Designation: C 614 – 74 (Reapproved 1999)

Standard Test Method for Alkali Resistance of Porcelain Enamels[1]

This standard is issued under the fixed designation C 614; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method[2] covers the measurement of the resistance of a porcelain enamel to a hot solution of tetrasodium pyrophosphate. Although the specific alkali mentioned herein is tetrasodium pyrophosphate the equipment and techniques are equally applicable to other alkali solutions.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 This test method is intended specifically for testing the resistance to alkaline attack of porcelain enamels to be used as a final finish on washing machines, dishwashers, combination washer-dryers, and similar appliances where the surface is normally exposed to an alkaline environment at elevated temperatures.

3. Apparatus

3.1 *Test Chamber*, of a type illustrated in Fig. 1, Fig. 3, and Fig. 6. This part of the equipment consists of a stainless steel beaker fitted with an O-ring sealed cover which supports an immersion heater, a stirring device, thermometer and thermistor wells; three specimen holders; a reflux condenser; a flow channeling shield to slide over and be supported by the immersion heater; and a stirring motor.[3] The beaker, heater sheath, stirring device, thermometer and thermistor wells, specimen holders, and the flow channeling shield shall be made from stainless steel alloys from the AISI 300 Series.

3.2 *Temperature Controller*, thermistor-actuated, capable of maintaining the temperature of the alkali solution in the beaker at 96.00 ± 0.20°C.

3.3 *Thermometer*, calibrated, approximately 15 in. (380 mm) long and 9⁄32 in. (7.1 mm) in diameter, with 0.1° divisions.

3.4 *Drying Oven*, electrically heated.

3.5 *Balance*, having a sensitivity of 0.1 mg and a capacity of not less than 200 g.

3.6 *Desiccator*.

3.7 *Hot Plate*, or burner suitable for heating the test solution.

3.8 *Sponge*, soft, cellulose.

3.9 *Aluminum Alloy Sheets*, approximately 3½ by 3½ by ⅛ in. (89 by 89 by 3.1 mm).

4. Reagents and Materials

4.1 *Tetrasodium Pyrophosphate*, hydrated ($Na_4 P_2 O_7 \cdot 10H_2 O$), recrystallized, reagent grade.

4.2 *Trisodium Phosphate* ($Na_3 PO_4 \cdot 12H_2 O$).

5. Test Specimens

5.1 *Preparation of Specimens*— The test specimens shall be 3½ in. (89 mm) square and may be prepared by enameling metal blanks of that size or by cutting from a larger piece. Hanging holes, if necessary for firing or weighing or both, may be placed at the corners. The specimens shall be sufficiently flat to permit sealing in the holders. The edges of cut specimens shall be stoned or filed to remove any sharp or loosely adhering fragments of metal or porcelain enamel. It is recommended that the specimens be permanently marked on the back side for identification.

5.2 *Number of Specimens*—Six specimens shall be tested simultaneously.

5.3 *Cleaning of Specimens Before Alkali Exposure*—The specimens shall be rinsed with running tap water and washed with a soft cellulose sponge dampened with a 1 % solution of trisodium phosphate. A light pressure and a back-and-forth motion in two directions, 90° apart, shall be used in the washing. After washing, the specimens shall be rinsed first with running tap water, next with distilled water and then allowed to drain in a near vertical position prior to oven-drying at 110°C for 15 min. The specimens shall be cooled in a desiccator for 30 min prior to weighing.

5.4 *Weighing of Test Specimens*—The specimens shall be weighed immediately upon removal from the desiccator. Weights shall be recorded to 0.0001 g. The weighed specimens shall be stored in a desiccator until tested.

