Designation: E 866 – 00

An American National Standard

Standard Specification for Corrosion-Inhibiting Adhesive Primer for Aluminum Alloys to Be Adhesively Bonded in Honeycomb Shelter Panels[1]

This standard is issued under the fixed designation E 866; 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 specification is for sprayable, pigmented liquid primers for use on aluminum alloys that are to be adhesively bonded in the fabrication of honeycomb sandwich panels for tactical shelters. When applied to a properly cleaned surface of aluminum alloy, the primer imparts corrosion resistance and forms a surface suitable for structural bonding using adhesives complying with Specification E 865 and for coating with shelter paint finishes.

1.2 The values stated in SI units are to be regarded as the standard where only SI units are given or where SI units are given first followed by inch-pound units; where inch-pound units are given first followed by SI units, the inch-pound units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

B 117 Practice for Operating Salt Spray (Fog) Apparatus[2]

D 522 Test Methods for Mandrel Bend Test of Attached Organic Coatings[3]

D 1002 Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal)[4]

D 3167 Test Method for Floating Roller Peel Resistance of Adhesives[4]

E 864 Practice for Surface Preparation of Aluminum Alloys to be Adhesively Bonded in Honeycomb Shelter Panels[5]

E 865 Specification for Structural Film Adhesives for Honeycomb Sandwich Panels[5]

E 1749 Terminology of Rigid Wall Relocatable Shelters[5]

2.2 *Federal Specifications:*

QQ-A-250/8d Aluminum Alloy 5052H34, Plate and Sheet[6]

QQ-A-250/11d Aluminum Alloy 6061T6, Plate and Sheet[6]

3. Materials and Manufacture

3.1 The primer shall be a pigmented liquid composed of a modified epoxy resin system, compounded so that it can be spray-applied to produce a continuous uniform coating without addition of solvent.

4. Physical Requirements

4.1 The uncured primer must meet the requirements of Table 1. The cured primer must meet the requirements of Table 2 and Table 3. These requirements shall be verified by tests described in Section 5.

4.2 *Properties of Cured Film*—When applied and cured in accordance with 5.1.7, the cured film shall meet the requirements of Table 2 and Table 3 when tested as specified in 5.2-5.3.8.

4.3 *Storage Stability*—Primer stored in accordance with the manufacturer's instructions shall meet the requirements of Table 1 and when cured in accordance with 5.1.7 shall meet the requirements of Table 2 and Table 3 when tested as specified in 5.2-5.3.8.

4.4 *Shelf Life*—The shelf life of the primer at 25 ± 7°C (77 ± 12°F) shall be at least four days. After four days, when tested in accordance with 5.1.4, the primer shall be capable of meeting all of the requirements of this specification.

4.5 *Film Thickness*—The film thickness for all tests shall be between 0.003 and 0.007 mm (0.0001 and 0.0003 in.).

5. Test Methods

5.1 *Uncured Primer*:

5.1.1 *Solids Content*—Determine the solids content by heating a 5 g sample of thoroughly mixed primer in an aluminum weighing dish in a circulating air oven at 105 ± 2°C (221 ± 3°F) for not less than 3 h. Calculate the mass of solids

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[2] *Annual Book of ASTM Standards*, Vol 03.02.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 15.06.

[5] *Annual Book of ASTM Standards*, Vol 04.11.

[6] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

TABLE 1 Physical Properties of Uncured Liquid Primer

Test	Requirement
Solids content, %	10 ± 1
Inhibitor Content,[A] %	15 ± 3
Color	easily visible film
Sprayability	film is of uniform thickness and color

[A] Based on mass of nonvolatile content.

