Designation: A 239 – 95 (Reapproved 2004)

Standard Practice for Locating the Thinnest Spot in a Zinc (Galvanized) Coating on Iron or Steel Articles[1, 2]

This standard is issued under the fixed designation A 239; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice covers the procedure for locating, by the use of a solution of copper sulfate, the thinnest spot in a zinc coating (hot dipped, electroplated, or sprayed) on iron or steel articles that are coated after the shape is produced by casting, drawing, pressing, or other forming methods. Examples are: electrical metallic tubing and rigid conduit pipe, castings and forgings, and structural steel; on special hardware, such as poleline, builder's, and farm implement hardware; bolts, nuts, screws, and other miscellaneous general hardware.

1.2 The use of this practice with zinc coating deposited through different processes (such as hot dipped, electroplated, or sprayed) requires caution in interpretation since the end point may vary considerably between different zinc-coating systems.

1.3 Excluded from this practice is sheet steel from hot-dip or electrocoating lines as the sheet products are normally subject to additional forming after the coating process. Also excluded from this practice are all zinc-coated wire and wire products either continuously or batch coated before or after forming. **Warning—**Past research (dating from around 1963) has indicated that this practice can be influenced by operator technique. Variations can be due to the difference in hand pressure used to wipe the sample or the inability of the operator to recognize the end point.

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. Significance and Use

2.1 This practice is designed to locate the thinnest portions of the zinc coating on newly coated items (see Appendix X1). Variations in coating thickness can be due to the process by which the zinc is applied (hot dipped, electroplated, or sprayed) or by the geometry of the part that is coated. During hot-dip galvanizing, the coating thickness is affected by the drainage pattern of the molten zinc, while during zinc spraying (metallizing), coating thickness can be dependent on the operator's manipulation of the spray nozzle. The geometry of the part can also influence coating thickness especially during hot-dip galvanizing, where peaks and valleys on the part can cause molten zinc to build up or thin out. This practice is designed to identify those areas of the part where the coating is thin.

3. Copper Sulfate Solution

3.1 The copper sulfate solution shall be made by dissolving approximately 36 parts by weight of commercial cupric sulfate crystals ($CuSO_4 \cdot 5H_2O$) in 100 parts by weight of distilled water (see 3.1.1). Heat may be used to complete the solution of cupric sulfate crystals. If heated, the solution shall be allowed to cool. The solution shall then be agitated with an excess of powdered cupric hydroxide ($Cu(OH)_2$), about 0.13 oz/gal [1.0 g/L] of solution. The presence of an excess of cupric hydroxide will be shown by the sediment of this reagent at the bottom of the vessel. The neutralized solution shall be allowed to stand for 24 h and then filtered or decanted.

3.1.1 Cupric oxide (CuO), 0.11 oz/gal [0.8 g/L] may be substituted for cupric hydroxide, provided the solution is allowed to stand not less than 48 h after this addition before decanting or filtering.

3.2 The solution shall have a specific gravity of 1.186 at 18°C [65°F]. To adjust a solution of improper specific gravity, add distilled water when the specific gravity is high, and add a

[1] This practice is under the jurisdiction of ASTM Committee A05 on Metallic-Coated Iron and Steel Products and is the direct responsibility of Subcommittee A05.07 on Methods of Testing.

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[2] This practice is used to locate the thinnest spot only, and is not intended as a test for coating weight. This practice is to be used only within the limits of Section 1, Scope.

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copper sulfate solution of a higher specific gravity when the solution is low in specific gravity.

4. Specimen Rinse Water

4.1 Ordinary, clean tap water may be used for rinsing specimens. If no running tap water is available, the rinse water shall be changed after each dip. The temperature of the rinse water shall be from 15 to 21°C [60 to 70°F].

5. Quantity of Copper Sulfate Solution

5.1 *Hardware and Similar Articles*— For evaluating hardware and similar articles, the quantity of copper sulfate solution required for each evaluation will depend on the superficial area of the specimen being evaluated and the weight of coating. The quantity should not be less than 1.2 qt/oz/ft^2[40.5 mL/g/m^2] of zinc coating on the specimen (Note 1) and shall be sufficient to cover the specimen so that the top surface of the solution is at least ½ in. [12.7 mm] above the top of the section of the specimen under evaluation. The solution shall be discarded after completion of the evaluation, and fresh solution shall be used for any additional evaluations.

