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Magnesium Alloys, Anodic Treatment of

RATIONALE

This document has been reaffirmed to comply with the SAE five-year review policy.

NONCURRENT NOTICE

This specification has been declared "NONCURRENT" by the Aerospace Materials Division, SAE, as of April, 2007. It is recommended, therefore, that this specification not be specified for new designs.

"NONCURRENT" refers to those specifications which have previously been widely used and which may be required for production or processing of existing designs in the future. The Aerospace Materials Division, however, does not recommend these specifications for future use in new designs.

"NONCURRENT" specifications are available from SAE upon request.

NOTICE

This document has been taken directly from U.S. Military Specification MIL-M-45202C, Notice 1, and contains only minor editorial and format changes required to bring it into conformance with the publishing requirements of SAE technical standards. The initial release of this document is intended to replace MIL-M-45202C, Notice 1. Any part numbers established by the original specification remain unchanged.

The original Military Specification was adopted as an SAE standard under the provisions of the SAE Technical Standards Board (TSB) Rules and Regulations (TSB 001) pertaining to accelerated adoption of government specifications and standards. TSB rules provide for (a) the publication of portions of unrevised government specifications and standards without consensus voting at the SAE Committee level, and (b) the use of the existing government specification or standard format.

Under Department of Defense policies and procedures, any qualification requirements and associated qualified products lists are mandatory for DOD contracts. Any requirement relating to qualified products lists (QPL's) has not been adopted by SAE and is not part of this SAE technical document.

1. SCOPE:

1.1 Scope:

This specification covers the requirements for equipment, materials and procedures to be used in anodic treatments for magnesium base alloys for the purpose of increasing their corrosion resistance or producing surfaces suitable for the application of a protective organic finish.

1.2 Classification:

The types, classes, and grades of anodic finishes covered by this specification are as follows:

Type I - Light coating.

- Class A - Tan coating (HAE).
  - Grade 1 - Without post treatment (dyed).
  - Grade 2 - With bifluoride-dichromate post treatment.
- Class C - Light green coating (Dow 17).

Type II - Heavy coating.

- Class A - Hard brown coating (HAE).
  - Grade 1 - Without post treatment.
  - Grade 3 - With bifluoride-dichromate post treatment.
  - Grade 4 - With bifluoride-dichromate post treatment including moist heat aging.
  - Grade 5 - With double application of bifluoride-dichromate post treatment including moist heat aging.
- Class D - Dark green coating (Dow 17).

2. APPLICABLE DOCUMENTS:

The following publications, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

2.1 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

QQ-A-250/8 Aluminum Alloy, 5052, Plate and Sheet

MIL-M-3171 Magnesium Alloy, Processes for Pretreatment and Prevention of Corrosion on

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-649 Aluminum and Magnesium Products Preparation for Shipment and Storage

MIL-HDBK-132 Protective Finishes

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM B 244 Measurement of Thickness of Anodic Coatings on Aluminum and Other Non-conductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments
ASTM B 487 Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section

3. REQUIREMENTS:

3.1 Materials:

3.1.1 Preparation of parts: All parts shall be free from broken edges before anodizing and unless otherwise specified, shall have all machining, drilling or other metal removal done prior to anodizing.

3.1.2 Cleaning and treatment materials: All materials used in cleaning and treating magnesium base alloys shall be free from salts of heavy metals and suitable for the purpose intended.

3.2 Equipment:

3.2.1 Materials of construction: All tanks and all pipes, valves, pumps, and other items of equipment which come in contact with the processing bath shall comply with table I, with respect to material construction.

3.2.2 Processing tanks:

3.2.2.1 Dimensions: The effective dimensions of tanks shall allow a minimum distance of two inches (51 millimeters) between the sides (including cooling coils) of the tank and the articles being treated, making due allowance for any position in which the work must be suspended.

TABLE I. Materials for construction of equipment

Classification	Recommended	To be avoided
Type I, class A and type II class A (HAE)		
Anodize tank	Black iron	Galvanized iron, brass, bronze, tin, zinc, rubber, all oxidizable materials
Valves	Black iron or copper	
Bifluoride-dichromate tank	Tank lined with polyethylene or similar inert material	
Racks and clamps	Magnesium or magnesium alloy	
Bus bars	Copper	
Type I, class C and type II class D (Dow 17)		
Tank	Unlined steel tanks or tanks lined with synthetic rubber or vinyl base materials	Copper, nickel, lead chromium, zinc, aluminum, monel
Racks and clamps	Magnesium, magnesium alloys or 5052 and 5056 aluminum alloys	

3.2.2.2 Heating and cooling equipment: Tanks shall be equipped, when required, with adequate facilities for heating or cooling in order to maintain the contents within the temperature limits specified for the type of coating being applied. This may be accomplished by pumping the electrolyte through an external heat exchanger or by means of coils or plates suitably located within the tank, through which cold water, steam, refrigerant or any suitable heating or cooling medium may be passed.

3.2.2.3 Lubrication of moving parts: Grease and other lubricants shall not be used in valves, pumps or other moving parts which are in contact with the bath since they may contaminate or chemically reduce the electrolyte.

