table also are results on virgin specimens and on the same test pieces after recleaning. This demonstrates the non-destructability of this test procedure and shows, as well, that a part of the specimen-to-specimen variability reflects real differences in specimen cleanability characteristics.

The original soil A (Table 3) was prepared with BBOT[1/], powdered graphite and mineral oil. The graphite and BBOT were premixed by dry ball-milling and the mineral oil was mixed with the dry ingredients on the specimen. However, the weighing of the small amounts of soil needed for each test was a rather tedious and time consuming operation. It was felt that mixing all the ingredients in the soil would produce a fluid soil so that uniform amounts could be dispensed onto the specimens with a hypodermic syringe. A two milliliter syringe, without a needle, was successfully used to dispense 0.15 ml quantities of the soiling agent to a series of six specimens without refilling.

1/ BBOT is 2,5,-bis-5' - tert-butylbenzoxazolyl (2') -thiophene.

The percentage of graphite was increased in soils C-E as well as changing to a more viscous oil, reagent grade paraffin oil. These changes were made to increase the time required for the graphite to settle out. A series of tests made over a two-week period showed that the reporducibility from day-to-day was not adequate, as illustrated in Table 4.

The desirability of a stable soil which would not require thorough mixing before use led to the use of colloidal graphite in oil, "Oildag"[1/] to replace the powdered graphite colorant.

The original calibration of the fluorometer was applicable to BBOT used with non-fluorescent ingredients (mineral oil and graphite). The use of "Oildag" required a recalibration of the fluorometer to include the combined fluorescence of the BBOT tracer and the petroleum oil of the "Oildag". The recalibration, showing the relation of the fluorescence to concentration of soil G is shown in Figure 1.

Soil G, employing Oildag as a colorant, was used in cleanability tests on a series of specimens from a single lot of a glossy white porcelain enamel for appliances. The amounts of soil G retained on typical groups of six test pieces of this enamel are given in Table 5 in the order in which they were soiled and cleaned. Within any of these groups the first value was high, the second was of intermediate soil retention, followed by four somewhat uniform

1/ Oildag is a product of Acheson Colloids Company.

values. The extraction of the retained soil for one group was performed in reverse order from that in which they were soiled. This demonstrated that the cause of high values for the first two specimens in a group was in the soiling or cleaning procedure and not in the extraction technique. It will be shown that the most probable cause of these anomolous results is associated with a settling out of undissolved BBOT from this rather fluid soil composition during storage in the syringe, which was not encountered with the powdered graphite colorant which settled more slowly.

The first experiment to support this premise consisted of a cleanability test of six specimens from the same lot reported in Table 4. The soil used was withdrawn from the top of the bottle of soil G without the usual shaking of the bottle. This procedure utilized soil from which any undissolved fluorescent tracer had been allowed to settle (overnight). The amounts of retained soil on this series of specimens, in the order of soiling, were: 0.08, 0.12, 0.10, 0.10, 0.08, and 0.12 $\mu g/cm^2$. It can be seen that the first two specimens retained amounts of soil that did not differ significantly from the other four.

The extent of the solubility of BBOT in the fluid portion of soil G was then approximated. A series of soil compositions was prepared with amounts of BBOT ranging from 0.1 to 1.0 weight percent. The "Oildag" and paraffin oil were maintained in the same ratio as in the original soil G. These preparations were thoroughly mixed.

The fluorescence of toluene solutions of this series of soils, after dilution to a constant concentration, was measured with no allowance for settling before taking a sample and again after three days' settling time. About 60 to 80 milligrams of the soils were diluted with about 400 grams of toluene; an 0.8 gram aliquot was again diluted with an additional amount of toluene calculated to yield a concentration of 1.9 micrograms of soil per gram of solution. The results are given in Table 6.

Figure 2 shows the fluorescence of this series as a function of the nominal BBOT concentration. The dilute soils GH, GG, GF, and GE, with no settling time, show a fluorescence proportional to the tracer content. The determinations after three days settling for those soils containing more than 0.3 weight percent BBOT, indicate considerably less fluorescence than was determined after little or no settling time. The solubility of BBOT in this combination of oils is seen to be not over 0.3 weight percent. These results suggest that a stable soil of this sort should contain not over 0.3 percent BBOT so that "fall out" difficulty will be avoided.

PLANS FOR NEXT REPORT PERIOD

There is no impelling reason for using a soil consisting of two varieties of oil which require thorough mixing before use. It would seem desirable to use a soil which looks "dirty". It is planned to compound a soil using about 0.2 weight percent of BBOT in "Oildag". Another possibility is to use the BBOT in mineral oil. This preparation would be colorless unless an oil-soluble stain or dye was added. It is believed that one of the above mixtures will be suitable and that extensive trials on specimens of porcelain enamels and

other finishes covering a wide range of cleanability will demonstrate its adequacy.

When a soil has been selected a correlation of cleanability values obtained with the procedure developed in this work with other estimates of the cleanability characteristic will be sought.

II. WEATHERING TESTS OF PORCELAIN ENAMELS

INTRODUCTION

One of the projects undertaken by the research associates over the past several years has been exposure testing of porcelain enamels and the associated development of accelerated tests to separate enamels with good and poor weatherability as indicated by the exposure test results.

