Designation: E 1659 – 00

Standard Test Methods for Coating Weight and Chemical Analysis of Zinc-Nickel Alloy Electrolytically Coated on Steel Sheet[1]

This standard is issued under the fixed designation E 1659; 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 These test methods cover independently the chemical analysis of each surface of zinc-nickel alloy electrolytically coated on steel sheet. The coatings have chemical compositions within the following limits:

Analyte	Concentration Range
Coating Weight	0.0 to 80 g/m^2
Nickel	7.0 to 17.0 %

1.2 These test methods are in the following sections:

	Sections
Coating Weight, by the Weigh-Strip-Weigh Method (20.0 to 45.0 g/m^2)	8-18
Nickel by the Atomic Absorption Method (11.0 to 13.5 % of Coating Weight Ranging from 20 to 45 g/m^2)	19-29

1.3 The values stated in SI units are to be regarded as standard. In some cases, exceptions allowed in Practice E 380 are also used.

1.4 *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:*

A 917 Specification for Steel Sheet, Coated by the Electrolytic Process for Applications Requiring Designation of the Coating Mass on Each Surface (General Requirements)[2]

A 918 Specification for Steel Sheet, Zinc-Nickel Alloy Coated by the Electrolytic Process for Applications Requiring Designation of the Coating Mass on Each Surface[2]

D 1193 Specification for Reagent Water[3]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[4]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[5]

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[6]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[4]

E 663 Practice for Flame Atomic Absorption Analysis[7]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[8]

E 1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry[8]

E 1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analyses[8]

3. Significance and Use

3.1 These test methods for the chemical analysis of zinc-nickel alloy coating on sheet steel are primarily intended as referee methods to test such materials for compliance with compositional specifications such as found in Specification A 918, particularly those under the jurisdiction of ASTM Committee A-5 on Metallic Coated Iron and Steel Products. It is assumed that all who use these test methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory under appropriate quality control practices such as those described in Guide E 882.

3.2 These test methods must be applied twice, once to each side of the specimen if coating weight and composition are required for both sides of a coated sheet. Two separate specimens are required for this purpose.

4. Apparatus, Reagents, and Instrumental Practices

4.1 *Apparatus*—Specialized apparatus requirements are listed in the apparatus section in each individual test method.

4.2 *Reagents*:

4.2.1 *Purity of Reagents*—Unless otherwise indicated, all reagents used in these test methods shall conform to the

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[1] These test methods are under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.05 on Zn, Sn, Pb, Cd, Be, and Other Metals.

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[2] *Annual Book of ASTM Standards*, Vol 01.06.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] *Annual Book of ASTM Standards*, Vol 03.05.

[6] Discontinued; see *1997 Annual Book of ASTM Standards*, Vol 03.05.

[7] Discontinued; see *1997 Annual Book of ASTM Standards*, Vol 03.06.

[8] *Annual Book of ASTM Standards*, Vol 03.06.

Reagent Grade Specifications of the American Chemical Society.[9] Other chemicals may be used, provided it is first ascertained that they are of sufficiently high purity to permit their use without adversely affecting the expected performance of the determination, as indicated in Section 28.

4.2.2 *Purity of Water*—References to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

4.3 *Photometric Practice*—Photometric practice prescribed in these test methods shall conform to Guide E 1024 and Practice E 1452.

5. Hazards

5.1 For precautions to be observed in the use of certain reagents and equipment in these test methods, refer to Practices E 50.

6. Sampling

6.1 *Zinc-Nickel Alloy Coated Sheets*—Samples for determining weight and composition of coating shall be secured in accordance with Specification A 917, which is referred to in Specification A 918. Test specimens shall be of squares with sides of 50 ± 5 mm. One test specimen is required for each side to be analyzed. The backside which is not to be analyzed shall be marked "X".

7. Interlaboratory Studies and Rounding Calculated Values

7.1 These test methods have been evaluated using Practice E 173, except for the update in the stripping solution, 15.1, 15.8, 18.1.2, 18.2, 29.1.1, and 29.2, as well as Tables 2, 3, 5, and 6.

7.2 Calculated values shall be rounded to the desired number of places in accordance with the rounding method of Practice E 29.

WEIGHT OF COATING ON ZINC-NICKEL ALLOY-COATED SHEET BY WEIGH-STRIP-WEIGH METHOD

8. Scope

8.1 This test method provides a procedure for determining independently the weight of coating on each surface of zinc-nickel alloy-coated sheet steel, in coating masses from 20 to 45 g/m^2 (Note 1).

