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NATIONAL BUREAU OF STANDARDS REPORT

9583

PROGRESS REPORT



April 1 through June 30, 1967

Development of Methods of Test For Quality Control of Porcelain Enamels

by

M. D. Burdick and M. A. Rushmer

7/20/07

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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NATIONAL BUREAU OF STANDARDS REPORT

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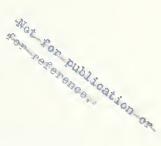
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U.S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

SUMMARY

Work has continued toward the modification of the oil-based soil currently used in the proposed test for cleanability of porcelain enameled surfaces. Investigations are currently under way to determine whether the oil-based soil can be replaced with a water-soluble soil to make the test applicable to more surfaces. Many of the short-comings of the water-soluble soil are discussed.

The six-months inspection of the 1966 Exposure Test of Nature-Tone Enamels on Steel was completed for the storage enamels and those exposed at Kure Beach and South Florida. This inspection confirmed previous findings that the nature-tone enamels have excellent color stability but are often plagued with inadequate coverage.

Further work on the Scratch-abrasion test indicated that the method of measuring change in soil retention of a specimen, before and after abrasion seems to correlate with visual concepts of abrasion damage.

I. CLEANABILITY

INTRODUCTION

Previous work reported in this series of Progress Reports led to a proposed method for evaluating the cleanability of various porcelain enameled surfaces. The current goal is to modify the proposed test in order to make the test applicable not only to porcelain enamel but to a variety of materials with different surface textures.

The oil-based soil employed in the proposed method can be mechanically cleaned in a controlled manner by the process of "film thinning". The fluorescent tracer technique is adequate to sensitively determine thin film residues well below the level of visibility. Consequently, the proposed test method appears to have great potential for determining the cleanability of surfaces, but its applicability is limited to inert surfaces because the method depends on the necessity for removal of oil films with an aggressive organic solvent which is not compatible with many organic surfaces.

The work described in this report was aimed at the development of a water-soluble soiling agent with a controlled removal or cleaning rate from surfaces of widely varying compositions and textures.

RESULTS AND DISCUSSION

The glycerol-based water-soluble soiling agent, described in the previous progress report, was used with a series of polished olate glass specimens to determine the degree of reproducibility one might expect when this type of surface was mechanically soiled and cleaned. The results obtained in repeated tests of the same specimens, spaced over a period of seven weeks are given in Table I. In all of these trials the specimens were carefully cleaned and conditioned, a uniform amount of soiling agent was applied and mechanical cleaning treatment was continued for six minutes before extraction of the remaining soil. It can be seen that the high and low values obtained on the same specimen, at different times, differ by a factor of as large as 3.5. This degree of reproducibility leaves much to be desired. The results of one analysis of these divergent values is shown in the lower part of Table I, where the signs of the differences between succeeding determinations are shown. On the second day (determination B) five out of six of the values were lower than in determination A, so A minus B is indicated as positive in those cases. A similar pattern, e.g., a nearly unanimous change in one direction, was found on all subsequent days.

One would not be surprised if some specimens resulted in higher and some in lower values (random error) than in the preceeding determination, but when five out of six were consistently lower (or higher) than the previous value, one suspects some unknown systematic variable was operative. Attempts were made to isolate and avoid these variables.

A. Possible Causes of Non-reproducible Results.

Two situations which might contribute to the difficulties mentioned above are: (1) the settling-out of colloidal graphite from the soil before use and failure to homogenize the soiling agent before use; and (2) the insufficient cleaning of fluorescent solutions from the specimens or from the utensils used in the extractions before reuse.

- 3 -

1. A calibration curve for the soil shows the fluorescence of homogeneous suspensions of known concentrations in water. Suspensions from which a part of the inert colloidal graphite had settled-out would be expected to show more fluorescence than calculated from the calibration curve. Fluorescence analyses of the settled soiling agent after various shaking times might show positive departures from the calibration value and would allow an estimation of the extent of mixing required to reestablish a homogeneous soiling agent. Soiling agent VIII contained one percent of uranine, as shown in Table 2. The intensity of the fluorescence of uranine is such that one drop of the soil must be diluted with about 500 ml of water in order to obtain a measure of its fluorescence with the instrument used. Soil VIII was allowed to stand, undisturbed, overnight. Triplicate samples were taken after 0, 0.5, 2 and 6 minutes of mechanical shaking. The weighed samples were diluted to 500 ml and the fluorescence measured. The departures of the analysed samples from the calibration value at that dilution (measured minus calculated) are shown below:

Summary of Analyses of Diluted Soil VIII After the Indicated Mixing Times.

