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NATIONAL BUREAU OF STANDARDS REPORT

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Progress Report
April 1 Through June 30, 1968

**DEVELOPMENT OF METHODS OF TEST
FOR QUALITY CONTROL OF PORCELAIN ENAMELS**



U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS

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DEVELOPMENT OF METHODS OF TEST FOR QUALITY CONTROL OF PORCELAIN ENAMELS

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U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

SUMMARY

A number of variables in the cleanability test procedure were investigated and the optimum conditions were selected to give a reproducible test procedure as evidenced by triplicate determinations on 24 different surface finishes.

The variation in overvoltage curves determined in different laboratories in the round-robin test was re-examined and found to be a function of the individual test equipment, not the operator or the laboratory in which they were determined. Therefore, provision is being made to compare all overvoltages to a "standard" instrument when the test is written.

The scratch-abrasion test under development was modified to use an abrasive paper and water soluble soil instead of loose grit and the oil-base soil.

I. CLEANABILITY

INTRODUCTION

The appearance of articles of commerce is an important attribute and it plays a major role in the marketing process. The appearance of meats in the grocer's display case may be enhanced by special lighting. The motorist may "turn in" to an attractively-sheathed gasoline station or eating place. A home-seeking family may be influenced to purchase by the attractive appearance of appliances within a display home. Somewhat more inquiring and less impulsive individuals may show concern for the degree of maintenance required to restore the original appearance of cleanliness, once the "honeymoon of the market place" is over. In an attempt to assist these latter individuals, we have been developing a method to numericalize the measurement of cleanability; to determine an estimate of the effort required to restore the original attractive appearance of soiled finishes, whether they be organic or inorganic, on appliances within the home or on the exterior of buildings.

RESULTS AND DISCUSSION

The following discussion summarizes the results in over 760 determinations of the cleanability of various finishes obtained during the period covered by this report. Some of the experimental designs performed to test the effect of a particular variable in the cleanability operation are summarized. The salient steps in the selected procedure are enumerated. Detailed results, in triplicate,

are given for a large number of various finishes tested, together with a discussion of the reproducibility that was obtained.

A. The Drying of Specimens for Testing

To select a suitable drying procedure, an experiment was performed to compare oven-dried specimens with those dried at room temperature. Specimens for test were washed with a detergent solution, rinsed with flowing tap water, with distilled water and finally with alcohol. Triplicate determinations, on different days, were made on a series of specimens which had been washed and rinsed as above, oven dried at 100°C and cooled in a desiccator. Triplicate determinations on the same test pieces were made on different days following washing, rinsing and drying for 15 minutes at room temperature.

No significant difference in the cleanability indices was shown between these rather extreme methods of drying. These results led to the adoption of a one-hour drying time at room temperature.

B. The Effect of Mechanical Agitation During the

Soil Extraction Process

The testing procedure under development subjects clean and dry specimens to a mechanical soiling and cleaning treatment. Following these steps the soil retained is extracted with water from a known area and the fluorescence of the extract is measured. It was desired

to determine whether a more complete removal of soil would be effected if the extraction cell were mechanically agitated during the extraction step. An experiment similar to that described in paragraph A was used to determine the need for mechanical agitation during extraction. No significant improvement in the degree or uniformity of soil extraction was achieved by the use of mechanical agitation and for this reason its use was not adopted.

C. The Effect of Close Temperature and Humidity Control for the Conditioning of Clean, Dry Specimens before Cleanability Measurement

The laboratory space assigned to the research associates had air temperature controlled at $25 \pm 5^{\circ}\text{C}$ and air humidity at 45 ± 5 percent. Other laboratory space was available where the temperature was controlled at $25 \pm 1^{\circ}\text{C}$ and the relative humidity at 50 ± 2 percent. A comparison of results obtained in these two locations showed that no improvement in results was obtained when the equipment and test materials were located in the laboratory with more closely controlled air temperature and humidity.

D. Improved Control of the Amount of Water used in the Mechanical Cleaning of Soiled Specimens

The previous report in this series (No. 9826) indicated the need for accurate and precise measurement of the small amounts of water

used in the mechanical cleaning operation. The most reproducible results quoted in that report were obtained by using one drop of water from an 18 gage hypodermic needle on each cleaning tissue and a cleaning cycle of 22 seconds. It has been found that more uniform amounts of water can be expected when measured and dispensed through the use of a repeating pipette of one to twenty-five lambda (0.025 ml) capacity. Table 1 illustrates the uniformity of drop sizes from an 18 gage needle and from the pipette at full capacity (25 lambda). The amount of water used in later work was 21 lambda but there was no reason to suspect less precision when the slightly smaller amount of water was used. An experimental pattern, in triplicate, showed no significant difference between the cleanability indices obtained with the 18 gage needle and those in which the 21 lambda pipette was used. Test results on six finishes are given in Table 2. Method I involved the use of approximately 0.021 g of water from an 18 gage needle while in Method II 21 lambda of water was dispensed from the pipette. This table clearly shows that the variance, s^2 , was less when the cleaning water was dispensed from the pipette than in Method I. The analysis of variance showed that the reduction in variability was significant, at the 95 percent confidence level, only for the two finishes (17 and 32) that were more difficult to clean. The pipette used in Method II was selected to dispense the 0.021 ml of water

previously determined to be an appropriate amount.

E. The Procedure Used in the Testing

The procedure used in the testing described in the next paragraph (F) was as follows:

Standard Soil

The soiling agent had the following composition:

Polyethylene glycol	98.0 percent
Water soluble dye	1.0
Uranine	1.0

Washing of Specimens

The specimens were scrubbed with a soft sponge and a warm solution of a laboratory detergent.

Rinsing

The specimens were rinsed immediately with flowing tap water, distilled water, and then with absolute alcohol.

Drying

The specimens were dried in a near vertical position for at least one hour at room temperature.