NOTE 1—The upper limit of the scope has been set at 45 g/m^2 because test materials with higher coating weight were not available for testing in accordance with Practice E 173. However, recognizing the simplicity of the weigh-strip-weigh technique, materials with higher coating weights can be tested following this procedure. Users of this test method are cautioned that use of it for coating weight determinations above 45 g/m^2 is not supported by interlaboratory testing.

9. Summary of Test Method

9.1 The coating on the sheet steel is stripped by using hydrochloric acid solution containing an inhibitor to prevent the attack on the base steel. The coating weight is determined from the weight difference of the specimen before and after stripping.

10. Interferences

10.1 The hexamethylene tetramine inhibitor used in this test method permits the dissolution of some base metal, which could lead to higher than expected coating weight determinations. Since Zn/Ni coatings contain no appreciable amounts of Fe, the effects of this bias are corrected by determining the mass of iron stripped with the coating and subtracting that value from the raw weigh-strip-weigh data.

11. Apparatus

11.1 *Analytical Balance*, capable of weighing to 0.1 mg.

11.2 *Electroplater's Tape*, capable of protecting one side of a coated piece of sheet steel while the other side is being stripped in a hydrochloric acid solution. It must not contaminate the acid solution or interfere with the coating weight determination by gaining or losing weight.

11.3 *Vernier Calipers*, calibrated to an international standard and capable of measuring to at least 0.05 mm.

12. Reagents

12.1 *Hexamethylene Tetramine, USP Grade*—Used as an inhibitor to prevent acid attack of the base metal while stripping the coating from the base steel.

12.2 *Stripping Solution*—Add 340 mL hydrochloric acid to 1660 mL of water. Add 7.0 g of hexamethylene tetramine, mix, and cool before use.

13. Precautions

13.1 **Warning**—Hydrogen gas, which can form explosive mixtures with air, is evolved in the stripping process. Therefore, this test method should be performed under conditions of adequate ventilation, such as a fume hood.

14. Sample Preparation

14.1 Clean the specimens with acetone using a soft paper towel, then dry with oil-free compressed air.

14.2 Cover the side of the specimen from which the coating is not to be stripped with electroplater's tape.

14.3 Use a roller to press the tape firmly against the sheet, making sure to remove all air bubbles or wrinkles.

14.4 Trim off the excess tape.

14.5 Press the tape firmly near the edge to protect the taped side from acid attack.

14.6 Write the sample identification on the taped side with a marker.

15. Procedure

15.1 *Specimen Area*—Using the calipers, measure and record the length of all four sides of the test specimen.

15.1.1 Check that the measuring face and reference edge of the calipers are clean. Check that the calipers read "0" when

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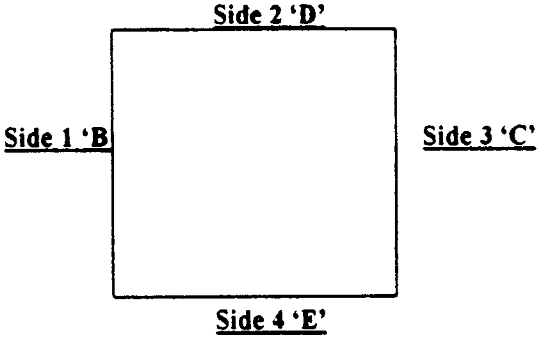
[9] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States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

the measuring surface is closed. If it does not, correct the problem according to the manufacturers instructions.

15.1.2 Place the calipers at the corners of the specimen and measure the length of each side to the nearest 0.005cm.

15.1.3 Record the length of each side as shown in the diagram below.

NOTE 2—To ensure that the calculated area is accurate even if the specimen does not have 90° angles, all four sides are measured. In 16.2, the average of opposite sides is calculated and used to determine specimen area.



15.2 Weigh the prepared specimen to the nearest 0.1 mg and record the weight as the original weight of the specimen.

15.3 Place the sample in a 600-mL beaker with the taped side down.

15.4 Add 25 mL of stripping solution slowly. As the coating is stripped, the color changes from gray to black and back to gray (see Note 3).

NOTE 3—Stripping time will depend on the nickel composition and weight of the coating.

