

MIL-M-3171C

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SUPERSEDING

MIL-M-3171B

16 October 1964

MILITARY SPECIFICATION

MAGNESIUM ALLOY, PROCESSES FOR PRETREATMENT AND
PREVENTION OF CORROSION ON

This specification is mandatory for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope - This specification covers general requirements for the apparatus, material, and procedures to be used in the processing of magnesium base alloys for the purpose of increasing their corrosion resistance and by producing surfaces suitable for organic paint finish systems.

1.2 Classification -

1.2.1 Types - The magnesium corrosion preventive treatments shall be of the following types:

Type I - Chrome Pickle Treatment

Type III - Dichromate Treatment

Type IV - Galvanic Anodizing Treatment

Type VI - Chromic Acid Brush-on Treatment

Type VII - Fluoride Anodizing Process plus Corrosion Preventive Treatment

Type VIII - Chromate Treatment

2. APPLICABLE DOCUMENTS

2.1 The following documents 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.

FSC MFFP

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SPECIFICATIONS

Military

MIL-C-46079	Coating, Epoxy, Baking Type for Magnesium Casting
MIL-M-46080	Magnesium Castings, Process for Anodic Cleaning and Surface Sealing of

STANDARDS

Military

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
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(Copies of specifications and standards required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

3. REQUIREMENTS

3.1 General requirements -

3.1.1 Materials - All materials used in the cleaning or treating of magnesium base alloys shall be of good quality and suitable for the purpose intended. All chemicals used in cleaning and treating shall be free from metallic impurities such as salts of heavy metals (lead, nickel, zinc, arsenic, antimony, etc.).

3.1.2 Workmanship - The treatments covered by this specification shall be controlled and operated to give a uniform surface conforming to the requirements specified herein. The surface shall be free from breaks, discontinuities, scratches, and other damage. The size and number of contact marks shall be held at a minimum.

3.2 Cleaning and pickling operations (see 3.12)-

3.2.1 Solvent cleaning - Grease or oil shall be removed by means of vapor degreasing, ultrasonic cleaning, organic solvents, or by emulsion cleaning which employs a mineral oil distillate and an emulsifying agent. For this cleaning operation, chlorinated solvents, petroleum spirits, naphtha, lacquer thinners, and similar grease solvents which do not attack, magnesium may be used. Methyl or wood alcohol (CH_3OH) is specifically prohibited as a solvent cleaner for magnesium alloys.

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- # 3.2.2 Mechanical cleaning - Mechanical cleaning shall consist of sand, shot, grit, or vapor blasting, sanding, hard bristle brushing, grinding and rough polishing. When sand, shot, or grit blasting methods are employed, there is danger of surface contamination which will greatly increase the initial corrosion rate of magnesium. In such case, subsequent pickling operations, as specified in 3.2.4.2, or fluoride anodizing as specified in 3.9.1 shall be employed
- # 3.2.2.1 New sand castings - New sand castings shall be sand or grit blasted and then pickled in conformance with 3.2.4.2 or fluoride anodized as specified in 3.9.1.
- # 3.2.2.2 Discolored or corroded castings - Discoloration or corrosion products shall be removed from rough unmachined castings. This operation may be performed by blasting and subsequent pickling as specified in 3.2.4.2. Machine parts or sections, which must be held to close dimensional tolerances, may be cleaned by use of a chromic acid pickle solution conforming to 3.4.1.
- # 3.2.2.3 Welding flux - When using flux in the welding operations, the welded sections shall be thoroughly cleaned immediately after the operation to remove all traces of flux. Where possible, hot water and a hard bristle brush shall be used to insure thorough flux removal. Inaccessible welded sections shall be washed with a high velocity stream of water (preferably hot). They shall then be immersed for 1 hour in a 2 to 5 percent sodium dichromate solution maintained at 180 to 212° F. The parts shall be thoroughly rinsed in cold running water.
- # 3.2.3 Alkaline cleaning - Cleaning prior to application of treatments other than Type I when used for protection during shipment or storage, shall be done by cleaning the parts in an alkaline cleaner recommended for steel or in a cleaning solution as specified in 3.3.2. Any alkaline cleaner used shall have a pH that is maintained above 8. Alkaline cleaning, prior to application of Type I when used for protection during shipment or storage only, may be omitted provided the parts are free from grease, oil, or other deleterious deposits at the time of application. Alkaline cleaners containing more than 2 percent caustic, such as sodium hydroxide will etch ZK60A, ZK60B, and some magnesium-lithium alloys with resultant decrease in dimensions. If such a dimensional change is not desirable, the alkaline cleaners used on these alloys should not contain more than 2 percent caustic.
- # 3.2.3.1 Graphite lubricant removal - Graphite base lubricants shall be removed from hot formed magnesium sheet parts by soaking the parts for 10 to 20 minutes in an aqueous sodium hydroxide (NaOH) bath containing 13 oz/gallon of NaOH and maintained at 190 to 212° F. This bath shall be maintained at a pH

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above 13. One-tenth ounce per gallon of soap or wetting agent may be added to this solution if removal of heavy films of mineral oil is required. The parts shall be thoroughly rinsed in cold water and then immersed in a chromic-nitrate pickle as specified in 3.4.2 for approximately 3 minutes. If complete cleaning is not obtained the first time, this operation shall be repeated until the parts are clean. Because of the difficulty of removing graphite from chrome pickled sheet, such sheet shall not be used for forming unless the chrome pickle coating has been completely removed as specified in 3.2.3.2.