Soiling

One drop of soil was dispensed from a hypodermic syringe without a needle. The drop size for this soil was 0.044 gram. The soil was mechanically distributed over an area of seven square inches at the center of the specimen with a teflon-faced brass head.

Cleaning

A brass head, 2-1/4 inches in diameter, was covered with four thicknesses of a laboratory tissue. Twenty-one milliliters of distilled water were dispensed to the center of the tissue covered head, which was then mechanically rubbed over the specimen to absorb and remove the soiling agent during a 22-second cycle. The operation was then repeated.

Extraction of Soiling Agent

An extraction cell with an O-ring seal was clamped against the soiled specimen surface. A measured amount of distilled water was dispensed into the extraction cell. After three minutes in contact with the specimen surface, the water was poured out and its fluorescence measured.

Calculation of Soil Retained

The amount of retained soil was calculated:

Soil Retained, $\mu\text{g}/\text{cm}^2 =$

$$\frac{\text{Weight of Water Solvent, g, x Calibration Factor, } \mu\text{g/g}}{\text{Extraction Area, cm}^2}$$

F. Cleanability Tests of Various Porcelain Enamels and Competitive Finishes

The results given in Tables 3 and 4 were obtained during the period May 27 to July 1, 1968. Each number given in the body of Table 3 is the mean of determinations on six specimens of each

finish on a particular day. In each case the determinations were repeated on the same test pieces for a second and third day to give an indication of the reproducibility of the results. Five of the finishes were tested on more than three days. It can be seen that generally those finishes which retained the least soil were the most repeatable. Those finishes which retained larger amounts of soil had a wider range of determined mean values. In order to compare the reproducibility of various finishes, a coefficient of variation (the standard deviation expressed as a percentage of the mean value) was calculated and given in Table 4.

Toward the end of this program, it was noted that the results obtained were more variable than those in the earlier determinations. This correlated well with the observed wear on the water dispensing devices. Three of the 25 lambda pipettes were purchased and have been used, two at a time, for about 1280 cleanability determinations. During each use, a stainless steel needle was moved twice through a teflon air-sealing orifice. As the teflon seal become worn, particularly if the needle was slightly bent, there was a marked tendency for air bubbles to enter during the filling operation and somewhat erratic drop sizes were obtained during the latter tests. It is believed that the use of worn pipette seals made a material contribution to the variable results obtained during the last week of the testing

program. Tests on the last 10 code numbers at the bottom of Table 4, all having coefficients of variation of over 12 percent, were completed during the last week of the work while the last four varieties with coefficients of 22 percent and over were completed during the last four days. It seems reasonable to assume that, with properly maintained pipettes, the method is capable of a reproducibility of less than 10 or 12 percent.

PLANS FOR THE NEXT REPORT PERIOD

From the work described in this report one might conclude that many finishes are easy to clean when they are new. Another important characteristic is how well is its virgin ease-of-cleaning retained after a period of use (or misuse). It is proposed to simulate the type of damage suffered during use, by a scratch-abrasion treatment. This should permit one to compare surface finishes which had nearly equal ease-of-cleaning in their virgin condition after various degrees of a simulated service use.

CONTINUITY OF COATING

INTRODUCTION

One of the prime reasons for using porcelain enamels is to protect the substrate from corrosion. In the past, glossy porcelain enamels have been applied in two or more coats. The coverage thus obtained was sufficient to protect the base metal from corrosion.

If discontinuities existed they could be readily detected by visual observation. However, the introduction of matte and one-coat enamels, introduced discontinuities that were extremely difficult to locate visually. This indicated a need for a continuity of coating test that was capable of locating minute discontinuities.

Early in the development of this test it was noted that many enameled specimens which rusted in service appeared to have continuous coatings when tested by methods which would locate only those defects open through the enamel to the base metal. This suggested that a continuity of coating test for porcelain enamels needed to be capable of locating areas of light coverage as well as areas with no coverage. Of all the test methods investigated, only the high-voltage probe appeared to have this capability.

RESULTS AND DISCUSSION

A. Test Procedure and Terms Used in High-Voltage Continuity of Coating Testing

The test requires high-voltage equipment - either ac or dc - whose output voltage is continuously variable from 0 to 4 kV. Before any testing can be done, it is necessary to prepare a calibration curve for the test equipment. This is done by taking a set of enamels of varying thicknesses (4 to 20 mils) turning the high-voltage equipment to its maximum output and puncturing as many

of the enamels as possible. The punctured enamels are then re-probed to determine the voltage necessary to arc across the air gap created by the previous puncturing operation. The length of the air gap is estimated by the thickness of the enamel. The voltage needed to arc across the gap is defined as the air gap voltage.

The thickness of these enamels is measured and a calibration curve is fitted for the air gap voltage as a function of enamel thickness.

When other enamels are tested, the thickness is measured first.

This thickness is referred to the calibration curve to determine the air gap voltage for that thickness. The air gap voltage is the minimum voltage to be used when testing an enamel. If a higher voltage is used to probe the enamel, then the difference between the higher voltage and the air gap voltage is the overvoltage.

B. Round-Robin Testing of High-Voltage Test Equipment

After the development work on the high-voltage continuity of coating test had progressed to a point where a tentative test procedure could be prepared, a round-robin test was conducted among three laboratories and the PEI Research Associateship at the National Bureau of Standards. The goals of this test were twofold: to determine the calibration curve for each instrument and to determine if the same enamel would be punctured at the same overvoltage regardless of the instrument used.

The calibration curves for the four laboratories participating in the round-robin test are given in figures one through four. The solid lines in figures two and four are the curves determined by the cooperating laboratory while the dashed lines were determined by the research associate using the laboratory's high-voltage equipment. These figures illustrate that while the calibration curve was not exactly reproduced, it had shifted only about 0.1 kV which is the smallest sub-division on the scale of the high-voltage instrument. This slight shift in the calibration curve is not considered to be significant at this time.