NOTE 1—The theoretical minimum amount of copper sulfate solution required to dissolve zinc is 0.4 qt/oz [13.5 mL/g]. The quantity specified in 5.1 is three times this minimum to ensure adequate rate of solution.

6. Preparation of Specimens

6.1 The specimens selected for evaluation shall be free of abrasion or cuts in the zinc coating, except those which may occur during manufacture of the specimen. Where the area of uncoated surface to be immersed is more than 10 % of the total immersed surface area, precautions, such as plugs for tubular material, or lacquer, paraffin, or other suitable coatings for the uncoated surfaces must be taken so as not to deplete the strength of copper sulfate solution.

6.2 Clean the specimens with a suitable volatile organic solvent and finally, thoroughly wash with clean water and wipe dry with a clean cotton cloth. Unless otherwise specified, remove lacquer or varnish coatings with a suitable clean, volatile organic solvent such as acetone, which will not attack the zinc coating or leave a greasy or waxy deposit. Thoroughly rinse the specimens in clean water and wipe dry with a clean cotton cloth. Bring the specimens to a temperature between 15 and 21°C [60 and 70°F] prior to the beginning of the immersion series.

6.3 Abnormal cases may arise when, by reason of unusual surface conditions, the copper sulfate solution will not act normally on the zinc coating. For example, the solution may have no apparent attack on all or part of the surface, or false deposits of copper may appear on the zinc coating. If there is any question of abnormality of performance of specimens, discard the specimens and select new ones. Clean the new specimens in alcohol, rinse, and wipe dry. Then immerse for 3 min in a solution consisting of 1 part by volume of ammonium hydroxide (sp gr 0.90) and 9 parts of water. The specimens may be scrubbed with a cotton cloth during this immersion. After cleaning, wash the specimens and wipe dry, then subject them to the procedure described in Section 7.

7. Procedure

7.1 Immerse the specimens in the copper sulfate solution, which shall be maintained at a temperature of 16 to 20°C [61 to 68°F]. Allow the specimens to remain in a fixed position in the solution for exactly 1 min. Do not agitate the solution during the immersion period, nor allow the specimens to touch each other or the sides of the container. After each 1-min immersion, wash the specimens immediately in the rinse water, and use a fiber bristle brush to remove any copper deposit that may have formed on the zinc coating. Before returning the specimens to the copper sulfate solution, drain thoroughly of excess rinse water and wipe dry with a clean cloth.

7.2 Continue successive immersions of 1 min each, washing and wiping the specimens after each immersion until the end point has been reached (see Appendix X2).

NOTE 2—Zinc coatings on threads are usually difficult to test. It has been found advantageous to saw female-threaded parts longitudinally, exposing the threads to full view and facilitating cleaning to remove sponge copper after each immersion in the copper sulfate solution. Brushing should be parallel to the threads.

8. End Point

8.1 The end point shall be recognized by the appearance of bright, adherent copper deposits. However, if such a deposit fails to form after the complete disappearance of the zinc coating, the immersion succeeding the disappearance of the zinc shall be recognized as representing the end point.

8.2 Confirm adherence of the bright copper deposit by a peeling test. If it is possible to remove the copper with an ink eraser or to peel the copper with the edge of a blunt tool such as the back of a knife blade, and zinc appears underneath the copper, such an appearance of deposited copper shall not be construed as an end point.

8.3 A fine line appearance of copper on the top of screw threads or on sharp edges of articles, or within 1 in. [25 mm] of a cut portion of a specimen, shall not be judged as an end point. Likewise, the indication of bright adherent deposits of copper at or adjacent to any cut or abrasion present on the original specimen shall not be considered an end point.

9. Supplementary Tests

9.1 If at any time during the immersion procedure there is any doubt as to the presence of exposed base metal, as determined by visual inspection, there are several supplementary tests described in Section 10, one or more of which may be used depending on the type of coating being evaluated.