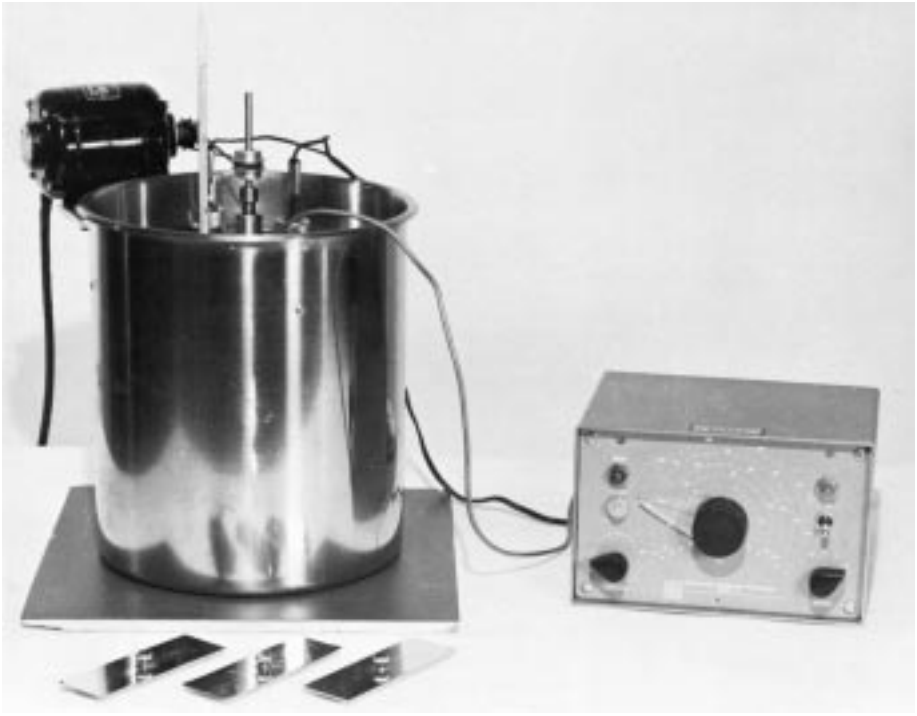
---

[1] This test method is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.

Current edition approved Feb. 27, 1974. Published May 1974. Originally published as C 614 – 68. Last previous edition C 614 – 68.

[2] This test was developed at the National Bureau of Standards under the sponsorship of The Porcelain Enamel Institute, Inc., and published as P. E. I. Bulletin T-25.

[3] See the appendix for drawings of the equipment. This equipment may be purchased from the Hoover Instrument Service, Inc., 401 N. Home Rd.,Mansfield, OH 44906.



NOTE 1—Test solution is in stainless steel beaker.
FIG. 1 Alkali Equipment in Operation

6. Procedure

6.1 *Preparation of Test Solution*—Prepare the test solution by adding 260 g of tetrasodium pyrophosphate to 4.940 litres of distilled water. Pour the entire amount of prepared solution into the beaker and cover the rectangular slots with lids. Use a freshly prepared solution for each set of six specimens.

6.2 *Preheating Test Solution*—Place the temperature controller and stirrer in operation and preheat the solution to 96°C. Heating may be expedited by placing the beaker on a hot plate or over a gas burner. When a temperature near 96°C has been reached, remove the beaker from the hot plate and place on an insulated surface. Allow an equilibration period of 15 min for all parts of the equipment to achieve a stable temperature, and adjustment of the control point to yield the desired temperature.

6.3 *Preparation for Alkali Exposure*— Place two clean, weighed specimens in each specimen-holder box. Shims may be useful in the bottom of the specimen boxes to center the specimens vertically about the exposure openings. Back-up each specimen with a stress distributing aluminum alloy backing sheet and insert the clamping device between the two sheets. Tighten the clamping device only enough to give a leakproof seal. Test the seal by filling the specimen box with water to confirm the absence of leaks. Next, remove the water and preheat the specimen holder assembly in an oven at 110°C for about 15 min.

6.4 *Exposure of Specimens*—When the test solution has reached 96°C and is under control, remove the lids and insert preheated specimen holder assemblies in the proper openings. Expose six specimens for 6 h at 96 ± 0.20°C. The exposure time shall start upon insertion of the specimen holders and shall end with their withdrawal. After removal from the holders clean, dry and weigh the specimens as specified in 5.3 and 5.4.

