Designation: ~~E864 – 12~~ E864 – 20

Standard Practice for Surface Preparation of Aluminum Alloys to Be Adhesively Bonded in Honeycomb Shelter Panels[1]

This standard is issued under the fixed designation E864; 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 U.S. Department of Defense.

1. Scope

1.1 This practice covers the preparation of clean uniform surfaces of aluminum alloys suitable for formation of durable adhesive bonds to nonmetallic honeycomb materials in the manufacture of sandwich panels for tactical shelters.

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 regarded as ~~the~~ standard.

1.3 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific warning statement, see 6.2.1.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2674 Methods of Analysis of Sulfochromate Etch Solution Used in Surface Preparation of Aluminum

D3167 Test Method for Floating Roller Peel Resistance of Adhesives

E631 Terminology of Building Constructions

E865 Specification for Structural Film Adhesives for Honeycomb Sandwich Panels

E866 Specification for Corrosion-Inhibiting Adhesive Primer for Aluminum Alloys to Be Adhesively Bonded in Honeycomb Shelter Panels

E1749 Terminology Relating to Rigid Wall Relocatable Shelters

2.2 *APHA Standard:*[3]

APHA Standard Methods for the Examination of Water and Waste Water (15th Edition, 1980), Sections 402, 403, and 408

3. Terminology

3.1 *Definitions*—~~See Terminologies~~ —For definitions of general terms related to building construction used in this practice, refer

[1] This practice is under the jurisdiction of ASTM Committee E06 on Performance of Buildings and is the direct responsibility of Subcommittee E06.53 on Materials and Processes for Durable Rigidwall Relocatable Structures.

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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~~standard's Document Summary page on the ASTM website.

[3] Available from the American Public Health Association (APHA), 800 I Street, NW, Washington, DC ~~20001-3710,~~20001, http://www.apha.org.

to Terminology E631, and ~~E1749 for definitions of general terms used in this practice.~~for general terms related to rigid wall relocatable shelters, refer to Terminology E1749.

4. Significance and Use

4.1 Durable adhesive bonds to aluminum alloys can be obtained reliably only through proper selection and careful control of the materials used and the steps in the bonding process. The preparation of the aluminum alloys to obtain clean, uniform surfaces with appropriate characteristics is a critical step. This practice describes how such surfaces can be obtained.

5. Apparatus

5.1 *General Processing:*

5.1.1 All heated tanks shall be equipped with automatic temperature controls and shall have means for agitation to prevent local overheating of the solution. Solutions may be heated by any internal or external means that do not change their compositions. Steam shall not be introduced into any solution. Compressed air introduced into any solution or equipment shall have been filtered to remove oil and moisture.

5.1.2 Tanks shall be made from, or lined with, materials that have no adverse effects on the solutions used or the parts being treated. All tanks shall be of sufficient size to allow complete immersion of the largest part or assembly to be treated.

5.2 *Rinse Tanks*—Immersion rinse tanks shall be equipped with a means for skimming or overflowing or both to remove surface contamination. The tanks shall be equipped with a means for flushing hollow sections.

5.3 *Rinses*—Rinses, other than final rinses, shall be maintained in such a manner to prevent carryover of materials that would adversely affect the next solution (for example, using a fog water rinse as the aluminum part/assembly is being withdrawn from the rinse tank).

6. Materials

6.1 *Water*—Water used for makeup of processing solutions and final rinsing shall be deionized water or shall meet the requirement of Table 1. Analyses shall be performed as often as necessary to assure that the water meets the requirements. Samples for analysis shall be collected at the processing tanks.

6.2 *Etch Solution:*

6.2.1 *Method I, Sulfo-Chromate Etch (FPL)*—The chemical analysis of the etch solution shall be maintained at approximately 30 parts by mass of water, ten parts by mass of sulfuric acid (sp gr 1.84), and one to four parts by mass of sodium dichromate. Prior to use, a minimum of 0.06 part by mass of dissolved 2024 aluminum shall be added. (**Warning—**It is recognized that chromates present a hazard to health. Use and disposal procedures are governed by Federal and Local EPA and DER limitations.)