Two of these exposure test programs have required a portion of the research associates' time during this report period. These are A) a new exposure test of Nature-Tone enamels on steel and B) the end of the six-months inspection and the beginning of the one-year inspection of porcelain enamels on aluminum and the addition of eight Nature-Tone enamels on aluminum to the aluminum test program.

A. Nature-Tone Enamels on Steel

The exposure test of Nature-Tone enamels on steel includes twenty-five different enamels colors. Sixteen of these enamels, are the original colors selected by the Architects Advisory Council of the Porcelain Enamel Institute. These colors are illustrated in the pamphlet, "Natural Colors for Architecture".

The remaining nine colors have been selected from a group of samples submitted by the frit companies. The enamels in this test will be exposed at three sites: Kure Beach-80, North Carolina; Gaithersburg, Maryland (new location of the National Bureau of Standards approximately 20 miles north of Washington); and a commercial test location near Biscayne Bay, Florida. Six specimens of each enamel are to be exposed at each site. In addition to the exposed specimens, three specimens of each enamel will be kept in dry, dark storage. These specimens will be inspected at the same time the exposed specimens are inspected to determine whether the enamels change during cleaning and storage.

B. Porcelain Enamels on Aluminum

The fifty-one enamels in the exposure test of porcelain enamels on aluminum have been described in previous reports. Triplicate specimens of these enamels are exposed at Kure Beach-80, Los Angeles, New York City, Montreal, and Washington and three specimens of each enamel have again been kept in dry, dark storage.

There are two sizes of specimens included in both tests. Four and seven-sixteenths inch square specimens are exposed at all sites except Kure Beach where four by six inch specimens are exposed in the racks belonging to the International Nickel Company. The specimens are exposed at 45° and face south at all locations except Kure Beach where they are exposed at 30° and face the ocean at east-south east.

INSPECTION PROCEDURE

1. Cleaning of Specimens

Previous exposure tests [2,3] have indicated the need to scour the specimens exposed at one or more of the sites before meaningful gloss and color measurements could be made. These scouring treatments usually tended to increase the gloss readings on the enamels scoured making comparisons with enamels exposed at the other sites invalid. Therefore, it was decided to scour the specimens in these tests both before and after exposure. The cleaning procedure adopted was 1)to scour 30 strokes with a sponge that had been moistened with a one percent, by weight, solution of trisodium phosphate and sprinkled with calcium carbonate, 2)rinse with tap water, 3)rinse with distilled water and 4)rinse with alchol.

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 gloss retained after exposure.

The change in color is measured with a color difference meter. One of the three storage specimens of each enamel is used as the color standard to obtain the maximum efficiency with this type of instrument. The storage standards is, in turn, measured against calibrated NBS standards to determine whether the enamels change color during storage. The color change after exposure is reported as color retention which is 100 minus the color change in NBS units.

RESULTS

A. Nature-Tone Enamels on Steel

1. Specimens Received

At the end of this report period, 16 of the 25 Nature-Tone enamels had been received at the National Bureau of Standards. Eleven of these enamels have been subjected to cleaning and laboratory tests.

2. Cleaning and Initial Thickness Measurements

All specimens submitted were subjected to the cleaning treatment outlined above. After cleaning, the thickness was determined with magnetic thickness gage for 12 of the 48 specimens of each enamel. The average thickness of these twelve specimens is reported in Table 7.

3. Continuity of Coating

Since the first set of Nature-Tone enamels exhibited quite poor coverage, it seemed desirable to determine the usefulness of the continuity of coating test, now under study, in eliminating specimens with poor coverage. Six specimens of each enamel will be exposed at each site in the coming test; three of these will have passed the continuity of coating test and the other three will be untested in this respect. For these enamels of which 90 or 100 percent passed the electrical inspection, (see Table 7) one would expect little or no difference in the tendency to rust during exposure, while the enamels of which only 25-50 percent passed might be expected to show a marked difference in the tendency to rust between tested (and passed) and untested specimen groups. It was shown in a previous report that the best separation of the Nature-Tone enamels, approximately eight mils thick, with acceptable and

unacceptable coverage occurred at a test voltage of 2 kV. Therefore, 2 kV was selected as the nominal test voltage. However, when testing the specimens for continuity of coating indicated a rather marginal quality (as indicated by more than 50% failures) the test voltage was relaxed to 1.5 kV to insure obtaining three specimens for exposure at each site with no large discontinuities. It was also noted that many of the current group of enamels were thicker than the earlier Nature-Tone enamels. The voltage was raised to 2.5 kV for the thicker enamels because this voltage was previously found [4] to give the best separation for enamels in the 9.5 - 11 mil range.

4. Acid Resistance

The boiling acid solubility and the acid spot test ratings were determined for triplicate specimens of each enamel. These results are given in Table 7.

5. Gloss and Color

The initial gloss and color measurements were made as previously described. The initial gloss values are reported in Table 7.

6. Edge Coverage

Since the enamel coverage of the edges of the specimens was light, the edges were coated with two coats of paint; one zinc chromate primer and one black exterior paint. This was done to prevent rust from forming on the edges and covering the surface of the enamel with a thin rust film.