Mixing Time	Measured M	inus Calculated	Fluorescence
	Individual	Average	Standard Deviation
minutes	(fluo	rescence units)	
0	+2.3; +2.3; +1.0	+1.9	0.8
0.5	+1.6; -1.4; +0.8	+0.3	1.6
2.0	-0.9; -4.4; -0.7	-2.0	2.1
6.0	-5.1; -0.5; -1.9	-2.5	2.4

Figure 1 shows the average values together with the magnitude of the standard deviations indicated above and below the average. Two conclusions may be drawn from these analyses: (1) the fluorescence of the undisturbed soil was indeed higher than that calculated

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from the calibrated values and dropped rapidly with shaking time; (2) six minutes of mechanical shaking does not appear to be sufficient to reestablish a homogeneous soil. These indications of the hazards of the settling-out of colloidal graphite led to the preparation of a black soiling agent using a water soluble colorant. Experiments will be made to determine the suitability of this soiling agent.

2. The Cleaning of Specimens and Utensils

In a series of tests such as shown in Table 1, it is reasonable to suspect that some of the variability of the results may be attributed to the use of initially "dirty" specimens or to non-uniformly "clean" utensils used in the extraction of soil from the specimen surface.

The procedure used in the extraction of small amounts of soil retained on mechanically cleaned specimens was as follows: 10 ml of distilled water was dispensed from an automatic pipette into the brass bottle, Figure 2, which had been clamped to the specimen surface. A rubber 0-ring, fitted into a groove in the bottom edge of the bottle, defined the area from which soil was extracted and provided a leak-proof seal against the specimen. The water remained in contact with the specimen surface for three minutes after which the water was poured through the top opening into a 100 ml transfer beaker. The fluorescence of the water extract was measured in a 5 ml cuvette in the fluorometer.

Blank determinations, made on specimens, omitting the soiling and cleaning operations, were quite variable and often showed between one quarter and one-half of the total fluorescence measured from specimens soiled and cleaned for six minutes. These high blank values represented the combined contributions from the pipette, the brass bottle and 0-ring, the specimen surface, the transfer beaker, and the cuvette. The pipette, beaker and cuvette were easily tested separately, and found to give blank readings.

In cleaning the brass bottles between determinations they were given a final alcohol rinse to avoid oven-drying and cooling. It was found that a reaction between the rubber 0-rings and alcohol resulted in a highly fluorescent, difficult to remove reaction film on the 0-ring. The alcohol rinse was discontinued. It was also found that distilled water

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at room temperature extracted fluorescent fillers or plasticizers from ordinary rubber O-rings. Soaking rubber O-rings in acetone for 48 hours removed most of the objectionable material but the rings were reduced in size by about 10 percent and became hard and brittle. A search was made for O-rings fabricated from other polymers that might have low fluorescent water extracts. Table 3 gives results on the polymers tested. Teflon O-rings were more readily available and somewhat cheaper than fluorosilicone but were found to be so inflexible that they could not easily be inserted in the O-ring grooves whose size was designed for flexible rubber. The teflon rings were also difficult to seal against matte surfaces. Fluorosilicone O-rings have been ordered.

The brass bottles used were bored out of a solid brass cylinder on a laboratory lathe. The interior surfaces, particularly the sloping portion, were poorly machined and quite rough and therefore difficult to clean. Distilled water held in contact with these interior surfaces for three minutes picked up a surprizing amount of fluorescence. These surfaces were polished with a series of abrasive papers down to No. 600 and were improved in appearance but not sufficiently improved in cleanability. A final coating of about one mil of self-leveling, bright nickel was electro-deposited on all surfaces of the bottles and a marked improvement in cleanability (and appearance) resulted.