15.5 After the coating is stripped, remove the sample with a poly (tetrafluoroethylene) coated magnet attached to one end of an approximately 150-mm long flexible polyethylene tube. Holding it over the same beaker, rinse it carefully with water and police the stripped side to remove the last traces of nickel.

15.6 Dry the stripped specimen with oil-free compressed air. Weigh it to the nearest 0.1 mg and record the weight.

15.7 Warm the beaker on a hotplate until all the stripped coating is dissolved. Transfer the solution to a 100-mL volumetric flask, dilute to the mark, and mix thoroughly. Reserve this sample stock solution for iron determination.

15.8 *Determination of Stripped Iron*:

15.8.1 Calibrate the instrument in accordance with the manufacturer's instructions following the guidelines set forth in Guide E 1024 and Practice E 1452. Choose the appropriate wavelength and calibration solutions that provide a calibration curve that includes up to 2.0 mg Fe/100 mL of solution.

15.8.2 Verify the calibration curve with an iron control solution that falls in the mid-range of the calibration curve. If this control standard is not within ±10 % of its assumed true value, identify the problem, recalibrate, and verify the curve. If the control standard is within ±10 %, analyze the solutions from 15.7 for iron.

15.8.3 After the final sample stock solution is analyzed, read the iron control standard and check that the reading is within ±10 % of the assumed true value. If it is not, identify the problem and repeat 15.8.1-15.8.3. If it is within ±10 %, record the results of all sample solutions.

NOTE 4—The typical amount of iron stripped from the base steel when performing this test method is equivalent to a coating weight of about 1 g/m^2. Paragraph 15.8 allows a 10 % error in this Fe determination, which is equal to a coating weight of 0.1 g/m^2. On a sample with the minimum coating weight of 20 g/m^2, this is an error in the coating weight determination of 0.5 %. At higher coating weights, the percent error would decrease. The reproducibility of the test method by Practice E 173 at a coating weight of 20 g/m^2 is 1.56 g/m^2, or an error of 7.8 %. The error in the iron determination was considered statistically insignificant and therefore Paragraph 15.8 did not undergo a formal interlaboratory evaluation of precision and bias.

16. Calculation

16.1 Calculate the mass of the zinc-nickel alloy coating as follows:

$$M = [(W_1 - W_2) - (E/1000)] \tag{1}$$

where:

M = mass of stripped coating, g,
W_1 = original weight of specimen, g,
W_2 = weight of stripped specimen, g, and
E = iron stripped, mg.

16.2 Calculate the area of the test specimen as follows:

$$A = 2.5(B + C)(D + E) \times 10^{-5} \tag{2}$$

where:

A = area of test specimen, m^2,
B = length of Side 1, cm,
C = length of Side 3, cm,
D = width of Side 2, cm, and
E = width of Side 4, cm.

16.3 Calculate the coating weight (W_c) in g/m^2 as follows:

$$W_c = M/A \tag{3}$$

where:

W_c = coating weight, g/m^2,
M = mass of stripped coating, g, from 16.1, and
A = area of test specimen, m^2, from 16.2.

17. Report

17.1 Report the weight of zinc-nickel alloy coating to the nearest 1 g/m^2.

18. Precision and Bias [10,11]

18.1 *Precision*:

18.1.1 Ten laboratories, one of which reported a second pair of values, participated in the original testing of this test method and obtained the data summarized in Table 1. The data in Tables 2 and 3 were generated by one laboratory comparing the effectiveness of a stripping solution containing hexamethylene tetramine as a replacement for NEP312-S (used in Test Methods E 1659 – 95), an inhibitor that is no longer commercially available. Along with a revised stripping solution, this

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[10] Supporting data are available from ASTM Headquarters. Request RR:E01-1009.

[11] Supporting data are available from ASTM Headquarters. Request RR:E01-1028.

TABLE 1 Statistical Information—Coating Weight-Original Test Data from E 1659 – 95

Test Material	Number of Laboratories Included	Average Coating Weight, g/m^2	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
20	9	22.93	1.06	1.56
30	10	36.52	1.76	1.92
40	9	41.78	1.00	1.91

TABLE 2 Statistical Information—Comparison of Stripping Solutions on 20/20 Material

Test Material	Original Stripping Solution, g/m^{2A}	Current Stripping Solution, g/m^{2A}	Iron Stripped, g/m^{2B}	Adjusted Coating Weight, g/m^{2C}
1	23.45	23.74	0.68	23.06
2	23.18	24.29	0.83	23.46
3	23.54	24.33	1.13	23.20
4	23.11	23.94	0.64	23.30
Average	23.320	24.075	0.820	23.255
2 Standard Deviations	0.415	0.568	0.445	0.337

[A] Results obtained following the original test method with no adjustments for iron stripped.