B. Porcelain Enamels on Aluminum

1. Status of the Test

The first inspection of the specimens included in this test was scheduled after six months' exposure. However, there was a delay in the return of specimens exposed at Los Angeles. By the time these specimens were returned, they had been exposed for eight months. These specimens, the specimens exposed for one year at Washington, and the specimens that had been stored for one year were examined during this report period.

Also, as previously reported, eight Nature-Tone enamels on aluminum are to be added to the test after the one year inspection. Six of the eight Nature-Tone enamels have been received and prepared for exposure.

2. Cleaining of Specimens

The cleaning treatment previously described was sufficient to clean all the specimens examined during this report period.

The tan stains that were easily detected on two white enamels, AA-D and AD-D, after six months' exposure had diffused evenly over the enamel surface and were quite difficult to see, especially on enamel AD-D, after one year's exposure. It seems likely that the stains were caused by a substance peculiar to these two enamels that has reacted with an ingredient of the atmosphere at Washington to produce the stain. These enamels will be carefully observed to see if further exposure results in either removal or darkening of the stain. Additional specimens of enamel AD-D were added to the racks after the six months inspection. These specimens can be studied in detail, including sectioning, without interrupting the exposure test, if the stains get darker.

3. Gloss and Color

The gloss and color were measured as described above. The percentage gloss retained and the color retention for the enamels exposed 8 months' at Los Angeles and one year at Washington as well as the one year inspection of the storage enamels are given in Table 8. The six months' data for Washington and storage specimens are also given in Table 8. AD-D and AT-C are the only enamels whose color change is relatively large and opposite to what one would expect. The increase in color retention of enamel AD-D is undoubtedly caused by the lightening of the tan stains, while the increase in color retention for enamel AT-C is unexplained.

Shortly after the gloss on the specimens exposed at Los Angeles had been measured, it was noticed that the gloss meter was out of adjustment. It is possible that this instrument was not in proper adjustment while the Los Angeles specimens were being measured. If such was the case it will be evident after the one year inspection at which time the six months' gloss data will be discarded.

4. Comparison of Exposure Sites

The relative severity of the changes in gloss and color of the enamels exposed at Los Angeles and the other sites was determined by a two-sided sign test [5]. A significant difference between Los Angeles and Kure Beach existed for both gloss and color while a significant difference between Los Angeles and both New York City and Washington existed only when color was considered. Therefore, Kure Beach is the only site considered significantly more severe than Los Angeles after this short exposure period.

5. Comparison of Color, Gloss, and Number of Coats

These comparisons were reported for the six-months inspection of all the sites except Los Angeles. The comparison has been made with Los Angeles added but the results reported previously still stand. It is felt that no conclusions can be drawn about the one year inspection on the basis on one site so this will be reported after more sites have been examined.

6. New Nature-Tone Enamels on Aluminum

Six of the eight Nature-Tone enamels have been received from the suppliers. Five of the six sheets of enameled aluminum had been cut into exposure specimens prior to shipment to the Bureau. These enamels have been measured for their initial gloss, color, thickness, acid solubility, and acid spot test ratings.

PLANS FOR NEXT REPORT PERIOD

A. Nature-Tone Enamels on Steel

The remaining nine enamels should be received at the laboratory during the next report period. The laboratory tests will be made on these enamels and the specimens should be on the exposure racks at the end of the next report period.

B. Porcelain Enamels on Aluminum

The specimens exposed at New York City, Montreal, and Kure Beach should be inspected after one-year's exposure.

The remaining specimens of Nature-Tone enamels on aluminum should arrive at the Bureau of Standards, be cut into exposure specimens if necessary, and the initial gloss, color, thickness, and acid resistance of these enamels measured.

III. CONTINUITY OF COATING

INTRODUCTION

Continuity of coating is a very important aspect of porcelain enamels that will be placed in corrosive surroundings.

Preliminary studies have indicated that a high-voltage discharge probe is capable of locating many defects that are not readily observed by visual examination but which have led to early corrosion of the base metal.

RESULTS

1. Nature-Tone Enamels on Steel

A field weathering test has been undertaken to determine if fewer rusting failures occur among specimens from which the defective ones have been culled by electrical probing, than among similar specimens not so tested. The details of this program are described in Section II of this report.

Our previous evaluation of the electrical testing method of locating defects which permit early corrosion was based on the presence or absence of rust on weathered specimens compared with the defect count on "duplicate" specimens which had not been exposed to the weather. There is some question as to whether the electrical testing of duplicate specimens gives a valid characterization of the defects of other specimens from the same production lot. The field test is designed to circumvent this question.

2. High-Voltage Probes on Specimens Submitted by Manufacturers

Two companies have submitted specimens with typical porcelain enamel defects such as blisters, fishscale, pits, pinholes, surface contamination, onionskin, crawling, burnoff, hairlining, and tearing. All of these defects except hairlining and tearing were located with the high-voltage probe.

In addition to these enamels containing defects, five good, two-coat enamels were submitted. One of these had a defect located at 2 kV, and another had a defect located at 3.5 kV, while the remaining enamels had no defects located up to 5 kV.

When a washing machine top was examined with the high voltage probe there was only one area in which discharges occurred even when the voltage was raised to 5 kV and this area was on a small radius that forms the depression for the cover. It is likely that this radius had received some impact or torsion damage but not enough to remove the chip that had formed.