3.2.3 Racks and clamps: Racks and clamps for holding magnesium articles during anodizing shall be made of materials recommended in table I. They shall be protected at the liquid-air interface with vinyl type electroplater's tape which resists the action of the electrolyte.

3.2.3.1 Stripping racks: The coating which forms on the magnesium racks during anodizing may be removed with a 20 percent chromic acid solution. However, the anodic coating need not be thus removed after each use provided that the points of contact are filed to provide sufficient contact area. There is usually no necessity for cleaning aluminum contacts.

3.3 Preparation for anodizing:

3.3.1 Cleaning: Cleaning prior to anodizing shall be accomplished by an alkaline cleaning solution as specified in 3.3.1.2. Before alkaline cleaning, excessive amounts of grease or oil shall be removed by a solvent method described in 3.3.1.1.

3.3.1.1 Solvent cleaning: Solvent cleaning may be accomplished by soaking, spraying, vapor degreasing, or ultrasonic cleaning, using organic solvents or an emulsion consisting of a mineral oil distillate, water, and an emulsifying agent. Petroleum spirits, naphtha, chlorinated hydrocarbons, lacquer thinners and similar grease solvents, which do not attack magnesium, may be used. Methyl (wood) alcohol should not be used in cleaning formulations.

3.3.1.2 Alkaline cleaning: Alkaline cleaning shall be accomplished by soaking, preferably accompanied by some form of agitation, in an alkaline cleaner recommended for steel or in a cleaning solution which has a pH above 8. The cleaning solution may be a proprietary type, in which case the operation shall be performed according to the vendor's instructions. A cleaner of the following formulation may be used under the indicated operating conditions.

	Solution Make-up	
	oz/gal	g/L
Sodium hydroxide	2-8 oz	15-60 g
Sodium phosphate ($Na_3P0_4.12H_20$)	1.3 oz	10 g
Soluble soap or wetting agent	0.1 oz	0.8 g
Water to make	1 gal	1 L

Operating conditions

Tank construction	Steel
Temperature	190-212 F (88-100 C)
Time	3 to 10 minutes

NOTE: Alkaline cleaners containing more than 2 percent caustic (sodium hydroxide) will etch ZK60A, ZK60B, and some magnesium-lithium alloys with resultant change in dimensions.

3.3.1.2.1 Cathodic cleaning: If available, direct current (dc) may be used with the alkaline cleaning solution to reduce the time required for cleaning. The parts are made cathodic in the bath and a current density of 10 to 40 amperes per square foot A/ft^2 at 6 volts is applied (current density in amperes per square decimeter is 1.1 to 4.3 A/dm^2).

3.3.1.2.2 Rinsing: After alkaline cleaning, the parts shall be given a thorough rinse in cold running water.

3.3.2 Acid pickling:

3.3.2.1 Type I, class A and type II, class A anodizing: Acid pickling is not required for these coatings. Acid pickling is recommended for type I, class A, where maximum corrosion resistance is required.

3.3.2.2 Type I, class C: The magnesium base alloy shall be pickled using the appropriate procedure as described in MIL-M-3171.

3.3.2.3 Type II, class D: The magnesium base alloy shall be pickled when necessary using the appropriate procedure as described in MIL-M-3171.

3.4 Anodizing procedures:

3.4.1 Process conditions: The composition of processing baths and the operating conditions shall be as stated in tables II to III, inclusive.

TABLE II. Process conditions for type I, class A and type II, class A anodizing (HAE).

Cleaning (see 3.3.1)		
Bath composition	Solution make-up	
	oz/gal	g/L
Potassium hydroxide	22 oz	165 g
Aluminum hydroxide (see 3.5.1)	4.5 oz	34 g
Potassium fluoride, anhydrous	4.5 oz	34 g
Trisodium phosphate ($Na_3PO_4.12H_2O$)	4.5 oz	34 g
Potassium manganate (or permanganate, see 7.1)	2.5 oz	19 g
Water to make	1 gal	1 L
For preparation and replenishment of bath, see para. 3.5.1		
Anodizing conditions	Type I, class A	Type II, class A
Temperature	Room temp.	Room temp. (cooling required)
Current density	18-20 A/ft^2	18-25 A/ft^2
Voltage	0-60 ac	0-85 ac
Time (approx)	8 min.	60 min.
Tank	See table I	See table I

TABLE II A. Operating data for type 1, class A post treatments.

	Grade 1	Grade 2
Solution	No post treatment; dye this coating as indicated below.	Bifluoride-dichromate solution oz/gal g/L Ammonium bi fluoride (NH_4HF_2) 10.8 oz 81 g Sodium dichro mate ($Na_2Cr_2O_7 2H_2O$) 2.7 oz 20 g
Conditions -		
Temperature	---	Room temperature
Time	---	1 Minute
Rinse	After tan coating is applied, rinse thoroughly in cold then hot water	No rinse
Dye solutions	Sandoz Aluminum Black 3 B or equal	
Strength	1.3 to 1.6 oz/gal (10 to 12 g/L)	
Temperature	150 F (66 C)	
Time	5 Minutes	
Sealing	Rohm and Haas Acryloid B66 or equal 1/	

1/ When specified, the dyed coating shall be sprayed lightly with an acrylic ester resin lacquer in order to seal the pores.

