# NATIONAL BUREAU OF STANDARDS REPORT

8535

PROGRESS REPORT

April 1 through June 30, 1964

Development of Methods of Test
For Quality Control of Porcelain Enamels

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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* NBS Group, Joint Institute for Laboratory Astrophysics at the University of Colorado.

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NBS

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## SUMMARY

A modification of the P.E.I. Alkali Test Equipment was devised to assure equality of alkali attack in any of the six specimen positions. In addition, the reproducibility of three cleaning methods was evaluated and one was selected for recommendation as the Standard Test Method. The recommended method will involve the exposure of a limited area of either three or six porcelain enameled specimens to a five percent solution of sodium pyrophosphate maintained at 96° ± 0.2°C for six hours. Weight loss will serve as a measure of the alkali resistance.

An analysis was made of citric acid spot test grading procedures. A questionnaire was sent to 44 appliance manufacturers. Replies indicated that the majority of inspectors used the architectural wet-rubbing method for grading A and B enamels rather than the PEI T-21 test method. A laboratory study in which six individuals graded 18 enamels by both procedures showed that the architectural method (complete removal of pencil marks from the treated area) gave better reproducibility than the T-21 test procedure in which the end point is the difference in ease of removal of pencil marks in the treated and untreated areas.

Final arrangements were made for the five exposure sites for the weathering of production-type aluminum enamels. Procurement of the necessary specimens and the exposure racks progressed on schedule.

Measurements of specimens exposed for seven years in the PEI-NBS weathering test were completed. Reduction of these data was nearly completed. Results are given for green enamels at all sites. Boiling citric acid tests were made on storage specimens of several enamels. The correlation with weather resistance was comparable to the correlation obtained by the citric acid spot test.

A round-robin test was made to determine the reproducibility of boiling citric acid test which is used for architectural porcelain enamels on aluminum. An analysis of variance showed that the data obtained by the three laboratories did not differ significantly at the 95% confidence level.

## I. STANDARD ALKALI TEST

### Introduction

Laboratory work was started during the previous quarter to improve the design and reliability of the Porcelain Enamel Institute alkali test equipment. The capacity of the device was increased from three to six specimens per test. A small but statistically significant difference was found, at that time, in the severity of the alkali attack between specimens facing the inside and those facing the outside of the equipment.

## Results and Discussion

1. Increasing Specimen Capacity

The specimen holder boxes were altered by cutting an opening in the back side of the box and inserting an "O" ring seal to permit two specimens rather than one to be tested in each box. A simple modification has been devised during the present quarter to insure complete equality of attack in any of the six specimen locations available. This was done by increasing the stirring speed from 1400 to 1800 r.p.m. and, at the same time, placing an open-ended stainless steel cylinder, 3½" in diam. by 4" high,around the outside of the heating element in such a way that the lower open end of the cylinder was at the same elevation as the bottom of the heater. With this arrangement, the stirrer moves the test solution down through the inside of the stainless steel cylinder after which it flows upward around the specimen holders. A series of alkali test results with this modified arrangement at both 96° and 99°C on a group of specimens of an alkali-resistant enamel is given in Table 1. These results show the uniformity of attack between specimens facing the center and those facing the outer beaker wall. A statistical "t" test confirmed that the attack was the same in either position. Further evidence on this point may be seen in the three groups of alkali test data in the top half of Table 2. The use of these data in connection with methods of cleaning specimens will be discussed later. Within each of these three specimen groups representing the three cleaning methods (top of Table 2) one half of the specimens were exposed to the alkali solution facing the center of the apparatus while the other half faced the outside.

(Of the method No. 1 specimens 3, 4, 7, 33, 35 and 37 faced the inside.) When the test results were grouped in this manner the following average values were obtained for weight-loss in mg/in.$^2$

| | Cleaning Method #1 | Cleaning Method #2 | Cleaning Method #3 |
|---|---|---|---|
| Facing inside | 7.11 | 8.33 | 8.32 |
| Facing outside | 7.17 | 8.20 | 8.37 |

An analysis of variance confirmed the equivalence of attack by any method of cleaning in either position.

The data in Table 2 were also used to establish if alkali test results were affected by the box into which the specimen was placed. The determinations of alkali resistance performed in box 1 were separated from those in box 2 and box 3. This separation for the test equipment using brass boxes gave the following results:

| Box No. | Cleaning Method #1 | Cleaning Method #2 | Cleaning Method #3 |
|---|---|---|---|
| 1 | 7.02 mg/in.$^2$ | 8.25 mg/in.$^2$ | 8.22 mg/in.$^2$ |
| 2 | 7.20 | 8.35 | 8.40 |
| 3 | 7.21 | 8.20 | 8.40 |

Here again an analysis of variance confirmed what one can readily see - that alkali resistance measured in weight loss per unit area has no significant dependence on the holder into which the specimen is placed. Also, the data showed that uniform exposure to alkali solution was obtained in all of the six possible positions in which the specimen might be inserted for testing. Because of this finding all specimens tested by a single cleaning method could justifiably be consolidated as in Table 2.

2. Selection of a Cleaning Method

In making a test of alkali resistance there are two specimen-cleaning steps which must be specified if gross errors resulting from the cleaning method are to be eliminated. The specimen must be prepared for testing before the initial weight is obtained, and at the conclusion of the treatment the corrosion products which cling to the treated surface must be removed to a reproducible extent. It was hoped to specify one cleaning method which would serve in both situations. The three methods selected for study represented a wide range of severity. The first method required only rinsing with a fine jet of distilled water from a wash bottle; the second method involved using a small amount of a 1% solution of trisodium phosphate on a soft cellulose sponge and rubbing with a light pressure followed by a distilled water rinse; while the third involved rubbing with a nylon-bristled hand-brush using a heavy pressure, followed by a distilled water rinse.

An experimental study was designed to permit evaluation of the cleaning methods and, at the same time, to compare results in the equipment modified to accommodate two specimens per box with those obtained in the stainless steel boxes containing only one specimen. The latter equipment is similar to that now in use in industry. Similar circular baffles were installed in both devices in the hope of obtaining comparable results. A series of alkali tests was made on comparable specimens of a single porcelain enamel. The results are given in Table 2. Following the analysis of these data, method #2 (wet rubbing) was selected. This selection was based on the following considerations:

a. Wet rubbing exhibited the least scatter as indicated by low coefficients of variation, V.

b. Method No. 1 was eliminated from consideration because it did not provide a sufficiently clean surface prior to testing and it was not sufficiently severe to remove "O" ring marks which sometimes occurred on specimens after the alkali treatment.

c. A nylon-bristled hand brush (Method No. 3), while not an expensive item, is less likely to be found in industrial testing laboratories than is a soft cellulose sponge.

d. The wet-rub was apparently sufficiently severe to remove the major portion of the adhering corrosion product.

3. The Temperature Dependence of Alkali Resistance

Tests of alkali resistance of a single enamel were performed at 96°C and at 99°C. It was desired to determine whether or not the larger weight losses at 99° would contribute to increased reproducibility. It was found that the standard deviations were reduced from 6 to 2 percent at the higher temperature. However a test temperature of 99° is not recommended because crystalline deposits that form on the exterior of the specimen boxes marking the solution level during the test make it quite difficult to remove the boxes at the end of the test period. The results in Table 1 allow one to calculate an increase in weight-loss of 12 percent per degree Centigrade between 96° and

99°C, which exactly confirms the figure referred to in the preceding report.[1/]

4. Temperature Controller

Previous work with the thermistor-actuated, on-off controller currently used with the equipment established short range control (up to six hours) of plus or minus 0.15°C. A recent 20-hour test continuously recorded the e.m.f. of a copper-constantan thermocouple located in the mercury at the bottom of the thermometer well which was immersed in the circulated alkali solution. The on-off cycling was indicated by the thermometer to be of the order of ±0.15°C. This cycling was continuous and uniform during the twenty-hour period. No larger temperature excursions were recorded and no long-time drift could be seen. These results confirm the adequacy of this controller for use in a standard test method.

<u>Plans for the Next Report Period</u>

The laboratory work which was aimed at selection of a standard procedure for alkali testing of porcelain enamels, is essentially complete. It is proposed that the standard procedure involve exposing a limited area, on either three or six specimens, to a five percent solution of sodium pyrophosphate maintained at 96° ± 0.2°C for six hours. Specimen weights before and after this treatment will permit calculation of loss-in-weight, expressed in $mg/in.^2$ of exposed surface, as a measure of alkali resistance.

No further work on alkali test development is planned.

## II. ANALYSIS OF SPOT-TEST GRADING PROCEDURES FOR THE ACID RESISTANCE OF PORCELAIN ENAMELS

### Introduction

The PEI spot test for acid resistance has enjoyed wide use in the porcelain enamel industry ever since it was first promulgated in 1939. The general consensus within the industry is that the test has been reasonably satisfactory in predicting the degree to which the enamel finishes on appliances, sanitary ware and even architectural panels will resist the type of acid attack encountered in service.