6.2.2 *Method II, Sulfo-Ferric Etch (P_2)*—The chemical analysis of the etch solution shall be maintained at approximately 27 to 36 % by weight of sulfuric acid (sp gr 1.84) and 2.9 to 4.7 oz/gal of ferric iron or 18 to 22 oz/gal of ferric sulfate. This is the equivalent of 2 gal of concentrated sulfuric acid and 12.5 lb of ferric sulfate in every 10 gal of solution. Two gallons of a 50 % ferric sulfate solution may be used in place of the 12.5 lb of the powdered ferric sulfate.

NOTE 1—Only virgin ferric sulfate solution shall be used in this process. Impurities in reclaimed ferric sulfate will cause unwanted reactions when the aluminum is treated.

TABLE 1 Requirements for Water to Be Used in Solutions and Rinses

	Requirements	
	Min	Max
pH	6.0	8.0
Total solids, ppm	...	200
Total alkalinity as $CaCO_3$, ppm	...	125
Chloride content, ppm	...	15

6.3 *Alkaline Cleaning Solution*—Nonetch, alkaline cleaning solution shall be prepared in accordance with the ~~manufacturer's~~manufacturer's recommendations, or as indicated in 8.1.2. When the aluminum being cleaned is immersed in the alkaline cleaner for the time and at the temperature used for processing, there shall be no evidence of gas evolution, etching, or metal removal. The alkaline solution shall not contain silicates.

6.4 *Quality Assurance Adhesive*—The adhesive system used for the quality assurance tests of 9.6 shall meet Specification E865. The adhesive shall be changed only when a batch is almost exhausted so the results of any particular day's testing can be directly compared to the results of previous tests. When a change is made in the batch or lot of adhesive used, an additional set of tests shall be made to compare the old batch or lot with the new one to establish a basis for comparison between the results obtained with each.

7. Test Methods

7.1 *Chemical Analyses*—Perform chemical analyses of the water and solutions as indicated in 7.2 and 7.3. Analyze as often as necessary to maintain the required concentrations at a minimum of every day of operation. If a process is not in use during the normal analysis period, note this on the analysis record and analyze the solution prior to further use.

7.2 *Water Analyses*—Analyze as described in APHA Standard Methods for the Examination of Water and Waste Water.

7.3 *Etch Solution Analysis:*

7.3.1 *Sulfo-Chromate Etch*—Perform the analyses in accordance with Methods D2674.

7.3.2 *Sulfo-Ferric Etch*—Perform the analyses in accordance with 7.3.2.1 and 7.3.2.2.

7.3.2.1 *Sulfuric Acid*—Pipet a ~~1-mL (0.03-oz)~~ 1 mL (0.03 oz) sample into a ~~250-mL (8.5-oz)~~ 250 mL (8.5 oz) Erlenmeyer flask containing 100 mL (3.4 oz) of distilled water. Add 1 g (0.03 oz) of tribasic sodium citrate and 1 g (0.03 oz) of sodium fluoride. Add 2 to 3 drops of phenolphthalein indicator. Titrate with 0.4 N sodium hydroxide to clear or faint pink. Calculate the normality of sulfuric acid in accordance with Table 2.

7.3.2.2 *Ferric Ion*—Pipet a ~~2-mL (0.06-oz)~~ 2 mL (0.06 oz) sample into a ~~250-mL (8.5-oz)~~ 250 mL (8.5 oz) Erlenmeyer flask containing 100 mL (3.4 oz) of distilled water. Add 10 mL (0.3 oz) of concentrated hydrochloric acid. Add 1 g to 2 g (0.03 oz to 0.06 oz) of potassium iodide titrate with 0.10 N sodium thiosulfate to a greenish color. Add about 2 mL of starch solution. Continue titration to a clear green end point. Calculate ferric sulfate as follows:

$$\text{mL sodium thiosulfate used} \times 2.79 = \text{g/L ferric ion} \quad (1)$$

7.4 *Visual Inspection*—Inspect parts visually for stains and water break.

7.5 *Floating Roller Peel Test*—Test in accordance with Test Method D3167.

TABLE 2 Calculation of Normality of Sulfuric Acid to Percent by Weight (1 g/100 g)

NOTE 1—N sulfuric acid = mL 0.4 N NaOH × 0.4

N %	*N* %
4.65 = 20	7.76 = 31
4.91 = 21	8.06 = 32
5.18 = 22	8.37 = 33
5.45 = 23	8.68 = 34
5.73 = 24	8.99 = 35
6.01 = 25	9.31 = 36
6.29 = 26	9.64 = 37
6.58 = 27	9.96 = 38
6.86 = 28	10.3 = 39
7.16 = 29	10.6 = 40
7.46 = 30	

8. Procedure

8.1 *Surface Preparation*—Perform the steps of the procedure in accordance with 8.1.1 – 8.1.5 and Table 3 or Table 4 as appropriate. Observe the restrictions listed in 8.2.