[B] Iron stripped from base metal from 15.8, expressed in g/m^2.

[C] Coating weight results obtained by using the current test method, adjusting for the stripped iron.

TABLE 3 Statistical Information—Comparison of Stripping Solutions on 30/30 Material

Test Material	Original Stripping Solution, g/m^{2A}	Current Stripping Solution, g/m^{2A}	Iron Stripped, g/m^{2B}	Adjusted Coating Weight, g/m^{2C}
1	35.21	38.19	1.03	37.16
2	37.47	35.83	0.50	35.33
3	35.64	37.86	0.55	37.31
4	37.65	36.05	0.51	35.54
Average	36.493	36.983	0.648	36.343
2 Standard Deviations	2.494	2.429	0.512	2.074

[A] Results obtained following the original test method with no adjustments for iron stripped.

[B] Iron stripped from base metal from 15.8, expressed in g/m^2.

[C] Coating weight results obtained by using the current test method, adjusting for the stripped iron.

study also used a more accurate procedure to determine sample area, thus improving the precision of this test method. This improved measurement procedure is described in 15.1.

18.1.2 The effectiveness of the current stripping solution containing hexamethylene tetramine was compared to that of the original stripping solution by analyzing four specimens from each of two test materials, one material with approximately 20g/m^2 of coating (20/20 material) and the other with approximately 30g/m^2 of coating (30/30 material). The data, obtained by testing done in one laboratory, is summarized in Tables 2 and 3 and shows that the precision of the current stripping solution is at least as good as that of the original stripping solution. Therefore, a complete interlaboratory test of the revised procedure was not carried out.

NOTE 5—The 30/30 material showed poor precision when using both stripping solutions. While the precision was poor for these samples, the average coating weight was comparable for both stripping solutions.

18.2 *Bias*—Using the hexamethylene tetramine (12.1) as the new inhibitor, the coating weight determination is biased due to iron being stripped from the base metal. This bias is corrected in the procedure by determining the amount of iron stripped and adjusting the coating weight result accordingly. The data, summarized in Columns 2 and 5 of Tables 2 and 3 shows that there is no bias between stripping solutions after adjusting the coating weight for stripped iron.

NICKEL BY THE ATOMIC ABSORPTION METHOD

19. Scope

19.1 This test method covers the determination of nickel in concentrations from 11.0 to 13.5 % in zinc-nickel coatings (Note 6).

NOTE 6—The upper limit of the scope has been set at 13.5 % because sufficient test materials containing higher nickel were unavailable in accordance with Practice E 173. However, recognizing the calibration technique of atomic absorption spectrophotometer, materials with higher nickel content may be tested following this procedure. Users of this test method are cautioned that use of it for nickel determination out of the scope is not supported by the interlaboratory testing.

20. Summary of Test Method

20.1 The solution from the coating weight test is diluted to a fixed volume and then aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. Spectral energy at 231.1 nm from a nickel hollow cathode lamp is passed through the flame, and the concentration is measured.

21. Concentration Range

21.1 The recommended concentration range is from 10 to 300 mg of nickel per litre of solution.

22. Interferences

22.1 There are no known interferences.

23. Apparatus

23.1 *Atomic Absorption Spectrophotometer*, capable of resolving the 231.1-nm line, equipped with a nickel hollow-cathode lamp and a microprocessor for direct readout in concentration. The performance of the instrument must meet the guidelines of Practice E 663 and Guide E 1024.

23.2 *Automatic Sampler (Optional)*, capable of reproducibly aspirating a sample synchronized with the read cycle of the instrument.

23.3 *Printer Sequencer*, capable of providing at least five significant digits of information.

23.4 *Auto-Sampler Tubes (Optional)*—15-mL polystyrene centrifuge tubes or equivalent compatible with the autosampler.

24. Reagents

24.1 *Nickel, Standard Stock Solution* (1 mL = 10 mg Ni)—A certified reference solution produced by or directly traceable to a National Metrology Institute with a nominal concentration of 10 mg/mL. NIST SRM 3136 nickel standard solution has been found to be satisfactory.