TABLE 2 Physical Properties of Cured Film on Primed Surfaces

Test	Requirement
Adhesion to metal	no cracking or loss of adhesion
Impact resistance	no cracking or loss of adhesion (on either side)
Pencil hardness	4H minimum
Water resistance	no blistering, cracking, softening, or loss of adhesion
Corrosion resistance	no blistering, cracking, or substrate degradation more than 3 mm from scribe mark
Humidity resistance	no blistering, cracking, or loss of adhesion
Heat resistance	no blistering, cracking, softening, or loss of adhesion
Low-temperature shock	no cracking or loss of adhesion

TABLE 3 Physical Properties of Bonded Specimens

Test	Requirements (min)
Normal-temperature lap shear, 25°C (77°F), MPa (psi)	20.0 (2900)
Low-temperature lap shear, −55°C (−67°F), MPa (psi)	20.0 (2900)
High-temperature lap shear, 93°C (200°F), MPa (psi)	13.0 (1890)
Humidity-exposure lap shear, 93°C (200°F), MPa (psi)	5.0 (725)
Salt-spray exposure lap shear, 35°C (95°F), MPa (psi)	16.0 (2320)
Normal-temperature floating roller peel strength, 25°C (77°F), N/m (lbf/in.)	4400 (25.1)
Low-temperature floating roller peel strength, −55°C (−67°F), N/m (lbf/in.)	2625 (15.0)

remaining as a percentage of the initial sample mass. Weighings before and after heating shall be accurate to ±0.001 g.

5.1.2 *Inhibitor Content*—Determine the inhibitor content in accordance with the following procedure (burn out method):

5.1.2.1 Tare four ignition loss crucibles and covers.

5.1.2.2 Pipette 2 mL portions of primer (1.75 g) from a well mixed sample into each crucible, add about 2 mL of MEK to each, replace the lids, and devolatize the primer at 121 ± 3°C (250 ± 5°F) for 60 ± 5 min.

5.1.2.3 Cool to room temperature in a desiccator, weigh, and compute the mass of the nonvolatiles. The nonvolatile content requirement is 0.155 to 0.195 g/2 mL (10 ± 1 % by weight).

5.1.2.4 Place the crucibles with lids (and residue) in a furnace at 593°C (1100°F) for 60 ± 5 min.

5.1.2.5 Cool to room temperature in a desiccator, weigh, and compute average mass of the inhibitor. The inhibitor content requirement is 0.025 ± 0.003 g/2 mL (15 ± 3 % by mass of nonvolatile content).

5.1.3 *Storage Stability*—Store a sample of the primer for six months from the date of shipment at −18°C (0°F), or two months at 5°C (41°F). Test the sample for ability to meet the requirements of Tables 1-3.

5.1.4 *Shelf Life*—Store a sample of the primer for four days at 25 ± 7°C (77 ± 12°F) and then test for ability to meet the requirements listed in Tables 1-3.

5.1.5 *Color*—Spray the primer onto an aluminum alloy panel, leaving a portion of the panel bare. Compare the coated and uncoated areas visually against the color requirements in Table 1.

5.1.6 *Sprayability*—The primer shall be capable of being readily applied in accordance with the manufacturer's instructions to all test panels of either 6061T6 or 5052H34 aluminum alloy (Fed. Spec. QQ-A-250/8d or QQ-A-250/11d, respectively) and cleaned in accordance with Practice E 864 for each of the tests described in 5.1.7-5.3.8. Inspect the coating for uniformity of thickness and color.

5.1.7 *Curing Properties*—Spray the primer onto a test panel 100 by 150 by 0.05 mm (4 by 6 by 0.020 in.) and air dry for at least 30 min at 25 ± 5°C (77 ± 9°F); then heat in air for 75 to 90 min at 115 ± 5°C (239 ± 9°F). Test the cured film for ability to meet the requirements listed in Table 2.

5.2 *Cured Film*:

5.2.1 *Adhesion to Metal*—Test the adhesion of the primer to the metal by bending a primed metal sample over a conical mandrel in accordance with Test Methods D 522, except use an aluminum alloy panel.

5.2.2 *Impact Resistance*—Determine the impact resistance of both the coated and uncoated side of a panel by subjecting the panel to the impact of a 0.45 kg (1 lb) mass dropped from a height of 1 m (40 in.) using a Gardner 160-in·lbf capacity impact testing machine or equivalent. Use the 0.45 kg (1 lb) falling weight. Apply a strip of masking tape[7] over the most highly stressed area on the coated side of each impact spot. Remove the tape in one abrupt motion by pulling perpendicular to the panel.