Briefly, a small area of the enamel surface is exposed to 10% citric acid for 15 min. at room temperature and then, after removal of the acid, the treated area is evaluated for degree of etch. This evaluation is made visually with the aid of such criteria as (1) Is there a visible strain? (2) Does the etching cause blurring of a reflected image? and (3) Is a pencil mark placed across the tested area of the specimen more easily removed from the treated than the untreated area?

The evaluation procedure was believed to be reasonably well described in the original test pamphlet, but it was observed that some operators deviated from the specified grading procedure, probably because of a misinterpretation. This was especially true for the wet-rubbing test for removal of pencil marks, which is used to separate class A enamels from class B enamels. Many people that made the test graded the enamel class A if the pencil marks <u>could be removed</u> by wet rubbing rather than if there was <u>no difference in the ease</u> with which the marks could be removed from the treated and untreated areas.

A somewhat better correlation was observed with weather resistance when the "complete removal" grading system was used; hence, this type of wet-rubbing test was included in the PEI Architectural Specification S-100 (62).

In an effort to reduce the variation that apparently existed in application of the earlier PEI T-7 test procedure, an ad hoc committee was appointed to investigate and, if necessary rewrite the T-7 pamphlet to clarify the exact grading procedure to be followed. This was done and the revised version was adopted by the Standards Committee in 1962 as PEI Standard Test T-21. Table 3 defines the differences in the various grading systems, past and present.

At the Standards Committee meeting in February 1964 the grading method for class A and class B enamels was again discussed. The point was made at the meeting that a number of production enamels that have in the past been graded as A are now graded as B by the new test procedure (T-21). These enamels have performed satisfactorily in service yet they are no longer acceptable under most purchase specifications because they require an acid resistance of class A or better. In the ensuing discussion, the following questions were raised with regard to selection of the most suitable method for differentiating between class A and class B enamels:

(1) Which of the two grading methods (complete removal or difference in ease of removal) is used most widely at the present time in the appliance industry?

(2) Which method of grading gives the best reproducibility?

(3) What degree of temperature control is required?

It was decided that no action would be taken by the Committee on any revision of T-21 until answers were obtained to these questions. The progress that has been made towards obtaining these answers is reported below.

## Results and Discussion

1. Survey of Type of Wet-Rubbing Test Procedure Used by Appliance Manufacturers

A questionnaire was sent to forty-four companies representing the major manufacturers of porcelain enameled appliances and plumbing fixtures. Thirty-eight replies were received from twenty of these companies. These replies were tabulated and the results are shown on the sample questionnaire (Table 4).

The majority of the inspectors used the architectural wet-rubbing method where the end point criterion is complete removal of the pencil marks. Only three inspectors said they used the new standard PEI test T-21, and even this number is questionable since no one indicated that they had changed grading methods subsequent to promulgation of this new test.

Eleven replies stated that differences had arisen regarding grading of the wet-rubbing test. Of these eleven, eight employed either the ASTM (7) or the PEI (1) procedure for grading by the wet-rubbing test, while only three used the architectural method. Thus, it is indicated that fewer differences exist when complete removal is used as an end point criterion than when ease of removal is taken as the end point.

The tabulation also shows that nineteen of the inspectors used a dry-rubbing test. While the questionnaire was not worded to reveal what dry-rubbing method was used, it is felt that the inspectors used the same criteria as they reported using for the wet-rubbing test.

2. Laboratory Study of Reproducibility of Architectural and T-21 Grading Procedures for Wet-Rubbing Test

The reproducibility of the two wet-rubbing grading procedures was investigated by having six impartial observers rate 18 specimens of acid-resistant enamels. The specimens, which were 12-inches square, were submitted by three different frit manufacturers. The acid-resistant enamels applied to the specimens were selected so as to include some enamels that might be designated either A or B depending on the method of grading by the wet-rubbing test.

All spots were applied at 80° ± 2°F by one individual in accordance with the standard procedure. A fresh spot was applied for each observer who rated the enamel after carefully reading the respective test procedures. In the case of the architectural procedure, the enamel received an A rating if the No. 3B pencil marks could be completely removed and a B rating if they could not be removed. If none of the observers could detect a visable stain, the enamel was rated AA and not considered in the analysis. Seven of the 18 enamels were so rated, leaving only 11 enamels for study by the wet rubbing tests.

Observer No. 2 was unable to detect a stain on two enamels where a stain was observed to be present by the other five, and similarly, observer No. 1 did not observe a stain on one enamel. For purposes of the analysis the assigned rating in these three cases was class A rather than AA since this part of the study was not designed to investigate any part of the grading system except the wet-rubbing test. Had these AA ratings been included, the analysis would have been biased in favor of the architectural procedure.

A summary of the results is listed in Table 5. Perfect agreement between observers occurred for six of the 11 enamels when the architectural procedure for grading (complete removal) was used as against three of the 11 enamels for the T-21 procedure (difference in ease of removal). The total number of discrepancies (deviations from the majority rating) was only six for the architectural procedure while for the T-21 procedure it was three times as great (18). A point of some interest was that one observer (obs. No. 4) arrived at exactly the same ratings by both grading procedures for all 11 enamels. If all observers were like No. 4 there would be no need for concern about which wet-rubbing test to use.

Some variations in the appearance of the various stained areas were observed on several of the specimens, indicating that there were local variations in acid resistance across the surface. This effect could account for some of the disagreement among observers but the effect should be present to the same extent on the spots used for the T-21 grading as on the spots used for the architectural. A more important cause for the discrepancies is probably the interpretation that is placed by the various observers on the written procedure. It seems highly doubtful, with a test involving the judgement of an operator, that any better agreement could be achieved than is represented by the architectural grading procedure.

The data in Table 5 seem clear in showing that there is considerably more disagreement among observers when using the T-21 procedure than when using the architectural method, at least in the case of this particular series of enamels. The survey of appliance manufacturers also indicated more differences of opinion by inspectors when using T-21 than when using the architectural procedure. Thus, because both the laboratory analysis and the industry survey showed fewer disagreements among observers when complete removal of the pencil marks was used as the criterion, it may be concluded that the architectural grading method is a more reproducible test procedure for current production enamels.

Table 5 also shows that if the architectural method was incorporated into T-21, only two of the 11 enamels (Nos. 11 and 15) would be raised from B to A when majority ratings were considered and even for these two enamels two of the six observers graded the enamel A by the T-21 method. Thus there does not appear to be any clear-cut upgrading of B enamels when the architectural grading procedure is used.

Enamels nos. 4, 5 and 7 are of special interest in the comparison of the two test procedures. Using the architectural method, all observers rated these three enamels A; yet by the T-21 procedure three observers assigned A ratings and three assigned B ratings to each of the three enamels. This type of disagreement when using the T-21 method could undoubtedly be lessened by a special training program for inspectors. However, special training of a test operator does not seem to be a feasible approach for a standard test procedure.

3. Effect of Temperature on Spot-Test Rating

A preliminary attempt was made to determine the effect of test temperature on the acid-resistance rating for this particular series of 18 enamels. Test procedure T-21 was used. All of the testing and grading was done by operator No. 4.

Table 6 gives the results. Three enamels (Nos. 6, 7 and 13) increased in rating with temperature. This is believed to be due to local variations in acid resistance across the specimen surface which might possibly be eliminated by applying and grading a larger number of spots.

Of the remaining 15 enamels, eight showed a decrease in rating from 70° to 90°F, but the decrease was never greater than one AR step. Thus, the tendency is for decreasing acid resistance rating with increasing temperature. To obtain more meaningful results on the temperature effect would require a much more comprehensive study in which more than one observer would grade a minimum of three spots for each of the test temperatures. There is some question whether such a study would be worth the time since the present test procedure calls for a temperature of 80° ± 2°F. The only reason for considering it at all is to determine whether or not this degree of temperature control is essential.

4. Reproducibility of Spot-Test Rating Methods

The same group of 18 enamels was also rated by the six observers with respect to the other criteria used for assigning ratings by spot-test procedures. The results, which are summarized in Table 7, show that the wet-rubbing test when made by the architectural method, gives about the same degree of agreement among observers as the visible stain and blurring highlight tests. However, when the wet-rubbing test is made by the T-21 method, the degree of agreement drops markedly. Hence, these data show that, for these enamels, the architectural wet rub (but not the T-21 wet rub) is almost equally as reproducible a test criterion as the visible spot and the blurring highlight tests.

All six observers passed the five stain-free enamels in the dry-rubbing test irrespective of whether the testing was done by the architectural or T-21 method. From the results with this series of enamels, there would appear to be some question as to whether dry-rubbing is a worthwhile test to include in the standard test procedure since all five enamels passed. However, the returns from the field questionnaire showed that half of those replying used the dry-rubbing test in their regular grading procedures. Unfortunately, the question as to whether or not they ever observed a failure was not asked.

Plans for the Next Report Period

No further work is planned on the AR spot test.

