Designation: E1659 – 12

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

This standard is issued under the fixed designation E1659; 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 mass	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 mass, by the Weigh-Strip-Weigh Method (20.0 to 45.0 g/m^2)	10-20
Nickel by the Atomic Absorption Method (11.0 to 13.5 % of Coating mass Ranging from 20 to 45 g/m^2)	21-31

1.3 The values stated in SI units are to be regarded as standard. In some cases, exceptions allowed in Practice E380 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:*[2]

A917 Specification for Steel Sheet, Coated by the Electrolytic Process for Applications Requiring Designation of the Coating Mass on Each Surface (General Requirements)

A918 Specification for Steel Sheet, Zinc-Nickel Alloy Coated by the Electrolytic Process for Applications Requiring Designation of the Coating Mass on Each Surface

D1193 Specification for Reagent Water

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

E380 Practice for Use of the International System of Units (SI) (the Modernized Metric System) (Withdrawn 1997)[3]

E663 Practice for Flame Atomic Absorption Analysis (Withdrawn 1997)[3]

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry (Withdrawn 2004)[3]

E1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analysis (Withdrawn 2005)[3]

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E135.

4. Significance and Use

4.1 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 A918, particularly those under the jurisdiction of ASTM Committee A05 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

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

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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] The last approved version of this historical standard is referenced on www.astm.org.

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 E882.

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

5. Apparatus, Reagents, and Instrumental Practices

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

5.2 *Reagents:*

5.2.1 *Purity of Reagents*—Unless otherwise indicated, all reagents used in these test methods shall conform to the Reagent Grade Specifications of the American Chemical Society.[4] 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 30.

5.2.2 *Purity of Water*—References to water shall be understood to mean reagent water as defined by Type II of Specification D1193.

5.3 *Photometric Practice*—Photometric practice prescribed in these test methods shall conform to Guide E1024 and Practice E1452.

6. Safety Hazards

6.1 For precautions to be observed in the use of certain reagents and equipment in these test methods, refer to Practices E50.

7. Sampling

7.1 *Zinc-Nickel Alloy Coated Sheets*—Samples for determining mass and composition of coating shall be secured in accordance with Specification A917, which is referred to in Specification A918. 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."

8. Interlaboratory Studies and Rounding Calculated Values

8.1 Calculated values shall be rounded to the desired number of places in accordance with the rounding method of Practice E29.

9. Interlaboratory Studies

9.1 These test methods have been evaluated using Practice E173, except for the update in the stripping solution, 17.1, 17.8, 20.1.2, 20.2, 31.1.1, and 31.2 as well as Table 1, Table 2, Table 3, and Table 4.

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

10. Scope

10.1 This test method provides a procedure for determining independently the mass 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 mass were not available for testing in accordance with Practice E173. 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 mass determinations above 45 g/m^2 is not supported by interlaboratory testing.

11. Summary of Test Method

11.1 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 mass is determined from the mass difference of the specimen before and after stripping.

12. Interferences

12.1 The hexamethylene tetramine inhibitor used in this test method permits the dissolution of some base metal, which could lead to higher than expected coating mass 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.

TABLE 1 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 17.8, expressed in g/m^2.

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

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[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.

TABLE 2 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 17.8, expressed in g/m^2.

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

TABLE 3 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 4 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

13. Apparatus

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

13.2 *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 mass determination by gaining or losing mass.

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

14. Reagents

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

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

15. Precautions

15.1 **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.

16. Sample Preparation

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

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

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

16.4 Trim off the excess tape.

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

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

17. Procedure

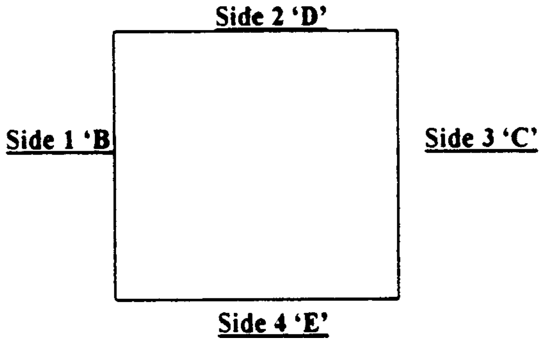
17.1 *Specimen Area*—Using the calipers, measure and record the length of all four sides of the test specimen.

17.1.1 Check that the measuring face and reference edge of the calipers are clean. Check that the calipers read "0" when the measuring surface is closed. If it does not, correct the problem according to the manufacturer's instructions.

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

17.1.3 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 18.2, the average of opposite sides is calculated and used to determine specimen area.



17.2 Weigh the prepared specimen to the nearest 0.1 mg and record the mass as the original mass of the specimen.

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

17.4 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 mass of the coating.

17.5 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.

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

17.7 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.

17.8 *Determination of Stripped Iron:*

17.8.1 Calibrate the instrument in accordance with the manufacturer's instructions following the guidelines set forth in Guide E1024 and Practice E1452. Choose the appropriate wavelength and calibration solutions that provide a calibration curve that includes up to 2.0 mg Fe/100 mL of solution.

17.8.2 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 17.7 for iron.