3.2.3.2 Previously applied chemical finishes - Previously applied chemical finishes shall be removed. Magnesium base alloys sometimes are furnished with Type I treatment which provides protection during shipment, storage, and machining. The coating resulting from Type I treatment remaining on the unmachined areas will impair the film produced by Type III and Type IV treatments and consequently, must be removed. This operation may be performed by use of alkaline cleaners such as the types specified in 3.2.3. If the finish is difficult to remove, the part should be immersed in the chromic acid pickle specified in 3.4.1. Alternate immersion in the alkaline cleaner and chromic acid pickle may be necessary to remove very old finishes.

3.2.3.2.1 The Type VI treatment may be applied over previously applied thin film by Type I or Type VI treatments without removing these finishes (see 3.8.1).

3.2.4 Acid pickling -

3.2.4.1 General pickling - General pickling to remove oxide layers, old chemical finishes, burned-on drawing and forming lubricants, and other water insoluble or non-emulsifiable substances shall be accomplished, preferably by using a chromic acid type pickle conforming to 3.4.1 or 3.4.2.

3.2.4.2 Pickling of magnesium sand and permanent mold castings - Magnesium sand and permanent mold castings which have been subjected to blasting operations shall be pickled in a sulfuric acid pickle conforming to 3.4.3 or in a sulfuric-nitric acid pickle conforming to 3.4.4. Pickling operations shall be continued until 0.002 inch of surface has been removed, whenever dimensional tolerances permit.

3.2.4.3 Pickling of magnesium sheet and plate - Magnesium sheet and plate up through 0.500 inch thick shall have the mill scale removed to insure a clean surface to which subsequent treatment will be applied. Where dimensional tolerances permit, 0.0005 inch to 0.001 of metal surface shall be removed. An acetic-nitrate pickle conforming to 3.4.7 or equivalent acid pickle shall be used for this operation.

3.2.4.4 Pickling of magnesium die castings - Die castings that are to be pickled for maximum corrosion resistance and a more uniform surface for treating shall be pickled in a chromic-nitric HF pickle conforming to 3.4.5 or a phosphoric acid pickle conforming to 3.4.6.

3.2.5 Retreatment of wrought and cast parts -

3.2.5.1 Grease and oil - Grease and oil shall be removed in conformance with 3.2.1.

3.2.5.2 Paint - Old paint coatings shall be removed, preferably by the use of an alkaline (caustic) liquid paint remover. Solvent type paint removers may be used provided the operation is followed by a wash with a wax-free solvent to remove any wax which may have been left on the surface. Light blasting may be used to remove paint from parts of substantial thickness, but only sanding shall be permitted on thin sheets. If sand-blasted, the part shall be pickled in conformance with 3.2.4.2.

3.2.5.3 Chemical finishes - Chemical finishes shall be removed as specified in 3.2.3.2 before retreatment.

3.2.5.4 Alkaline cleaning - Parts to be retreated shall be cleaned in an alkaline cleaning solution as specified in 3.2.3.

3.2.5.5 Corrosion or oxide films - Corrosion products or oxide films shall be removed, preferably by use of the chromic acid pickle or chromic-nitrate pickle as specified in 3.4.1 and 3.4.2, respectively.

3.3 Alkaline cleaning solutions -

3.3.1 Alkaline cleaning as required in 3.2.3 may be performed by using proprietary type cleaners, in which case the operation shall be as specified by the vendor. In no case will any such cleaner be used that has a pH lower than 8. An alkaline cleaner of the formulation given in Table I may be used quite satisfactorily. The parts are simply immersed in the solution, or, if direct current is available, the parts are made cathodes in the bath and a current density of 10 to 40 amp/sq. ft. at 6 volts, is applied. When cathodic cleaning is employed, the time required for adequate cleaning is reduced. Parts are given a thorough rinse in cold running water after alkaline cleaning. Parts shall be cleaned until no water break occurs in the rinse.

3.3.2 Anodic cleaning employing the work as the anode in the alkaline cleaning solution is generally not recommended because of the formation of undesirable oxide films as well as pitting of magnesium alloy surfaces from prolonged contact.

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TABLE I

ALKALINE CLEANER SOLUTION AND OPERATION

Composition	Ounces per gallon (water added to make one gallon)	Operating temperature °F	Treatment time- minutes	Tank construc- tion
Sodium hydroxide (NaOH)	8	190 to 212°	3 to 10	Steel
Trisodium phosphate (Na ₃ PO ₄ 12H ₂ O)	1-1/3			
Soluble soap or wetting agent	0.1			