## III. EXPOSURE TEST OF ALUMINUM ENAMELS

Introduction

A separate exposure test of current production enamels for aluminum was approved early in the calendar year by the Aluminum Council of the PEI. Activities since that time have been concerned with (a) selection of enamels, (b) selection and procurement of exposure sites, (c) procurement of the metal sheet and (d) application of the selected enamels to the sheets to provide the necessary specimens. Table 8 lists the enamels that will be included in the test.

Progress During Past Quarter

1. Arrangements for five exposure sites were completed. These five sites are Washington, Kure Beach, North Carolina, Los Angeles, New York City and Montreal.

2. The racks for each site were finished and placed in packing crates for shipment.

3. A total of 205 - 3 x 5' sheets of 0.064" thick aluminum alloy 6061 (H-12 temper) was received. These sheets were first divided into smaller lots and then the required number shipped to the ten cooperating fabricators.

4. Instruction sheets were sent to each fabricator at about the same time that 3 x 5 ft. sheets were shipped. These sheets gave explicit instructions on a) enamel type, b) suggested mill batch, c) thickness of coating, and d) desired gloss and color.

Plans for the Next Report Period

The enameled sheets upon return from the fabricators will be cut into $4\frac{1}{3}$ x $4\frac{1}{3}$" specimens and the gloss, color, and thickness measured. The target date for installation of the specimens at the 5 sites is October of this year.

## IV. PEI-NBS WEATHERING TEST

Introduction

The PEI-NBS exposure test is now entering its eighth year. At the end of seven years all specimens were returned to Washington and their gloss and color measured. These 7-yr data are now in process of being reduced and analyzed preparatory to writing a 7-yr report.

This does not constitute termination of the test but it appears likely that there will be no further complete inspections of specimens until 1971.

Results and Discussion

1. Correlation of Two Color Difference Meters

In February 1963 the National Bureau of Standards traded in their original Hunter Color Difference Meter in the purchase of the latest model of the instrument. The operating characteristics of the new instrument were checked when it was first received and they were believed to be satisfactory. Recently, however, when measuring the last two sets of specimens from the Kure Beach sites, it was discovered that the two color difference meters did not give the same readings on all enamels. Consultation with the Bureau's Colorimetry Section indicated that this is common in color difference meters produced at different times because they do not have the same filter-photocell response. This difference in the filter-photocell response is not noticeable if the "standard" has spectral characteristics close to those of the color being measured. This was true for the whites, blacks, and grey-blues in the test but the greens, reds, yellows and bright blues differed significantly from the NBS standards, which had been used as the "standard" for the color difference meter.

The difference between the NBS standard and the specimen color resulted in a measured color difference of as much as four NBS units between six and seven years' exposure on enamels that showed no appreciable color change between three and six years' exposure. Correction curves were established to eliminate these differences. These curves were based on the Rd, a, and b readings of the reference enamels after they had been stored for five and seven years. These enamels had spectral characteristics close to those of the exposed specimens and should have shown no color change between five and seven years' storage if the color difference meter had not been changed. Knowing this, the individual Rd, a, and b values for the reference enamels for five years (old meter) were plotted against those for seven years (new meter) for each color standard used in the test. A least squares line was then fitted through these points. The difference between this fitted line and a 45 degree line (see Figure 1) is the difference in the filter-photocell response of the two color difference meters. All data taken with the new instrument were corrected through use of curves of this type.

2. Effect of Exposure time

The gloss retention and the color stability index of all the green enamels exposed at all seven sites are given in Tables 9 and 10. The enamels exposed at Dallas, Washington and Kure Beach - 80 ft sites were chosen to represent the mild, moderate and severe sites as reported in 1963[2/]. It can be seen (Figure 2) that the gloss retention at the mild site, Dallas, and the moderate site, Washington, remains nearly constant after three years' exposure, while the gloss at the most severe site, Kure Beach - 80 ft, continues to decrease with exposure time.

The color continues to change slightly during all seven years' exposure with the greatest color change occurring at Kure Beach - 80 ft, and the least change at Dallas (see Figure 3).

3. Correlation Between Acid Resistance and Weather Resistance

The average color stability index, gloss retention and acid resistance for the mat steel enamels and the mat aluminum enamels are given in Table 11. These are the only two types of enamels evaluated in this section because they are the only types completely represented by green enamels (see Table 12). It can be seen from the data in Table 11 that the gloss retention does not show good correlation with the acid spot rating for the mat steel enamels at any of the exposure sites and it correlates for only four of the seven sites for the mat aluminum enamels. These results are similar to those reported by Potter[3/] after these enamels had been exposed for one year.

In the case of the color stability index, the correlation is somewhat better; correlation being noted for three of the seven exposure sites for the mat steel enamels, while the mat aluminum enamels showed a correlation for all seven exposure sites.

The boiling acid solubility had also been determined on the mat aluminum enamels to determine whether the weathering characteristics would correlate better with this measure of acid resistance than with the spot test rating. The results obtained from the boiling acid tests correlated with the gloss retention for only two of the seven sites and with the color stability index for six of the seven sites. Thus, for the limited number of enamels in the test, the spot test rating seems to correlate better with the weathering characteristics of the mat aluminum enamels than does the boiling acid solubility test.

4. Comparison of Acid Spot Test and Acid Solubility with the Weather Resistance of Steel Enamels after Five Years' Exposure at Washington

The acid resistance of all the enamels in the test has been determined by the acid spot test method[4]. This method of rating the enamels places them into five distinct classes. It has been shown[2,5] that the degree of weather resistance of porcelain enamels on steel is indicated by the acid spot test ratings only when averages are considered. In 1960, Sopp, et al[6] noted that the weather resistance of porcelain enamels on aluminum correlated better with a boiling acid solubility test than with the spot test ratings. This improved correlation resulted from rating the enamels on a continuous weight loss scale.

During this quarter the acid solubility was determined on twelve steel enamels in the weathering test. Six of these enamels were selected because their acid spot ratings correlated with their weather resistance, particularly their color stability index, while the other six were selected because they lacked any apparent correlation between weather resistance and acid spot rating. The results of the acid solubility and acid spot test are given in Table 13 and are plotted against gloss retention and color stability index after five years' exposure at Washington in Figures 4 and 5, respectively.

It can be seen in Table 13 that the acid solubility correlates with the spot test ratings for the enamels whose weathering characteristics correlated with the spot test rating. This indicates that the acid spot test and boiling acid solubility measure the same property of the enamel and those enamels whose weathering characteristics do not correlate with one method of determining acid resistance will not correlate when the other method is used.

A curve was fitted through the acid solubility values (for all 12 enamels) while the spot test values were averaged. When a straight line is fitted through the average spot test values, it becomes evident that the same trend is present whether the acid spot test or the boiling acid solubility is compared with weather resistance. It can be seen in Figure 4 that the individual acid solubility points scatter about the fitted curve in the same manner as the individual acid spot test points scatter about their average values. However the color stability values shown in Figure 5 correlate somewhat better with the acid solubility than with the average spot test ratings. It is difficult with this limited data to say which method of measuring acid resistance correlates better with weather resistance.

Plans for the Next Report Period

The rest of the seven-year data will be reduced and a paper will be prepared for presentation at the Ceramic-Metal Systems Division meeting in September and at the PEI Shop Practice Forum in October.

## V. ROUND-ROBIN TEST ON THE ACID RESISTANCE OF ALUMINUM ENAMELS BY THE BOILING-ACID TEST

Late in the report period, the Research Associateship cooperated in a round-robin test to explore the reproducibility of the ASTM C-283 boiling acid test. Three laboratories took part in the test: a frit manufacturer, a fabricator, and the Research Associates of the Porcelain Enamel Institute.

The results obtained are given in Table 14. The differences in results obtained between the most divergent laboratories represented approximately 17 percent of the total weight loss. These differences included equipment and operator variables, barometric pressure and elevations above sea level, interpretation of and technique in maintaining a "rolling boil" as well as the ever present sampling error among specimens.

It is known that not unreasonable differences in the rate of boil alone can effect the boiling acid weight-loss results by several milligrams per square inch.

An analysis of variance was made on the first three specimens tested by each laboratory. This analysis showed that the three laboratories did not differ significantly (95 percent confidence level). Hence, it is believed that reasonably good agreement between laboratories was obtained.

## VI. STANDARD REFERENCE MATERIALS

A new shipment of $4\frac{3}{8}$ by $4\frac{3}{8}$ by $\frac{1}{4}$ inch plate glass specimens was received and calibrated during this report period. The new glass plates did not differ significantly (at the 95% confidence level) from those calibrated in January 1963. However, the surface abrasion factor of 50 ± 0.5% (shown on p. 5, section II-C-4, PEI Bulletin T-2) will be changed to 49.2 ± 0.5% and the subsurface abrasion factor of 5.00 (shown on p. 8, section IV-C-7, PEI Bulletin T-2) will be changed to 5.058. These changes are identical to those used with the 1963 standards and an information sheet giving these changes will be sent with the new standard glass plates.