These same four laboratories were supplied with replicate specimens of 16 enamels to determine the overvoltage necessary to puncture them. Figure 5 illustrates the overvoltages obtained for each of the three ac instruments as a function of the overvoltage for the one dc instrument. It can be seen that the overvoltages for the dc instrument differed from those for the ac instruments. (If they were the same, the line would be at 45°). It can further be seen that the overvoltage obtained for the three ac instruments was not the same. This difference indicates the need to correlate each instrument used in the field to a "standard" instrument such as the one at NBS. This would be done by determining overvoltage curves such as those illustrated in figure 5 for each instrument used.

C. Comparison with Field Tests

When the term "high-voltage" is used in connection with a method for testing porcelain enamels, most enamelers are reluctant to try it because high-voltage testing is synonymous with destructive testing. However, data collected on storage specimens of the enamels exposed in the 1956 exposure test indicated that 50% of the enamels tested which had not rusted after seven years' exposure did not breakdown with overvoltages as high as 3.5 kV dc. This indicates that the "good" enamels - enamels that have withstood 7-years' exposure at Kure Beach 80 without rusting - can withstand high overvoltages without puncturing. Therefore, the test is non-destructive IF the quality of the finished product is good.

The effect of testing with lower overvoltages is demonstrated by the enamels in the 1966 Exposure Test of Nature-Tone Enamels on Steel. When this test was initiated it was decided to select one half of the exposed enamels on the condition that they pass the high-voltage probe. At this stage in the development of the continuity of coating test, the concept of overvoltage was not defined. Therefore, the specimens were probed with arbitrarily selected voltages. The table below indicates the number of enamels that rusted after one-year's exposure at Kure Beach and South Florida for various ranges of overvoltages.

<u>Range of Overvoltages (kV dc)</u>	<u>Number of Enamels Rusted</u>	
	<u>Passed High- Voltage Probe</u>	<u>Selected at Random</u>
0.1 - 0.5	6	8
0.6 - 1.0	1	3
1.1 - 1.5	2	15
1.6 - 2.0	0	2

The data in this table indicate that the high-voltage probe can be used to locate enamels with poor coverage IF the overvoltage is high enough. At the lowest overvoltages only the wide open holes are located and little, if any, improvement is noted between the probed and unprobed specimens. As the overvoltage is increased, the ratio of the untested enamels that rusted to the tested enamels that rusted increases. This again indicates that the high-voltage probe is capable of locating enamels with poor coverage and should serve as an invaluable tool in the development of porcelain enamels.

PLANS FOR NEXT REPORT PERIOD

The instruments used thus far in the continuity of coating work were produced by two manufacturers. The loan of an instrument from the third manufacturer of such equipment has been requested to determine if it behaves in a manner similar to those already tested. When this is determined a standard method of test for continuity of coating of porcelain enamels will be prepared.

SCRATCH-ABRASION

INTRODUCTION

The scratch resistance of porcelain enamels has long been of interest to the porcelain enamel industry. As early as 1942 a gouge test was developed to quantitatively define an enamel's resistance to subsurface crushing. This test was used with confidence until porcelain enamels began to be produced in thinner coatings. Then the gouge test no longer yielded meaningful results and work was begun on the development of a test where the surface of the enamel was scratched without crushing the subsurface enamel. Most of the work in the development of this test centered around the use of a diamond point to produce a single scratch. The force necessary for the diamond to just scratch the enamel's surface was used as a measure of scratch resistance. When this test was almost completed, it was found that the results obtained did not correlate with service results and further work on a scratch test was temporarily abandoned. However, a review of previous work by the research associates revealed two methods of scratching an enamel's surface had been developed that did correlate qualitatively with service but there was no way to quantitatively measure the extent of the abrasion damage. The first of these methods consisted of placing a one-inch square piece of 280 grit emory paper on a one-inch square brass head and pulling it across the specimen's surface

on the gouge testing apparatus, while the second method consisted of placing a slurry of 600 silicon carbide and water on the surface of a specimen which was taped to a standard polishing lap and scratching the surface with a reciprocating motion of an automatic polishing device. As previously mentioned, both of these methods produced scratched surfaces similar to those occurring in service, but there was no quantitative method available for determining the abrasion damage.

As the work on the development of a cleanability test progressed, it seemed possible that the amount of abrasion damage could be quantitatively determined by the change in soil retained between new and abraded surfaces. Exploratory work in this area using an oil-base soil indicated that the change in soil retained was a good quantitative measure of the scratch resistance of porcelain enamels. It ranked four enameled surfaces in the same order when they were abraded by three different methods and this ranking also agreed with the visual estimates of the abrasion damage by four different observers. However, this method of determining abrasion damage was limited to those surfaces not affected by toluene. The recent development of a cleanability test which utilizes a water soluble soil may open the way for a more universally usable method of determining abrasion damage. The work during this report period has

concentrated on determining if the water-soluble soil can be used in the same manner as the oil-base soil for determining abrasion damage.

RESULTS AND DISCUSSION

A. The Procedure Used for Scratch-Abrading Specimens

Unless otherwise stated, all specimens were abraded by the following procedure: the specimens were taped to a polishing lap that rotated at 163 rpm. A tenth of a milliliter of water was applied to the specimen's surface and was spread over the surface for 30 seconds with a teflon-faced, one-inch diameter brass head in an automatic polishing unit. The automatic polishing unit had a reciprocating motion of eleven cycles per minute. The teflon-faced head was then replaced with one that was laminated to a 1/4 inch thick piece of polyurethane foam, and then covered with a piece of 400 grit silicon carbide paper. The specimens were scratched for 2-1/2 minutes.

