Designation: C 810 – 90 (Reapproved 2000)

Standard Test Method for Nickel on Steel for Porcelain Enameling by X-Ray Emission Spectrometry[1]

This standard is issued under the fixed designation C 810; 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 measurement of the amount of nickel deposited on sheet steel during its preparation for porcelain enameling. It is an X-ray emission method used for testing sample panels or certain commercial parts.

NOTE 1—An alternative wet chemical method is Test Method C 715.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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.* For a specific hazards statement, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

C 715 Test Method for Nickel on Steel for Porcelain Enameling by Photometric Analysis[2]

3. Summary of Test Method

3.1 Steel samples coated with a light nickel deposit are inserted in the sample position of an X-ray spectrometer. The count rate for nickel is measured and converted by means of a calibration curve to g/m ^{2} (g/f^2).

NOTE 2—1 m^2 = 10.75 ft^2. Industry usage is typically in mixed units, grams per square foot. For example, 0.10 g/ft ^{2} equals a little more than 1 g/m^2.

4. Significance and Use

4.1 This test method is an accurate and rapid means for measuring nickel deposits on steel sample plates and such parts that can be fitted into the X-ray spectrometer. Its accuracy extends over a wide range of nickel deposits.

5. Interferences

5.1 There are no interferences from other elements present. However, low values can result from absorption of the X rays by overlaying material. Grease on the sample or rust due to storage in humid areas are examples of such material. Low results are also obtained on de-enameled samples because the nickel deposit is converted to a nickel iron alloy at enameling temperatures. The presence of the iron in the alloy layer absorbs some of the X radiation and accounts for the lower result.

6. Apparatus

6.1 *Suitable X-Ray Emission Spectrometer* complete with 50-kV power supply goniometer, detector with pressure-regulated gas flow attachments, scaler-counter, lithium fluoride analyzing crystal, and 0.02° Soller slit collimator is required.[3] About a 1-in. (25.4-mm) diameter area of the sample is irradiated.

6.2 *Special Sample Holder* (Fig. 1), to permit insertion of a 2 by 2-in. (51 by 51-mm) flat corner of a large flat sample. Alternatively, the standard sample holder supplied with the equipment may be used, but the sample must be cut to 1.5 by 1.25 in. (38 by 32-mm).

6.3 *Steel Sheets* with various amounts of nickel deposits are required for calibration and standardization.

6.4 *Nickel-Base Alloy Sample*, such as 18-8 stainless steel, for routine calibration.

7. Hazards

7.1 Equipment should be periodically checked for radiation leaks to ensure against exposure to X radiation.

8. Calibration and Standardization

8.1 Prepare approximately 18 standard plates by cleaning and pickling 4 by 6 in. (102 by 152 mm) commercial enameling iron stock (any steel used in commercial enameling operations may be used) and applying nickel in the conventional manner for varying treatment times to give a range of nickel deposition from 0 to 3 g/m^2 (0 to 0.4 g/ft^2).

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[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] *Annual Book of ASTM Standards*, Vol 02.05.

[3] Available from the following: (1) Philips Electronic Instruments, 750 S. Fulton Ave., Mount Vernon, NY 10550; (2) Siemens Corp., Medical Industrial Div., 186 Wood Ave., South, Iselin, NJ 08831; and (3) Diano Corp., X-ray Div., 2 Lowell Ave., Winchester, MA 01890.

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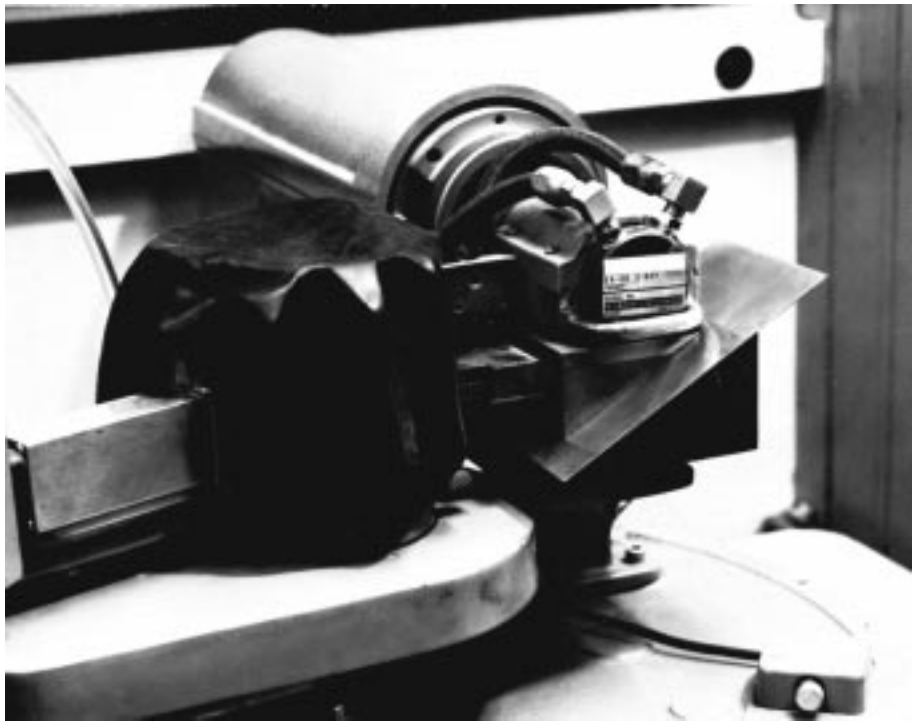

