Designation: E 864 – 03

An American National Standard

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 E 864; 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 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 *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.* For specific precautionary statements, see Note 1 of 5.2.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 2674 Test Method for Analysis of Sulfochromate Etch Solution Used in Surface Preparation of Aluminum

D 3167 Test Method for Floating Roller Peel Resistance of Adhesives

E 865 Specification for Structural Film Adhesives for Honeycomb Sandwich Panels

E 866 Specification for Corrosion-Inhibiting Adhesive Primer for Aluminum Alloys to Be Adhesively Bonded in Honeycomb Shelter Panels

2.2 *APHA Standard:*

Sections 402, 403, and 408 of Standard Methods for the Examination of Water and Waste Water (15th Edition, 1980)[3]

3. Significance and Use

3.1 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.

4. Apparatus

4.1 *General Processing*:

4.1.1 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.

4.1.2 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.

4.2 *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.

4.3 *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.

5. Materials

5.1 *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.

5.2 *Etch Solution*:

5.2.1 *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.

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[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 Document Summary page on the ASTM website.

[3] Available from the American Public Health Assn., 1015 15th St. N.W., Washington, DC 20005.

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

NOTE 1—It is recognized that chromates present a hazard to health. Use and disposal procedures are governed by Federal and Local EPA and DER limitations.

5.2.2 *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 2—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.

5.3 *Alkaline Cleaning Solution*—Nonetch, alkaline cleaning solution shall be prepared in accordance with the manufacturer's recommendations, or as indicated in 7.1.2. 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.

5.4 *Quality Assurance Adhesive*—The adhesive system used for the quality assurance tests of 8.6 shall meet Specification E 865. 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.

6. Test Methods

6.1 *Chemical Analyses*—Perform chemical analyses of the water and solutions as indicated in 6.2 and 6.3. 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.

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

6.3 *Etch Solution Analysis*:

6.3.1 *Sulfo-Chromate Etch*—Perform the analyses in accordance with Test Method D 2674.

6.3.2 *Sulfo-Ferric Etch*—Perform the analyses in accordance with 6.3.2.1 and 6.3.2.2.

6.3.2.1 *Sulfuric Acid*—Pipet a 1-mL (0.03-oz) sample into a 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.

6.3.2.2 *Ferric Ion*—Pipet a 2-mL (0.06-oz) sample into a 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 to 2 g (0.03 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:

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

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

6.5 *Floating Roller Peel Test*—Test in accordance with Test Method D 3167.

7. Procedure

7.1 *Surface Preparation*—Perform the steps of the procedure in accordance with 7.1.1-7.1.5 and Table 3 or Table 4 as appropriate. Observe the restrictions listed in 7.2.

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

7.1.2 *Alkaline Cleaning*—Immerse the aluminum in the alkaline cleaning solution held from 50 to 80°C (122 to 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). 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.

7.1.3 *Etching*:

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

7.1.3.2 *Sulfo-Ferric Etch*—Immerse the aluminum parts in the etch solution for 10 to 20 min from 49 to 65°C (120 to 149°F). Immerse the flat sheet for 10 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) maximum.

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

NOTE—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	

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

STEP 1—PRECLEAN (see 7.1.1) Vapor degrease or clean with safety solvent. Flush all hollow sections. Repeat as necessary.
STEP 2—ALKALINE CLEAN (see 7.1.2) Immerse in aluminum cleaner from 50 to 80°C (122 to 176°F) for 5 to 10 min. Repeat as necessary.
STEP 3—RINSE (see 7.1.2) Use water from room temperature to 70°C (158°F).
STEP 4—ETCH (see 7.1.3.1) 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.
STEP 5—RINSE (see 7.1.3.1) Use room temperature water.
STEP 6—FINAL RINSE (see 7.1.4) Use water from room temperature to 50°C (122°F) for 1 to 2 min.
STEP 7—DRY (see 7.1.5) Use air from room temperature to 65°C (149°F) for not more than 1 h.