17.8.3 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 17.8.1-17.8.3. 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 mass of about 1 g/m^2. Paragraph 17.8 allows a 10 % error in this Fe determination, which is equal to a coating mass of 0.1 g/m^2. On a sample with the minimum coating mass of 20 g/m^2, this is an error in the coating mass determination of 0.5 %. At higher coating weights, the percent error would decrease. The reproducibility of the test method by Practice E173 at a coating mass 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 17.8 did not undergo a formal interlaboratory evaluation of precision and bias.

18. Calculation

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

$$M = [(W_1 - W_2) - (E/1000)] \quad (1)$$

where:

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

18.2 Calculate the area of the test specimen as follows:

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

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.

18.3 Calculate the coating mass (W_c) in g/m^2 as follows:

$$W_c = M/A \quad (3)$$

where:

W_c = coating mass, g/m^2,
M = mass of stripped coating, g, from 18.1, and
A = area of test specimen, m^2, from 18.2.

19. Report

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

20. Precision and Bias[5, 6]

20.1 *Precision:*

20.1.1 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 5. The data in Tables 1 and 2 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 E1659 – 95), an inhibitor that is no longer commercially available. Along with a revised stripping solution, this 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 17.1.

20.1.2 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 1 and 2 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 mass was comparable for both stripping solutions.

20.2 *Bias*—Using the hexamethylene tetramine (14.1) as the new inhibitor, the coating mass 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 mass result accordingly. The data, summarized in Columns 2 and 5 of Tables 1 and 2 shows that there is no bias between stripping solutions after adjusting the coating mass for stripped iron.

TABLE 5 Statistical Information—Coating Mass-Original Test Data from E1659 – 95

Test Material	Number of Laboratories Included	Average Coating mass, g/m^2	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
20	9	22.93	1.06	1.56
30	10	36.52	1.76	1.92
40	9	41.78	1.00	1.91

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:E01-1009.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:E01-1028.

20.3 Practice E173 has been replaced by Practice E1601. The Reproducibility Index R_2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. Likewise the Repeatability Index R_1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601.

NICKEL BY THE ATOMIC ABSORPTION METHOD

21. Scope

21.1 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 E173. 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.

22. Summary of Test Method

22.1 The solution from the coating mass 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.

23. Concentration Range

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

24. Interferences

24.1 There are no known interferences.

25. Apparatus

25.1 *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 E663 and Guide E1024.

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

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

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

26. Reagents

26.1 *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.

26.2 *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 28.2.4).

26.3 *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 28.2.4).

26.4 *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 28.2.4). A commercially available atomic absorption nickel reference standard solution of the same matrix and nickel concentration is also satisfactory.

26.5 *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 28.2.4).

27. Instrument Performance and Linearity Check

27.1 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.

27.2 *Photometry:*

27.2.1 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.

27.2.2 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 27.1 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.

27.2.3 Aspirate the nickel solution used in 27.2.2 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:

$$s = (A - B) \times 0.40 \quad (4)$$

where:

s = standard deviation,

A = highest of the six values, and
B = lowest of the six values found.[7]

Repeat the procedure for the remaining calibration solutions prepared in 27.1. 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 27.2.1-27.2.3.

27.2.4 *Curve Linearity*—Aspirate the nickel solution used in 27.2.2 to ensure that the absorbance reading is stable. Read and record the absorbance readings of each of the six calibration solutions in 27.1. 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.

28. Procedure

28.1 *Test Solution*—Pipet 5 mL of the sample solution reserved in 17.7 into a 100-mL volumetric flask, dilute to the mark, and mix.

28.2 *Photometry:*

28.2.1 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 27.1. 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 26.5). Repeat this sequence as needed to complete the set of samples. At the end read the calibration standard solutions which were used for calibration.

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

28.2.3 When using an automatic sampler, load the auto sampler carousel in the same sequence in accordance with 28.2.1 (Note 9).

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

28.2.4 If the readings of the calibration standards read at the end as unknowns are not within ±2 % of their true values, recalibrate the instrument and run the samples. Record the instrument reading for the nickel control standard solution (see 26.5) for control charting. The value for the nickel control standard (see 26.5) 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.

29. Calculation

29.1 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 18.1, and
N = nickel concentration read by the instrument, mg/L.

30. Report

30.1 Report the percent nickel to the nearest 0.1 %.

31. Precision and Bias[5, 6]

31.1 *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 6. All testing met the requirements of Practice E173. The data in Tables 3 and 4 were generated by one laboratory comparing the effectiveness of the stripping solution containing hexamethylene tetramine as a replacement for NEP312-S (used in E1659 – 95), an inhibitor that is no longer commercially available.

31.1.1 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.[6] The data summarized in Tables 3 and 4 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.

31.2 *Bias*—Table 3 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 4 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.

TABLE 6 Statistical Information—Nickel

Test Material	Number of Laboratories Included	Nickel Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
20	9	12.18	0.99	1.43
30	10	11.52	0.64	0.96
40	9	12.29	0.81	1.13

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[7] 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.

During the study, there was no material of known homogeneity sufficient to test this further.

31.3 Practice E173 has been replaced by Practice E1601. The Reproducibility Index R_2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. Likewise the Repeatability Index R_1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601.

32. Keywords

32.1 atomic absorption spectrophotometry; coated steel sheet; coating mass; nickel; stripping solution; weigh-strip-weigh; zinc

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