Specimens of a "good" and "bad" system of direct-on enamel were submitted in both 4 and 8 mil thicknesses. Two specimens of these enamels were probed with the high-voltage equipment, but no clear cut separation of these enamels was found.

PLANS FOR NEXT REPORT PERIOD

Test probes are going to be made on enamels, supposedly having good and bad continuity of coating which have been submitted by manufacturers.

It is also planned to measure the spark gap of the test equipment to see if these measurements might be used to calibrate similar test equipment in other laboratories.

IV. BACKGROUND MATERIAL FOR NEW PROJECTS

INTRODUCTION

At the November 18, 1965 meeting of the Standards Committee, it was suggested that future projects to be undertaken by the Research Associates should include the development of tests for: Scratch resistance, impact resistance, measuring stress in enamels, thermal shock, and abrasion.

RESULTS AND DISCUSSION

Since scratch resistance, impact resistance and abrasion resistance are somewhat related, a literature survey covering hardness, scratch resistance, abrasion and impact was initiated.

1. Hardness and Scratch Resistance.

The surface of an enamel or glaze has been found [6,7] to be harder than the underlying structure. This makes testing the hardness of an enamel by indentation [8,9] as used for metals somewhat unreliable.

As early as 1929, Westinghouse [10] had developed a method for testing the scratch resistance of production enamels by moving the enameled surface underneath a weighted phonograph needle. A similar test was used by Peterson [11] in 1947. He stated that this test might be used successfully in one plant but expressed doubts about its being used as a standard test since the phonograph needles were either dulled, or broken as soon as the enamel was scratched. It was also difficult to determine when the first scratching had occurred.

Bailey [12] developed a test for determining the scratch-resisting power of glass by rolling a 1/8 inch diameter ball over the glass surface. The pressure at which the first conchoidal breaks occurred was taken as a measure of the hardness of the glass. Ghering and Turnbull [13] conducted a similar study using 1/4 inch diameter rods with a hemispherical end. The rods were clamped at a 45° angle to the specimen surface and the specimen was pulled at a uniform rate under the rod. The pressure at which the crescent shaped cracks occurred indicated failure. The principle of the test methods used by Bailey and Ghering and Turnbull was incorporated, with modifications, into the test for gouge resistance for porcelain enamels [14, 15]. However, the end point in this test was taken as the pressure at which 50 percent of the ball track was gouged, and not the occurrence of the first crescent or conchoidal fracture.

Many investigators, [16,17,18,19] have studies the effect of scratching glasses, glazes or enamels with diamond points using diamonds of various shapes and sizes. The endpoint in these tests is usually defined in terms of the width of the scratch after a given load has been applied. The main difficulty in using this method of test occurs in measuring the width of the crack since spalling or flaking of the enamel away from the scratch often occurs.

2. Abrasion

Early abrasion tests for porcelain enamels and glazes were of the falling sand type. In one of the early abrasion tests [20] the weight loss was taken as a measure of the abrasion resistance after a given exposure to falling sand.

This was later modified [6] to weigh the amount of sand required to reduce the gloss by 10 percent of its initial value. There were many variations of this type of test including propelling the sand against the specimen from a rotating disc [21] or with a blast of air [22]. All of these methods were satisfactory for any given plant or laboratory but difficulty was encountered in getting the same results between plants.

The Tabor abrader has also been used [11,19,23] to measure the abrasion resistance of porcelain enamel but difficulties have been encountered in keeping the abrasive wheels free from the ground porcelain enamel.

In 1942 the Porcelain Enamel Institute issued a standard test [24] for the abrasion resistance of porcelain enamels. In this test a slurry of a standard grade of feldspar, water and a charge of steel balls was agitated on the specimen surface by means of a Ro-Tap machine for a given period of time. This method appeared to give reproducible results. Later findings, however, indicated some faults in the test procedure and it was modified [25] to the test method that is in use today. This method gives reproducible test results between laboratories and plants, but early investigations in the cleanability study indicated that the abrasion produced by the PEI abrader did not resemble many types that are encountered in service.

3. Impact

There have been two test methods developed for testing the impact resistance of porcelain enamels. One is the drop weight method [26] and the other is the pendulum method [27]. The drop weight method was designed especially for utensils while the pendulum method was designed to be used on specially

fabricated specimens. The preparation of the test pieces has been rather difficult and the use of this test has suffered accordingly.

PLANS FOR NEXT REPORT PERIOD

Because the assembling of background material for new projects is undertaken only in periods of inactivity caused by late specimen arrival or equipment failure, no definate activity is planned. However, it is most likely that the first new project to be undertaken will be one related to product performance such as the resistance to scratching or abrasion, and that the test method should be capable of evaluating competitive finishes as well as porcelain enamels.

STANDARD REFERENCE MATERIALS

The following stock of standards was on hand April 1, 1966:

Corundum abrasive, March 1960 issue,
for subsurface abrasion . . . . . 249 lbs., 61 jars.

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

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

Calibrated glass plates for abrasion testing . . . . . 26.5 doz.

Respectfully submitted,

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TABLE 1

Adjustment in Cleaning Procedure Necessary to Yield Comparable Results with Old and New Cleaning Equipment.