B. Comparison of Results with Water-Soluble and Oil-Base Soils

Specimens of three different surfaces were abraded and the change in cleanability was determined with both the water-soluble and the oil base soils. The results are given below:

<u>Surface</u>	<u>Change in Soil Retained</u> <u>Water Soil</u>	<u>Oil Soil</u>	<u>Ratio</u> <u>Water/Oil</u>
AZA	0.56	0.39	1.43
4	2.30	1.95	1.18
FGC	15.05	13.66	1.10

Thus it can be seen that while the absolute values obtained with the oil and water soils are not the same, the ratio remains almost constant. This indicates that the water soluble soil behaves much the same as the oil soluble soil and can be used as an indicator of the scratch-abrasion resistance of a surface.

C. Reproducibility of Water-Soluble Soil in

Measuring the Change in Soil Retained

In order to separate the variability in the cleanability determinations and the abrading process, duplicate cleanability determinations were made on the same abraded specimens and then new specimens were abraded and duplicate cleanability determinations were made again. These results are given in Table 5 where it can be seen that there is fairly good reproducibility in both the cleanability determinations and in the abrading process. These results further indicate that the mean of five or six specimens gives better duplication of the change in cleanability due to scratch abrasion than does the mean of only three specimens.

D. The Effect of Different Abrasive Papers

In selecting an abrasive for scratching the enamel's surface, it is desirable to choose one which causes the most damage in the least time without causing the enamel to fail by spalling. To date 240, 400 and 600 grit silicon carbide and 240 grit aluminum oxide papers have been tried - both wet and dry. In all cases the addition of water made the abrasion occur at an accelerated rate. This indicates that water should be specified in the abrasion process.

The 240 alumina and 240 silicon carbide papers both caused the enamels to spall along the scratch lines and were therefore eliminated from further consideration. Both the 400 and 600 silicon carbide papers produced similar scratch patterns, but the 400 did more damage and has been temporarily selected as the paper to be used in this test procedure.

PLANS FOR THE NEXT REPORT PERIOD

Future work on the scratch-abrasion test includes:

1) Investigating the effect of more abrasive papers, 2) investigating the effect of different abrasion times, and 3) comparing the results obtained with this abrasion method with the results obtained with other abrasion methods and with an individual's estimate of abrasion damage.

TABLE 1. SHOWING THE VARIABILITY IN THE AMOUNT OF
WATER DISPENSED FROM TWO DEVICES a/

	18 Gage Hypodermic Needle	25 Lambda Pipette
	g.	g.
	0.0191	0.9259
	.0194	.0261
	.0197	.0263
	.0199	.0263
	.0201	.0264
	.0206	.0265
	.0208	.0267
	.0210	.0268
	.0215	.0268
	<u>.0239</u>	<u>.0271</u>
mean	0.0206	0.0264
s	.00149	.00036
v	7%	1.3%
range	.0048	.0012

a/ Arranged in the order of increasing drop size, rather than
in the order dispensed and weighed.

TABLE 2. THE RELATIVE VARIANCE OF TWO CLEANING METHODS.a/

Code	Cleanability Index	Method	$s^2 \underline{b/}$	$F \underline{c/}$	Critical Value of F at the 95 Percent Confidence Level	Conclusion:
17	7.6	I II	1.433 0.332	4.35	2.47	Variance of Method II Significantly Less than of Method I.
32	4.3	I II	2.844 0.372	7.65	2.34	" " "
6	4.1	I II	0.389 0.700	0.56	2.69	Variance of Method II Not Significantly Less than of Method I.
5	1.3	I II	0.063 0.048	1.31	2.34	" " "
18	1.1	I II	0.091 0.042	2.16	2.28	" " "
16	0.9	I II	0.071 0.052	1.37	2.79	" " "

a/ Method I involved dispensing 0.021±0.001 g water from an 18 gage hypodermic needle.
Method II involved dispensing 0.021±0.0004 g water from a repeating pipette.

b/ Method I $\frac{SS_{\text{days}} + SS_{\text{error}}}{\text{total D.F.}} = s^2_I$ Values for the sum of squares (SS) for days, the sum of squares for error and the number of Degrees of Freedom (D.F.) were obtained from analyses of variance.

Method II $\frac{SS_{\text{days}} + SS_{\text{error}}}{\text{total D.F.}} = s^2_{II}$

$$\underline{c/} \ F = \frac{s^2_I}{s^2_{II}}$$

TABLE 3. CLEANABILITY INDICES OF VARIOUS SURFACE FINISHES. a/

Code <u>b/</u>	Mean of Six Specimens Determined on Different Days						Grand Mean
	1st	2nd	3rd	4th	5th	6th	
3	0.2	0.2	0.2	--	--	--	0.2
16	0.7	1.0	0.9	--	--	--	0.9
2	0.8	1.0	1.1	--	--	--	1.0
7	1.2	0.9	1.0	--	--	--	1.0
18	1.0	1.0	1.2	1.4	1.0	1.3	1.1
25	1.2	1.3	1.2	--	--	--	1.2
31	1.5	1.6	1.8	--	--	--	1.6
24	1.3	1.7	1.9	--	--	--	1.6
30	1.7	1.3	2.1	--	--	--	1.7
28	1.9	1.9	1.4	--	--	--	1.7
27	1.7	1.6	1.7	--	--	--	1.7
5	1.2	1.1	1.4	1.5	--	--	1.7
33	1.7	1.6	1.7	--	--	--	1.7
29	1.6	1.8	1.9	--	--	--	1.8
4	1.7	1.7	2.1	1.7	--	--	1.8
26	1.9	1.8	1.9	--	--	--	1.9
22	2.1	2.0	2.3	--	--	--	2.1
23	2.0	1.9	2.3	--	--	--	2.1
1	3.7	2.4	2.8	--	--	--	3.0
21	3.8	3.4	4.3	--	--	--	3.8
6	4.3	3.2	4.7	--	--	--	4.1
32	4.2	3.9	5.0	4.2	--	--	4.3
17	7.1	7.9	7.7	7.5	--	--	7.6
20	11.8	13.9	12.9	--	--	--	12.9

a/ All cleanability determinations were made on specimens in their virgin or unused condition.

b/ Code numbers 20 through 33 were porcelain enamels in various colors, acid resistance and gloss ranges.