24.2 *Nickel Standard Solution A* (1 mL = 1.0 mg)—Pipet 10 mL of Nickel Standard Stock Solution into a 100-mL volumetric flask, dilute to the mark, and mix. Transfer the solution into a polyethylene bottle. Prepare fresh as needed upon failure of control standard solution performance (see 26.2.4).

24.3 *Nickel Standard Solution B* (1 mL = 0.1 mg Ni)—Pipet 10 mL of Nickel Standard Solution A into a 100-mL volumetric flask, add 1 mL of HCl, dilute to the mark, and mix. Transfer the solution into a polyethylene bottle. Prepare fresh as needed upon failure of control standard solution performance (see 26.2.4).

24.4 *Nickel Control Standard Stock Solution* (1 mL = 1.0 mg Ni)—Prepare this solution with a different metallic source than that used for preparing the Nickel Standard Solution A. In a 400-mL beaker, carefully moisten 1.00 g of 99.9 % nickel metal with a small amount of water. Add 10 mL of HCl to dissolve the nickel and transfer the solution to a 1-L volumetric flask. Dilute to the mark and mix. Transfer the solution into a polyethylene bottle. Prepare fresh as needed upon failure of the control standard solution performance (see 26.2.4). A commercially available atomic absorption nickel reference standard solution of the same matrix and nickel concentration is also satisfactory.

24.5 *Nickel Control Standard Solution* (1 mL = 0.015 mg Ni)—Transfer 3 mL of the Nickel Control Standard Stock Solution by pipet into a 200-mL volumetric flask. Add 2 mL of HCl, dilute to the mark with water, and mix. Transfer the solution into a polyethylene bottle. Prepare fresh as needed upon failure of control standard solution performance (see 26.2.4).

25. Instrument Performance and Linearity Check

25.1 To each of six, 100-mL volumetric flasks pipet 0, 3, 6, 9, 12, and 15 mL of the Nickel Standard Solution B. Add 1 mL of HCl to each, dilute to the mark, and mix. The resulting solutions will have nickel concentrations of 0, 3.0, 6.0, 9.0, 12.0, and 15.0 mg/L, respectively.

25.2 *Photometry*:

25.2.1 With the nickel hollow-cathode lamp in position, energized, and stabilized, adjust the wavelength to maximize the energy response of the 231.1-nm line.

25.2.2 Following the manufacturer's instructions, light the burner, allow it to reach thermal equilibrium, and adjust the instrument to zero while aspirating water. Aspirate the nickel solution with the highest concentration from the series prepared in 25.1 and adjust the burner position, air and fuel pressures, and flow rates and solution aspiration rates to obtain maximum response (Note 7).

NOTE 7—Recalibrate whenever one or more of these parameters are changed.

25.2.3 Aspirate the nickel solution used in 25.2.2 a sufficient number of times to ensure that the absorbance reading is repeatable. Record six readings, and calculate an estimate of the standard deviation, s, of the readings (if not provided from the microprocessor of the instrument) as follows:

$$s = (A - B) \times 0.40 \tag{4}$$

where:

s = standard deviation,
A = highest of the six values, and
B = lowest of the six values found.[12]

Repeat the procedure for the remaining calibration solutions prepared in 25.1. If the standard deviation is found to be greater than 0.005, repeat the measurement. If a problem is indicated, determine the cause, take appropriate corrective measures, and repeat 25.2.1-25.2.3.

25.2.4 *Curve Linearity*—Aspirate the nickel solution used in 25.2.2 to ensure that the absorbance reading is stable. Read and record the absorbance readings of each of the six calibration solutions in 25.1. Plot the absorbance readings against the concentration of the solutions. Check that the difference between the absorbance readings of the two highest calibration solutions (12 and 15 mg/L) is more than 0.7 times the difference between the blank solution and the lowest calibration solution (3 mg/L). If the linearity check fails, check whether the solutions are prepared correctly or there is evidence for instrument malfunction; correct it if present, and repeat the measurements (Note 8). In case no problem is found and still the linearity check fails, proceed with the test method but use only the linear portion of the calibration curve. If the sample concentration exceeds the linear range of the instrument, report the result as greater than the upper concentration limit of the linear range.

NOTE 8—Curve linearity needs to be done only once for each instrument upon start-up and repeated only when significant changes are made to the instrument.

26. Procedure

26.1 *Test Solution*—Pipet 5 mL of the sample solution reserved in 15.7 into a 100-mL volumetric flask, dilute to the mark, and mix.