TABLE II B. Operating data for type II, class A, post-treatment

	Grade 1	Grade 3	Grade 4	Grade 5 1/
Solution	No post treatment	Bifluoride-dichromate solution 10.8 oz/gal (8 g/L) ammonium bifluoride (NH_4HF_2) 2.7 oz/gal (20 g/L) Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$)	Same as grade 3	Same as grade 3
Condition				
Temp.	---	Room Temperature	Same as grade 3	Same as grade 3
Time	---	1 Minute	"	"
Rinse	---	No Rinse	"	"
Aging conditions				
Temp.	---	---	Approx. 185 F (85 C)	Approx. 185 F (85 C)
rh 2/	---	---	85 ± 5% rh	85 ± 5% rh
Time	---	---	6 hrs to overnight	3 to 4 hrs

1/ Repeat entire post-treatment including aging.
2/ Relative humidity (rh) - Moisture condensation on parts shall be avoided.

TABLE III. Process conditions for type I, class C and type II, class D anodize (Dow 17).

Cleaning (see 3.3.1)

Anodizing bath for use with alternating current (ac)

	Operating Range		Initial Composition
Ammonium bifluoride (NH_4HF_2)	30-60 oz/gal (225-450 g/L)	- -	32 oz/gal (240 g/L)
Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$)	6.7-16 oz/gal (50-120 g/L)	- -	13.3 oz/gal (100 g/L)
Phosphoric acid (concentration: 85% sp gr 1.69)	6.5-14 fl oz/gal (51-109 mL/L)	- -	11.5 fl oz/gal (90 mL/L)
Water	to make 1 gal or 1 L	-	to volume

Anodizing bath for use with direct current (dc)

	Operating range	-	Initial Composition
Ammonium bifluoride (NH_4HF_2)	40-60 oz/gal (300-450 g/L)	- -	48 oz/gal (360 g/L)
Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$)	6.7-16 oz/gal (50-120 g/L)	- -	13.3 oz/gal (100 g/L)
Phosphoric acid (concentration: 85%, sp gr 1.69)	6.5-14 fl oz/gal (51-109 mL/L)	- -	11.5 fl oz/gal (90 mL/L)
Water	to make 1 gal or 1 L	-	to volume

For preparation and replenishment (see 3.5.2)

TABLE III. Process conditions for type I, class C and type II, class D anodize (Dow 17). - Cont'd

	Type I, class C		Type II, class D	
Anodizing conditions				
Temperature	160-180 F(71-82 C)		160-180 F(71-82C)	
Current density	5-50 A/ft^2 (0.5-5.4 A/dm^2)		5-50 A/ft^2 (0.5-5.4 A/dm^2)	
Voltage	up to 75 V		up to 100 V	
	ac	dc	ac	dc
			(minimum)	
Quantity of electricity used (amp min/ft^2)	80-100	50-60	460	300
Time, min, based on 20A/ft^2 current density 1/	4-5	2.5-3	23	15
Rinse	Water			
After anodizing, coatings may be sealed as follows:				
Sealing bath composition:				
Sodium silicate solution: 40-42°Be (1.38-1.41 sp gr.)	10% by volume			
Water	Remainder			
Conditions				
Temperature	200-212 F (94-100 C)			
Time	15 minutes			
Rinse	Cold running water, then in hot water to facilitate drying.			

1/ The time varies inversely as the current density; e.g., for 10 A/ft^2 all times would be doubled.

3.4.2 Arrangement of electrodes: When alternating current is used, the parts shall be divided into two groups, each group serving as an electrode. The surface area of each group shall be approximately the same. When direct current is used (optional for type I, class C, and type II, class D), the tank shall be made the cathode and shall be grounded. However, if the tank is composed of or lined with a nonconductive material, the cathode shall consist of a steel plate immersed in the treating bath.

3.4.3 Regulation of current: Due to the increasing resistance of the anodic coating being deposited, the voltage required to produce a given current increases during the anodizing process. The voltage shall be regulated manually or automatically in order to maintain the desired current.

3.4.4 Frequency of alternating current: When alternating current is used, it shall have a frequency of 60 + 10 cycles per second. (60 ± 10 Hz)

3.4.5 Complex parts: When complex parts are treated, they shall be agitated while totally immersed in the solution in order to minimize the entrapment of air in pockets or blind holes. The parts should be repositioned periodically so as to bring the bath in contact with the uncoated areas and to prevent attacks at the solution-air interface at such pockets.

3.5 Preparation and Replenishing of Processing Baths:

3.5.1 Type I, class A and type II, class A treatment bath (HAE):

3.5.1.1 Preparation: Dissolve the ingredients in the water in the order given. Instead of aluminum hydroxide, scrap aluminum may be used at the rate of 1.5 oz/gal (11.3 g/L) of bath. However, to prevent contamination of the bath, the scrap must consist only of 1100 aluminum alloy, which contains 99 percent aluminum. In addition, the aluminum must be allowed to react in another container with part of the potassium hydroxide until dissolved, before transferring to the processing tank. Any undissolved residue shall be separated by decantation and discarded.