Old Equipment Cleaning Method 7	New Buehler-Olsen Equipment
Cleaning Time Before Tissue Changes	
Seconds	
15	30
15	30
30	60
120	120
120	120
Rubbing Pressure	
psi	
0.5	1.0

TABLE 2

Miscellaneous Cleanability Results on a
Brown, Glossy Porcelain Enamel.

Soil E (See Table 3), Method 10 (using Buehler-Olsen Mechanical Equipment)

Virgin Specimens		Redeterminations on Recleaned Specimens.	
Specimen No.	Soil Retained ug/cm^2	Specimen No.	Soil Retained ug/cm^2
13	0.22	13	0.29
14	.25	14	.25
15	.16	15	.17
16	.21	16	.21
17	.22	17	.[illegible]
19	.43	19	.20
Ave.	0.25	Ave.	0.23
V, percent	37.	V, percent	20.

Soil A, Method 7 (using old equipment)

Specimen No.	Soil Retained ug/cm^2
4	0.25
5	.32
8	.23
9	.22
10	.15
11	.28
Ave.	0.24
V, percent	23.

TABLE 3

Various Soiling Agents

Soil	Fluorescent Tracer		Colorant		Vehicle		Method of Mixing
	Type	Percentage	Type	Percentage	Type	Percentage	
A	BBOT	3.3	Powdered Graphite	32.8	Mineral Oil	63.9	BBOT & Graphite dry mixed, Oil mixed in on specimen surface.
B	BBOT	3.3	Powdered Graphite	32.8	Mineral Oil	63.9	Food Blender
C	BBOT	4.5	Powdered Graphite	44.7	Mineral Oil	50.8	Food Blender
E	BBOT	3.3	Powdered Graphite	55.7	Paraffin Oil	41.0	Vibratory Shaker
G	BBOT	3.3	Oildag	55.7	Paraffin Oil	41.0	Vibratory Shaker
GC	BBOT	1.0	Oildag	57.0	Paraffin Oil	42.0	Motorized Rotational Mixer
GE	BBOT	0.5	Oildag	57.4	Paraffin Oil	42.1	"
GF	BBOT	0.3	Oildag	57.4	Paraffin Oil	42.3	"
GG	BBOT	0.2	Oildag	57.9	Paraffin Oil	41.9	"
GH	BBOT	0.1	Oildag	57.6	Paraffin Oil	42.3	"

TABLE 4

Tests of Cleanability of Similar White Porcelain Enamels from the Same Lot.

Soil E, Method 10

Group No.	Number of Specimens	Average Soil Retained ug/cm^2	Date
I	12	0.27	2-10-66
II	12	.25	2-11-66
III	12	.16	2-15-66
IV	12	.18	2-16-66
V	6	.10	2-23-66
VI	6	.14	2-24-66

TABLE 5

Cleanability Experiments Using Soil G.

White Porcelain Enamel
Soiling and Cleaning Method 10

Specimen Number	Order of Extraction	Soil Retained ug/cm^2
79	1	0.86
114	2	.33
60	3	.12
76	4	.11
97	5	.14
101	6	.11
	Average of last four	0.12
42	1	0.49
108	2	.34
79	3	.10
114	4	.14
60	5	.12
11	6	.11
	Average of last four	0.12
88	6	0.35
100	5	.18
111	4	.11
75	3	.05
115	2	.08
37	1	.11
	Average of last four	0.09

In the above experiments, the bottle of Soil G was shaken thoroughly before filling a hypodermic syringe with an amount sufficient for six specimens. The sequence for each specimen, in turn, involved (1) dispensing 0.15 ml of soil on the specimen, (2) mechanically smearing the soil on the specimen for one minute, and (3) mechanically cleaning the specimen for a total cleaning time of six minutes.

TABLE

The Effect of Settling Time on the Fluorescence of Some Soiling Agents Containing Various Amounts of BBOT.

Soil No	BBOT Content	Settling Time [a/]	Concentration of Diluted Soil [b/]	Fluorescence Reading
	Weight percent	hours	ug soil/g. soln	
GC	1.0	0	1.9	off scale
GE	0.5	0	1.9	69.0
GF	0.3	0	1.9	48.5
GG	0.2	0	1.9	36.0
GH	0.1	0	1.8	29.5
GC	1.0	0.17	1.9	94.0
GC	1.0	1.5	1.9	80.5
GC	1.0	72.	1.9	61.5
GE	0.5	72.	1.9	56.0
GF	0.3	72.	1.8	49.0
GH	0.1	72.	2.1	32.0

a/ Time between mixing and sampling for dilution.

b/ $\text{Concentration, ug soil/g. soln} = \frac{\text{Sample, ug, x aliquot}}{\text{Wt. 1st soln. x wt. 2nd soln.}}$

TABLE 7

Summary of Initial Date for Nature-Tone Enamels on Steel

Enamel	45° Specular Gloss	Acid Resistance		Thickness mils	Continuity of Coating	
		Spot Test	Solubility mg/in^2		Test Voltage kV	Percent Passed
101	13.8	B	14.4	8.8	1.5	43
102	13.5	B	9.5	8.0	2.0	56
103	13.3	A	8.3	7.5	1.5	25
104	17.4	B	7.4	9.2	2.0	100
108	19.2	A	1.2	9.4	2.5	90
109	16.6	A	0.7	9.2	2.5	100
110	20.7	AA	0.8	9.0	2.0	100
111	18.5	A	1.0	10.8	2.5	90
112	13.5	A	0.9	10.7	2.0	100
113	10.1	A	1.0	10.8	2.5	100
4	11.3	A	2.8	8.8	2.0	82