3.4 Acid pickling solutions -

3.4.1 Chromic acid pickle - Chromic acid pickling may be used where parts with close tolerance limits are to be finished since this pickle causes no dimensional change. It can be used in removing old chemical finishes by alternate immersion in an alkaline cleaner specified in 3.3.2 and the chromic acid pickle. This pickle is satisfactory for removal of surface oxidation, corrosion products, and for general cleaning of parts. It is not satisfactory for the removal of sand or the effects of blasting and shall not be used for parts containing copper based inserts unless masked off. Excessive amounts of anions, such as chlorides, sulfates, and fluorides must not be allowed to build up in the solution as these tend to coat or etch the metal rather than clean the surface. Silver nitrate (AgNO₃) may be used to precipitate out any chloride buildup, thus extending the life of the bath, but it is better to discard the contaminated solution and replace with a new solution. The solution composition is indicated in Table II

3.4.2 Chromic-nitrate pickle - The chromic-nitrate pickle is not generally used where surface oxidation or corrosion products are to be removed but can be substituted for the chromic acid pickle specified in 3.4.1. It is used, as specified in 3.2.3.1, primarily for removing burned-on graphite lubricants. It is not satisfactory for the removal of sand or the effects of blasting and shall not be used for parts containing copper base inserts unless masked off. Lack of chemical action and a pH of 1.7 or higher indicates depletion of the bath. The bath may be controlled by additions of chromic acid to bring the solution back to the original pH range of 0.5-0.7. Large tanks may be revived economically in this manner about 4 times after which tanks should be drained and refilled with new solution. Excessive revivification

TABLE II
Acid Pickling Solutions

Treatment	Composition 1/		Immersion Time	Operating Temperature °F	Tank Construction	Metal Removed, Mils 2/	Ref Para
	Material	oz					
Chromic acid	Chromic acid (CrO ₃)	24	1 to 15 min	190° - 202° 3/	Lead-lined steel, or stainless steel, or 1100 aluminum	None	3 4 1
Chromic-nitrate	Chromic acid (CrO ₃) Sodium nitrate (NaNO ₃)	24 4	2 to 20 min 4/	60° - 90°	Ceramic, stainless steel, lead-lined, or synthetic rubber lined tanks or tanks lined with vinyl based materials	0 5	3 4 2
Sulfuric acid	Sulfuric acid (sp gr 1.84)	4	10 to 15 sec	70° - 90°	Ceramic, rubber-lined, lead, or other suitable tanks	2 0	3 4 3
Nitric-sulfuric acid	Nitric acid (sp gr 1.42) Sulfuric acid (sp gr 1.84)	10-1/4 2-1/2	10 to 15 sec	70° - 90°	Ceramic, rubber-lined or other suitable tanks	2 0	3 4 4
Chromic-nitric-HF	Chromic acid (CrO ₃) Nitric acid (sp gr 1.42) Hydrofluoric acid (60% HF)	37-1/2 3-1/4 1	1/2 to 2 min	70° - 90°	Synthetic rubber lined tank or tanks lined with vinyl base materials	0 5 to 1	3 4 5
Phosphoric acid	Phosphoric acid (85% H ₃ PO ₄)	11 5 2	1/2 to 1 min	70° - 80°	Ceramic or tank lined with lead, glass, or rubber	0 5	3 4 6
Acetic-nitrate	Glacial acetic acid (CH ₃ COOH) Sodium nitrate (NaNO ₃)	25-1/2 6-2/3	1/2 to 1 min	70° - 80°	3003 Aluminum, ceramic, or rubber lined tank	0 5 to 1	3 4 7
Glycolic-nitrate	Glycolic acid (70% CH ₂ OHCOOH) Magnesium nitrate Nitric acid	32 27 3 8	3 to 4	70° - 80°	Stainless steel, ceramic or other suitable tanks	0 5 to 1	3 4 8
Spot weld chromic-sulfuric	Chromic acid (CrO ₃) Sulfuric acid (sp gr 1.84)	24 0 065	3 min	70° - 90°	Stainless steel, 1100 aluminum, ceramic or synthetic rubber	0 3	3 4 9

NOTE.

- 1/ Avoid splashes or fluid ounces per gallon. Water is added to make one gallon.
 2/ Average removed per surface during immersion time in pickle solution.
 3/ Bath can be operated at room temperature for a longer time if desired.
 4/ Bath maintained at pH of 0.0 to 1.7

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results in too great a consumption of chromic acid, excessive etching and staining of the magnesium. Actual time and temperature used should be regulated to produce satisfactory results. The solution composition is indicated in Table II

- # 3.4.3 Sulfuric acid pickle - Sulfuric acid pickling is used on magnesium sand castings to remove the effects of blasting operations. The pickle should be applied before any machining operation, since the amount of metal removed is likely to exceed permissible tolerances. The solution composition is indicated in Table II
- # 3.4.4 Nitric-sulfuric acid pickle - As an alternate for the sulfuric acid pickle (see 3.4.3) the nitric-sulfuric pickle may be used. The solution composition is indicated in Table II
- # 3.4.5 Chromic-nitric - HF pickle - A chromic-nitric - HF pickle may be used for pickling of castings, particularly die castings. This pickle bath removes about 0.0005 inch per minute per surface. The solution composition is indicated in Table II
- # 3.4.6 Phosphoric acid pickle - The phosphoric acid pickle may be used for pickling all types of castings, particularly die castings. It is especially effective in removing aluminum segregation from the surfaces of AZ91A and AZ91B alloys. It has been used for some wrought alloys, such as HK31A, prior to plating. This pickle bath removes about 0.0005 inch per minute per surface. The solution composition is indicated in Table II
- # 3.4.7 Acetic-nitrate pickle - The acetic-nitrate pickle is suitable for removal of mill scale and other surface contamination from magnesium sheet to ensure maximum protective finishing. This pickle can be used on other wrought forms and on solution heat treated magnesium castings. Castings in the as-cast condition (-F) or in the solution heat-treated and aged condition (-T6) should not be pickled by dipping in an acetic-nitrate pickle bath as a loose gray smut is formed. Magnesium castings in these conditions should be treated with a chromic-nitric - HF pickle (see 3.4.5). For most effective results the pickle should be allowed to remove 0.0005 inch to 0.001 inch metal per surface and therefore may not be suitable for parts requiring close tolerances. The solution composition is indicated in Table II
- # 3.4.8 Glycolic-nitrate pickle - In cases where spray pickling is used or fumes are a problem, the glycolic-nitrate pickle may be preferred to the acetic-nitrate pickle (3.4.7) to reduce acid loss from vaporization. The solution composition is indicated in Table II