6.5 *Measurement of Exposed Area*—Calculate the area of the etched portion of the specimen using the average of two diameters, approximately 90° apart, which have been measured to the nearest 0.01 in. (0.25 mm).

7. Calculation

7.1 Calculate the weight loss for each specimen as follows:

$$L, \text{mg/in.}^2 = [(B - C) \times 10^3]/D \quad (1)$$

or

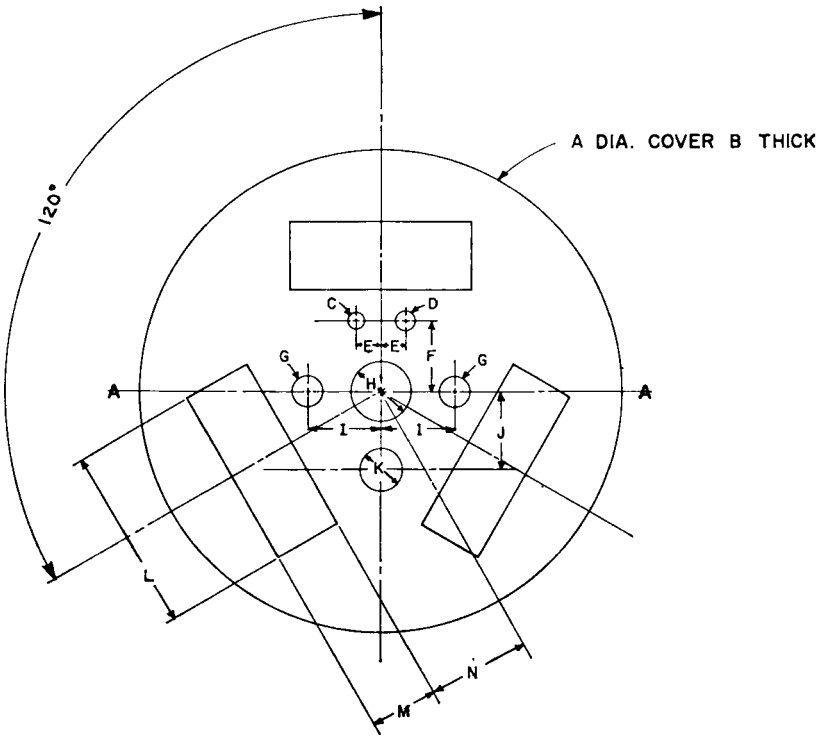
$$L, \text{mg/cm}^2 = [(B - C) \times 10^3]/E \quad (2)$$

where:
L = weight loss,
B = original weight of specimen, g,
C = final weight of specimen, g,
D = area of etched attack, in.^{2} (0.155 × cm^2),
E = area of etched attack, cm^2 (6.45 × in.^{2}).

8. Treatment of Data

8.1 *Single Determination*—Six specimens shall constitute a sample. The average of six values of weight loss *(L)*, shall be termed the mean weight loss, L, for the sample. The statistical error of the determination shall be computed by the use of the equation:

$$e = 1.15\,\sigma \quad (3)$$



Dimension	in.	mm
A	9	229
B	¼	6.4
C—Thermistor well	11/32	8.7 OD
	8⅛	206 length
D—Thermometer well	⅜	9.2 OD
	8⅛	206 length
E	½	12.7
F	1½	38.0
G—Heater chuck	⅝	15.9
H	1¼	32.0
I	1 17/32	39.0
J	1⅝	41.5
K	⅞	22.2
L	3⅞	98
M	1 15/32	37.5
N	2⅛	54

FIG. 2 Cover for Alkali Equipment

where:

e = the statistical error of the mean value for the sample, at the 95 % confidence level, and

σ = the standard deviation of the six individual weight losses from the average of all six weights.

NOTE 1—The factor 1.15 is applied only when the number of specimens is six, and the confidence level is 95 %.