CAUTION: The reaction of aluminum with potassium hydroxide produces hydrogen gas. Precaution should be taken to dissipate the envolved gas and to keep flames out of the room.

If potassium manganate is not available, an equal quantity of potassium permanganate may be used. The permanganate shall be completely dissolved in hot water before adding to the tank.

3.5.1.2 Replenishment: Depletion of the manganate is indicated by the lightening of the characteristic brown color of the coating. Chemical analysis of the bath is not necessary. Adjustments of aluminum and manganese content is normally made when 15 sq. ft. (1.4 m^2) of magnesium for type II, class A, or 140 sq. ft. (13 m^2) of magnesium for type I, class A, per gallon of bath have been coated. To replenish, add 50 percent of the original quantities of aluminum and manganate. The aluminum shall be dissolved separately in a sufficient amount of potassium hydroxide before adding. Potassium mangagate shall be first dissolved in 5 percent potassium hydroxide solution. If permanganate is used, it shall be dissolved in hot water, and twice the amount of potassium hydroxide added. The free alkali content shall be maintained at a level of 10 to 12 percent free potassium hydroxide. (See appendix for methods of analysis). Fluoride and phosphate deplete at a very slow rate.

3.5.2 Type I, class C and type II, class D (Dow 17) bath:

3.5.2.1 Preparation: Heat one-half of the required amount of water to 160 F (71 C). Add the ammonium bifluoride slowly with constant stirring, then add the other chemicals and the rest of the water. Heat to 180 F (82 C) and stir vigorously 5 to 10 minutes. Reheat and stir after each cooling.

3.5.2.2 Replenishment: Adjustment of the bath should be made when about 20 sq. ft. (1.9 m^2) of surface per gallon of bath have been treated, or when the final operating voltage exceeds the values established by actual practice by 5 volts ac or 10 volts dc. The amounts of the various ingredients to be added are determined by analysis according to standard methods. If the operating voltage attained after the addition of make-up chemicals is lower than normal, add sodium (not ammonium) acid fluoride until the normal voltage is established.

3.6 Requirements of Anodizing Surfaces:

3.6.1 General requirements: The coating produced by anodizing of the magnesium surface shall be uniformly applied over the entire surface, and shall be free from scratches or other damage or irregularities, and from breaks other than at the point of electrical contact. The procuring activity may also require the color to be a reasonably close approximation to that of a sample consisting of an anodized piece or pieces of the same magnesium alloy. Color may vary with different alloys.

3.6.2 Dimensional change: Unless otherwise specified, the dimensional change per surface resulting from anodizing shall comply with the requirements stated in table IV when tested in accordance with 4.4.4.1. It should be noted that maximum corrosion resistance can be achieved even with the minimum value of the range for each type. No significant advantages are gained by deposits heavier than the specified range.

TABLE IV. Dimensional change requirements of anodized coatings.

	Dimensional increase per surface	
	Range	Typical1/
Type I, class A (HAE)	0.1-0.3 mil(2.5-7.6 um)	0.2 mil(5.1 um)
Type I, class C (Dow 17)	0.1-0.5 mil(2.5-12.7 um)	0.3 mil(7.6 um)
Type II, class A (HAE)	1.3-1.7 mil(33-73 um)	1.5 mil(38 um)
Type II, class D (Dow 17)	0.9-1.6 mil(23-41 um)	1.2 mil(30 um)

1/ On AZ31B magnesium alloy.

3.6.3 Alternative weight requirements: If the dimensional change of the anodic coating cannot be determined accurately because of the irregular shape or variable dimensions of the items, a coating weight per unit of surface area requirements may, with the approval of the procuring activity, be substituted for the dimensional change requirement. The required weight of coating shall be based on a correlation between weight and coating thickness established to the satisfaction of the procuring activity by tests performed on suitable specimens anodized to various coating thicknesses over the required range of thickness. The weight of the anodized coating shall be determined according to 4.4.5.

3.6.4 Special requirements: In addition to the requirements specified above, anodized coating shall conform to any special requirements specified in the contract or purchase order.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for inspection:

Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Materials:

In case of doubt, the contractor may be required to furnish satisfactory evidence of compliance of materials used in processing with the requirements of this specification.

4.3 Process inspection:

4.3.1 Process control: Process inspection shall consist of the observation of voltage, current density, temperature, time of treatment, and all other pertinent process conditions.

4.3.2 Compliance: The process shall be inspected for compliance with the requirements of this specification at the beginning of each production run and at the beginning of the re-use of any equipment following any period of one week or longer during which the equipment is not used for production in accordance with this specification. In addition, sufficient periodic inspections of the process shall be made to insure compliance with the requirements of this specification.

4.3.2.1 Records: The contractor will maintain a permanent record of the history of each anodizing bath, showing all additions of chemicals, results of analyses, and quantity and kinds of items processed in the bath. These records shall be made available to the Government for examination.