- # 3.4.9 Spot weld chromic-sulphuric pickle - A chromic-sulphuric pickle may be used where parts are to be cleaned prior to spot welding. Parts are first treated by immersion in an alkaline cleaner and then given a thorough rinse in cold running water as specified in 3.3.2. Parts are then immersed in an alkali neutralizing pickle consisting of an aqueous solution of either 0.5 to 1 percent of sulphuric acid by volume or a 1 to 2 percent solution of sodium bisulphate (sodium acid sulphate) (weight by volume). Following the neutralizing dip, the parts are immersed in the chromic-sulphuric acid pickle. This treatment gives a low resistance surface for spot welding. The solution composition is indicated in Table II
- # 3.5 Type I treatment - Cleaning of parts to receive the Type I or chrome pickle treatment shall be in accordance with other requirements of this specification
- # 3.5.1 Procedures for wrought parts - Wrought magnesium parts shall be treated in a chrome pickle bath whose solution composition and operation is stated in Table III. The parts shall be immersed and agitated in the bath for a period of time ranging from 1/2 minute in a solution containing the full quantity of nitric acid to 2 minutes in a solution in which the nitric acid is nearing depletion. Usually a one minute dip will be sufficient in a properly controlled bath. After the immersion period, the parts shall be held above the tank for 5 seconds. This allows the adhering solution to drain off and produce a better colored coating. The parts shall then be washed in cold running water followed by a dip in hot water, to facilitate drying, or by exposure to heated air. Other methods, such as spraying may be used to expose the parts to the chrome pickle solution, provided the other requirements of this specification are met.
- # 3.5.2 Procedures for sand, permanent mold, and die castings - Magnesium sand, permanent mold, and die castings shall be treated in a chrome pickle bath as given in Table III. Die castings and aged sand castings shall be given a 15 to 30 second hot water dip followed immediately by immersion in the solution specified in Table III. A 10 second immersion is sufficient if the bath is operated at 120 to 140° F. If the bath is held at a lower temperature a longer time is needed. Excessive treatment time results in a powdery coating and failure to pre-heat the castings in hot water may result in no coating. If the bath for castings is not available, the bath for wrought parts may be used for die castings and aged sand castings. Sand castings in the solution heat treated condition can be chrome pickled with the solution in Table III at room temperature. After dipping, parts shall be rinsed as described above in 3.5.1.
- # 3.5.3 Precautions - Chrome pickle baths (see Table III) may remove as much as 0.0006 inch of metal per surface during treatment and, therefore, cannot be used on machined surfaces unless tolerances will permit or proper allowances have been made. Magnesium parts containing steel inserts can be

TABLE III
TYPE I CHROME PICKLE BATHS FOR TREATING MAGNESIUM ALLOYS

Types for	Composition 1/		Immersion time	Operating temperature °F	Tank construction	Rack and basket construction
	Material	Oz.				
Wrought parts	Sodium dichromate (Na ₂ Cr ₂ O ₇ · 2H ₂ O)	24	30 sec. to 2 min. followed by 5 sec. draining	70 to 110°	Stainless steel or tanks lined with glass, ce- ramics, synthe- tic rubber, or vinyl base materials	Stainless steel or monel.
	Nitric acid, concentrated (sp gr 1.42)	24				
Sand, permanent mold, and die castings	Sodium dichromate (Na ₂ Cr ₂ O ₇ · 2H ₂ O)	24	30 sec. to 2 min. followed by 5 sec. draining	70 to 140°	Type 316 stain- less steel pre- ferred. Tanks lined with synthetic rub- ber or vinyl base materials.	Type 316 stain- less steel.
	Nitric acid, concentrated (sp gr 1.42) Sodium, potassium, or ammonium acid fluoride (NaHF ₂ , KHF ₂ , NH ₄ HF ₂) ^{2/}	16 to 24				

1/ Avoirdupois or fluid ounces per gallon. Water is added to make 1 gallon. When available, water from steam condensate or treated by ion exchange should be used in preference to well or hard tap water.

