



Designation: E1659 – 05

Standard Test Methods for Coating Weight and Chemical Analysis of Zinc-Nickel Alloy Electrolytically Coated on Steel Sheet¹

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 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)	10-20
Nickel by the Atomic Absorption Method (11.0 to 13.5 % of Coating Weight 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 Metals³
- E380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)³
- E663 Practice for Flame Atomic Absorption Analysis
- 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 Spectrophotometry³
- E1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analysis³
- 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 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 weight and composition are

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 Withdrawn.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

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 weight 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.

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 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/m².

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

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/m².

^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 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

WEIGHT 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 weight of coating on each surface of zinc-nickel alloy-coated sheet steel, in coating masses from 20 to 45 g/m² (Note 1).

NOTE 1—The upper limit of the scope has been set at 45 g/m² because test materials with higher coating weight were not available for testing in accordance with Practice E173. However, recognizing the simplicity of

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

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/m² 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 weight is determined from the weight 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 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.

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 weight determination by gaining or losing weight.

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

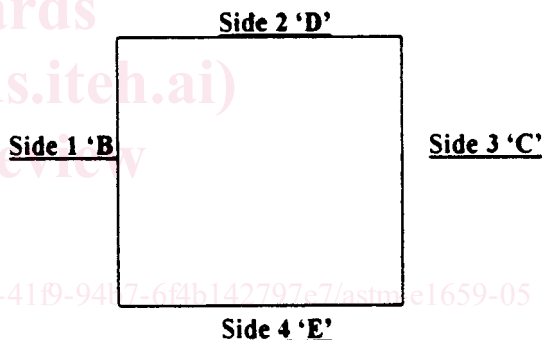
17.1 *Specimen Area*—Using the calipers, measure and record the length of all four sides of the test specimen.

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 weight as the original weight 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 weight 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 weight.

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.