5.2.3 *Pencil Hardness*—Determine the pencil hardness of the cured film using the following procedure:

5.2.3.1 *Preparation of Pencils*—Prepare a set of drawing pencils[8] ranging in hardness from 6B to 5H by stripping the wood away from the end approximately 10 mm (⅜ in.) without damaging the lead. Square the tip of the lead by holding the pencil in a vertical position and moving the lead back and forth over 400-grit or finer abrasive paper. Square the tip of the lead after each trial.

5.2.3.2 *Procedure*—Place the test panel with the cured primer applied in a horizontal position. Push pencils of increasing hardness into the coated surface of the panel at a 45° angle until one is found that will cut or scratch the coating. Use the number of this pencil to express the primer hardness.

5.2.4 *Environmental Resistance Test*—For each test described in 5.2.5-5.2.9, clean five test panels of approximately 75 by 125 mm (3 by 5 in.) of either 6061T6 or 5052H34 aluminum alloy (Fed. Spec. QQ-A-250/8d or QQ-A-250/11d, respectively) in accordance with Practice E 864. Apply the primer and cure in accordance with 5.1.7. These panels when tested in accordance with the procedures described in 5.2.5-5.2.9 shall meet the requirements of Table 2

5.2.5 *Water Resistance*—Immerse the panels with the cured primer applied in distilled water for seven days at 24 ± 3°C (75

[7] No. 250 masking tape, 3M Co., has been found satisfactory.

[8] KOH-1-NOOR 1500, Venus Drawing Pencil, A. W. Faber Castell, or Eagle-Turquoise have been found satisfactory.

± 5°F) and then expose to 100 % relative humidity at 50 ± 3°C (121 ± 5°F) for 30 days. After this exposure, inspect the film for blistering and cracking and test for loss of adhesion as described in 5.2.10 and for pencil hardness as in 5.2.3.

5.2.6 *Heat Resistance*—Heat the five test panels prepared as described in 5.2.4 at 120 ± 3°C (249 ± 5°F) for 70 h. After this exposure, inspect the film for blistering and cracking and test for loss of adhesion as described in 5.2.10 and for pencil hardness as in 5.2.3.

5.2.7 *Low-Temperature Shock*—Subject the five test panels prepared as in 5.2.4 to 24 cycles, each consisting of 25 min at 65 ± 3°C (150 ± 5°F) then, following transfer within 5 s, 5 min at −55 ± 3°C (−66± 5°F). On completion of the last cycle the panel shall remain in a cold box maintained at −55 ± 3°C (−66 ± 5°F) for 5 h. Bend the panel rapidly over a 100-mm (4-in.) diameter mandrel that has been conditioned at the same condition for a minimum of 45 min. Panel evaluation after exposure shall include inspection for cracking and the adhesion test in accordance with 5.2.10. When a normal work day will not allow completion of this test, the cycle shall be interrupted at the end of the low temperature exposure (store overnight at room temperature) and start again at the hot cycle.

5.2.8 *Corrosion Resistance*—Scribe the five test panels prepared in accordance with 5.2.4 through the coating to the base metal with two diagonal scribe marks extending from corner to corner and exposed to 5 % salt spray in accordance with Practice B 117 for 40 days.

5.2.9 *Humidity Aging*—Expose the five test panels prepared in accordance with 5.2.4 to a relative humidity of 100 % at 50 ± 3°C (121 ± 5°F) for 30 days. After this exposure, inspect the film for blistering and cracking and test for loss of adhesion as described in 5.2.10.

5.2.10 *Loss of Adhesion*:

5.2.10.1 Thoroughly dry the panel by wiping with a clean dry cheesecloth. Conduct the test at 20 to 27°C (68 to 80°F).

5.2.10.2 Scribe two parallel scratches 25 mm (1 in.) apart, through the coating down to the metal, with a sharp-edge scribe.