2/ If sodium acid fluoride(NaHF₂) is used, add it before the nitric acid or dissolve in small quantity of water before adding since sodium acid fluoride does not go into solution with nitric acid present.

given the Type I treatment. The color, luster, and etch produced by the Type I treatment will vary with age of the solution, with the alloy composition, and with the condition of heat treatment of the alloy. The most desirable paint base is a matte gray to yellow-red, iridescent coating, which exhibits a network of pebbled etch finish as viewed under magnification. Bright, brassy coatings, which show a relatively smooth surface with only occasionally rounded pits when examined with magnification, shall be unsatisfactory where the treatment is used as a paint base, but shall be acceptable for protection during shipping and storage. This shade indicates an excess of nitric acid or nitrate salt buildup in the bath.

- # 3.5.4 Brush application - Articles too large to be immersed shall be carefully brushed with a generous amount of fresh pickle solutions specified in Table III. The solution shall be allowed to remain on the surface for at least 1 minute while brushing and shall then be washed off immediately with plenty of cold running water. The coating thus formed is less uniform in color than that produced by immersion but is equally good as a paint base. Powdery coatings are not good as paint bases and indicate poor rinsing or failure to keep the surface wet with solution during the one minute treatment time by continuous brushing over the same area. In treating riveted assemblies, care must be taken to avoid entrapping solution in the joints. Brush application is suitable for touchup of all types of treatments which have been damaged. Type I coatings are best for touchup of base areas before making an electrical bonding joint and where no primer can be used to seal the treatment.

- # 3.5.4.1 Parts such as tanks which take a long time to fill and empty may be treated with chrome pickle solution for wrought parts diluted with an equal quantity of water. The time of treatment shall be such as to produce a complete coating on all areas. Similarly, if a more dilute solution is required to treat parts by automated time cycle, more water may be used in the pickle bath.

3.5.5 Control of Type I treatment -

- # 3.5.5.1 Sodium dichromate determination - The sodium dichromate determination shall be performed as follows, or by recognized analytical methods:

Pipette 1 ml of the chrome pickle solution into a 250 ml beaker containing 150 ml of distilled water. Add 5 ml of concentrated hydrochloric acid (HCl) and 5 gms of potassium iodide (KI). Allow at least 2 minutes for the reaction to take place. Next, stir and titrate the liberated iodine with N/10 sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) until the

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yellow color is almost gone. Add several drops of starch indicator solution, then continue titration with N/10 sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) until the purple starch iodine color disappears. CAUTION: Do not add starch indicator solution until the yellowish iodine color is almost gone or incorrect results will be obtained. The final solution varies from pale green to blue in color.

Calculation:

$$\text{ml N/10 sodium thiosulphate (Na}_2\text{S}_2\text{O}_3) \times 0.6635 = \text{oz./gal. sodium dichromate (Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O)}$$

- # 3.5.5.2 Nitric acid determination - The nitric acid determination shall be accomplished as follows:

Pipette 1 ml of the chrome pickle solution into a 250 ml beaker containing 50 ml of distilled water. Standardize a glass electrode equipped pH meter with a buffer solution of approximately 4.0 and immerse the glass electrode system in the solution. Stir and titrate with N/10 sodium hydroxide (NaOH) to a pH of 4.0 to 4.05.

Calculation:

$$\text{ml N/10 sodium hydroxide (NaOH)} \times 0.8109 = \text{fl. oz./gal. nitric acid}$$

- # 3.5.5.3 Control limits of chrome pickle bath (Table III) - Depletion of the solution is indicated by the paleness of the color, shallowness of etch, and slowness of action on the metal. The paleness of color should not be confused with that obtained by insufficient exposure to air between removal from the bath and the wash in water. The chrome pickle bath shall be revived only once when non-aluminum alloys are to be processed. It may be revived 7 times for other alloys. The bath may be revived at the end of each run (the end of the run is that point at which the nitric acid has been depleted to or near 8 fl. oz./gal.) according to the following schedule.

Run Number	Chemical Content of Bath	
	$\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ oz./gal.	HNO_3 (sp gr 1.42) fl. oz./gal.
1	24	24
2	24	21
3	24	18
4 to 7	24	14

If the chrome pickle solutions are used to produce a protective treatment for shipment and storage only, it is recommended that they be discarded after 30 to 40 revivifications. Baths from which there is considerable drag out, necessitating frequent additions of new solution, may be revivified without limit provided that the coated articles conform to this specification.

3.5.5.4 Operational difficulties and possible explanation - The following operational difficulties may be encountered during chrome pickling:

(a) Brown, non-adherent, powdery coating:

1. The part was in the air too long before rinsing. The air interval shall be as specified in 3.5.1.
2. The ratio of acid concentration to sodium dichromate content may be too high.
3. The solution may be too hot due to a small quantity of solution and a large number of parts going through the bath. Cool the bath or use a larger quantity of solution.
4. The metal is not properly degreased. Brown powder will occur at spots where oil exists.
5. Solution has been revivified too many times. The nitrate buildup causes powdery coatings.