The following stock of standards was on hand as of July 1, 1964.

Corundum Abrasive, March 1960 issue, for subsurface
abrasion test 324 lbs - 81 jars

Standard Pennsylvania Glass Sand for surface abrasion,
July 1963 issue 336 lbs - 112 jars

Distinctness of image gloss standards - 20 sets

Calibrated glass plates for abrasion testing - 39 dozen.

Respectfully submitted,

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## REFERENCES


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2. D. G. Moore and Alan Potter, "Effect of Exposure Site on Weather Resistance of Porcelain Enamels Exposed for Three Years", N.B.S. Monograph 44 (1962) U. S. Government Printing Office, Washington, D.C. 20234

3. Alan Potter, "NBS Progress Report No. 6346, Nov. 16, 1958 - Feb. 15, 1959, National Bureau of Standards, Washington, D. C. 20234.

4. "Test for Acid Resistance of Porcelain Enamels, Pt. 1, Flatware", Issued by the Porcelain Enamel Institute, Inc., 1145 19th St. N. W. Washington, D. C. 20036.

5. D. G. Moore and W. N. Harrison, "Fifteen-Year Exposure Test of Porcelain Enamels", National Bureau of Standards Building Materials and Structures Report 148, June 1957, U. S. Govt. Printing Office, Washington, D. C. 20234.

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Table 1. Dependence of Alkali Resistance on Specimen Position

Tests at 96 C., 6 Hours, 5% Pyro

| Facing Inside | | Facing Outside | |
|---|---|---|---|
| Specimen No. | Weight Loss mg/in$^2$ | Specimen No. | Weight Loss mg/in$^2$ |
| A-70 | 2.6 | A-71 | 2.7 |
| A-73 | 2.6 | A-72 | 2.4 |
| A-75 | 2.7 | A-74 | 2.6 |
| A-77 | 2.8 | A-76 | 2.7 |
| A-79 | 2.6 | A-78 | 2.6 |
| A-81 | 3.0 | A-80 | 2.7 |
| A-83 | 2.8 | A-82 | 2.9 |
| A-85 | 2.7 | A-84 | 2.8 |
| Average | 2.72 | | 2.68 |
| Std. Dev. | 0.1643 | | 0.1761 |
| V, Percent | 6.0 | | 6.6 |

Tests at 99 C., 6 Hours, 5% Pyro

| | | | |
|---|---|---|---|
| A-86 | 3.7 | A-87 | 3.6 |
| A-88 | 3.6 | A-89 | 3.6 |
| A-90 | 3.6 | A-91 | 3.7 |
| A-93 | 3.6 | A-92 | 3.6 |
| A-95 | 3.7 | A-94 | 3.8 |
| A-97 | 3.8 | A-96 | 3.7 |
| Average | 3.67 | | 3.67 |
| Std. Dev. | 0.0812 | | 0.0812 |
| V, Percent | 2.2 | | 2.2 |

Table 2. Specimen Cleaning Procedures in Alkali Testing

| | Method #1 | | | Method #2 | | | Method #3 | | |
|---|---|---|---|---|---|---|---|---|---|
| | RINSE ONLY | | | LIGHT WET RUB | | | HARD RUB | | |
| | Specimen No. | Weight Loss | Gloss Retained | Specimen No. | Weight Loss | Gloss Retained | Specimen No. | Weight Loss | Gloss Retained |
| | | $mg/in^2$ | percent | | $mg/in^2$ | percent | | $mg/in^2$ | percent |
| Brass Boxes | B-02 | 7.3 | 4.5 | B-08 | 8.3 | 5.9 | B-50 | 8.5 | 4.6 |
| | B-03 | 7.0 | 5.0 | B-09 | 8.1 | 6.9 | B-51 | 8.3 | 5.2 |
| 2 Specimens | B-04 | 7.1 | 5.0 | B-10 | 8.6 | 6.2 | B-52 | 8.1 | 4.9 |
| | B-05 | 7.2 | 4.5 | B-11 | 8.2 | 6.6 | B-53 | 8.7 | 5.4 |
| Per Box | B-06 | 6.7 | 5.4 | B-12 | 7.9 | 5.7 | B-54 | 8.5 | 4.8 |
| | B-07 | 7.0 | 4.7 | B-13 | 8.7 | 5.4 | B-55 | 8.1 | 5.7 |
| Exposed Area | B-32 | 7.4 | 4.7 | B-38 | 8.4 | 5.1 | B-44 | 8.9 | 4.2 |
| | B-33 | 7.1 | 4.2 | B-39 | 8.0 | 6.5 | B-45 | 8.1 | 5.6 |
| Each Specimen | B-34 | 7.5 | 4.5 | B-40 | 8.2 | 5.1 | B-46 | 7.9 | 4.9 |
| | B-35 | 7.0 | 5.0 | B-41 | 8.4 | 5.8 | B-47 | 8.2 | 6.4 |
| $8.6\ in^2$ | B-36 | 6.9 | 4.8 | B-42 | 8.0 | 4.7 | B-48 | 8.5 | 4.5 |
| | B-37 | 7.5 | 4.4 | B-43 | 8.4 | 6.1 | B-49 | 8.3 | 5.6 |
| | Average | 7.14 | 4.72 | | 8.27 | 5.83 | | 8.34 | 5.15 |
| | Std. Dev. | 0.2466 | 0.3330 | | 0.2462 | 0.6708 | | 0.2875 | 0.6186 |
| | V, Percent | 3.5 | 7.0 | | 3.0 | 11.5 | | 3.4 | 12.0 |
| Stainless Steel | B-17 | 7.7 | 6.0 | B-21 | 8.2 | 8.6 | B-24 | 8.9 | 7.4 |
| Boxes 1 Specimen | B-19 | 7.5 | 6.2 | B-22 | 8.3 | 10.0 | B-25 | 9.2 | 7.1 |
| Per Box | B-20 | 6.9 | 6.9 | B-23 | 7.7 | 9.6 | B-26 | 8.8 | 8.5 |
| Exposed Area | B-27 | 8.0 | 7.2 | B-30 | 8.1 | 9.2 | B-14 | 8.7 | 8.8 |
| Each Specimen | B-28 | 7.7 | 7.7 | B-31 | 8.6 | 8.2 | B-15 | 8.5 | 11.7 |
| $6.38\ in^2$ | B-29 | 7.9 | 7.7 | B-32 | 8.1 | 9.2 | B-16 | 7.8 | 9.0 |
| | Average | 7.62 | 6.95 | | 8.17 | 9.13 | | 8.65 | 8.75 |
| | Std. Dev. | 0.3920 | 0.7287 | | 0.2943 | 0.6531 | | 0.4764 | 0.8216 |
| | V, Percent | 5.2 | 10.5 | | 3.6 | 7.2 | | 5.5 | 9.4 |

Table 3. Procedural Differences Between Three Standard Methods for Determining Citric Acid Spot Test Ratings.

| Method / Procedures | PEI T-7 ASTM C-282 | PEI T-21 | Architectural Specification S-100 (62) |
|---|---|---|---|
| Marking Pencil | No. 1 | 3B | No. 1 |
| Temperature of Test | 80 ± 10°F | 80 ± 2°F | 80° ± 10°F |
| Wet and Dry Rubbing Endpoint | 1/ | 2/ | 3/ |
| Rubbing Pressure | Not Specified | 4/ | Vigorous |

1/ "If the marks are retained more tenaciously on the treated area than on the protected area the specimen fails the test."

2/ "If the marks are removed from either of the two areas more readily than from the other the specimen fails the test."

3/ "If the marks are removed completely by this wet-rubbing treatment the specimen passes the test."

4/ "Starting with gentle pressure and then applying gradually increasing pressure rub repeatedly across the lines...in such a manner that the marks in the treated and protected areas are rubbed at the same time."

Table 4. Questionnaire and Tabulation of Replies On the Grading of Porcelain Enamel Using the Citric Acid Spot Test[a].

1. Do you use the dry-rubbing test on enamels that show no visible stain?

Yes 19 No 19

2. In the wet-rubbing test, do you, after applying the pencil marks, grade the enamels as class B if:

| | | |
|---|---|---|
| (architectural method) | a. 21 | pencil marks in the treated area cannot be completely removed by rubbing, |
| (T-21 method) | b. 3 | pencil marks are removed from either of the two areas more readily than from the other area, |
| (ASTM method) | c. 14 | pencil marks over the treated area are definitely more difficult to remove than those over the protected area. |

3. Have you changed your method of grading A and B enamels in the past year?

Yes 0 No 38

In the past five years? Yes 0 No 36 (2 did not answer)

4. If your answer to question 3 is yes, which of the methods in question 2 did you previously use for the wet-rubbing test?

2a NA

2b NA NA = No answer

2c NA

5. Have you graded enamels where a difference of opinion arose between different people as to whether the enamel should be rated "A" or "B"?