8.1.1 This standard deviation shall be computed from the generalized equation:

$$\sigma = \sqrt{(\Sigma X^2/n) - \bar{X}^2} \quad (4)$$

or if the number of specimens, n, is six, from:

$$\sigma = \sqrt{(\Sigma X^2/6) - \bar{X}^2} \quad (5)$$

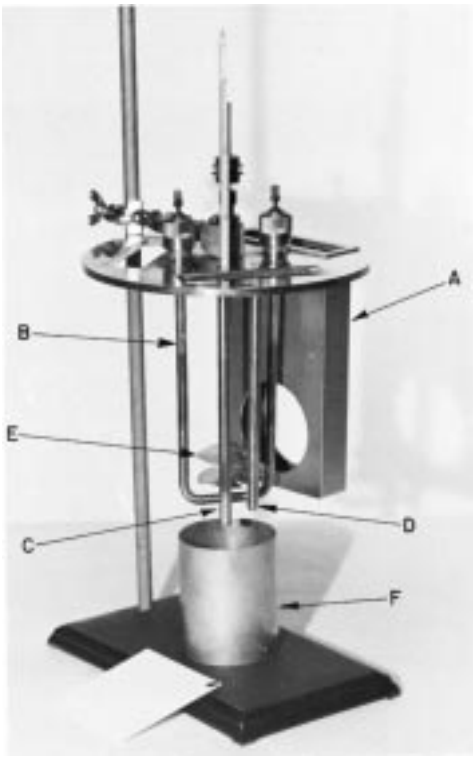
where:

ΣX^2 = sum of the squares of the weight loss values of the six individual specimens, and

$\bar{X}^2$ = the square of the mean weight loss of the six specimens.

8.1.2 Illustrative calculations are given below. The recommended number of decimal places is given in each case:

Specimen No.	L, mg/in.^{2}	L^2
1	7.7	59.29
2	7.5	56.25
3	6.9	47.61
4	8.0	64.00
5	7.7	59.29
6	7.9	62.41
Sum	45.7	348.85
Mean	7.6167	58.1417



A— Empty specimen holder in position.
B— Immersion heater.
C— Thermometer well.
D— Thermistor well.
E— Stirrer.
F— Circular shield (lowered for clarity).

FIG. 3 Cover Assembly

from Eq 3:

$$\sigma = \sqrt{58.1417 - (7.6167)^2} = \sqrt{0.1276} = 0.357 \quad (6)$$

from Eq 1:

$$e = 1.15 \times 0.357 = 0.411 \quad (7)$$

The weight loss $\bar{L}$ is reported as 7.6± 0.4 mg/in^2

8.2 *Difference Between Two Determinations*—The significance of a difference between two mean values shall be tested through the use of the ratio $d:e'$

where:
d = the difference in means, and
e' = the statistical error in the determination of d.

8.2.1 The statistical error, e', of the difference, d, between two means shall be determined from the following equation:

$$e' = \sqrt{(e_1)^2 + (e_2)^2} \quad (8)$$

where:
e' = the error of the difference in means,
e_1 = the error of one mean value, and
e_2 = the error of the other mean value.

8.2.2 An example to illustrate the use of Eq 4 is given below:

Assume:
$L_1 = 7.6 \pm 0.5$,
$L_2 = 8.2 \pm 0.3$, and
$d = 0.6$.

Then:

$$e' = \sqrt{(0.5)^2 + (0.3)^2} = \sqrt{0.34} = 0.58 \quad (9)$$

$$d/e' = 0.6/0.58 = 1.03$$

When $d:e'$ is equal to or greater than 1.00, the difference is significant at the 95 % confidence level.