FIG. 1 Special Sample Holder for X-Ray Beam Exposure

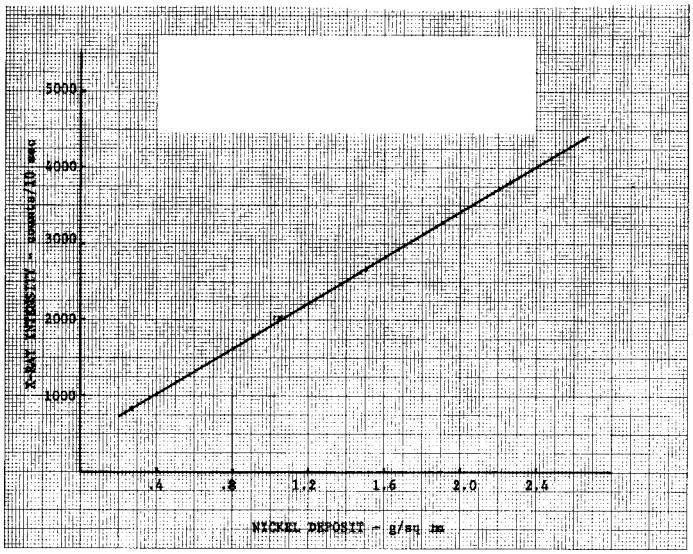

FIG. 2 Example of Conversion of X-Ray Intensity to Nickel Deposit

8.2 Prepare a parer mask which, when placed over each plate, will show the areas measured by the X-ray spectrometer. The mask is used later to indicate the same areas for wet determination of nickel deposition by Test Method C 715. In this way, comparative data are obtainable by both measurement methods on the same areas of the standard plates.

8.3 The spectrometer is set to the *K-alpha* line of nickel at 1.66 A and the X-ray intensity in counts/s is scaled for each measurement. A reference plate of Type 321 stainless steel (Cr 18, Ni 10) is measured before and after each measurement on the standard plates. The two counts on the reference plate are averaged.

8.4 The numbered standard plates are measured on one side at two areas, top and bottom, and are corrected for instrument drift by relating the count rates on the standard plates to a fixed average count rate on the reference plate.

8.5 On the basis of X-ray count data, ten standard plates are selected for the best distribution of counts covering the full range of nickel on the plates. Nickel deposition on the ten selected plates is then determined at one area on each plate by Test Method C 715. The wet results are then plotted versus X-ray count rate on linear graph paper and a smooth curve drawn through the plotted points. The curve may be used to prepare a chart which lists count ranges for each increment of 0.1 g/m^2 (0.01 g/ft^2) nickel.

9. Procedure

9.1 *Standardization of Equipment*—Insert the reference standard (6.4) which has a known X-ray count determined when equipment was standardized, in the special sample holder. After the equipment is warm, set the voltage to obtain X-ray counts characteristic of the reference standard.

9.2 *Nickel Determination*:

9.2.1 Insert a 4 by 6-in. (10 by 152-mm) sample in the special sample holder (or cut a piece to fit a regular holder).

9.2.2 Using an X-ray tube with suitable target, adjust the power supply to provide 50 kV at 25 mA and set the scaler for a 10-s count. Align the spectrogoniometer to 48.65°, 2-θ angle for nickel determination using a lithium fluoride crystal. Start the apparatus and record the count from the counter scales.

9.2.3 Repeat the determination on a minimum of two areas on each side of each sample. Average the counts so recorded and read the nickel concentration from the standard plot.

9.2.4 Specific details of operation of the X-ray apparatus are not included herein due to the complexity of such equipment and the slight variations in procedure between different types of apparatus. These details can be provided by the manufacturer.

9.3 *Verification of Standard Equipment*— After completing determinations of unknown samples, recheck the machine calibration by reinserting the reference standard and checking the reading.

10. Report

10.1 Convert X-ray counts to grams per square metre by using the calibration curve.

NOTE 3—The result in grams per square metre can be converted to grams per square foot by dividing by 11.

11. Precision and Bias

11.1 The precision and bias of this test method is being established.

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