(b) Gray, non-adherent, powdery coatings on castings:

1. This may be minimized or virtually eliminated by treatment with the fluoride modified chrome treatment bath as specified in Table III.
2. The coating is pyrophoric and may flash violently upon impact with the rough surfaces of harder materials such as steel or concrete, or even during grinding (see 6.6.5).
3. The part may have been excessively over-treated by keeping in the bath too long. Extreme care should be taken in handling such parts during further treatment. Upon removal the part should be thoroughly rinsed in cold water, dipped in machine or motor oil and scrapped. If parts have not been too severely

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damaged by over-pickling and salvage is desirable, the powdery coating may be removed by immersing in a 10 to 20 percent hydrofluoric acid solution for 5 to 10 minutes. This will remove the powder and render the part safe to handle.

3.5.6 To give a smoother surface on some alloys after pickling in the chrome pickle baths, magnesium sulfate may be added to the chrome pickle solutions specified in Table III to the extent of 4 ounces per gallon. Such a modification should be used for temporary storage and shipment but not where the coating will be used as a base for painting. Type I treatment to which magnesium sulfate has been added leaves a surface which is unfit for organic finish systems.

3.6 Type III treatment - The Type III treatment or dichromate treatment provides satisfactory paint base and protective qualities on all standard alloys except EK30A, EK41A, HM31A, HM21A, HK31A, La141A and M1A. No coatings form on these alloys. Properly applied coatings vary from light to dark brown depending on the alloy. On AZ91C-T6 and AZ92A-T6 castings, the coating produced is gray. The treatment causes no appreciable dimension changes and normally is applied after machining. Castings and other parts containing bearings, studs, and inserts of brass, bronze, cadmium plate, and steel shall not be treated, although these metals are not affected by the treatment, unless the dissimilar metal parts are sealed and demonstrated to the procuring activity that the treatment will not adversely affect the characteristics of the parts. Aluminum, however, is rapidly attacked during the hydrofluoric acid dip (see 3.6.1) which is an important step in this treatment. Where aluminum inserts are used or wrought parts are assembled with aluminum rivets, the acid fluoride dip (see 3.6.2) shall be used in place of the hydrofluoric acid dip. Close control of the Type III treatment is essential when applying this treatment to AZ31B-H24. Acid pickling by the methods specified in 3.4.2, 3.4.7, or 3.4.8 should be used on magnesium alloy sheet where maximum corrosion resistance is required. On parts that have previously been Type I treated, the Type III treatment will show the general marking characteristics of the Type I treatment, but with no decrease in effectiveness.

3.6.1 Hydrofluoric acid treatment - After parts have been cleaned in accordance with the other requirements of this specification, they shall be pickled in the hydrofluoric acid treatment. The solution composition and operation are contained in Table IV. This operation both cleans and activates the magnesium surface. It is recommended that AZ31B parts be immersed for 30 seconds and that all other wrought alloys and castings be immersed for 5 minutes. After the immersion period, the parts shall be thoroughly rinsed in cold running water. Thorough rinsing is important in order to minimize fluoride carry-over which will make the dichromate bath inoperative.

TABLE IV
TREATMENT AND DICHROMATE SOLUTIONS FOR TREATING MAGNESIUM ALLOYS BY TYPE III TREATMENT

Solutions	Composition 1/		Metal removed (mils)	Immersion time	Operating temperature	Tank construction	Rack and basket construction
	Material	Oz.					
Hydrofluoric acid treatment	Hydrofluoric acid (as 60% HF)	38	0.1	1/2 to 5 min.	70 to 90 ° F	Lead, rubber, synthetic rubber, or polyethylene lined tanks.	Monel, Type 316 stainless steel or vinyl plastic coated steel.
Acid fluoride treatment	Sodium, potassium, or ammonium acid fluoride (NaHF ₂ , KHF ₂ or NH ₄ HF ₂)	6-2/3	0.1	5 min. (minimum)	70 to 90 ° F	Lead, rubber, or synthetic rubber lined tanks.	Monel or Type 316 stainless steel.
Dichromate	Sodium dichromate (Na ₂ Cr ₂ O ₇ · 2H ₂ O) Calcium or magnesium fluoride (CaF ₂ or MgF ₂)	16 - 24 1/3	--	30 to 45 min.	Boiling	Steel	---

1/ Avoid dross or fluid ounces per gallon. Water is added to make one gallon. When available, water from steam condensate or treated by ion exchange should be used in preference to well or tap water.

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3.6.2 Acid fluoride treatment - The acid fluoride treatment shall be used for all parts containing aluminum inserts, rivets, etc., and may be used as an alternate treatment for hydrofluoric acid treatment (see 3.6.1). The solution composition and operation are contained in Table IV. It is preferred for AZ31B and AZ31C. The acid fluoride treatment may not only be more economical to operate, it is also safer than the hydrofluoric acid treatment. However, the acid fluoride treatment will not remove the dark smudge which forms on some castings after sandblasting and pickling. Then the hydrofluoric acid treatment must be used. After immersion in the solution, the parts shall be thoroughly rinsed in cold running water. Other acid fluoride solutions may be used upon approval by the procuring agency.

3.6.3 Dichromate treatment - After the parts have been given the fluoride treatment in the solution specified in either 3.6.1 or 3.6.2, they shall be boiled for 30 minutes in the bath whose solution composition is listed in Table IV. Properly applied coatings vary from light to dark brown depending upon alloy. After the immersion period, the parts shall be rinsed in cold running water followed by a hot water dip, to facilitate drying, or by exposure to heated air. The specified paint coating shall be applied as soon as practical after the treated parts are thoroughly dry. Because ZK60A takes the dichromate treatment faster, a 15 minute boil of ZK60A parts is equivalent to 30 minutes on other alloys.