B. Fluorometer Calibration

The fluorometer was originally calibrated with Soil VIII in February 1967. This involved determining the instrument response to suspensions prepared to contain between 0.5 and 100 micrograms of the soil per gram of suspension. Recently the same suspensions were remeasured, after storage in tightly stoppered clear glass bottles. It was found that the fluorescent intensity had decreased by 10 to 50 percent of the original values. Freshly prepared suspensions from the original supply of soil VIII, however, duplicated the original calibration. One may conclude that the undiluted soil is stable with respect to

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photo-decomposition and that the diluted calibration suspensions are not. These conclusions are in agreement with similar findings by Udenfriend in connection with the use of dilute solutions as reference standards. The analytical method used in fluorometer calibration would be expected to be more reliable and accurate than the short-cut method described earlier in Section A.1. for soil analyses. The calibration method used a 0.5 to 1.0 gram sample of soil which was weighed on an analytical type balance in a 100 ml volumetric flask as a weighing bottle. The short-cut method started with one drop of soil, about 45 milligrams, and weighings were made in a 500 ml flask on a less sensitive balance of large capacity, in order to obtain an analysis after a single dilution.

PLANS FOR THE NEXT REPORT PERIOD

A bank of various metal finishes and coatings for metals has been assembled. Many of these finishes are used in appliance manufacture as substitutes for porcelain enamel. It is proposed to make cleanability tests on these competitive surface finishes with the best water-soluble soil currently available for a report at the PEI Forum. It is hoped that this effort will serve to evaluate both the method of test and the various finishes tested. II. 1966 Exposure Test of Nature-Tone Enamels on Steel

INTRODUCTION

Early in 1966, an exposure test of Nature-tone enamels on steel was initiated by the Porcelain Enamel Institute's Research Associateship at the National Bureau of Standards. The goals of this test were 1) to determine the weather resistance of these new matte enamels and 2) to determine whether the high-voltage method of test for continuity of coating was capable of separating enamels with good coverage from those with poor coverage. Six specimens of each of twenty-five nature-tone enamel colors have been exposed for six months at Kure Beach, North Carolina - 80 feet from the ocean, and at South Florida Test Service, Miami, Florida - where the specimens were exposed 175 feet from the ocean. The remainder of this section of the report will discuss the results of the six months' inspection of these enamels.

INSPECTION PROCEDURE

A. Cleaning of Specimens

The procedure used to clean the specimens in this test has been to 1) scour 30 strokes with a sponge that has 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 alcohol.

B. Visual Inspection

After the above cleaning process, the specimens were examined visually for discontinuities as evidenced by either irridescent or rust colored spots.

C. 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 was measured with a color difference meter. One of the three storage specimens of each enamel was used as the color standard to obtain the maximum efficiency possible with this type of instrument. The color change is reported as color retention which is 100 minus the color change in NBS units.

RESULTS AND DISCUSSION

A. Color and Gloss Retention

The average color and gloss retention data for the enamels exposed for six months at Kure Beach and South Florida and also the six months data for the storage enamels are $\frac{2}{}$ presented in Table 4. A two-sided sign test showed no significant differences in color retention between the enamels exposed at Kure Beach and South Florida and, in fact, no significant difference between either Kure Beach or South Florida and the storage enamels.

When the gloss data were analyzed in the same manner, significant differences between the enamels exposed at Kure Beach-South Florida, Kure Beach-Storage, and South Florida-Storage were indicated. This suggests that gloss and color do not deteriorate in the same manner. This may be caused by relatively small changes in gloss resulting in large changes in percentage gloss retained since the initial gloss values are so low. It is currently believed that consumers are more aware of color changes than gloss changes, particularly when the initial gloss is low. Therefore, the major criterion for an enamels' weatherability will be the color change.

B. Discontinuities in Nature-Tone Enamels

The visual inspection of the exposed specimens revealed rust occurring around pinholetype defects on 19 and 7 specimens exposed at Kure Beach and South Florida, respectively. Although this figure is high it represents only 12.5% of the enamels exposed at Kure Beach while 25% of the enamels in a preliminary exposure test rusted in the same exposure period at Kure Beach. This indicates that there has been an improvement in the quality of these enamels since they were first introduced.

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C. The Use of High-Voltage Testing for Continuity of Coating.

When this test was initiated, one half of the exposed enamels (three specimens of each enamel) was selected at random from those submitted while the other half was required to be free from discontinuities when probed with a high-voltage dc probe. A voltage of 2kV was arbitrarily selected as the test voltage to be used. However, variations in thickness and continuity altered that aim. The lowest voltage that all specimens of each enamel passed is listed in Table 5. This table also includes information about the number of enamels that show rust after six months' exposure and the amount of overvoltage to which each enamel was exposed. While the continuity of coating test does an excellent job of sorting "good" and "bad" enamels exposed at the South Florida site, it failed to eliminate seven specimens that rusted during exposure at Kure Beach. Of these specimens only two (111 and 102) are cause for concern since the test voltage for the other five enamels was lowered significantly to pass enough enamels for exposure. If the enamels tested with less than 0.6 kV over-voltage are eliminated from consideration, then the continuity of coating test reduced the failures due to rust by 50% at Kure Beach and 100% at South Florida. While not perfect, this method of test still shows promise.