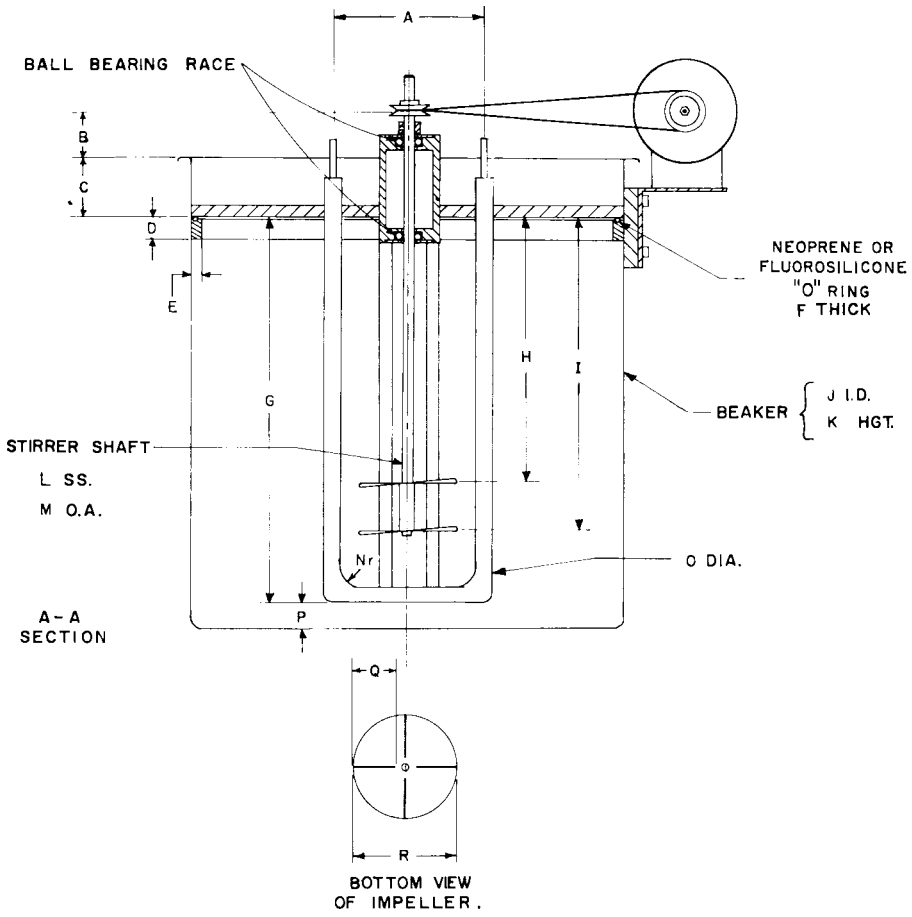
8.2.3 The following table shows the relation between values of $d:e'$ and the confidence level, for samples of six specimens.

$d:e'$	Confidence Level, %	Interpretation
Over 1.50	99 or higher	highly significant
Over 1.00 but less than 1.50	95 to 99	significant
Over 0.80 but less than 1.00	90 to 95	indicative

The basing of conclusions on differences in which less than 90 % confidence can be placed is not recommended.

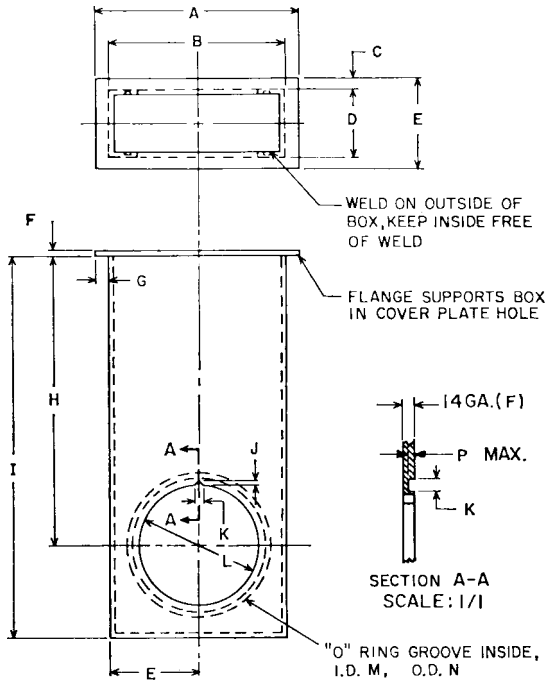
9. Precision and Bias

9.1 The precision and bias of this method are being developed.



Dimension	in.	mm
A	3 1/16	78
B	1	25.4
C	1 7/8	47.5
D	7/16	11.1
E	3/16	4.75
F	1/16	1.59
G	7 1/2	190
H	5 1/2	140
I	6 1/2	165
J	9	229
K	9.8	249
L	3/16	4.75
M	10 1/4	260
N	1/2	12.7
O	5/16	7.9
P	1/2	12.7
Q	3/4	19.0
R	2	51