Code numbers 18 and below were competitive organic and metallic finishes, except code 16 which was a porcelain enamel on aluminum.

TABLE 4. THE VARIABILITY OF REPEATED DETERMINATIONS
OF THE CLEANABILITY OF VARIOUS FINISHES

Code	Cleanability Index $\mu\text{g}/\text{cm}^2$	Range of Determined Values $\mu\text{g}/\text{cm}^2$	$\frac{\text{Range}^{\text{a/}}}{\sqrt{n}}$	Coefficient of Variation Percent
33	1.7	0.1	0.06	3
25	1.3	0.1	0.05	4
26	1.8	0.15	0.09	5
17	7.6	0.8	0.48	6
3	0.2	0.02	0.01	6
27	1.7	0.2	0.13	8
22	2.1	0.3	0.16	8
29	1.8	0.3	0.17	9
20	12.9	2.1	1.21	9
31	1.6	0.3	0.16	10
23	2.1	0.4	0.23	11
21	3.8	0.8	0.48	12
18	1.1	0.32	0.13	12
7	1.0	0.22	0.13	12
32	4.3	1.1	0.54	13
4	1.8	0.5	0.24	13
5	1.3	0.4	0.21	16
28	1.7	0.5	0.29	17
16	0.9	0.3	0.16	18
2	1.0	0.3	0.18	19
24	1.6	0.6	0.36	22 $\frac{\text{b/}}{\text{b/}}$
6	4.1	1.5	0.89	22 $\frac{\text{b/}}{\text{b/}}$
30	1.7	0.75	0.43	26 $\frac{\text{b/}}{\text{b/}}$
1	3.0	1.4	0.79	26 $\frac{\text{b/}}{\text{b/}}$

a/ The range divided by \sqrt{n} is a good estimate of the population standard deviation for samples of $n = 3$ to 10. See Experimental Statistics, NBS Handbook 91, pages 2-6. The estimated standard deviation times 100 divided by the mean (cleanability index) yields a coefficient of variation shown in the last column.

b/ Tests completed on the last four days of testing.

TABLE 5. REPRODUCIBILITY OF THE SCRATCH-ABRASION PROCESS

Enamel	Change in Soil Retained		Number of Specimens
	1st Determination	2nd Determination	
	$\mu\text{g}/\text{cm}^2$	$\mu\text{g}/\text{cm}^2$	
4	2.1	2.5	6
4	2.8	2.6	5
4	1.9	3.2	3
4	2.9	3.0	3
AZA	0.6	0.6	5
AZA	0.7	0.2	3