TABLE 8

Summary of Exposure Data for Porcelain Enamels on Aluminum

Enamel	Los Angeles		Washington				Storage				Visual Color	Acid Solubility mg/in^2
	Gloss	Color	Gloss		Color		Gloss		Color			
	8 mo.	8 mo.	6 mo.	1 yr.	6 mo.	1 yr.	6 mo.	1 yr.	6 mo.	1 yr.		
AA-A	95.6	99.5	93.2	91.6	99.5	99.6	98.8	98.2	99.9	99.9	White	5.5
AA-B	103.0	99.3	92.7	91.5	99.5	99.3	99.2	98.4	99.7	99.8	White	5.9
AA-C	96.0	99.0	90.7	90.8	98.8	98.8	99.1	98.7	99.9	99.8	White	5.0
AA-D	101.0	98.2	94.6	92.5	97.8	97.9	99.2	98.7	99.8	99.7	White	12.7
AB-A	84.9	98.7	82.8	80.9	98.8	98.8	99.6	98.7	99.9	99.9	White	7.2
AB-C	83.6	99.4	81.0	83.6	99.5	99.5	98.1	96.9	99.8	99.8	White	4.9
AB-D	79.6	98.4	93.3	92.6	98.9	98.8	99.2	97.1	99.9	99.9	White	7.9
AC-A	102.6	99.1	93.0	91.8	99.3	99.2	99.0	98.5	99.4	99.6	White	6.4
AC-B	102.1	98.6	92.8	90.2	99.1	98.8	99.3	98.8	99.8	99.7	White	11.3
AC-C	98.2	98.9	93.7	92.1	98.6	98.6	99.8	99.5	99.7	99.8	White	9.9
AD-A	90.3	99.4	91.7	91.1	99.4	99.4	99.6	98.5	99.7	99.8	White	6.2
AD-B	92.4	99.4	89.8	88.9	99.6	99.6	99.6	99.2	99.8	99.9	White	6.7
AD-C	87.5	98.5	90.9	90.3	99.0	98.8	98.8	98.0	99.7	99.9	White	7.1
AD-D	88.6	98.4	98.0	96.5	96.9	98.1	99.6	98.8	99.7	99.9	White	12.4
AE-A	80.5	99.8	81.3	80.5	98.6	98.8	97.8	96.5	99.6	99.7	Black	6.5
AE-B	87.9	99.7	86.5	82.9	99.8	99.8	98.8	97.7	99.8	99.7	Black	10.1
AE-C	91.1	99.4	89.4	85.6	99.6	99.6	98.4	97.4	100.0	99.9	Black	12.1
AE-D	85.5	99.4	82.2	78.4	98.0	96.4	98.6	97.1	99.5	99.6	Black	15.5
AF-A	85.7	99.6	85.7	80.0	99.2	99.1	99.0	98.6	99.7	99.9	Black	14.2
AF-B	96.1	99.3	93.2	91.7	98.8	99.0	98.5	98.1	99.8	99.9	Black	9.0
AF-C	87.5	99.6	84.5	82.6	98.8	99.1	98.8	97.9	99.6	99.7	Black	10.1
AG-B	79.5	99.3	100.5	97.8	98.6	98.7	98.6	96.8	99.7	99.7	Black	12.5
AG-C	33.2	99.4	105.7	100.8	99.4	99.5	96.2	92.3	99.4	99.6	Black	7.5
AH-A	103.7	97.4	95.8	96.6	98.1	97.6	100.3	100.1	99.3	99.4	Red	8.1
AH-B	76.8	95.5	72.3	70.0	94.6	94.0	100.1	99.6	99.7	99.7	Red	8.8
AH-C	77.6	91.9	70.8	68.5	91.9	91.5	100.1	99.7	99.4	99.7	Red	6.5
AH-D	83.2	91.0	74.7	70.6	88.9	87.2	99.6	98.9	99.6	99.6	Red	10.5
AO-A	83.3	99.9	82.1	79.3	99.1	99.0	99.5	98.8	99.7	99.9	Dk. Green	19.9
AO-B	84.6	99.8	84.0	82.7	99.5	99.7	99.2	98.3	99.7	99.6	Dk. Green	10.1
AO-D	87.0	98.9	83.8	80.7	98.5	98.0	98.9	97.3	99.7	99.8	Dk. Green	17.0
AP-A	90.2	99.3	95.5	93.4	99.4	99.2	99.2	98.3	99.8	99.8	Lt. Green	12.3
AP-B	77.0	99.6	82.9	81.9	99.4	99.4	98.7	98.0	99.8	99.8	Lt. Green	6.4
AP-C	72.5	99.2	85.7	83.9	99.0	99.1	98.2	97.5	99.8	99.8	Lt. Green	8.2
AP-D	88.2	99.2	93.9	93.4	99.0	99.0	98.1	97.8	99.8	99.9	Lt. Green	10.0
AR-A	25.5	99.6	111.8	104.8	99.5	99.6	94.5	90.8	99.7	99.7	Lt. Green	4.4
AR-B	0.0	99.6	82.5	74.7	99.6	99.6	100.0	91.4	99.8	99.7	Lt. Green	5.5
AR-C	0.0	99.5	85.7	75.6	99.6	99.6	100.5	89.9	99.7	99.8	Lt. Green	8.1
AS-A	92.1	99.5	90.2	86.4	99.4	99.0	98.7	97.7	99.9	99.8	Gray	13.4
AS-B	82.5	99.3	83.0	82.3	99.3	99.1	99.1	98.9	99.6	99.6	Gray	7.4
AS-C	93.3	99.8	91.8	92.2	99.6	99.7	99.0	98.9	99.7	99.6	Gray	5.4
AT-A	71.4	99.1	83.8	83.2	98.9	98.9	98.8	98.8	99.8	99.9	Blue	6.2
AT-B	90.9	97.9	93.8	94.1	98.9	99.0	99.4	98.8	99.9	99.9	Blue	7.0
AT-C	82.0	99.0	78.8	79.2	97.1	99.1	98.8	98.2	99.8	99.7	Blue	6.1
AU-A	87.7	99.7	84.3	84.9	99.7	99.7	99.1	98.7	99.6	99.6	Brown	5.3
AU-B	80.3	99.8	92.4	91.8	99.6	99.6	98.2	97.6	99.8	99.6	Brown	7.5
AU-C	91.4	99.8	94.5	93.9	99.5	99.4	98.6	98.2	99.8	99.6	Brown	7.6
AW-A	85.3	99.6	82.7	81.5	99.4	99.4	98.8	98.2	99.9	99.9	Yellow	7.8
AW-B	93.7	99.3	93.3	92.4	99.2	99.0	98.5	98.1	99.8	99.7	Yellow	8.7
AW-C	91.7	99.4	84.6	81.4	99.4	99.0	99.7	99.0	99.9	99.8	Yellow	18.6
AZ-A	104.1	98.9	93.5	91.5	99.3	99.2	100.7	100.2	99.9	99.8	White	9.5
AZ-B	99.1	98.8	90.6	91.2	99.0	98.9	100.0	99.7	99.7	99.6	White	5.2
Average	82.9	98.9	88.8	86.7	98.6	98.6	99.0	97.8	99.7	99.7		