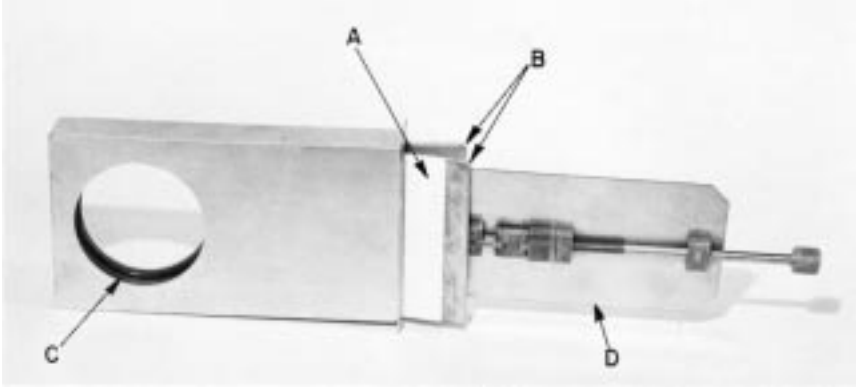
FIG. 4 Alkali Test Equipment



Dimension	in.	mm
A	4¼	108
B	3¾	95
C	17/64	6.7
D	1 11/32	34.0
E	1⅞	47.5
F	0.079	2.01
G	¼	6.4
H	6	152
I	8	203
J	3/32	2.40
K	⅛	3.18
L	2½	64
M	2¾	70
N	3	76
P	0.060	1.52

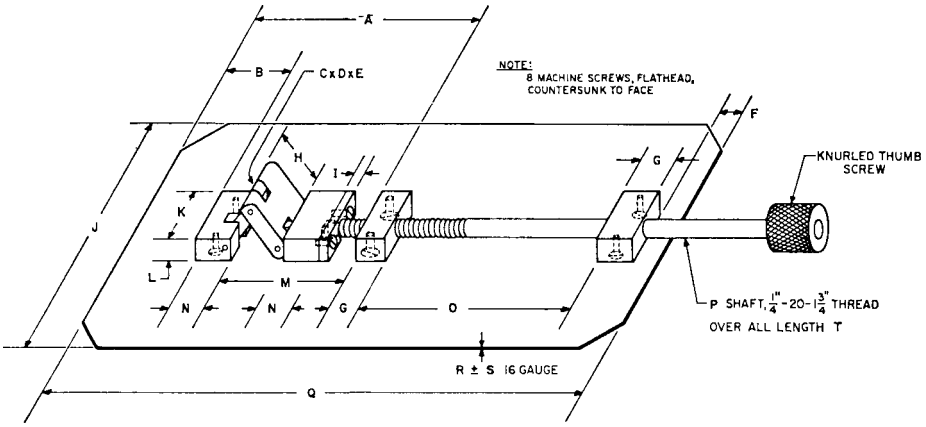
NOTE 1—Box welded from 14-gage (0.079 in.) stainless steel.

FIG. 5 Detail Specimen Holder



A—Showing a specimen.
B—The back-up plate.
C—O-ring interior.
D—Clamping plate (partly withdrawn).

FIG. 6 Specimen Holder Assembly



Dimension	in.	mm
A	3¼	83
B	1⅛	28.5
C	⅛	3.18
D	¼	6.4
E	11/16	17.5
F	¼	6.4
G	⅜	9.5
H	¾	19.0
I	⅛	3.18
J	3 7/16	87
K	¾	19.0
L	5/16	7.9
M	1⅝	41.5
N	½	12.7
O	2⅝	67
P	¼	6.4
Q	7⅛	181
R	0.060	1.52
S	0.003	0.076
T	6¼	159

FIG. 7 Stainless Steel Specimen Backup Plate