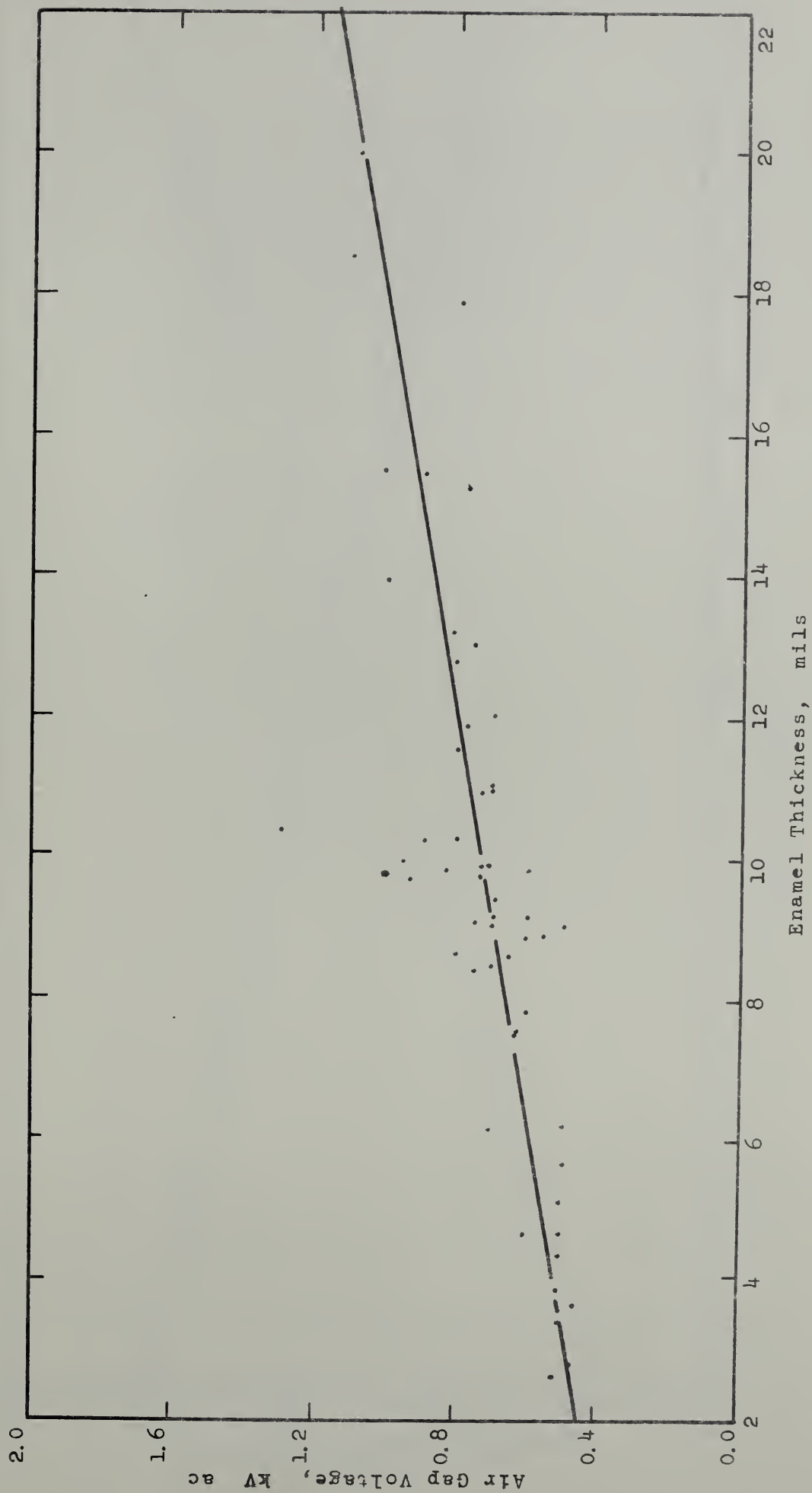


Figure 3. Calibration Curve for Laboratory C, Hypot ac Test Equipment.

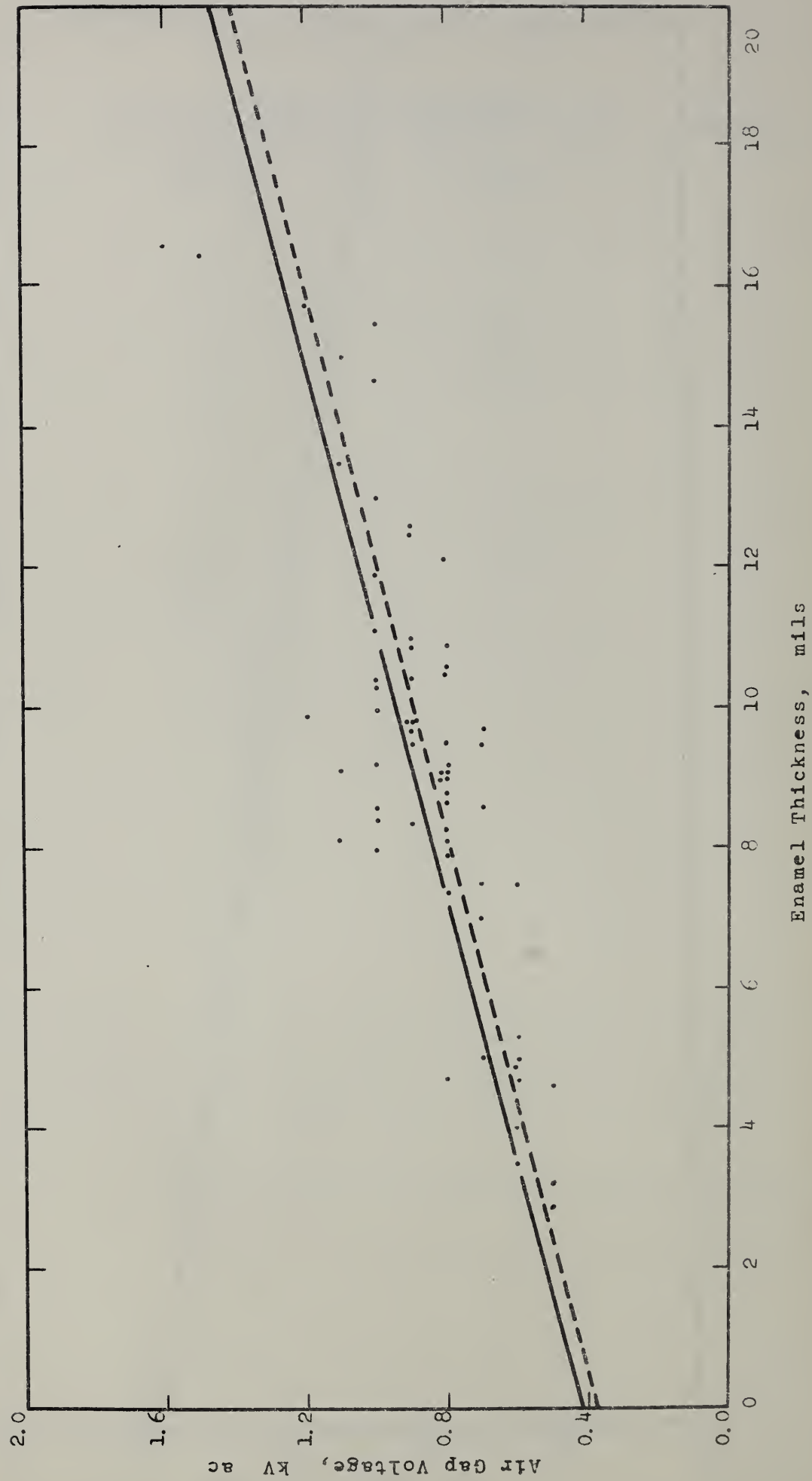


Figure 4. Calibration Curve for Laboratory D, Hypot ac High-Voltage Test Equipment. The solid curve was determined at Laboratory D, while the dashed line was determined at Laboratory A using Laboratory D's Test Equipment.