TABLE 4 Procedure for Surface Preparation of Aluminum Alloys Using the Sulfo-Ferric Etch

STEP 1—PRECLEAN (see 7.1.1) Vapor degrease or clean with safety solvent. Flush all hollow sections. Repeat as necessary.
STEP 2—ALKALINE CLEAN (see 7.1.2) Immerse in aluminum cleaner at 50 to 80°C (122 to 176°F) for 5 to 10 min. Repeat as necessary.
STEP 3—RINSE (see 7.1.2) Use water from room temperature to 70°C (158°F).
STEP 4—ETCH (see 7.1.3.2) Immerse in the sulfo-ferric etch for 10–20 min from 49 to 65°C (120 to 149°F). Spray rinse heavy sections as they emerge from the solution as necessary to prevent staining.
STEP 5—RINSE (see 7.1.3.2) Rinse for 2 min. minimum in water at 55°C (131°F) maximum
STEP 6—FINAL RINSE (see 7.1.4) Use water from room temperature to 55°C (131°F) for 1 to 2 min.
STEP 7—DRY (see 7.1.5) Use air from room temperature to 65°C (149°F) for not more than 1 h.

7.1.4 *Final Rinse*—Rinse the aluminum for 1 to 2 min in water from room temperature to 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 7.1.4.1).

NOTE 3—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).

7.1.4.1 *Rework*—Parts that are rerun, because of water break, stains, or unprimed parts that have exceeded the permitted storage time shall be reworked no more than two times. Do not exceed a total immersion time of 34 min.

7.1.5 *Drying*—Air dry the aluminum for not more than 1 h at a temperature not exceeding 65°C (149°F) prior to movement to the controlled area.

7.2 *Restrictions*:

7.2.1 Immerse parts completely in all solutions.

7.2.2 Do not allow rinsing stains and dichromate stains on the bonding surfaces.

7.2.3 Place cleaned dried parts within ½ h in a controlled area with filtered air that is maintained at a relative humidity of 50 % maximum. Prime or bond the cleaned parts within 16 h.

7.2.4 Do not allow the solution temperature to exceed 63°C (145°F) when changing a new sulfo-ferric etch tank.

7.2.5 Take precautions to prevent precipitation (keep spent solution warm in transport) when taking spent sulfo-ferric etch solution to a waste disposal facility.

8. Quality Assurance

8.1 *Handling*—Parts that have been processed shall be handled using clean cotton gloves.

8.2 *Marking and Storing*—All processed parts shall be marked with the date or time of processing, or both. The processed parts shall not be marked on the faying surfaces.

8.3 *Records*—Records of all tests required by this practice shall be maintained by the processor for a minimum period of three years or, at the option of the processor or the request of the purchaser, the records may be forwarded to the purchaser.

8.4 *Written Process Procedures*—Each processor shall keep a written description of the process procedure in the process area. The description shall include, as a minimum, the following information: (*a*) processing time for each step, (*b*) processing temperatures for each step, and (*c*) materials used for each step. The description of the procedure shall be signed by the individual responsible for process control and shall be updated as necessary to ensure conformance with requirements.

8.5 *Visual Inspection*—Parts shall be inspected for compliance with 7.1.4 and 7.2.2.

8.6 *Quality Assurance Test*—Control specimens are required when processing for structural bonding. One set of test specimens shall be processed each day with a typical production load. A test coupon shall be bonded, cut into specimens, and tested in accordance with 8.6.1. Failure of these specimens to meet the minimum acceptable average peel strength shall warrant an immediate investigation to determine the cause.

8.6.1 *Floating Roller Peel Specimen*—A minimum acceptable average floating roller peel strength for the adhesive shall be established prior to its use in quality assurance tests. Thereafter, five unprimed specimens cut from one test coupon shall be prepared and tested at the start of each day's production as described in 6.5. The adhesive used shall be as defined in 5.4, and the average peel strength for the five specimens shall not be less than that minimum established for that adhesive.

NOTE 4—If the customer does not require use of a primer (Specification E 866) in shelter panel manufacture, then only five unprimed specimens shall be prepared and tested each day.

9. Certification

9.1 At the request of the purchaser in the contract or order, the processor shall certify in writing that the surface preparation of the aluminum parts to be bonded was performed in accordance with this practice.

10. Keywords

10.1 adhesive bond; aluminum; bonding process; clean surfaces; honeycomb sandwich panels; relocatable shelters; surface preparation