Yes 11 No 26 (1 did not answer)

6. If yes, please describe how these differences are resolved. (Reply summary: Differences arise on whether or not pencil marks must be completely removed and amount of pressure and time used in wet rubbing test. Differences resolved by re-examination, by referral to supervisor, or by customer's decision.)

7. Have you encountered many field complaints directly attributable to lack of adequate acid resistance?
(Only two affirmative answers, one being a single case, and the other involving Government specifications.)

COMPANY ______________________

ADDRESS ______________________

a/ This is a sample questionnaire that was sent to the manufacturers. The numbers indicate the replies received.

Table 5. Wet-Rubbing Test Ratings by Six Observers for Those Enamels That Showed Visible Staining After the Acid Treatment

| | Acid Resistance Rating by Wet Rubbing Test | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Architectural Method[a] | | | | | | | T-21 Method[b] | | | | | | |
| Enamel No. | Obs. No.1 | Obs. No.2 | Obs. No.3 | Obs. No.4 | Obs. No.5 | Obs. No.6 | No. of Discrepancies | Obs. No.1 | Obs. No.2 | Obs. No.3 | Obs. No.4 | Obs. No.5 | Obs. No.6 | No. of Discrepancies |
| 3 | A | A | A | A | A | A | 0 | B | A | A | A | A | B | 2 |
| 4 | A | A | A | A | A | A | 0 | B | A[c] | B | A | B | A | 3 |
| 5 | A | A | A | A | A | A | 0 | B | A[c] | B | A | A | B | 3 |
| 7 | A[c] | A | A | A | A | A | 0 | B | B | A | A | A | B | 3 |
| 9 | B | A | B | B | B | B | 1 | B | B | B | B | B | B | 0 |
| 10 | B | B | B | B | A | B | 1 | B | B | B | B | B | B | 0 |
| 11 | A | A | A | B | A | B | 2 | B | B | A | B | A | B | 2 |
| 12 | B | B | B | B | B | B | 0 | B | B | A | B | B | B | 1 |
| 15 | A | A | A | A | A | B | 1 | B | B | B | A | A | B | 2 |
| 17 | A | A | A | A | A | B | 1 | B | A | A | A | A | B | 2 |
| 18 | B | B | B | B | B | B | 0 | B | B | B | B | B | B | 0 |
| | | | | | | | 6 | | | | | | | 18 |

a/ Complete removal of pencil marks from treated area.

b/ Difference in ease of removal of pencil marks from treated and untreated areas.

c/ No visible stain detected by observer but on A rating designed to prevent bias in comparison.

Table 6. AR Ratings by Observer No. 4 when Using T-21 Grading Procedure On 18 Enamels at Three Test Temperatures.

| Enamel No. | Test Temperature 70°F | 80°F | 90°F |
|---|---|---|---|
| 1 | AA | AA | AA |
| 2 | AA | AA | A |
| 3 | AA | A | A |
| 4 | A | A | A |
| 5 | A | A | A |
| 6 | A | A | AA |
| 7 | AA | A | AA |
| 8 | AA | AA | AA |
| 9 | B | B | B |
| 10 | A | B | B |
| 11 | A | B | B |
| 12 | A | B | B |
| 13 | A | A | AA |
| 14 | AA | AA | A |
| 15 | A | A | A |
| 16 | AA | AA | AA |
| 17 | A | A | B |
| 18 | B | B | C |

Table 7. Results by Six Observers for 18 Enamels When Using Various Spot-Test Criteria.

| | Visible Spot[a/] | | Dry-Rubbing | | Wet Rubbing[a/] | | Blurring Highlight[a/] | |
|---|---|---|---|---|---|---|---|---|
| Enamel No. | No.1 | No.2 | Arch. Method | T-21 Method | Arch. Method | T-21 Method | No.1 | No.2 |
| 1 | Pass (1) | Pass | Pass | Pass | Pass | Pass(1) | Pass | Pass |
| 2 | Pass | Pass | Pass | Pass | Pass | Pass | Pass | Pass |
| 3 | Fail | Fail (1) | - | - | Pass | Pass(2) | Pass | Pass |
| 4 | Fail | Fail (1) | - | - | Pass | Pass(3) | Pass | Pass |
| 5 | Fail | Fail (1) | - | - | Pass | Pass(3) | Pass | Pass |
| 6 | Fail | Fail (2) | - | - | Pass | Pass(1) | Pass | |
| 7 | Fail(1) | Fail | - | - | Pass | Pass(3) | Pass | Pass |
| 8 | Pass | Pass (1) | Pass | Pass | Pass | Pass | Pass | Pass |
| 9 | Fail | Fail | - | - | Fail(1) | Fail | Pass(1) | Pass(2) |
| 10 | Fail | Fail | - | - | Fail(1) | Fail | Pass | Pass(1) |
| 11 | Fail | Fail | - | - | Pass(2) | Fail(2) | Pass | Pass |
| 12 | Fail | Fail | - | - | Fail | Fail(1) | Pass(1) | Pass(2) |
| 13 | Fail(2) | Fail(1) | - | - | Pass | Pass(1) | Pass | Pass |
| 14 | Pass | Pass | Pass | Pass | Pass | Pass(1) | Pass | Pass |
| 15 | Fail | Fail | - | - | Pass(1) | Fail(2) | Pass | Pass |
| 16 | Pass(2) | Pass | Pass | Pass | Pass | Pass | Pass | Pass |
| 17 | Fail | Fail | - | - | Pass(1) | Pass(2) | Pass | Pass |
| 18 | Fail | Fail | - | - | Fail | Fail | Pass(2) | Pass |
| No.of enamels with no disagreement among observers | 13 | 12 | 5 | 5 | 13 | 6 | 15 | 15 |
| % of enamels with no disagreements | 72 | 67 | 100 | 100 | 72 | 33 | 83 | 83 |

a/ Figures in parenthesis indicate number of observers that did not agree with majority.

Table 8. Enamels in PEI-NBS Aluminum Weathering Test

| Enamel Color | Enamel Gloss | Number of Coats | Enamel Thickness (mils) |
|---|---|---|---|
| White | Full gloss | One | 2.5 - 3.5 |
| White | Full gloss | Two | 4 - 6 |
| White | Full gloss | Two | under 3 |
| White | Semi gloss | One | 2.5 - 3.5 |
| White | Semi gloss | Two | 4 - 6 |
| Black | Full gloss | One | 2.5 - 3.5 |
| Black | Semi gloss | One | 2.5 - 3.5 |
| Black | Mat | One | 2.5 - 3.5 |
| Red | Full gloss | One | 2.5 - 3.5 |
| Dark green | Full gloss | One | 2.5 - 3.5 |
| Pastel green | Semi gloss | Two | 4 - 6 |
| Pastel green | Mat | Two | 4 - 6 |
| Pastel blue | Semi gloss | Two | 4 - 6 |
| Pastel grey | Semi gloss | Two | 4 - 6 |
| Pastel brown | Semi gloss | Two | 4 - 6 |
| Pastel yellow | Semi gloss | Two | 4 - 6 |

Table 9. Gloss Retentions For Green Enamels

| | Gloss Retention at the Indicated Exposure Time | | | | | | |
|---|---|---|---|---|---|---|---|
| | 1 Yr | 2 Yr | 3 Yr | 4 Yr | 5 Yr | 6 Yr | 7 Yr |
| Dallas | 82.5 | - | 78.2 | - | - | - | 80.0 |
| Los Angeles | 72.1 | 69.1 | 69.7 | - | - | - | 81.9 |
| New Orleans | 76.1 | - | 77.5 | - | - | - | 58.9 |
| Pittsburgh | 74.3 | 73.0 | 70.5 | - | - | - | 74.1 |
| Kure Beach - 800 ft | 76.8 | 64.4 | 68.0 | 60.8 | 64.4 | 61.1 | 61.8 |
| Washington | 71.2 | 65.8 | 65.7 | 63.5 | 63.3 | 63.2 | 60.1 |
| Kure Beach - 80 ft | 70.5 | 63.1 | 54.9 | 46.0 | 45.1 | 43.4 | 37.4 |

Table 10. Color Stability Index for Green Enamels

| | Color Stability Inded at the Indicated Exposure Time | | | | | | |
|---|---|---|---|---|---|---|---|
| | 1 Yr | 2 Yr | 3 Yr | 4 Yr | 5 Yr | 6 Yr | 7 Yr |
| Dallas | 97.9 | - | 97.6 | - | - | - | 97.0 |
| Los Angeles | 97.2 | 96.8 | 97.3 | - | - | - | 97.7 |
| New Orleans | 97.1 | - | 97.3 | - | - | - | 95.2 |
| Pittsburgh | 96.0 | 96.5 | 96.6 | - | - | - | 96.5 |
| Kure Beach-800 ft | 96.7 | 92.8 | 96.0 | 95.3 | 93.9 | 94.5 | 93.4 |
| Washington | 96.7 | 94.2 | 96.0 | 95.8 | 95.3 | 94.9 | 94.8 |
| Kure Beach-80 ft | 97.0 | 93.9 | 95.6 | 94.9 | 94.2 | 93.6 | 93.1 |