4.4 Inspection of processed items:

4.4.1 Lot: A lot shall consist of all processed articles of the same kind presented for acceptance at the same time, or completed within a period of time specified by the procuring activity. Lot size may also be determined by the procuring activity as a number of square feet (square meters) of surface treated per unit volume of the treating bath.

4.4.2 Sampling:

4.4.2.1 For visual examination: Random sampling for visual examination shall be conducted in accordance with MIL-STD-105, inspection level II, with an acceptable quality level (AQL) of 1.5 percent defective.

4.4.2.2 For determination of dimensional change: A number of pieces corresponding to inspection level S-4 of MIL-STD-105 shall be selected from the lot prior to anodizing, provided however that the sample shall consist of not less than ten articles, or of the entire lot, whichever is smaller. The AQL shall be 1.5 percent unless otherwise specified.

4.4.2.3 For coating weight determination: When the thickness of the coating on the anodizing articles cannot be accurately determined, five specimens shall be selected from the lot determination of the weight of the anodized coating according to 4.4.5.

4.4.2.4 Concurrently anodized specimens: When the use of a sample of anodized items for the determination of the dimensional change or the weight is impracticable, suitable specimens composed of the same basis metal and processed concurrently with the lot which they represent may be used for the dimensional change determination.

4.4.3 Visual examination of processed items: The sample of processed items selected according to 4.4.2.1 shall be visually examined for compliance with the requirements of this specification and any other requirements specified by the procuring activity. If the number of defective items exceeds the acceptance number for the given sample, the lot represented by the sample shall be rejected.

4.4.4 Dimensional change determination:

4.4.4.1 Measurement: The thickness of each piece selected prior to anodizing shall be determined by measuring with a micrometer which reads accurately to 0.0001 inch (2.5 um). The pieces shall then be processed concurrently with the remainder of the lot. The pieces shall be distributed throughout the various batches during processing so as to be representative of the entire lot. The thickness of each piece shall be determined after anodizing by measuring in the same location as before. The dimensional change per surface of each piece shall be determined by substracting the two measurements and dividing by two if both surfaces were anodizing.

4.4.4.1.1 Correction factor: In the case of the type II anodizing process, the anodizing bath removes some of the basis metal at the same time as it deposits the anodize coating, therefore the dimensional change as measured must be multiplied by a factor of 1.3 to give the true thickness. For type I anodizing, no basis metal or only a negligible amount is removed, and a correction factor is not necessary.

4.4.4.2 Alternative procedures: In the event that the procedure of 4.4.4.1 is not applicable because of the shape of the anodized articles or by reason of excessive or uncontrolled variability of the thickness of the item, or for any other reason, a coating weight requirement shall be substituted for dimensional change as provided in 3.6.3, and the coating weight shall be determined according to 4.4.5. If the determination of coating weight is not practicable because of the shape or size of the anodized articles, suitable specimens composed of the same basis metal and anodized concurrently with the lot which they represent may be substituted for anodized pieces. Other methods that can be used to evaluate the quality and thickness of the anodic coating are the ASTM B 487 (microscopical) and ASTM B 244 (Eddy current) methods.

4.4.5 Determination of weight of anodizing coating:

4.4.5.1 Type I coatings: Weigh a specimen of known area. Immerse it for about 5 minutes in a solution containing 300 grams of chemically pure, sulfate free, chromic acid per liter at room temperature. During this operation, maintain a specimen of commercially pure aluminum in the stripping solution in order to minimize the loss of magnesium metal. Prevent contact between the aluminum and magnesium. Rinse, dry and weigh again. Repeat this operation until constant weight is obtained. For purposes of this test, a weight difference of not more than 0.25 milligram per square inch (3.9 milligram per square decimeter) shall be considered constant.

4.4.5.2 Type II coatings: Use the same procedure as in 4.4.5.1 except that the temperature of the chromic acid solution may be 120° ± 5°F (49 ± 3 C) for faster stripping, the initial period of immersion is 10 minutes and subsequent immersions shall be 5 minutes until the weight difference is not more than 1.0 milligram per square inch. (15.5 milligram per square decimeter)

4.4.6 Other tests: The anodized pieces shall be tested for conformance to any special requirements called out in the contract or purchase order. The tests shall be performed according to the methods specified therein.

4.5 Rejection:

4.5.1 For nonconformance of process: In the event that the process conditions fail to conform to the requirements of this specification, the contractor shall be promptly notified, and all work which is readily obtainable and which was processed under doubtful conditions shall be rejected unless the contractor can demonstrate to the satisfaction of the procuring activity that the coatings thus produced are equivalent, for the intended purpose, to coatings produced under the specified conditions. Doubtful cases shall be referred to the procuring activity for decision.

4.5.2 For nonconformance of coating: If any sample of coated articles fails to conform to the requirements of this specification, including any special requirements of the contract or purchase order, the lot represented by the sample shall be rejected. Rejected lots may be resubmitted for acceptance after the lot has been reprocessed or subjected to 100 percent inspection by the contractor in order to remove any defective items and after any deviations from the specified process conditions responsible for the failure of the coating have been corrected by the contractor.