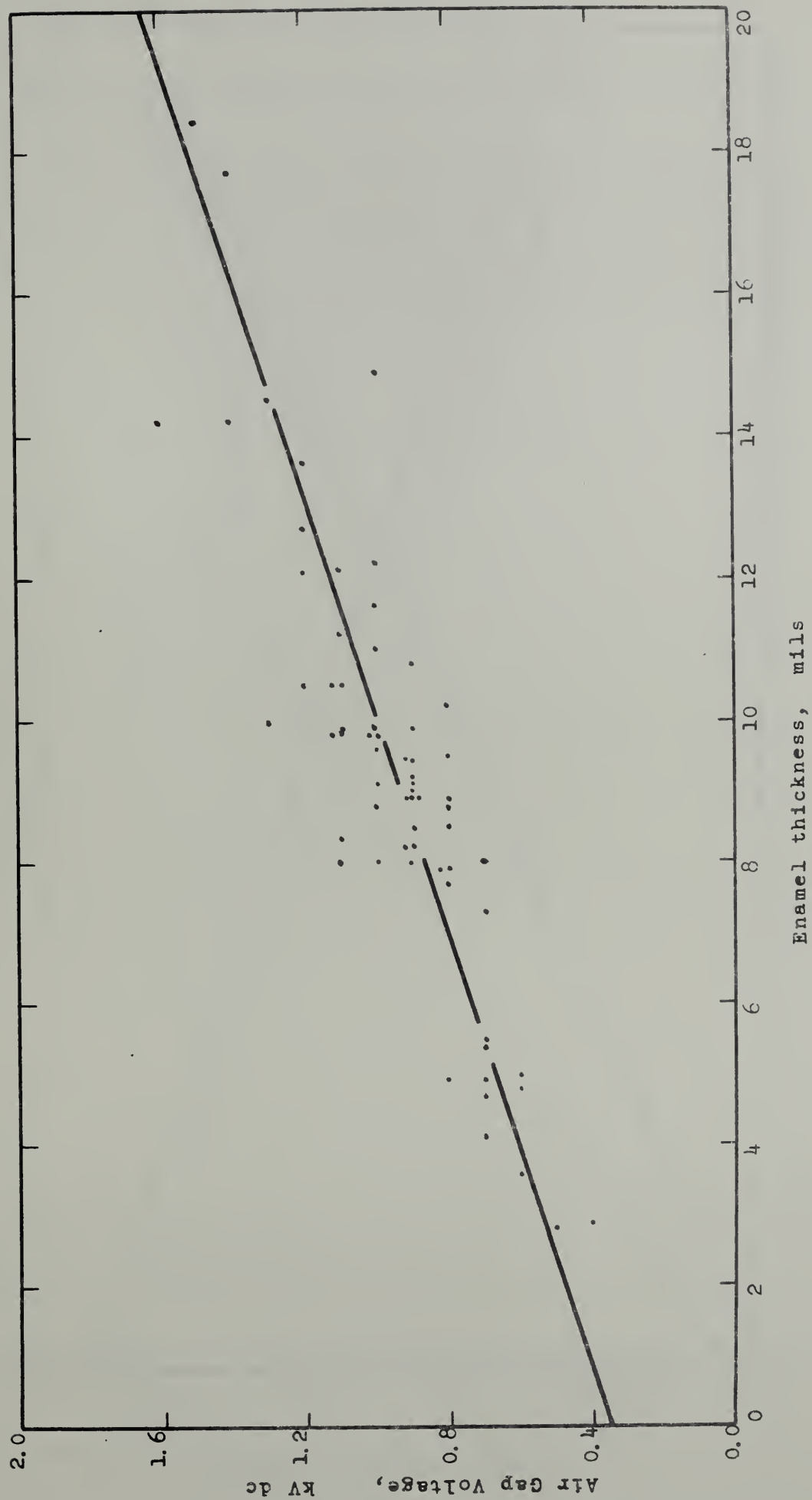


Figure 1. Calibration Curve for Laboratory A, Uvral dc High-Voltage Test Equipment.