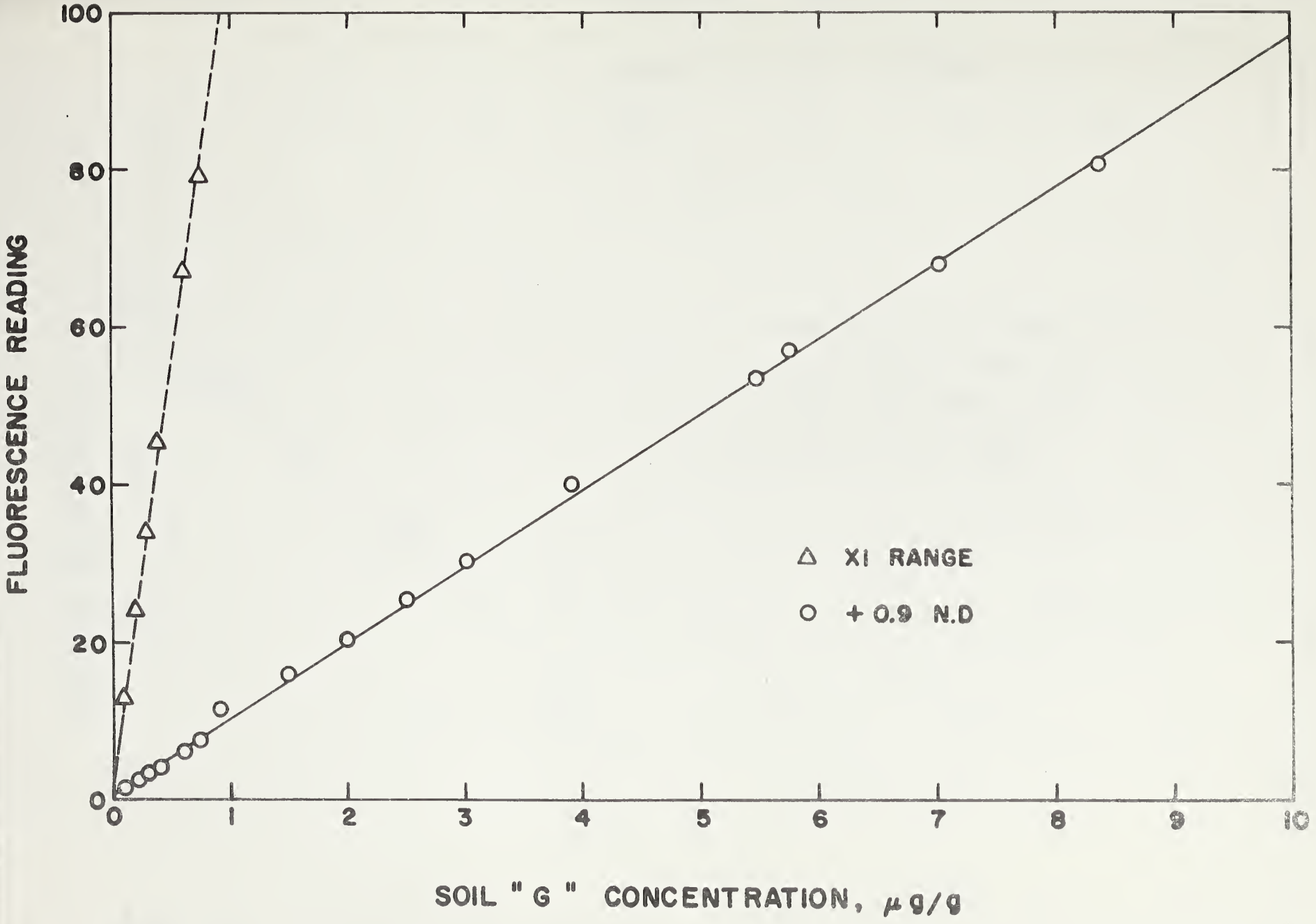

FIG. 1 Calibration of Fluorometer for Use With Soil G

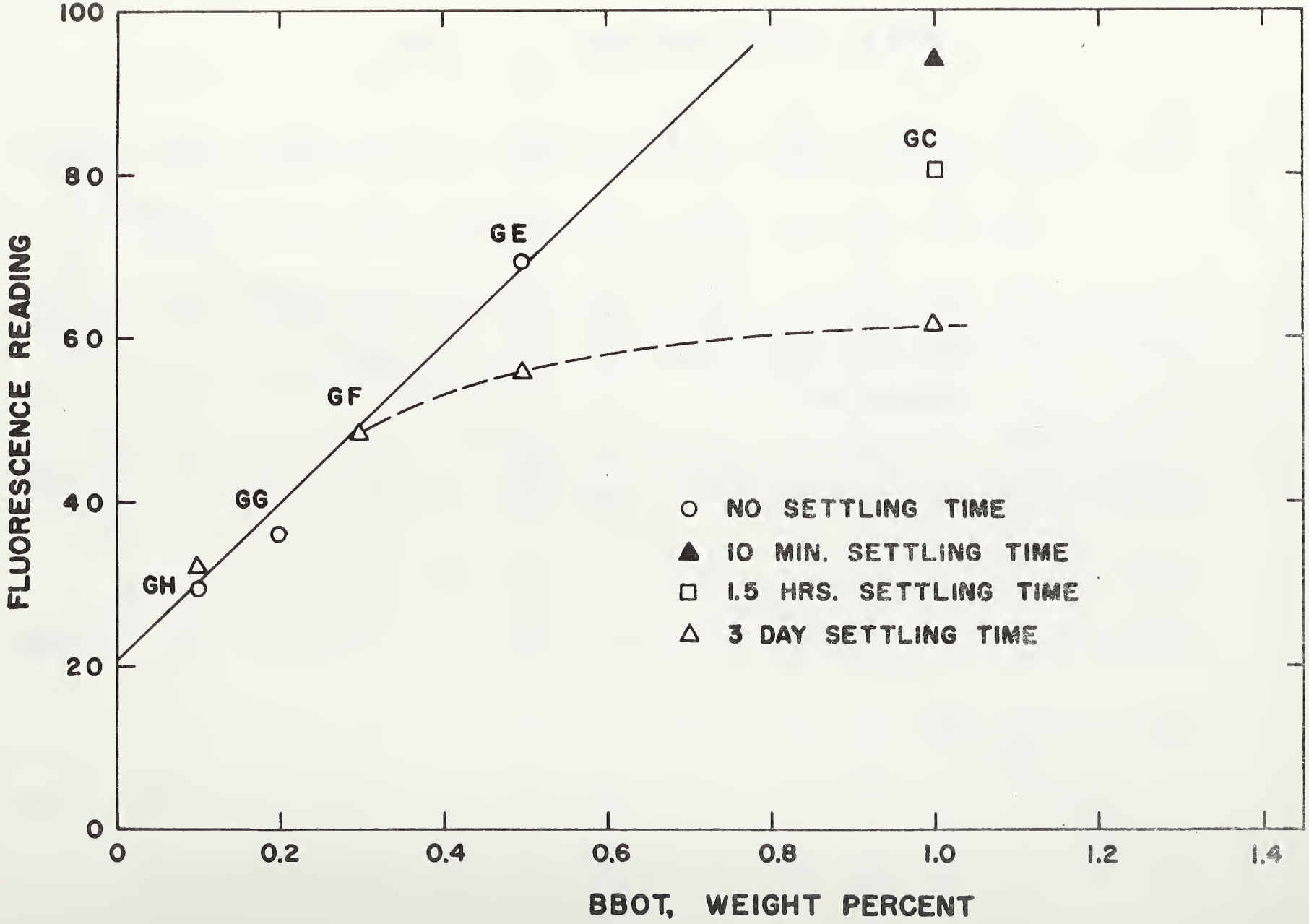

FIG. 2 The Effect of Settling Time on The Fluorescence Response of Several Soil Preparations.


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