Table 11. Comparison of Acid Resistance with Weather Resistance after Seven Years' Exposure

## A. MAT STEEL ENAMELS

| | Color Stability Index | | | | Gloss Retention | | | |
|---|---|---|---|---|---|---|---|---|
| Acid Spot Rating | A | B | C | D | A | B | C | D |
| Kure Beach 80 ft | 96.1 | 94.6 | 94.8 | 93.8 | 49.4 | 36.2 | 40.7 | 22.5 |
| Kure Beach 800 ft | 97.9 | 94.0 | 94.1 | 93.7 | 78.3 | 51.6 | 55.5 | 40.0 |
| Washington | 98.3 | 96.7 | 97.3 | 88.8 | 72.9 | 63.8 | 51.0 | 56.5 |
| Pittsburgh | 98.7 | 96.1 | 97.4 | 96.1 | 74.9 | 75.7 | 73.1 | 31.6 |
| Los Angeles | 99.0 | 96.9 | 98.0 | 98.2 | 77.6 | 75.7 | 89.3 | 80.2 |
| New Orleans | 96.8 | 97.5 | 96.3 | 98.0 | 78.1 | 42.2 | 49.4 | 40.5 |
| Dallas | 98.9 | 95.7 | 96.8 | 98.4 | 73.7 | 72.7 | 87.3 | 104.6 |
| Number of Enamels | 1 | 8 | 8 | 1 | 1 | 8 | 8 | 1 |

## B. MAT ALUMINUM ENAMELS

| | Color Stability Index | | | | | | Gloss Retention | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Acid Spot Rating | AA | AA | AA | A | A | A | AA | AA | AA | A | A | A |
| Acid Solubility (mg/in.$^2$) | 6.3 | 6.5 | Ave. | 8.1 | 20.7 | Ave. | 6.3 | 6.5 | Ave. | 8.1 | 20.7 | Ave. |
| Kure Beach 80 ft | 91.7 | 93.0 | 92.3 | 90.6 | 90.9 | 90.7 | 29.8 | 69.2 | 49.5 | 22.5 | 64.8 | 43.6 |
| Kure Beach 800 ft | 91.4 | 93.0 | 94.5 | 91.2 | 90.3 | 90.7 | 101.2 | 90.2 | 95.7 | 51.7 | 52.8 | 52.3 |
| Washington | 96.9 | 95.6 | 96.3 | 95.2 | 93.5 | 94.3 | 56.5 | 59.1 | 57.8 | 43.1 | 73.9 | 58.5 |
| Pittsburgh | 98.9 | 95.2 | 97.1 | 96.4 | 96.4 | 96.4 | 78.7 | 65.7 | 72.2 | 78.1 | 112.6 | 95.3 |
| Los Angeles | 99.2 | 96.9 | 98.1 | 97.7 | 96.8 | 97.2 | 80.4 | 73.8 | 77.1 | 70.8 | 78.1 | 74.4 |
| New Orleans | 93.9 | 89.0 | 91.4 | 90.0 | 90.3 | 90.1 | 79.3 | 13.3 | 46.3 | 42.4 | 0.0 | 21.1 |
| Dallas | 99.0 | 96.6 | 97.8 | 96.6 | 98.1 | 97.4 | 73.6 | 79.8 | 76.7 | 79.8 | 92.7 | 86.2 |
| Number of Enamels | 1 | 1 | 2 | 1 | 1 | 2 | 1 | 1 | 2 | 1 | 1 | 2 |

Table 12. Enamels in the PEI - NBS Weathering Test

| Type of Enamel | Number of Enamels in Test | Number of Green Enamels in Test |
|---|---|---|
| Regular Glossy Steel Acid-Resistant | 24 | 0 |
| Mat Aluminum | 4 | 4 |
| Mat Steel | 18 | 18 |
| Regular Glossy Steel Non-Acid-Resisting | 16 | 0 |
| Glossy Aluminum | 10 | 4 |
| 1000°F Steel | 2 | 1 |
| 1300°F Steel | 4 | 2 |
| Red and Yellow Screening Pastes | 13 | 0 |
| Total Number of Enamels | 91 | 29 |

Table 13. Comparison of Boiling Acid Solubility with the Acid Spot Test Ratings of Enamels Exposed Five Years at Washington

A) Enamels whose acid spot test ratings correlated with their weather resistance

| Enamel | Acid Spot Test Rating | Boiling Acid Solubility (mg/in.$^2$) | Gloss Retention (%) | Color Stability Index |
|---|---|---|---|---|
| A4 | AA | 0.6 | 81.3 | 99.3 |
| H4 | A | 2.1 | 79.8 | 98.9 |
| M1 | A | 2.7 | 78.2 | 98.3 |
| B1 | B | 13.4 | 43.0 | 98.5 |
| N1 | C | 21.1 | 67.5 | 92.1 |
| K4 | D | 63.9 | 59.6 | 83.3 |

B) Enamels whose acid spot test ratings did not correlate with weather resistance

| Enamel | Acid Spot Test Rating | Boiling Acid Solubility (mg/in.$^2$) | Gloss Retention (%) | Color Stability Index |
|---|---|---|---|---|
| D3 | AA | 3.4 | 73.2 | 98.4 |
| D4 | AA | 4.0 | 63.4 | 98.5 |
| L3 | A | 3.2 | 73.4 | 99.1 |
| N4 | C | 41.5 | 73.7 | 91.1 |
| K1 | C | 20.9 | 70.0 | 98.8 |
| E4 | D | 22.3 | 71.6 | 99.0 |

Table 14. Results of Round-Robin Boiling Acid Test (ASTM C-283) on Two Aluminum Enamels

| Test No. | A) Light Gray Enamel Fabricator mg/in.$^2$ | A) Light Gray Enamel Frit Mfger. mg/in.$^2$ | A) Light Gray Enamel PEI mg/in.$^2$ | B) Light Brown Enamel Fabricator mg/in.$^2$ | B) Light Brown Enamel Frit Mfger. mg/in.$^2$ | B) Light Brown Enamel PEI mg/in.$^2$ |
|---|---|---|---|---|---|---|
| 1 | 10.9 | 10.9 | 12.0 | 11.1 | 15.7 | 10.1 |
| 2 | 12.3 | 14.9 | 10.7 | 10.2 | 14.1 | 15.8 |
| 3 | 14.1 | 13.7 | 10.4 | 10.3 | 13.9 | 15.0 |
| 4 | 11.8 | - | - | 9.9 | - | - |
| 5 | 12.2 | - | - | 10.4 | - | - |
| 6 | 12.7 | - | - | 10.8 | - | - |
| 7 | - | - | - | 10.9 | - | - |
| 8 | - | - | - | 12.3 | - | - |
| 9 | - | - | - | 11.8 | - | - |
| 10 | - | - | - | 12.1 | - | - |
| 11 | - | - | - | 11.4 | - | - |
| Average | 12.3 | 13.2 | 11.0 | 11.9 | 14.6 | 13.6 |