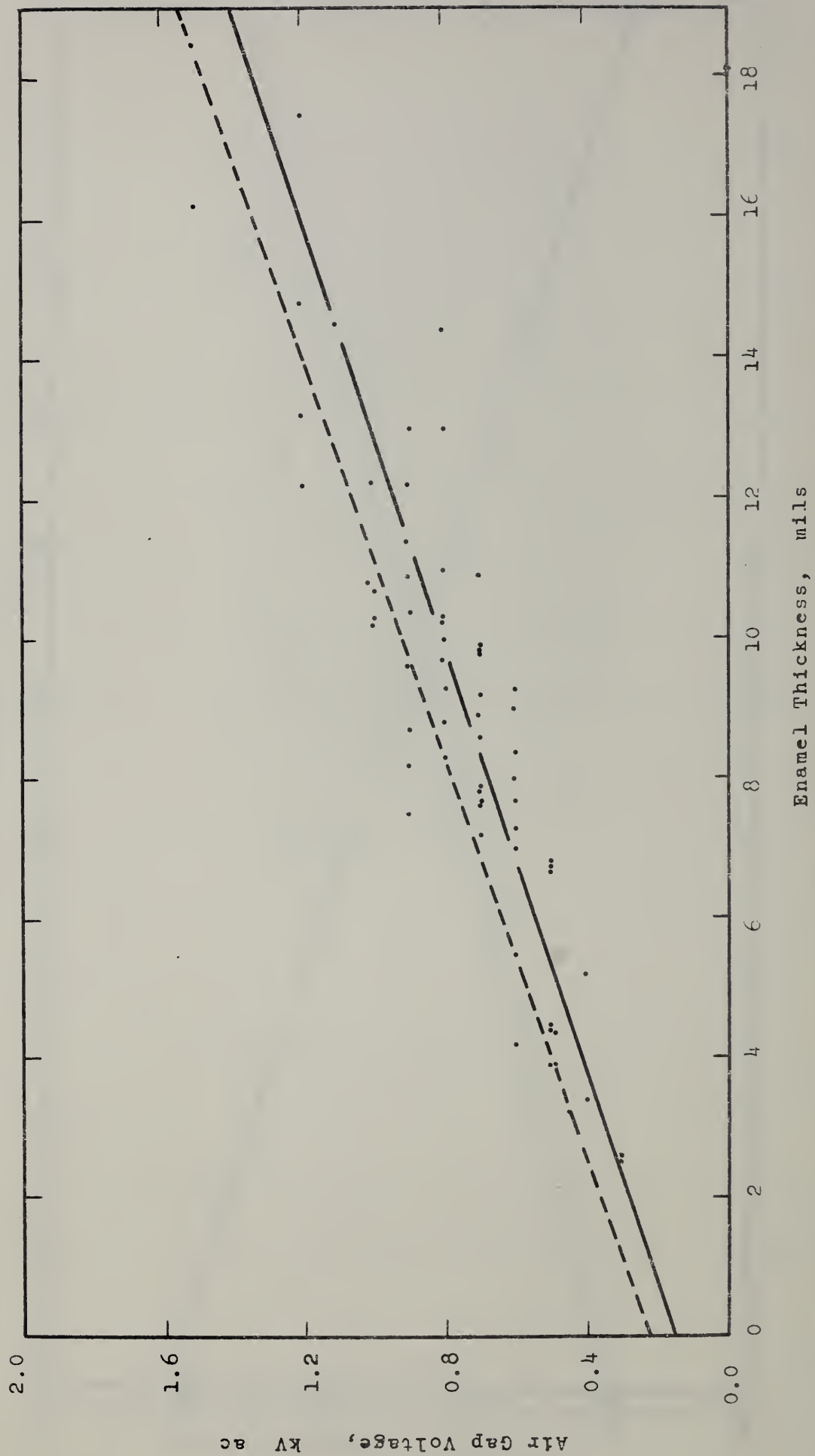


Figure 2. Calibration Curve for Laboratory B, Hypot ac Test Equipment. The solid curve was determined at Laboratory B, while the dashed line was determined at Laboratory A using Laboratory B's Test Equipment.

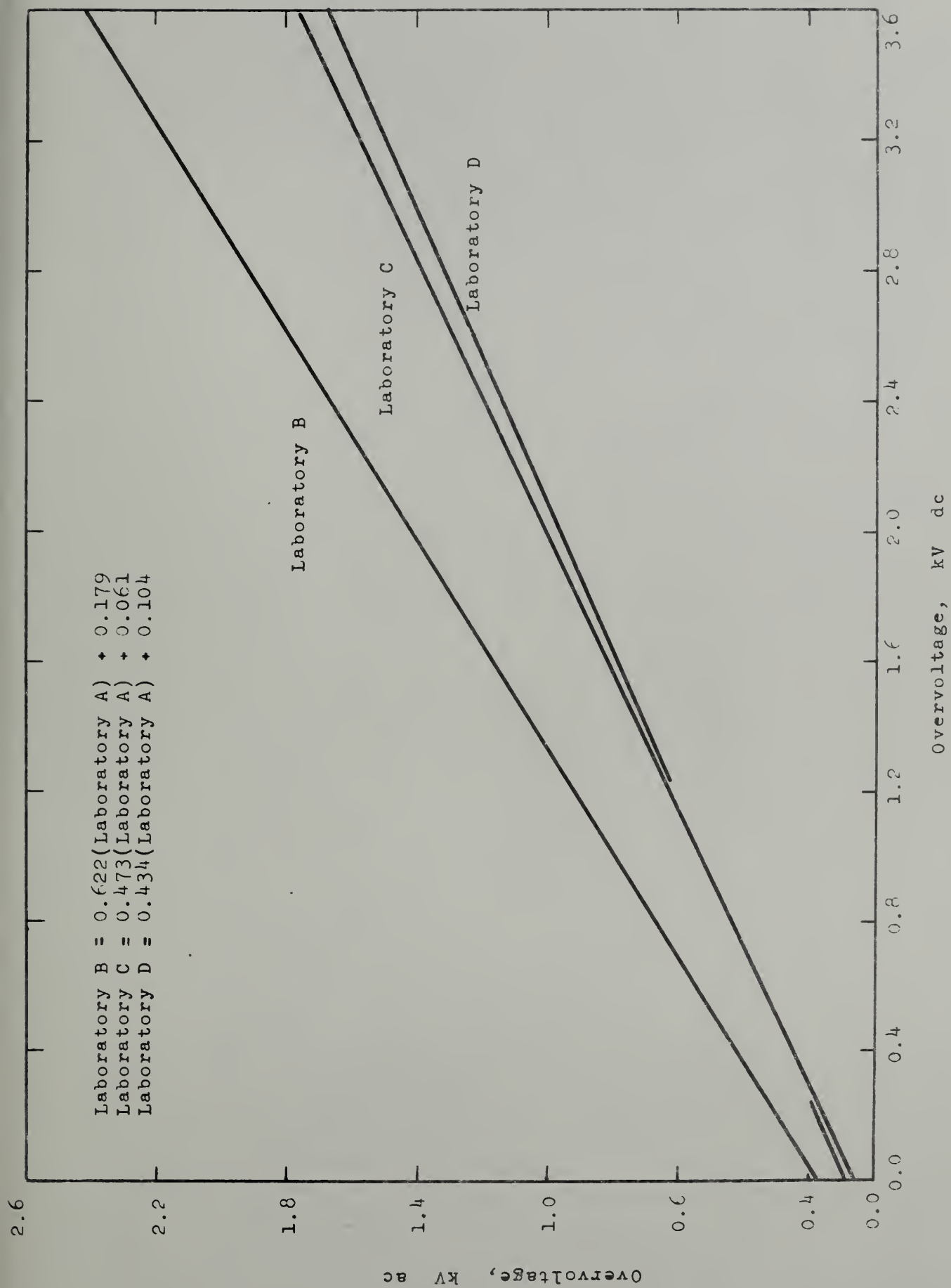


Figure 5. The Relationship between the overvoltages obtained at Laboratories B, C, and D and that obtained on the "Standard" instrument at Laboratory A.