8.1.1 *Precleaning*—Remove visible oil and grease from the aluminum by vapor degreasing, solvent cleaning, or other suitable method to ensure a clean, nonoily surface.

8.1.2 *Alkaline Cleaning*—Immerse the aluminum in the alkaline cleaning solution held from ~~50~~50 °C to ~~80°C (122~~80 °C (122 °F to ~~176°F)~~176 °F) for a minimum of 5 min. Follow the alkaline cleaning by thoroughly rinsing in water from room temperature to ~~70°C (158°F).~~70 °C (158 °F). When the immersion does not completely clean the aluminum, alkaline cleaning shall be repeated. Keep the parts wet between the alkaline treatment and immersion in the rinse tank.

8.1.3 *Etching:*

8.1.3.1 *Sulfo-Chromate Etch*—Immerse the aluminum parts in the etch solution for 9 min to 15 min from ~~65~~65 °C to ~~70°C (149~~70 °C (149 °F to ~~158°F).~~158 °F). Keep the parts wet between the etch tank and the rinse tank. Follow the etching by thoroughly rinsing with room temperature water.

8.1.3.2 *Sulfo-Ferric Etch*—Immerse the aluminum parts in the etch solution for 10 min to 20 min from ~~49~~49 °C to ~~65°C (120~~65 °C (120 °F to ~~149°F).~~149 °F). Immerse the flat sheet for 10 min to 12 min. Extrusions may require up to 20 min. Keep the parts wet between the etch tank and the rinse tank. Follow the etching by thoroughly rinsing in water at ~~55°C (131°F)~~55 °C (131 °F) maximum.

8.1.4 *Final Rinse*—Rinse the aluminum for 1 min to 2 min in water from room temperature to ~~55°C (131°F).~~55 °C (131 °F). Check parts that can be readily observed for water break and recycle if a water break occurs. Verify the water break-free surface for 30 s. If the water film does not remain continuous for 30 s, repeat surface preparation procedure (see 8.1.4.1).

NOTE 2—A water break-free surface maintains a continuous water film (no beads) for a period of at least 30 s after being sprayed or immersion rinsed in clean water at a temperature below ~~38°C (100°F).~~38 °C (100 °F).

TABLE 3 Procedure for Surface Preparation of Aluminum Alloys Using the Sulfuric Acid-Sodium Dichromate Etch

STEP 1—PRECLEAN (see 8.1.1) Vapor degrease or clean with safety solvent. Flush all hollow sections. Repeat as necessary.
STEP 2—ALKALINE CLEAN (see 8.1.2) ~~Immerse in aluminum cleaner from 50 to 80°C (122 to 176°F) for 5 to 10 min. Repeat as necessary.~~ Immerse in aluminum cleaner from 50 °C to 80 °C (122 °F to 176 °F) for 5 min to 10 min. Repeat as necessary.
STEP 3—RINSE (see 8.1.2) Use water from room temperature to 70°C (158°F).
STEP 4—ETCH (see 8.1.3.1) ~~Immerse in sulfuric acid-sodium dichromate etch for 9 to 15 min from 65 to 70°C (149 to 158°F). Spray rinse heavy sections as they emerge from the solution as necessary to prevent staining.~~ Immerse in sulfuric acid-sodium dichromate etch for 9 min to 15 min from 65 °C to 70 °C (149 °F to 158 °F). Spray rinse heavy sections as they emerge from the solution as necessary to prevent staining.
STEP 5—RINSE (see 8.1.3.1) Use room temperature water.
STEP 6—FINAL RINSE (see 8.1.4) ~~Use water from room temperature to 50°C (122°F) for 1 to 2 min.~~ Use water from room temperature to 50 °C (122 °F) for 1 min to 2 min.
STEP 7—DRY (see 8.1.5) ~~Use air from room temperature to 65°C (149°F) for not more than 1 h.~~ Use air from room temperature to 65 °C (149 °F) for not more than 1 h.