5.2.10.3 Apply a strip of 25-mm (1-in.) wide masking tape (not more than five months old from date of manufacture) across the scratches at 90° in the panel area to be tested. Press the tape down using two passes of a 2-kg (4.5-lb) rubber-covered roller approximately 90 mm (3.5 in.) in diameter by 45 mm (1.75 in.) in width. The Durometer of the roller surface shall be 70 to 80 Shore A.

5.2.10.4 As an alternative to 5.2.10.3, the tape shall be pressed down by rolling the tape roll along the strip of applied tape using a firm hand pressure of approximately 2 kg (4 to 5 lb).

5.2.10.5 Remove the tape in one abrupt motion perpendicular to the panel. No primer shall be removed from the panel, other than that which was previously removed during the scribing procedure (see 5.2.10.2).

5.3 *Bonding Property Tests*:

5.3.1 *Preparation of Test Specimens*— Prepare and test five test specimens, in accordance with Test Method D 1002 and Practice E 864, for each of the required lap-shear tests in Table 3. In addition, prepare five test specimens for each floating roller peel test in Table 3 in accordance with Test Method D 3167 and Practice E 864. The adherends shall be of either 6061T6 or 5052H34 aluminum alloy. Cure the primer by air drying for not less than 30 min at 25 ± 5°C (78 ± 9°F); follow by heating at 115 ± 5°C (239 ± 9°F) for 75 to 90 min. Bond the primed panel with an adhesive meeting Specification E 865.

5.3.1.1 *Tests*—The primer shall be subjected to all the tests described in this section. Lap-shear tests (see 5.3.2-5.3.5) shall be performed in accordance with Test Method D 1002.

5.3.2 *Normal-Temperature Lap Shear*—Subject each specimen to a lap shear test at 25 ± 3°C (77 ± 5°F).

5.3.3 *Low-Temperature Lap Shear*—Test each specimen for low-temperature lap shear at −55 ± 3°C (−66 ± 5°F). Bring the temperature of the specimen to −55± 3°C (−66 ± 5°F) as indicated by a thermocouple at the bond area and stabilize for 10 min just prior to test.

5.3.4 *High-Temperature Lap Shear*—Test each specimen for high-temperature lap shear at 93 ± 3°C (200 ± 5°F). Bring the temperature of the specimen to 93 ± 3°C (200± 5°F) as indicated by a thermocouple at the bond area and stabilize for 10 min just prior to test.

5.3.5 *Humidity Exposure Lap Shear*—Test each specimen at 93± 3°C (200 ± 5°F) for lap-shear strength after two weeks' exposure at 95 ± 5 % relative humidity at 93 ± 3°C (200 ± 5°F). Bring the temperature of the specimen to 93 ± 3°C (200 ± 5°F) as indicated by a thermocouple at the bond area. Apply the load within 2 min after reaching the test temperature. Start the strength test no later than 30 min after removing the specimens from the humidity chamber.

5.3.6 *Salt Spray Exposure Lap Shear*— Test each specimen in accordance with 5.3.2 after exposure for two weeks to salt using 5 % NaCl solution at 35 ± 3°C (95 ± 5°F) in accordance with Practice B 117.

5.3.7 *Normal-Temperature Floating Roller Peel Strength*—Test each specimen in accordance with Test Method D 3167. The test temperature shall be 25 ± 3°C (77 ± 5°F).

5.3.8 *Low-Temperature Floating Roller Peel Strength*—Test each specimen in accordance with Test Method D 3167. The test temperature shall be −55 ± 3°C (−66 ± 5°F) as indicated by a thermocouple attached to the thin member. Stabilize the specimen at temperature for 10 min just prior to test.

6. Inspection

6.1 *Responsibility for Inspection*— Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use any facilities suitable for the performance of inspection requirements specified herein unless disapproved by the purchaser. The purchaser has the right to perform any of the inspections described in this specification where such inspections are necessary to ensure compliance with the requirements.