3.6.4 Control for Type III treatment -

3.6.4.1 Hydrofluoric acid determination - The hydrofluoric acid bath is depleted very slowly in use. It shall not be allowed to go below 10 percent HF as determined by titration for free acid with 1N.NaOH and phenolphthalein indicator. A solution weaker than 10 percent will attack magnesium quite severely. Hydrofluoric acid shall be added to the batch as needed to maintain the titration between 10 and 20 ml of 1N. NaOH per 2 ml of sample. This corresponds approximately to 10 and 20 percent HF by weight. A pipette for obtaining a bath sample may be made by lining a small (3 or 4 ml) glass pipette with paraffin wax and calibrating to a 2 ml volume. To avoid error by reaction of the HF with glass, the sample shall be discharged into at least 100 ml of distilled water in the vessel used for titration, and titration shall be done immediately.

3.6.4.2 Bifluoride determination - If bifluoride (acid fluoride) is used instead of the hydrofluoric acid, it shall be controlled by titration with caustic. The bifluoride content shall be maintained so that a 100 ml sample requires 45 to 55 ml of 1N. NaOH to produce pink color with phenolphthalein indicator.

3.6.4.3 Control of dichromate bath - The sodium dichromate determination shall be performed as specified in 3.5.5.1. The dichromate content of the bath will be maintained within the limits of 16 to 24 ounces per gallon.

The pH of the dichromate bath must be controlled carefully within the limits of 4.1 to 5.5 by the addition of chromic acid. However, in the treatment of AZ31B parts, the pH of the bath must be held within 4.1 - 5.2. Chromic acid is conveniently added to the bath as a 10 percent solution. The pH determination shall be made as follows:

Standardize a glass electrode equipped pH meter with a buffer solution of approximately 4.0 and check the pH of the dichromate bath. The pH of the bath is brought back to 4.1 with chromic acid. Close control of this factor is required when treating parts of large surface area. Low aluminum-containing alloys shall be processed on the low side of the pH range to obtain a good coating.

3.6.4.4 Operational difficulties and possible explanation - The following operational difficulties may be encountered during the application of the Type III treatment:

(a) Abnormally heavy and loose powdery coatings:

1. The hydrofluoric acid or acid fluoride bath is too dilute. Concentration of free HF shall be controlled as described in 3.6.4.1 and 3.6.4.2.
2. The pH of the dichromate bath is too low. It should not fall below 4.1. The pH is adjusted by additions of NaOH.
3. Treatment of oxidized, corroded, or flux contaminated parts result in gray to yellow coatings of a loose nature. Parts shall be cleaned as specified in 3.2.
4. Powdery coatings may form when the work contacts the tank or is in an electrical circuit with the tank through metal holding bars, baskets, or other pieces of equipment which contact the tank.
5. Prolonged treatment in the dichromate bath.

(b) Failure to coat or nonuniform coatings:

1. The pH of the dichromate bath is too high. This is very important when HF is used as a pre-dip on low aluminum-containing alloys such as the AZ31B.

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Adjust the pH back to 4.1 by addition of chromic acid. The pH change is quite rapid from the lower limit and frequent checks are necessary.

2. The dichromate concentration is too low in the bath. The dichromate concentration should not fall below 16 oz./gal. (12 percent by weight).
3. Oily matter has not been properly removed, resulting in a spotted coating where some areas are coated and others are not. Insufficient cleaning alone may not be at fault. Properly cleaned parts may become contaminated by oily matter existing as a film on the fluoride or dichromate bath. This oily film builds up due to inadequate rinsing after alkaline cleaning and perhaps other reasons such as oil in the atmosphere or dropping from overhead equipment, etc.
4. Previous chrome pickle coating was not completely removed. Use of either solution specified in 3.4.1 or 3.4.2 to supplement the alkaline cleaning may be required.
5. The part was not fluoride treated.
6. Alloy treated is one as stated in 3.6 where the Type III treatment is not suitable for coating deposit.
7. Too long an HF dip with alloys such as AZ31B produces a fluoride film that does not break down evenly in the normal time, producing a spotty coating. For these alloys treatment time should be 30 seconds to 1 minute.
8. The bath was not kept boiling during the treating period. This is particularly important when processing AZ31B. Minimum bath temperature is 200° F.
9. Improper rinsing after the HF dip. No coating forms if the amount of the HF or maximum soluble fluoride carried over into the dichromate bath exceeds 0.2 percent. Streaked coatings will be observed before this point is reached. A 0.2 percent solution of calcium chromate (CaCrO_4) can be added to the bath to precipitate the excess fluoride out as insoluble calcium fluoride (CaF_2). When this addition is made, the bath need not be discarded.

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3.7 Type IV treatment - The Type IV treatment or galvanic anodizing treatment may be applied to all alloys. It is also used specifically for those alloys which do not react to give a protective film with the Type III treatment. It causes no dimensional change and may be applied after machining operations. After the parts have been cleaned in accordance with the other requirements of this specification, they shall be treated in the hydrofluoric acid pickle as specified in 3.6.1 or in the acid fluoride pickle as specified in 3.6.2.