5. PACKAGING:

5.1 Preparation for delivery:

When the contract or purchase order calls for the contractor to deliver the anodized items to the procuring activity, the anodized articles shall be prepared for shipment as specified therein or in such a manner as to afford protection against damage prior to or during shipment from the supply source to the first receiving activity. The supplier shall conduct such inspection as is necessary to assure compliance with the above requirements. Reference may be made to MIL-STD-649 for product preparation for shipment and storage.

6. NOTES:

6.1 Intended use:

Dow No 17 coatings range from thin clear to light gray-green, to thick dark green coatings, depending on the intended use. The clear coatings are used as a base for subsequent clear lacquers or paints to produce a final appearance similar to clear anodizing on aluminum. The light gray-green coatings are used in most applications which are to be painted. The thick, dark green coating offers the best combination of abrasion resistance, protective value and paint base characteristics.

The HAE anodic finish is probably the hardest coating currently available for magnesium. These coatings exhibit stability at high temperatures and good dielectric strength. This coating provides an excellent paint base. The coating requires a resin seal or paint for maximum corrosion protection.

6.2 Acid pickling:

Normal cleaning and pickling procedures are recommended when applying the light gray to green coating (type I, class C) but may not be required for the dark green coating (type II, class D). Because of the relatively high voltage used to obtain the thick dark green coating and the bath's excellent polarization characteristics, the bath has a strong tendency to clean the magnesium surface during the application of this coating. Graphite, corrosion product, surface impurities and other non-metallic films are removed.

6.3 Post treatments:

The term "post-treatments" means any treatment used after anodizing which causes a chemical change within the anodic film such as the post-treatments which are characteristic of each of grades 3, 4 and 5 of type II, class A finishes.

6.4 Rinsing:

Rinse tanks used in a countercurrent flow arrangement are highly desirable in minimizing the amount of water required to obtain a desired rinsing criterion and facilitate meeting the EPA standard.

6.5 This paragraph was deleted as it did not pertain to the converted SAE document.

PREPARED UNDER THE JURISDICTION OF AMS COMMITTEE

APPENDIX
METHODS OF ANALYSES

10. SCOPE:

10.1 Instrumentation:

The commercial use of instrumental devices for the analyses of anodizing solutions is not necessary. Unless otherwise specified in the contract, the contractor may use his own or any other facilities suitable for performing solution control analyses. Analytical methods requiring the photometric techniques of spectrophotmetry and modern atomic absorption spectroscopy can be adopted for solution control analyses.

10.2 Appendix:

This appendix contains the detailed methods for the analyses of the HAE and the Dow No. 17 Anodizing baths by wet chemistry techniques.

10.3 Methods:

Methods are included for determining the concentrations of aluminum, phosphorus, fluoride, manganese, free alkali and hexavalent chromium.

10.4 Sampling:

Samples should be taken from a well mixed bath at operating temperature. A freshly stirred bath should be allowed to stand a few minutes to allow for settling of insoluble constituents.

10.5 Equipment:

Polyethylene equipment should be used for handling the concentrated solution.

HAE ANODIZING SOLUTION

20. PHOSPHATE:

20.1 Introduction:

The separation of the phosphorus as ammonium phosphomolybdate followed by titration with standard alkali appeared to be the most logical approach to this determination. The only possible interference seemed to be the fluoride which retards formations of the precipitate. The fluoride can be removed by volatilization.

20.2 Reagents:

20.2.1 Ammonium molydate solution: Mix 100 g of 85 percent molybdic acid with 400 mL of water; add 80 mL of ammonium hydroxide and filter when solution is complete. Prepare a second solution containing 400 mL of nitric acid and 600 mL of water. Vigorously agitate the latter by means of a current of air and add the molybdate solution very slowly through a tube dipping under the solution. When all has been added, continue the current of air for one hour. Let stand, filter if necessary, and keep in a glass stoppered bottle.

20.2.2 Sodium Hydroxide, 0.1 N: Standardized against acid potassium phthalate.

20.2.3 Nitric acid, 0.1 N: Standardized against standard sodium hydroxide.

20.2.4 Standard: National Bureau of Standards standard sample No. 186 - II, disodium hydrogen phosphate.

20.3 Procedure:

Pipette a 2 mL bath sample into a 250 mL beaker.

Add 20 mL of water and 20 mL of nitric acid (1:1) slowly.

Add 1 g of sodium nitrate, 10 mL of perchloric acid (70-72 percent) and evaporate to fumes. Fume for 5 minutes.

Cool, dilute to 100 mL, and filter through a Whatman No. 40 filter paper, or equivalent, catching filtrate in a 200 mL. Erlenmeyer flask fitted with a ground glass stopper. Wash well with water.

Make the solution just alkaline to methyl red with ammonium hydroxide; then just acid with concentrated nitric acid and add 10 mL in excess.

Add 10 g of ammonium nitrate, heat solution to 40 C and add 60 mL of ammonium molybdate reagent.