PLANS FOR NEXT REPORT PERIOD

During the next report period, it is planned to return the specimens to the exposure racks at Kure Beach and South Florida and to install the new exposure racks and Nature-Tone specimens at the Gaithersburg, Maryland Site.

The next inspection of these enamels is scheduled after one-year's exposure.

III. SCRATCH ABRASION

INTRODUCTION

Porcelain enamels as well as other finishes are subjected to many types of abrasion during use. A literature survey will reveal a variety of abrasion tests, each designed for a specific use or product. The porcelain enamel industry currently has such a test. However, the results obtained with this test do not always correlate with industry service records. One of the current goals of the Research Associateship is to develop a "scratchabrasion" test method which will correlate better with the results obtained in service. RESULTS AND DISCUSSION

During the past report period, a method of abrasion was investigated in which a slurry of paraffin oil and abrasive was rubbed over the surface of a rotating porcelain enameled specimen for 4 1/2 minutes using a reciprocating one-inch diameter plastic-covered brass head. Hereafter this method of abrasion will be referred to as mechanical abrasion. The degree of abrasion damage was measured by both 1) the change in soil retention and 2) the change in haze.

Work has continued using both the mechanical and the PEI abrader. A summary of the results using the PEI abrader (Std. sand - 5 min abrasion time) and the mechanical abrader using both 400 SiC and PEI Std. sand as the abrasive is presented in Table 6. The amount of abrasion damage was also estimated visually by four observers and instrumentally by measuring the change in haze, the change in soil retention by the fluorometric cleanability procedure and the percentage gloss retained. Observation of this data will indicate that the change in soil retained ranked the four enamels in the same order regardless of the method used to abrade the enamels. The change in soil retention also agreed with the visual ranking when the enamels were mechanically abraded using PEI STANDARD sand. This indicated that the method of change in soil retention of abraded surfaces agrees well with the amount of abrasion damage people see. One question that has been raised is "why do the visual estimates of abrasion damage agree for only one type of abrasion?" This is currently thought to be caused by a lack of contrast in the areas abraded with the PEI abrader and the Mechanical abrader when 400 SiC is used as the abrasive. When Std. sand is used with the Mechanical abrader, a few long, deep scratches contrast with many shorter, shallower scratches to make visual estimates of abrasion damage easier.

Since these results showed some promise, six additional enamels were abraded using both the PEI abrader and the mechanical abrader with 400 SiC. The results of these tests are presented in Table 7. Even though there are some reversals in the rank of these specimens, good correlation (rank correlation coefficient of 0.93) does exist.

These limited comparative trials of the different abrasion methods indicate 1) the soil retention method is probably the best indicator of abrasion damage, and 2) the actual method of abrasion damage appears to be independent of the method used to determine the damage (change in soil retention) as long as all surfaces to be compared are abraded in the same manner.

PLANS FOR NEXT REPORT PERIOD

It is currently planned to 1) determine the reproducibility of the abrading process and 2) to abrade some specimens for different lengths of time on the PEI abrader to see if an optimum abrasion time can be established.

IV. CONTINUITY OF COATING

The results of one laboratory participating in the round-robin continuity of coating tests have been received. However, these will not be reported until the results from all three participating laboratories have been received.

REFERENCES

- Sidney Udenfriend; Fluorescence Assay in Biology and Medicine, Academic Press,
 N. Y. (1962) p. 104.
- M. G. Natrella; Experimental Statistics NBS Handbook 91, U. S. Government Printing Office, Washington, D. C. p. 16-9.

TABLE 1.DETERMINATIONS OF RETAINED SOIL (FORMULATION VIII)
REPEATED ON THE SAME POLISHED PLATE GLASS
SPECIMENS AT VARIOUS TIMES.