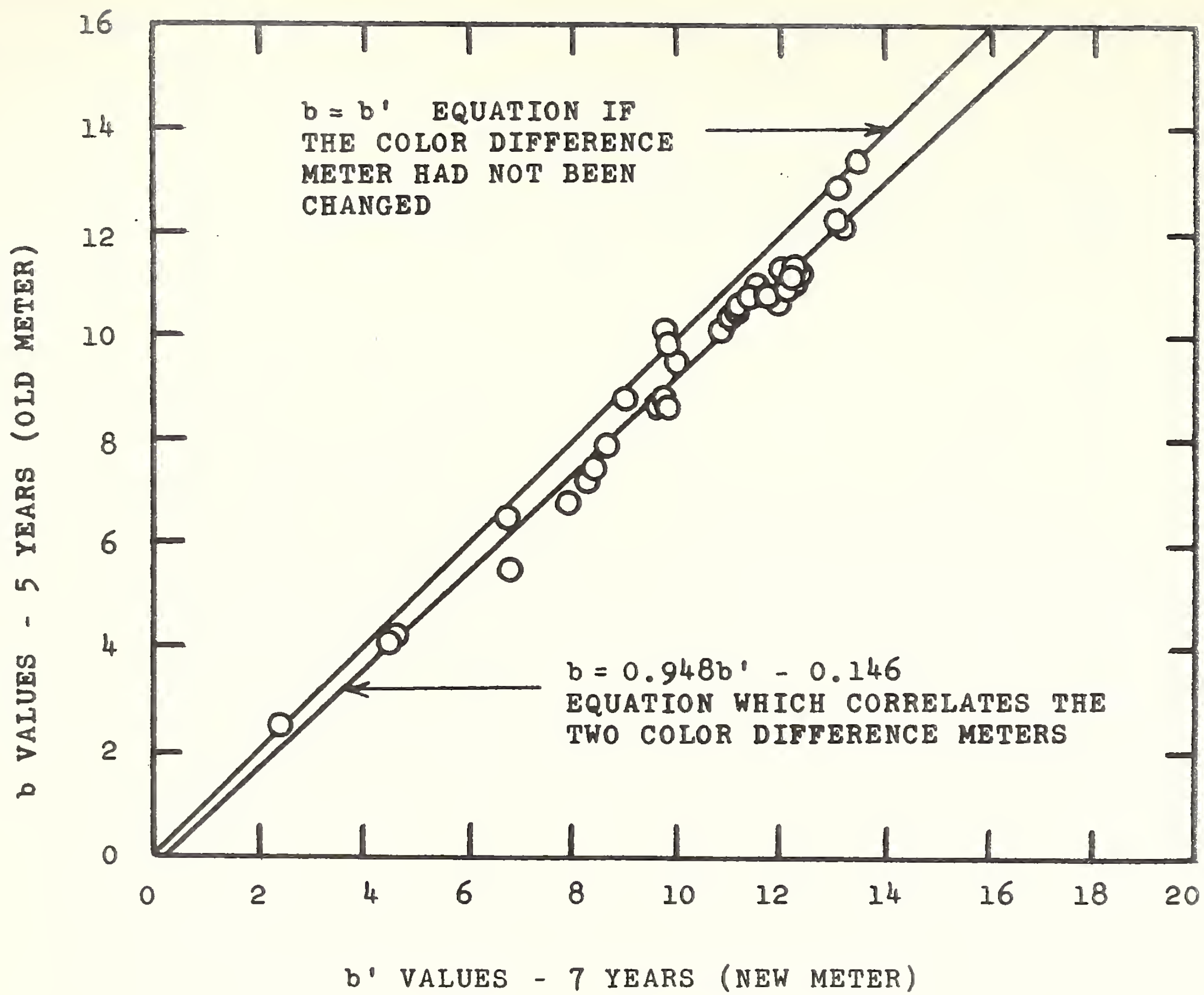


Figure 1. Correction curve for b when NBS standard number SKC-15 is used.

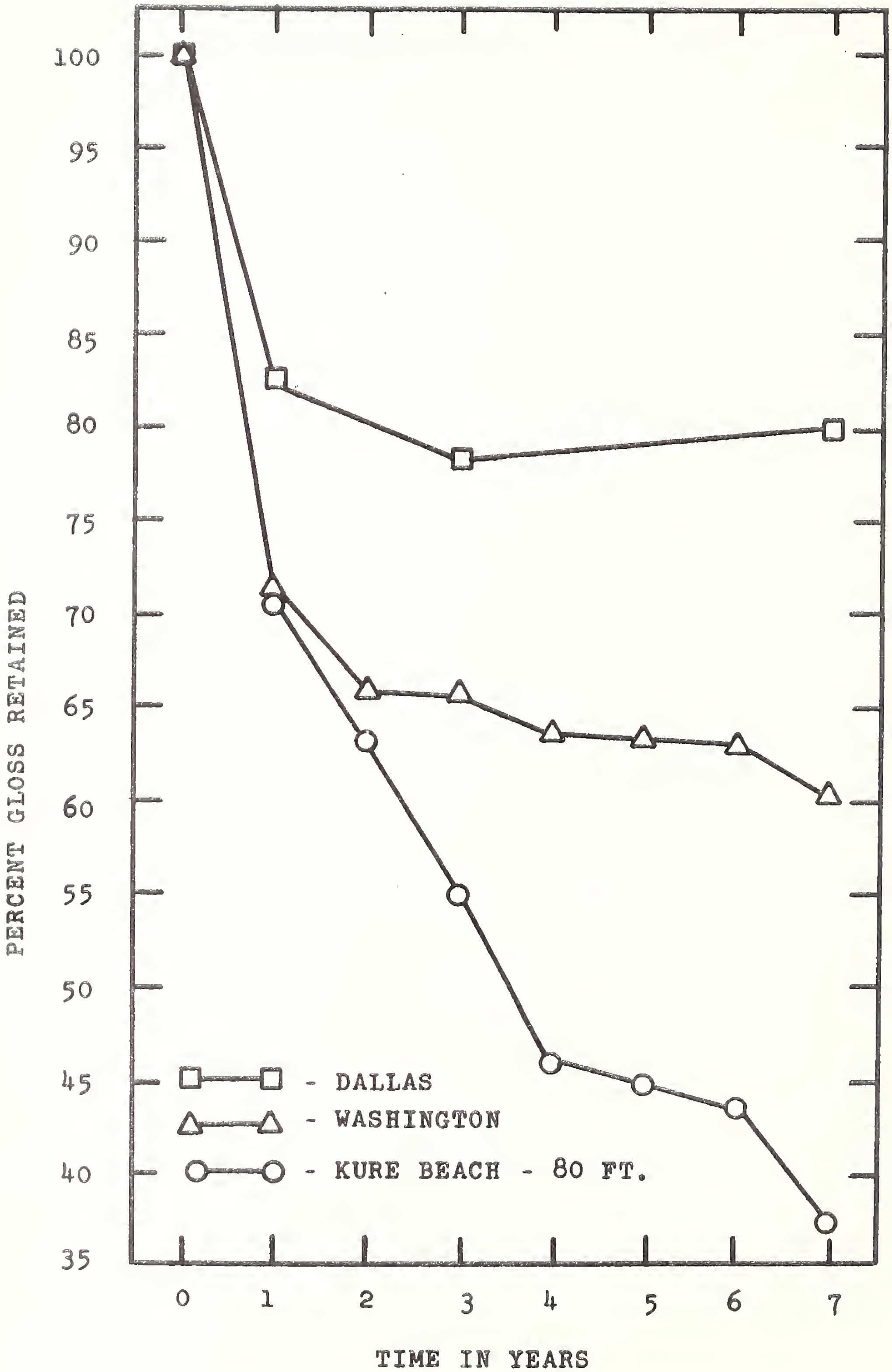


Figure 2. The effect of time on the gloss retention of all green enamels exposed at Dallas, Washington and Kure Beach - 80 ft.

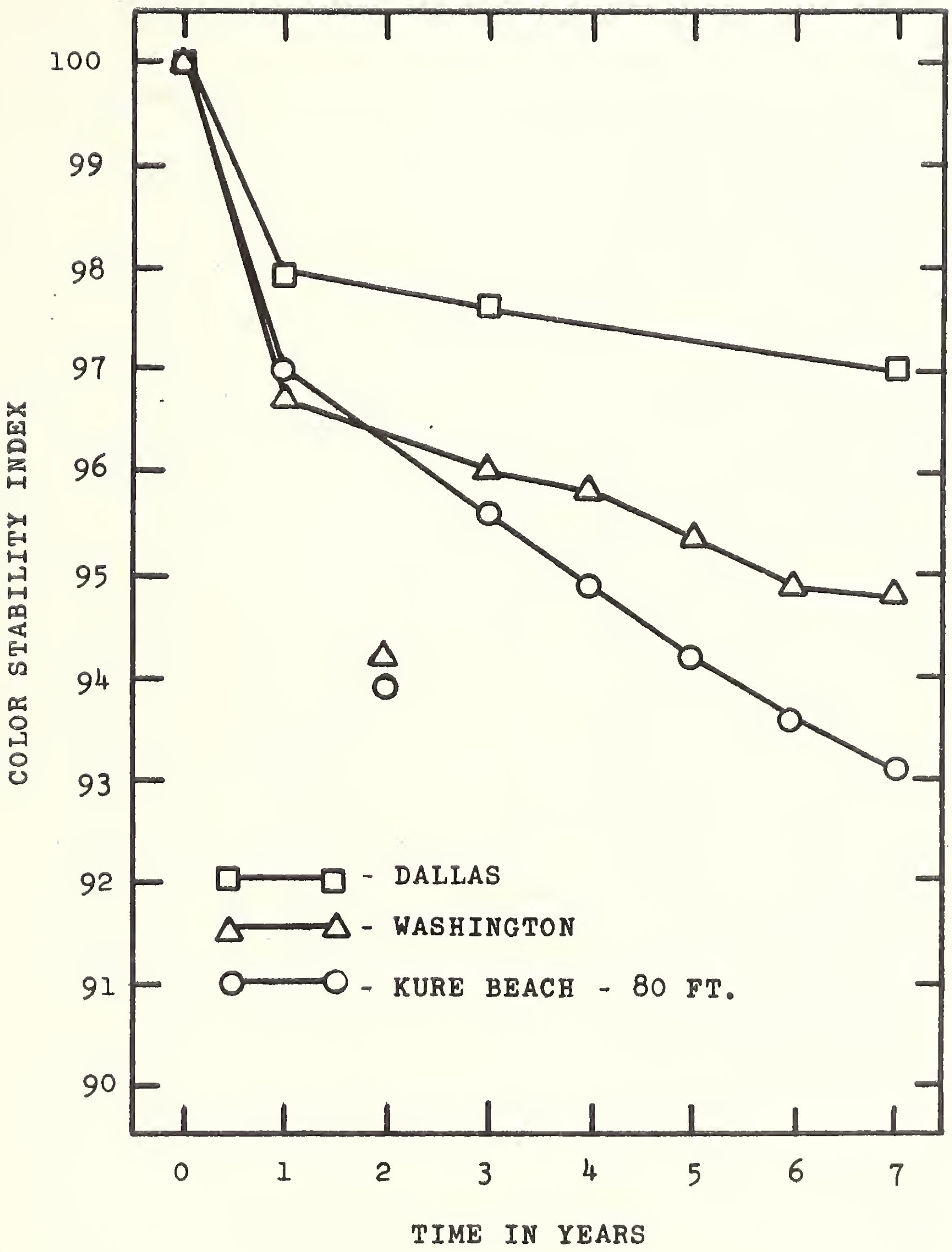


Figure 3. The effect of time on the color stability index of all green enamels exposed at Dallas, Washington, and Kure Beach-80 ft.

