Designation: C 715 – 90 (Reapproved 2006)

Standard Test Method for Nickel on Steel for Porcelain Enameling by Photometric Analysis[1]

This standard is issued under the fixed designation C 715; 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 covers the determination of the amount of nickel deposited on sheet steel during its preparation for porcelain enameling. It is a photometric method commonly used on production parts and is suitable for determining the heavier nickel deposits that may be obtained during the processing of steel for one-coat enameling.

NOTE 1—An alternative X-ray emission spectrometry method is Test Method C 810.

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. Referenced Documents

2.1 *ASTM Standards:* [2]

C 810 Test Method for Nickel on Steel for Porcelain Enameling by X-Ray Emission Spectrometry

E 30 Test Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron[3]

E 60 Practice for Analysis of Metals, Ores, and Related Materials by Molecular Absorption Spectrometry

3. Significance and Use

3.1 This test method is primarily used to control the nickel dipping operation to ensure that the desired level of nickel deposition is attained. It is also used to prepare test plates used for calibration in Test Method C 810.

4. Apparatus

4.1 *Photoelectric Photometer*, conforming to Practice E 60.

4.2 *Weighted Rubber Ring Assembly*, required to confine stripping agents to a definite area, consisting of a molded rubber ring and a metal outer ring. The rubber ring shall have an inside diameter of 1.35 in. (34.3 mm) in order to encircle an area of 0.01 ft^2 (0.000929 m^2), a wall configuration as shown in Fig. 1, and the lower portion beveled at a 45° angle (0.78 rad) to reduce the contact area and ensure a better seal. The metal outer ring shall weigh about 3.5 lb (1.5 kg), suitably machined to fit over the top of the rubber ring as shown in Fig. 1.

4.2.1 The exact area covered by the rubber ring will gradually increase as the rubber ring itself is consumed by the acid reagent used. In the most accurate analysis, the area etched by the rubber ring shall be calculated occasionally, factored against the prescribed area, and that number applied to the reading obtained from the graph.

4.3 *Aspirator*, consisting of a calibrated 500-mL flask, equipped with a twohole stopper, an aspirator bulb, and a suction tube formed from 0.079-in. (2-mm) inside diameter capillary glass tubing.

5. Reagents and Materials

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee of Analytical Reagents of the American Chemical Society.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Use distilled or deionized water.

5.3 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH_4OH).

5.4 *Ammonium Persulfate*—$(NH_4)_2S_2O_8$.

5.5 *Dimethylglyoxime*—Prepare a 1 % solution of dimethylglyoxime in methyl alcohol or a 2.62 % solution of sodium dimethylglyoximate in water (store in a polyethylene bottle).

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

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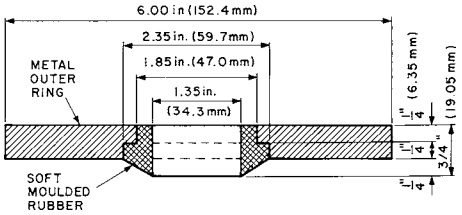
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn.

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



NOTE 1—The outer ring is a steel disk approximately 6 by ½ in. (152 by 13 mm), weighing approximately 3½ lb (1.58 kg).

FIG. 1 Detailed Drawing of Rubber Ring

5.6 *Hydrochloric Acid (1+5)*—Dilute 1 vol of concentrated hydrochloric acid (HCl, sp gr 1.19) with 5 vol of water.

5.7 *Nickel Sulfate, Standard Solution*— Dissolve 0.448 g of nickel sulfate ($NiSO_4 \cdot 6H_2O$) in water. When the material is completely dissolved, cautiously add 10 mL of concentrated H_2SO_4 and transfer the solution to the 1000-mL flask. When cool, make up to the mark with water. One millilitre of this standard solution is equivalent to 0.0001 g of nickel per millilitre.

5.8 *Nitric Acid (1+1)*—Dilute 1 vol of concentrated nitric acid (HNO_3, sp gr 1.42) with 1 vol of water.

5.9 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H_2SO_4).

6. Sampling

6.1 Sampling frequency shall be consistent with the objective of control of the nickel dipping operation.

7. Standardization of Photoelectric Photometer

7.1 The photoelectric photometer shall be standardized as follows:

7.1.1 Using a buret, accurately measure out 2, 4, 8, 12, 16, and 20 mL portions of nickel sulfate standard solution. These amounts are equivalent to 0.2, 0.4, 0.9, 1.3, 1.7, and 2.1 g of nickel per square metre (0.02, 0.04, 0.08, 0.12, 0.16, and 0.20 g of nickel per square foot) of surface when specimens from the steel surface are obtained as prescribed. (Iron in solution that is dissolved from the steel surfaces has a negligible effect on the nickel determinations.) Using the standard analytical procedures described in 8.2, determine the percent transmission at a wavelength of approximately 525 nm for each of the six increments of nickel sulfate standard solution. Then plot a graph on appropriate graph paper of the percent light transmission against the known nickel concentration representing 0.2 to 2.1 g of nickel deposit per square metre of surface. The resulting graph, which should be a straight line, will be used to obtain nickel-coating masses from light transmission results.

8. Determinations of Nickel Coating Masses

8.1 *Sampling a Nickel-Coated Steel Surface*:

8.1.1 Place the weighted, rubber ring assembly on the nickel-coated metal surface. Add 3 mL of warm (approximately 120°F (50°C)) HNO_3 (1+1). Allow the foaming reaction to proceed for about 10 s for light nickel coatings and about 15 s for heavier nickel coatings. If the warm acid does not react, scratch the steel surface or try another spot. After the acid has foamed for the prescribed time, add 5 mL of HCl (1+5) to stop the foaming reaction. Withdraw the solution from the steel surface with the aspirator into the calibrated flask. Rinse the test area twice with water and retain the washings in the flask.

8.1.2 Alternative methods for determining nickel are described in Test Methods E 30, Sections 62 to 70 for the gravimetric method and Sections 71 to 73 for the volumetric method.

8.2 *Analytical Procedure*:

8.2.1 Add chemicals in the order given below to the solution in the flask and mix thoroughly after each addition:

Additions for 23-mm Optical Path Cell[A]	Amount in Order of Use
Ammonium hydroxide (sp gr 0.90)	50 mL
Ammonium persulfate	4 g
Dimethylglyoxime	10 mL
Dilute with water to	500 mL

[A] Cells with other optical path lengths are available.

8.2.2 Filter a portion of the solution. Discard the first 10 to 20 mL from the filter and collect a sufficient amount of filtrate in the absorption cell for testing. Just before testing, set the wavelength as determined in accordance with Section 7 and adjust the instrument to 100 % transmission with a cell that contains only water. Place the cell containing the test solution in the photometer and read the percent transmission.

9. Calculation and Report

9.1 Refer to the graph developed in Section 7. Determine the mass of nickel that corresponds to the percent transmission reading shown by the photometer. Report the results in grams of nickel per square metre of steel surface.

10. Precision and Bias

10.1 The precision and bias of this test method is believed to be within 0.000929 g/ft^2 (0.1 g/m^2). Exact values are difficult to obtain because of sample inhomogeneity, and the effect of the acid etch on the rubber ring diameter (see 4.2.1).