



Designation: D3933 – 98 (Reapproved 2010)

Standard Guide for Preparation of Aluminum Surfaces for Structural Adhesives Bonding (Phosphoric Acid Anodizing)¹

This standard is issued under the fixed designation D3933; 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 This guide describes the requirements for phosphoric acid anodizing of aluminum and its alloys for structural adhesive bonding.

1.2 The procedure included herein is based on the commercial practice of numerous agencies and organizations. The method may be revised or supplemented, as necessary, to include methods based on proven performance.

1.3 The surface preparation of metal systems used for qualification and quality-control testing of the adhesive should be agreed upon between the manufacturer and the user.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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.* A specific precaution is given in 5.5.

2. Referenced Documents

2.1 *ASTM Standards:*²

D907 Terminology of Adhesives

D2651 Guide for Preparation of Metal Surfaces for Adhesive Bonding

2.2 *Federal Specifications:*

0-0-670 Orthophosphoric Acid, Technical³

¹ This guide is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.80 on Metal Bonding Adhesives.

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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 U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

3. Terminology

3.1 *Definitions*—Many terms in this guide are defined in Terminology D907.

4. Significance and Use

4.1 Durable adhesive bonds between metal to metal, metal to composites 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 metallic substrates to obtain surfaces with appropriate characteristics is a critical step. Improper surface preparation can produce seemingly acceptable bonds that can degrade rapidly with time. This guide describes one method on how to properly prepare aluminum surfaces can be obtained.

4.2 The formation of reproducible, durable, adhesive bonds in structural assemblies requires great care in the selection of materials, the preparation of the surfaces of the components to be bonded, the fit of the components, and the performance of the steps in the bonding process. Experience has shown that when adhesively bonded aluminum surfaces which have been prepared in accordance with this guide produce relatively reproducible, durable bonds.

5. Apparatus

5.1 Locate surface preparation and drying facilities separate from other activities or equipment, or both, to preclude contamination by substances detrimental to adhesion.

5.2 Facilities should be arranged such that parts can flow from the beginning of surface preparation to the priming operation without being touched.

5.3 Filters or traps, or both, should be installed for removing airborne dust, moisture, and oil from all air lines or ducts used for solution agitation and parts drying. Periodically check, clean, or replace filters to ensure proper operation.

5.4 Surface preparation facilities should not be used for purposes other than the preparation of parts for adhesive bonding and subsequent finishing.

5.5 Equipment requirements for this process include equipment normally used in the chemical processing of aluminum alloys, with the following special provisions:

5.5.1 Racks and frames that are electrically anodic shall be constructed of materials that will not cause corrosive reactions with the suspension wires or clips, or the parts, during the entire process. “Picture-frame”-type racks have performed satisfactorily.

5.5.2 Parts shall be attached to racks or frames with conductive material that anodizes, that is, aluminum or titanium in the form of wire, hooks, springs, clips, etc.

5.5.3 The phosphoric acid anodizing tank lining shall be of such material that interaction between the solutions, parts, frames, racks, or clips and hangers does not interfere with the anodic process and is suitable for use as a cathode. Tanks should be equipped with a surface skimming device to remove surface contamination, a filtering system, and an agitation system for mixing the tank contents.

5.5.4 Terminals for electrical current shall be designed and positioned such that parts cannot be “burned.”

5.5.5 The electrical system used during a *single-rack* anodic process shall be adequate for producing 10-V dc and maintaining any set voltage within ± 1 -V dc.

5.5.6 The electrical system used during a *multiple-rack* process shall be adequate for producing 15-V dc and maintaining any set voltage within ± 1 -V dc.

5.5.7 Rinse tanks should be placed adjacent to processing tanks in such a manner that parts can be easily and quickly moved from one solution to another so that the parts will not become dry during successive steps of the process.

5.5.8 Incoming solution makeup water and rinse water shall contain not more than the “recommended upper limit” for fluoride and not more than the “highest desirable level” of dissolved chemicals based on American Chemical Society standards for deionized or distilled water, or both (1.7 ppm fluoride, 500 ppm total dissolved solids, etc.) except that:

5.5.8.1 Chloride shall not exceed 25 ppm.

5.5.8.2 pH shall be between 5.5 and 8.0.

NOTE 1—Most regionally distributed potable water will meet these requirements.

5.5.8.3 If the above requirements cannot be met, the incoming water shall be deionized and maintained within the following limits:

(a) Total dissolved solids shall not exceed 150 ppm.

(b) pH shall be between 5.5 and 8.0.

6. Manufacturing

6.1 Parts shall be racked or suspended from frames such that they all have firm, reliable electrical contact with the anode connections, so that the parts do not contact each other and that rinse water contacts all surfaces and drains freely.

6.2 Take the utmost care that the parts are not touched at any time during the entire process, such as touching of adjacent parts, the rack or supporting frame, or any other item, since the surfaces and the phosphoric acid anodic coating are susceptible to contamination or physical damage prior to the application and cure of the primer.

6.3 If it becomes necessary to handle or touch parts, clean white gloves should be used and contact should be limited to surfaces not to be adhesively bonded.

6.4 If parts become contaminated, either prior to or subsequent to anodizing, corrective action shall be accomplished by reprocessing, beginning with alkaline cleaning.

6.5 To ensure solution homogeneity, agitate immediately prior to process use and after every addition of water or chemicals. (**Warning**—Solutions used in this process are corrosive and toxic. Avoid breathing solution mists or vapors. Do not allow solutions to get on the eyes, on the skin, or on clothing.)

6.5.1 Consult Industrial Hygiene for safety precautions.

6.6 Control the time interval between withdrawal from processing solutions and rinsing so that there is no drying of the solution on the details.

6.7 Parts must be water-break-free following rinsing after alkaline cleaning, after deoxidizing, and after anodizing.

7. Procedure

7.1 Perform the surface preparation process in a continuous operation as detailed in the flow chart, Fig. 1.

7.2 Complete all fabrication processes, inspections, prefit, etc. before the start of the preparation cycle.

7.3 Complete all “hand work” before solvent cleaning.

7.4 Once racked for alkaline cleaning, parts shall not be touched by anything except the applicable processing solution and rinse waters, until the parts are dried after primer application (5.2).

7.5 The “wet” processing steps, from alkaline cleaning through drying after phosphoric acid anodizing, should be performed in one continuous uninterrupted sequence of processes, with the parts not drying at any time until the oven drying after the rinse following phosphoric acid anodizing.

7.5.1 Should the parts become delayed prior to anodizing during this wet processing, hold the parts in the applicable rinse tank.

7.5.2 There shall be no holding of parts in the processing sequencing after anodizing.

7.6 Should the electric current fail or otherwise be interrupted during the phosphoric acid anodizing, anodizing may be continued for an additional 20 to 25 min, if the potential can be reestablished within 2 min. If the potential cannot be reestablished within 2 min, then rinse and reprocess starting with the deoxidizer.

7.7 Apply adhesive primer within 72 h after oven drying following the phosphoric acid anodizing. Parts should be held in a limited contamination area prior to priming and should not be handled (5.2). However, it is most desirable to prime parts as soon as possible to minimize the possibility of inadvertent contamination.

7.8 *Rinse Requirements:*

7.8.1 Rinsing may be by spray or immersion. Single spray or double counter-current immersion rinsing are commonly used.

7.8.2 Feed water to rinse operations may be direct from source water in accordance with 5.5.8 or from any subsequent