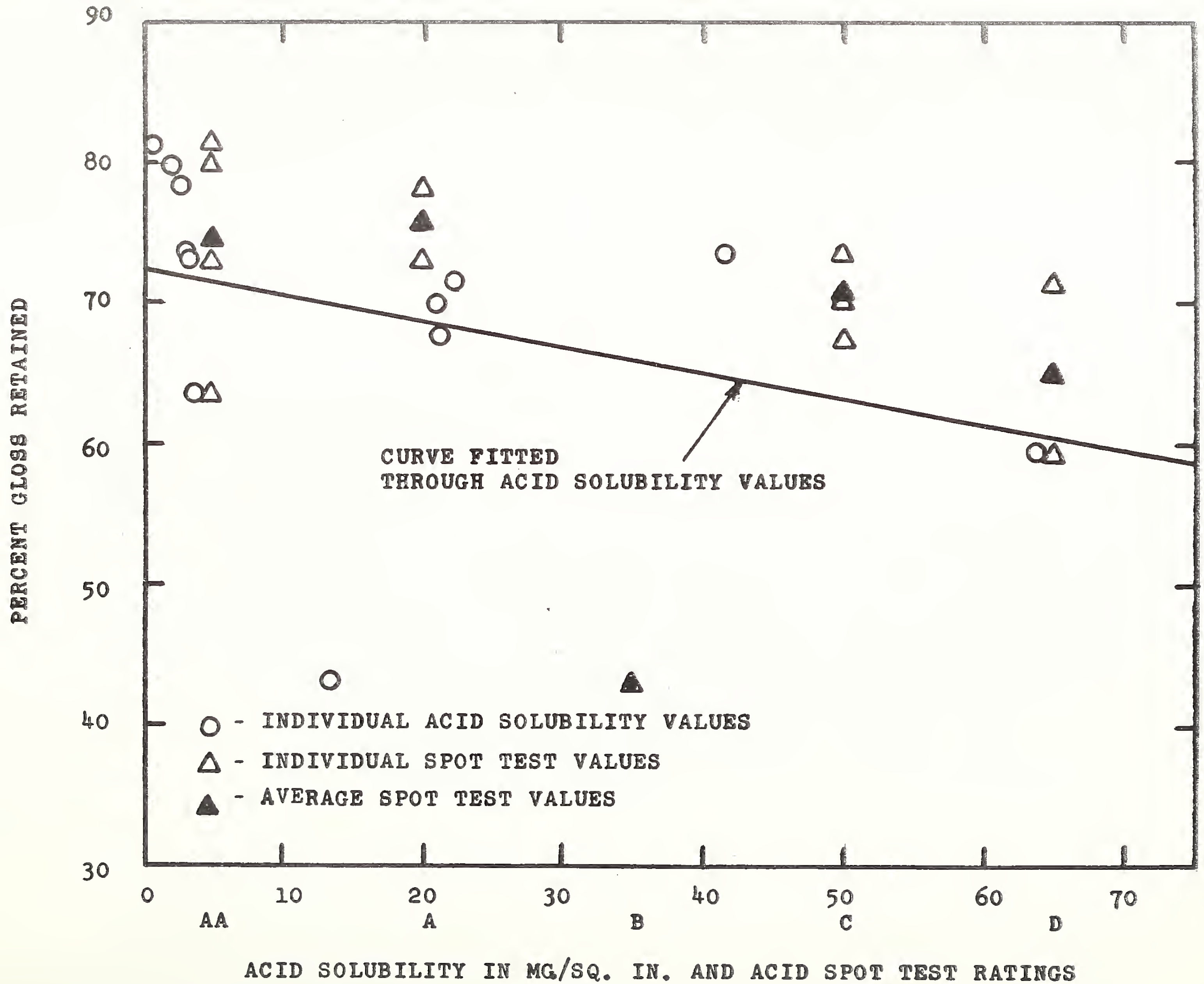


Figure 4. Comparison of boiling acid solubility and acid spot test rating with gloss retention of 12 enamels exposed 5 years at Washington.

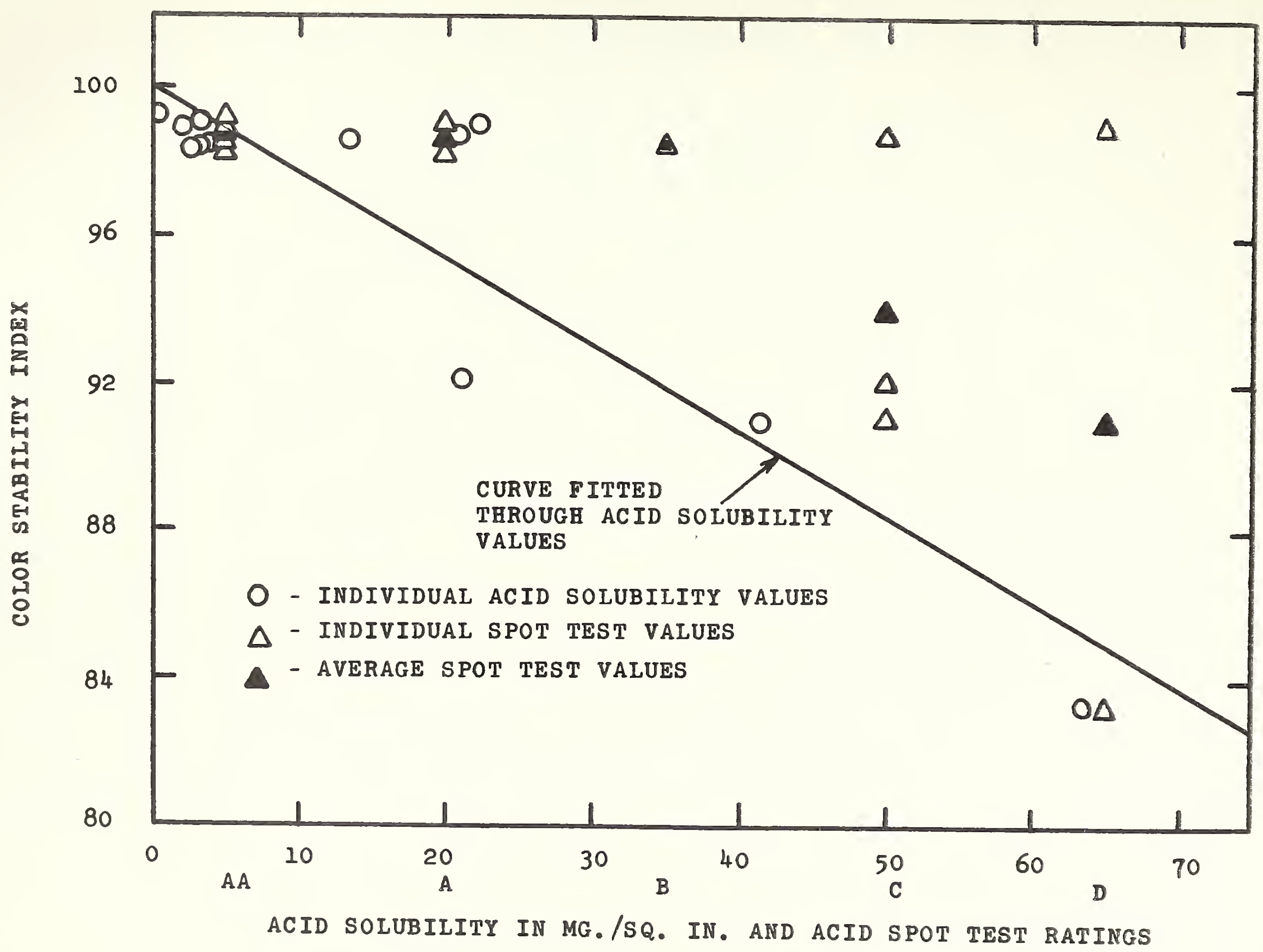


Figure 5. Comparison of boiling acid solubility and acid spot test rating with the color stability index of 12 enamels exposed 5 years at Washington.