10. Supplementary Test for All Types of Zinc Coatings

10.1 *Microscopical Test*—Section the specimen through the copper deposit, mount, and polish it for metallographic analysis. Etch the polished surface using an etching solution composed of 20 g of chromic acid, 1.5 g of sodium sulfate, and 100 mL of distilled water (Palmerton reagent). After etching, wash the specimen with alcohol. Examine the etched specimen under a microscope, using a magnification of 100 diameters or greater if necessary.

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10.2 *Qualitative Test for Zinc*[3]—Apply a drop (or several drops) of diluted hydrochloric acid to the area in question (depending on its size). The presence of zinc is indicated by immediate vigorous effervescence (evolution of hydrogen). If no appreciable zinc is present, the effervescence will be mild. By carefully removing the acid, a confirming test for zinc may be made as follows: neutralize the acid with ammonium hydroxide, acidify with acetic acid, and pass hydrogen sulfide into the solution; a white precipitate (zinc sulfide, ZnS) confirms the presence of zinc.

[3] Only an outline of the test is given. The details of the test may be found in technical literature.

11. Keywords

11.1 bolts; electroplated; hardware; hot dipped; nuts; screws; thinnest spot; zinc coating thickness

APPENDIXES

(Nonmandatory Information)

X1. APPLICATION OF THIS PRACTICE TO WEATHERED GALVANIZED WARE

X1.1 This practice is not applicable to aged or weathered material because of the corrosion film present on the zinc coating. If it is desired to use this practice on such material, the corrosion film should be removed before conducting this procedure by immersing the specimens for 3 min in an ammonium hydroxide solution (1 part by volume of ammonium hydroxide (sp gr 0.90) to 9 parts by volume of distilled water) then rinsing the specimens in clean water and wiping them dry.

X2. STANDARD FOR COMPARISON

X2.1 If it is desired to show the character of the bright metallic copper deposit on an exposed iron or steel surface, a reference standard for comparison may be prepared as follows: Partially submerge a zinc-coated specimen in strong hydrochloric acid until violent action ceases. Immediately remove the specimen, wash, and wipe it dry. The specimen with an area of bare surface thus exposed, including a portion with zinc coating intact, is then dipped for a few seconds in the copper sulfate solution at a temperature of 16 to 20°C [61 to 68°F], removed, washed, and wiped dry. This copper-coated reference standard should be prepared at the time this practice is being conducted.

X3. ACTION OF COPPER SULFATE SOLUTION ON ZINC COATINGS

X3.1 The fact that this practice cannot be used for determining the weight of zinc coating is due to the wide variation in the rate of solubility of the various types of coating resulting from differences in the composition and structure of the coatings. For instance, electroplated and sprayed-zinc coatings consist essentially of pure zinc; the outer part of hot-dipped coatings consists of nearly pure zinc; whereas, the portion next to the steel base is composed of zinc-iron alloys; in the case of hot-dipped coatings heated after galvanizing, the coating consists almost entirely of zinc-iron alloys.

X3.2 In view of these differences in the makeup of the zinc coatings applied by the different processes, it has been found by Groesbeck and Walkup[4] in tests on a limited number of samples that electroplated and flame-sprayed coatings, by reason of their high pure-zinc content, dissolve rather rapidly in the copper sulfate solution; that the conventional hot-dip coatings dissolve only about two thirds as fast as the pure zinc coatings; and that the hot-dip coatings heated after galvanizing dissolve only about half as fast as the pure zinc coatings.

[4] For further information on this test method, reference may be made to the following papers:

Walkup, H. H., and Groesbeck, E. C., "Some Factors Affecting the Preece Test for Zinc Coatings," *Proceedings*, ASTM, Vol 32, Part II, 1932, p. 453.

Groesbeck, E. C., and Walkup, H. H., "Preece Test (Copper Sulphate Dip) for Zinc Coatings," *National Institute of Standards and Technology Journal of Research*, Vol 12, No. 6, June 1934, p. 785 (Research Paper RP688).

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