26.2 *Photometry*:

26.2.1 Calibrate the instrument in accordance with the manufacturer's instructions in the concentration mode in the range from 0 to 15 mg/L using the required number of calibration standards from 25.1. In order to verify the calibration, read the calibration standard solutions used to calibrate the instrument as unknown and check that the readings fall within ±2 % of their true values. If the readings do not fall within ±2 %, find the problem, correct it, and recalibrate the instrument. When the calibration is verified, read a maximum of eight unknown sample solutions, followed by the nickel control standard solution (see 24.5). Repeat this sequence as needed to complete the set of samples. At the end read the calibration standard solutions which were used for calibration.

26.2.2 Record the average of two readings of each solution as nickel in mg/L.

26.2.3 When using an automatic sampler, load the auto sampler carousel in the same sequence in accordance with 26.2.1 (Note 9).

NOTE 9—Prepare the auto-sampler and printer for operation in accordance with the manufacturer's instruction manual.

26.2.4 If the readings of the calibration standards read at the end as unknowns are not within ±2 % of their true values,

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[12] The value 0.40, which is used to estimate the standard deviation from the range of six values, was published by Dixon, W. J., and Massey, F. J., *Introduction to Statistical Analysis*, McGraw-Hill, 1957, Table 8b(1), p. 404.

recalibrate the instrument and run the samples. Record the instrument reading for the nickel control standard solution (see 24.5) for control charting. The value for the nickel control standard (see 24.5) must be within the range of the control limits (Note 10). If the point is out of control, determine the cause, correct it, recalibrate the instrument, and rerun the samples.

NOTE 10—In absence of an existing control chart, assume upper and lower limits for the instrument reading of the nickel control standard solution (see 23.5) to be ±2 % of the true value.

27. Calculation

27.1 Calculate the percent nickel as follows:

$$\text{Nickel, \%} = 0.2N\,M \tag{5}$$

where:

0.2 = constant with dimensions of gL/mg,
M = mass of stripped coating, g, from 16.1, and
N = nickel concentration read by the instrument, mg/L.

28. Report

28.1 Report the percent nickel to the nearest 0.1 %.

29. Precision and Bias [10,11]

29.1 *Precision*—Ten laboratories, one of which reported a second pair of values, participated in the original testing of this test method and obtained the data summarized in Table 4. All testing met the requirements of Practice E 173. The data in Tables 5 and 6 were generated by one laboratory comparing the effectiveness of the stripping solution containing hexamethylene tetramine as a replacement for NEP312-S (used in E 1659 – 95), an inhibitor that is no longer commercially available.

29.1.1 The effectiveness of the current stripping solution containing hexamethylene tetramine was compared to that of the original stripping solution by analyzing specimens from each of two test materials.[11] The data summarized in Tables 5 and 6 shows that the precision obtained when using the current stripping solution is at least as good as that obtained when using the original stripping solution, and, therefore, a complete interlaboratory test of the revised procedure was not carried out.

TABLE 4 Statistical Information—Nickel

Test Material	Number of Laboratories Included	Nickel Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
20	9	12.18	0.99	1.43
30	10	11.52	0.64	0.96
40	9	12.29	0.81	1.13

TABLE 5 Statistical Information—Comparison of Stripping Solutions on 20/20 Material

Test Material	Original Stripping Solution, %Ni	Current Stripping Solution, %Ni
1	11.25	11.38
2	11.20	11.30
3	11.37	11.33
Average	11.27	11.34
2 Standard Deviations	0.175	0.081

TABLE 6 Statistical Information—Comparison of Stripping Solutions on 30/30 Material

Test Material	Original Stripping Solution, %Ni	Current Stripping Solution, %Ni
1	11.44	11.74
2	11.40	11.76
3	11.42	11.68
4	11.20	11.85
Average	11.37	11.76
2 Standard Deviations	0.222	0.141

29.2 *Bias*—Table 5 shows that the results obtained from using the current stripping solution are comparable to that of the old stripping solution when tested on the 20/20 material. Table 6 shows there was a 0.4 % difference when comparing the two stripping solutions. This difference is believed to have been due to the test material rather than the testing protocol. During the study, there was no material of known homogeneity sufficient to test this further.

30. Keywords

30.1 atomic absorption spectrophotometry; coated steel sheet; coating weight; nickel; stripping solution; weigh-strip-weigh; zinc