Determination	A	В	С	D	Е	F
Specimen No.			μg/c	m ²		
1	0.78	0.46	0.74	0.27	0.37	0.31
2	0.49	0.52	0.62	0.54	0.25	0.44
3	0.61	0.55	0.58	0.44	0.33	0.45
4	0.72	0.71	0.59	0.48	0.40	0.52
5	0.76	0.51	0.58	0.48	0.48	0.53
6	0.62	0.50	0.62	0.76	0.21	0.67
Average	0.66	0.54	0.62	0.50	0.34	0.49
	A-B	B-C	C-D	D-	E E	-F

	A-B	B-C	C-D	D-E	E-F
1	+	-	+	-	+
2	-	-	+	+	-
3	+	-	+	+	-
4	+	+	+	+	-
5	+	-	+	o	-
6	+	-	-	+	-

Ingredients	VIII
	percent
Glycerol	72.9
S-Lube <u>a</u> /	26.0
Uranine <u>b</u> /	1.0
Detergent	0.1

- <u>a</u>/ S-Lube is a commercially available dispersion of colloidal graphite in water and was obtained from Graphite Products Corporation, Brookfield, Ohio.
- <u>b</u>/ Uranine is the sodium salt of Fluorescein, $C_{20}H_{10}O_5Na_2$

TABLE 3. THE FLUORESCENCE OF WATER EXTRACTS FROM VARIOUS O-RING MATERIALS

Polymer	The Fluorescence of 10 ml water	in
	which the O-rings were soaked f	or:

	3 minutes	2 hours
Fluorosilicone	0.0	6.5
Teflon	1.0	
Viton	2.0	15.0
Butyl	2.0	16.0
Neoprene	3.0	
Ethylene Propylene	3.0	
Silicone	3.0	au
Rubber	10.0	
Nitrile	17.0	
SBR	19.0	

Summary of Six Months' Data for the 1966 Exposure Test of Nature-Tone Enamels on Steel. Table 4.

etained	Stora	103.15 102.52 99.54 102.61 103.58	105.03 100.67 102.54 102.78 103.66	103.43 101.97 99.68 95.43	99.08 101.75 394553 102.92	102.34 103.78 87.39 108.51 110.15	101.55
te Gloss R	South Florida	93.70 89.10 96.23 98.14	95.98 93.63 101.64 102.04	102.24 104.55 107.33 94.93 97.54	97.36 90.19 94.67 96.77 99.52	93.00 94.19 92.31 151.69 132.54	100.59
Percentag	ure each	85.48 82.59 89.89 87.53	88.40 88.98 102.09 104.81 103.18	99.78 101.45 105.26 89.22 91.60	93.06 87.14 93.85 93.40	86.42 88.04 89.23 145.22 127.25	96.20
цо	Storage	99.82 99.68 99.88 99.77 99.72	99.68 99.52 99.744 99.80	99.66 99.62 99.61 99.05	99.20 99.92 99.29 99.65	99.61 99.64 99.81 99.86	99.66
olor Retention	South Florida	99.51 98.94 99.49 99.80 99.74	99.67 99.38 99.70 99.47	99.74 99.64 99.59 99.59	99.74 99.65 99.13 99.73	99.79 99.45 99.80 99.49	99.57
Col	Kure Beach	99.14 98.77 99.64 99.64	99.42 99.55 99.45 99.79	99.35 99.72 99.63 99.78 99.56	99.5 ⁴ 99.27 99.59 99.72	99.53 99.41 99.39 99.76	e 99.52
Ename l		101 102 104 105	106 107 108 1109	1112 211 211 4111 211	116 117 118 119 120	しのやるて	Average

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EFFECT OF HIGH-VOLTAGE PROBE ON SORTING ENAMELS SUSCEPTIBLE TO EARLY RUSTING TABLE 5.