6.2 *Classification of Inspections*— The inspection requirements specified herein are classified as follows:

6.2.1 Qualification inspection (see 6.3).

6.2.2 Incoming shipment inspection (see 6.4).

6.3 *Qualification Inspection*:

6.3.1 *Qualification Test*—Unless otherwise specified by the purchaser, adhesive primers that have not passed the tests specified in Section 5, or that have previously passed the test but have subsequently been modified in any manner, must be tested against this specification and shown to meet its requirements.

6.3.1.1 *Instruction Sheet*—A dated, coded, and titled instruction sheet that outlines instructions for use of the adhesive primer shall be supplied by the manufacturer. These instructions shall be forwarded with the test report (see 6.3.2) when requesting qualification. In addition, a copy of the instruction sheet shall accompany each shipment of the adhesive primer. At the option of the purchaser, the tests as specified in Section 5 may be repeated at any time on material previously found satisfactory.

6.3.2 *Test Report*—In addition to the test specimens cited herein, the manufacturer shall furnish a dated and numbered report, certified by notarized affidavit, giving the results of all tests listed in Section 5. The individual and average values for the tests shall be reported. The test report shall cover all requirements of Section 4. Exact information as to how the individual specimens are fabricated, including component materials, component material preparation, and their processing into samples, as well as complete testing information for each test such as test apparatus, cross head speeds, etc. shall be reported. The report shall certify that the application of the primer conformed to the description given in the instruction sheet.

6.3.3 *Instruction Sheet*—The manufacturer shall submit with the test report (6.3.2) two copies of an instruction sheet that contains the information specified in 6.3.1.1.

6.4 *Incoming Shipment Inspection*—The inspection of each incoming lot of adhesive primer purchased under this specification shall consist of the following:

6.4.1 *Normal-Temperature Lap Shear*—In accordance with 5.3.1 and 5.3.2.

6.4.2 *Normal-Temperature Floating Roller Peel*—In accordance with 5.3.1 and 5.3.7.

6.4.3 *Solids Content*—In accordance with 5.1.1.

6.4.4 *Inhibitor Content*—In accordance with 5.1.2.

7. Packaging and Identification

7.1 *Packaging*—The product shall be packaged in airtight containers suitable for storage at a temperature of −18°C (0°F).

7.2 *Identification*—Each container shall be identified with the following information on durable labels, using clearly legible characters that will not be obliterated by normal handling:

7.2.1 Primer, adhesive, corrosion-inhibiting,
7.2.2 Specification E 866,
7.2.3 Date of manufacture (month and year),
7.2.4 Manufacturer's name and address,
7.2.5 Manufacturer's product trade name,
7.2.6 Manufacturer's product type designation,
7.2.7 Manufacturer's batch/lot designation,
7.2.8 Contract number,
7.2.9 Date of shipment,
7.2.10 Expiration date,
7.2.11 Unit quantity,
7.2.12 Perishable, STORE BELOW −18°C (0°F),
7.2.13 Appropriate materials safety data sheet (MSDS), and
7.2.14 Manufacturer's certification that the batch and lot meet the requirements of this specification. See Terminology E 1749.

7.3 *Packing*—The individual containers shall be packed in an exterior shipping container capable of protecting the product and maintaining the required temperature during transit.

7.4 *Exterior Marking*—Each exterior shipping container shall be legibly marked as in 7.2.

7.5 *Containers*—Containers shall be prepared for shipment in accordance with commercial practice to assure carrier acceptance and safe transportation to point of delivery. Packaging shall conform to carrier rules and regulations applicable to the mode of transportation. In addition to the exterior marking in 7.4, the exterior container shall be marked to comply with ICC regulations applicable to the mode of transportation.

8. Safety

8.1 *Precautions*—Because the primers covered by this specification may be toxic through contact or inhalation, special precautions in their handling and use are required.

8.2 *Spray and Cure Areas*—The spray and cure areas shall have adequate exhaust ventilation as determined by applicable Federal and local EPA regulations.

8.3 *Protective Equipment*—Protective clothing, and rubber or vinyl plastisol gloves and goggles should be worn to prevent personal contact with these materials.

9. Keywords

9.1 adhesive bond; aluminum; corrosion resistance; honeycomb sandwich panels; primer; relocatable shelters