Stopper flask, shake for 5 minutes and allow to stand at least 1 hour.

Filter through a Gooch crucible or equivalent fitted with a double No. 42 Whatman filter paper, or equivalent, using suction. The paper may be cut to correct size by means of a cork borer of an appropriate size.

Wash the flask, precipitate, and paper twice with 5 mL portions of 1:100 nitric acid, then wash the flask and precipitate 5 times with 15 to 20 mL portions of 1 percent sodium nitrate solution.

Wash the precipitate with 1 percent sodium nitrate solution until the washings give no acid reaction with methyl orange.

Transfer paper and precipitate, with the aid of a stirring rod and a stream of water from a wash bottle, to a 400 mL beaker.

Add 50 mL of 0.1 N sodium hydroxide from a pipette, using a small portion of this to dissolve any precipitate adhering to the flask. Transfer liquid from flask to beaker with the aid of some water from a wash bottle.

Stir by means of a magnetic stirrer until the precipitate is completely dissolved. Add 5 drops of 0.1 percent phenolphthalein solution and titrate with 0.1 N nitric acid to the disappearance of the pink color.

Calculation:

$$\text{Concentration of } Na_3P0_4.12H_20(g/L) = \frac{[(A \times B) - (C \times D)] \times 0.3802 \times 1000}{23 \times E}$$

where:

A = mL of alkali
B = normality of alkali
C = mL of nitric acid
D = normality of nitric acid
E = sample volume (mL)

30. ALUMINUM:

30.1 Introduction:

Aluminum can be quantitatively separated from phosphate and fluoride by precipitation with 8-hydroxyquinoline in an ammoniacal solution. The possible interferences are magnesium, which could arise from the processed material, and manganese. The former was shown by spectrographic examination to be absent from both fresh and used baths; the latter can be removed by precipitation as manganese dioxide using hydrogen peroxide in an alkaline medium.

30.2 Reagents:

30.2.1 8-hydroxyquinoline solution: Dissolve 5 g of 8-hydroxyquinoline in 100 mL of 2 N acetic acid.

30.2.2 Acetic acid, 2 N.: Dilute 60 mL of glacial acetic acid to 500 mL with water.

30.2.3 Ammonium hydroxide, 6 N.: Dilute 200 mL of ammonium hydroxide (sp gr 0.90) to 500 mL with water.

30.2.4 Standard: Aluminum wire, C.P. Baker’s Analyzed, label analyses 99.99 percent.

30.3 Procedure:

Pipette a 3 mL bath sample into a 250 mL beaker.

Dilute to 50 mL with water and heat to boiling.

To the boiling solution add 10 mL of 30 percent hydrogen peroxide dropwise. Continue boiling until the volume is reduced to about 25 mL.

Filter through a Whatman No. 42 filter paper, or equivalent, and wash well with cold water.

Dilute the filtrate to 150 mL and make just acid to litmus paper using hydrochloric acid (1:1).

Heat the solution to 75° to 80° and add 6 mL of 8-hydroxyquinoline solution by means of a pipette.

Add 6 N ammonium hydroxide slowly with stirring until just basic to litmus then add 1 to 2 mL in excess. Set the solution aside, without further heating, for one hour or more.

The supernatant liquid should be yellow when the precipitate has settled indicating that an excess of reagent is present. If the superantant liquid is not yellow., reacidify with 1:1 hyrdrochloric acid, add 1 mL of 8-hydroxyquinoline, and precipitate as before. This procedure is to be repeated until the supernatant liquid is yellow.

Filter off the precipitate through a tared sintered glass crucible of medium porosity, and wash thoroughly with cold water.

Dry for 1 hour at 130 C. Cool in a desiccator for a half hour and weigh.

Calculation:

$$\text{Concentration of aluminum (g/L)} = \frac{A \times 0.05872 \times 1000}{B}$$

where:

A = weight (g) precipitate
B = sample volume (mL)

40. FLUORIDE:

40.1 Introduction:

A number of methods were tried before adopting one which proved sufficiently accurate and reproducible. Among the former was a modification of the colorimetric method using the ferric compound of 7-iodo-8-hydroxyquinoline-5-sulfonic acid (ferron). Results with this method ran from 5 to 50 percent low. Another approach involved a distillation procedure followed by precipitation of the fluorine as PbC1F. Results in this case were also low and erratic. The method which finally proved to be satisfactory is a modification of that used by Hoffman and Lundell.

40.2 Reagents:

40.2.1 Phenolphthalein: Dissolve 1 g of the powder in 80 mL of 95 percent ethyl alcohol and dilute to 100 mL with water.

40.2.2 Brom phenol blue indicator: Grind 0.4 g of the dry powder with 6 mL of 0.1 N Na0H and dilute to 100 mL with water.

40.2.3 Lead chlorofluoride wash solution: Dissolve 10 g of lead nitrate in 200 mL of water, and pour the solution into 100 mL of a solution containing 1.0 g of sodium fluoride and 2 mL of hydrochloric acid. Mix thoroughly, let settle, decant the supernatant liquid with 200 mL, portions of water. Add 1 L of water to the precipitate, stir occasionally, let stand for at least 1 hour and filter. More wash solution can be prepared as needed by treating the precipitate with fresh portions of water.