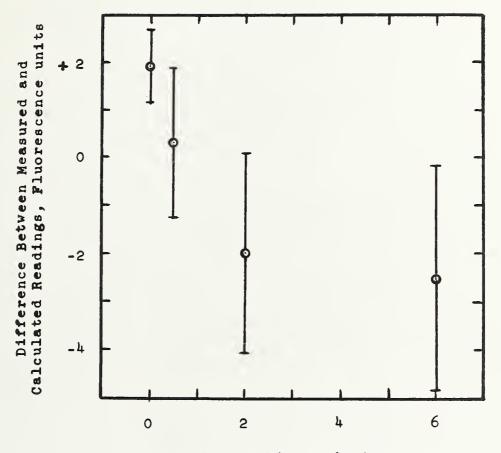
Vist 8* A. Me	R b* chan1	Rank Visuel Observation a* b* c* d* Mechanical Abrasi	ı uo	Change in Soil Retaine <u>Rank</u> 4 1/2 Minutes	ମ୍ଚ ଥିଲା ।	Change Haze <u>Rank</u> Standard Sand	nange in Haze ank % i Sand	Rank	Gloss Percent Retained
-		Ħ	1	1	0.39	e	7.6	2	84 .2
2		2	2	2	00.73	-1	3.3	Ч	88.8
e		ŝ	ę	ę	1.03	4	16.2	e	7.97
4		4	4	4	2.87	2	9.3	4	79.5
	80	e Abr.		 PEI Abrader	• •	•	•	•	•
7	4	4	4	1	6.9	2	27.9	4	42.5
	5	,	2	2	15 . 9	Ţ	24.8	1	61.4
.,	e	7	m	ę	17.3	4	30.5	n	53.4
		e	1	4	80.8	ŝ	29.8	2	53.8
•		•	•	•	•	•	•	•	•
ecl	Jan	fcal	Mechanical Abrasion -	4	1/2 Minutes - 4	400 Silic	Silicon Carbide	ø	
, ,		3	4	1	1.6	-1	2.0	1	113.6
7	4	4	2	2	4.3	ę	3.3	2	86.4
e		e	1	e	7.8	4	8 • 3	4	76.7
	2	1	e	4	11.5	3	2.5	e	83.1

TABLE 6. COMPARISON OF THE ABRADIBILITY OF 4 ENAMELS

* 4 different observers

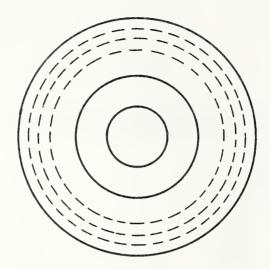
TABLE /		COMPARISON OF MECHANICAL AND FEL ABRADERS	MECHAI	NICAL A	ND FEL A	BKADEKS						
		Mechanical Abrasion, 400 SiC	Abra	sion, 4	00 SIC			PE	PEI Abrader	er		
Ename 1	Char Soil F Rank	Change in Soil Retention Rank ug	Change Haze Rank	Change in Haze Rank %	Percent Gloss Retained Rank %	ent etained %	Char So <u>f11</u> Rank	Change in Soil Retention Rank <u>ug</u>	Change Haze Rank	Change in Haze Rank %	Per Gloss Rank	Percent <u>Gloss Retained</u> <u>Rank %</u>
E-115	1	1.0	e	2.5	2	107 .5	2	7 .8	2	21.0	2	72.3
TN	2	1.3	Ч	-2.2	4	94 •6	1	7.4	Ч	17.0	Ļ	79.2
BA	e	1.6	7	2.0	1	113.6	S	11.8	9	27.9	6	42.4
ZB	4	2.4	8	5.6	5	88.3	4	11.1	10	52.0	10	40.3
U	ß	3.9	9	2.7	6	81.4	en E	9 * 3	'n	26.5	Ŋ	60.3
2	9	4.3	٢	3 • 3	9	86 .4	۲.	17.8	4	24.8	4	61.3
Λ	7	7.2	ო	2.5	7	84.0	9	16.9	ę	24.0	ę	63.0
2	8	7.8	10	8.3	10	76.7	8	21.8	œ	30 • 5	7	53.6
4	6	11.5	ო	2.5	8	83.1	6	84 .8	٢	29.8	9	55.5
R	10	42.5	6	5.8	ო	6°96	10	1047.7	6	44.0	80	47.0

COMPARISON OF MECHANICAL AND PEI ABRADERS TABLE 7.



Mixing Time, minutes

Figure 1. Fluorescence of Soil VIII as a Function of Mixing Time.



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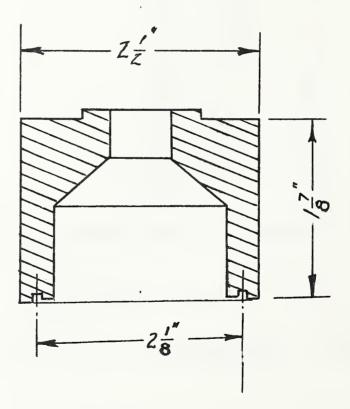


Figure 2. Brass Extraction Bottle.

Sec.