40.2.4 Standard: A thoroughly mixed and dried sample of Baker's C.P. sodium fluoride was used. (A sample was analyzed by precipitating the fluorine as PbC1F and found to be 100.0 percent NaF.)

40.3 Procedure:

Pipette a 5 mL bath sample into a 400 mL beaker.

Dilute to about 50 mL, bring to a boil, remove from hot plate and add 10 mL of 30 percent hydrogen peroxide dropwise to the hot solution.

Return beaker to the hot plate and continue boiling until the solution has been evaporated to about 25 mL.

Dilute to about 50 mL with water and filter through a Whatman #42 filter paper, or equivalent. Wash well with cold water, catching the filtrate in a 400 mL beaker.

Dilute the filtrate to about 200 mL, add 6.0 g of sodium.

Return the gelatinous mass to the original beaker twice and thoroughly disintegrate in the wash solution. (The mass can be returned to the beaker by rotating the funnel above the beaker and cutting the precipitate loose from the paper with a jet of hot water.)

Add 2 drops of brom phenol blue to the filtrate and the $HN0_3$ (1:1) until filtrate is nearly neutral, leaving the solution slightly alkaline. Boil until the solution is reduced in volume to about 250 mL.

Add $HN0_3$ (5:95) until the color of the solution just changes to yellow, add Na0H (10 percent) until the color just changes to blue and then add 3 mL of 10 percent NaC1.

Add 2 mL of HC1 (1:1) and 5 g of lead nitrate, and heat on a steam bath.

As soon as the lead nitrate is in solution, add 5 g of sodium acetate, stir vigorously and digest on a steam bath at least a half hour.

Allow to stand overnight at room temperature and filter through a tared, medium porosity, sintered glass crucible.

Wash beaker and precipitate once with cold water, then four or five times with saturated lead chlorofluoride solution after scrubbing beaker with a policeman. Finally wash precipitate once with cold water. (Excessive washing with water must be avoided since the precipitate is somewhat soluble in water.)

Dry in an oven for 1 hour at 130 C, cool in a desiccator for half hour and weigh.

Calculation:

$$\text{Concentration of potassium fluoride (g/L)} = \frac{A \times 0.2220 \times 1000}{B}$$

where:

A = weight of lead chlorofluoride in grams (g).
B = volume (mL) of sample.

50. FREE ALKALI:

50.1 Introduction:

A direct titration of free alkali with acid was not feasible since upon dilution or acidification K_2Mn0_4 decomposes and produces hydroxyl ions. The trisodium phosphate and carbonate present in the bath would also be titrated. It was found that $Ba(N0_3)_2$ could be used to precipitate the manganate, phosphate, carbonate, and fluoride from the bath without affecting the free alkali.

50.2 Reagents:

50.2.1 Sodium hydroxide, 0.25 N: Dissolve 50 g of C.P. sodium hydroxide in 50 mL of water and allow to cool. Centrifuge the solution for a short time. Transfer 13 mL of the clear carbonate-free alkali solution to a 1 L volumetric flask and dilute to 1 L with $C0_2$ free water.

Transfer this solution after thorough mixing to a pyrex or alkali resistant glass bottle fitted with a 2 hole stopper. Through one hole, the end of an ascarite tube is inserted, and through the other, a glass delivery tube is installed reaching to the bottom of the bottle. The ascrite tube is fitted with a double valve rubber bulb. When not in use, the end of the delivery tube is protected by covering with a rubber policeman.

Standardization. Accurately weigh out approximately 2 g of pure acid potassium phthalate and transfer to a 250 mL pyrex flask. Dissolve in 50 mL of $C0_2$ free water and titrate with Na0H solution, using phenolphthalein indicator.

50.2.2 Hydrochloric acid, 0.25 N: Standardize by titrating against 0.25 N sodium hydroxide to a pH of 10.5. The value obtained is empirical and slightly different from the true normality.

50.2.2.1 Apparatus: A pH meter fitted with a special glass electrode suitable for measurement in the range pH 9 to pH 13.5.

50.5 Procedure:

Pipette a 2 mL bath sample into a 25 mL glass-stoppered flask.

Add carefully from a burette 10.00 mL of 0.25 N Na0H.

Add 0.5 g $Ba(N0_3)_2$ crystals, stopper tightly and shake vigorously for at least one minute.

Filter through a Whatman #40 filter paper, or equivalent, being careful to rinse all material from the flask and the stopper into the funnel with cold water. Catch the filtrate in a 250 mL beaker which should be kept covered as much as possible.

Wash paper and precipitate seven or eight times with cold water.

Test for complete removal of alkali from precipitate by collecting 10 mL of filtrate and adding 1 drop of 0.1 N HC1 and 1 drop of phenolphthalein indicator. If the test indicates that alkali is still present in the precipitate, continue washing until the alkali has been removed.

Titrate the filtrate with 0.25 N hydrochloric acid to a pH of 10.5.