3.7.1 Galvanic anodizing solution - After the treatment in a fluoride solution, the work shall be galvanically anodized for at least 10 minutes and for as long as 30 minutes in the solution whose composition is specified in Table V. The steel tank shall be the cathode. If the tank is made of nonmetallic material, steel cathode plates shall be used. The magnesium parts must have good electrical connection with the tank or the cathode plates, care being taken that the work does not make contact with the tank except by an external connection. An ammeter and rheostat shall be in the electric circuit. The current density shall not exceed 10 amps per square foot of anode area at any time. A minimum of 70 ampere-minutes per square foot of anode shall be maintained to secure a uniform coating. Ordinarily 70 to 150 ampere-minutes per square foot will be sufficient. When the area of work is large, in respect to tank size, the anode may fall below 2 amperes per square foot, resulting in a poor coating being obtained. In this case, an external source of current should be used to maintain the anode current density within the required range. After the immersion period the parts shall be rinsed in cold running water, followed by a hot water dip, to facilitate drying, or by exposure to heated air. The specified paint coating shall be applied as soon as practical after the treated parts are thoroughly dry.

3.7.2 Control of the Type IV treatment - The determination of hydrofluoric acid and the acid fluoride solutions shall be performed as specified in 3.6.4.1 and 3.6.4.2 respectively. The sulfate-dichromate-hydroxide bath used for galvanic anodizing shall be controlled by maintaining the pH between 5.6 and 6.0 with additions of a solution containing 5 percent by weight each of chromic acid (CrO_3) and concentrated sulfuric acid (H_2SO_4). A properly applied Type IV treatment usually produces a coating that is uniformly dark brown to black. Time of treatment, condition of bath, and composition of the alloy influence the color of the coating. Gray and nonuniform coatings indicate that articles were not properly cleaned before treatment or that the solution was depleted. Non-adherent coatings are usually caused by too high a current density, too prolonged a treatment, or by too low a bath pH. An increase in time required to secure a uniform coating also indicates a depletion of the solution. Parts must be racked firmly for the proper galvanic action to take place. Monel, stainless steel, or phosphor bronze racks may be used.

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TABLE V
SOLUTION COMPOSITION AND OPERATION FOR TYPE IV TREATMENT

Composition 1/		Current density amp/sq. ft	Immersion time, min.	Operating temperature °F	Cathode	Tank construction	Rack construction
Material	Oz.						
Ammonium sulfate (NH ₄) ₂ SO ₄	4	2 to 10	10 to 30	120 - 140°	Steel	Steel	Monel, stainless steel or phosphor bronze.
Sodium dichromate (Na ₂ Cr ₂ O ₇ · 2H ₂ O)	4						
Ammonium hydroxide (sp gr 0.880)	1/3						

1/ Avordupois or fluid ounces per gallon. Water is added to make 1 gallon. When available water from steam condensate or treated by ion exchange should be used in preference to well or tap water.

3.8 Type VI treatment - The Type VI treatment or chromic acid brush-on treatment can be applied to magnesium parts that require touchup. This treatment, as well as Type I treatment, is generally used in refinishing procedures or where parts and assemblies are too large to be immersed. The Type VI treatment is less critical to apply than the Type I treatment by brush application as detailed in 3.5.4. Also, the Type VI treatment is relatively inexpensive, not harmful when trapped in faying surfaces and does not present the toxicity hazards of the Type I treatment. Normal methods for removing oil, grease, etc., as given in 3.2 are used prior to the treatment. It can be readily applied to all alloys by brushing, dipping or spraying. The solution composition and operation are specified in Table VI

TABLE VI

SOLUTION COMPOSITION AND OPERATION FOR TYPE VI TREATMENT

Composition <u>1/</u>		Operating temperature °F	Tank construction
Material <u>2/</u>	Oz.		
Chromic acid (CrO ₃) <u>3/</u> Calcium sulphate (CaSO ₄ · 2H ₂ O)	1-1/3 1	70 to 90°	Stainless steel, aluminum, vinyl polyethylene or rubber lined tanks.

1/ Avoirdupois or fluid ounces per gallon. Water is added to make one gallon. Most tap water or purified water may be used.

2/ Chemicals should be added to the water in the order shown, and then the solution stirred vigorously, either mechanically or by air agitation for at least 15 minutes.

3/ Technical grade chromic acid (99.5 percent purity) may be used.

3.8.1 Brush application - Proper application by the Type VI treatment requires that the surface of the parts be kept wet with the brush-on solution for a sufficient time, usually about 1 to 3 minutes, to produce a brown film. The parts shall then be rinsed in cold running water, and dried, either in an oven, or by exposure to a blast of hot air. In no case, should the parts be rinsed in hot water. Unlike brush application by Type I treatment, the time between the pickling and cold water rinsing is not critical. In fact, where running water is not feasible, the rinse step can be eliminated without materially altering the coating effectiveness. The paint base properties are substantially equal to that of the Type I treatment. The brown color of the coating is not altered by chemical composition of the magnesium alloys. As with the Type I treatment, Type VI treatment may be applied over previously prepared thin films of all the Type treatments.

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- # 3.8.2 Operational explanations - Facilities that use and are familiar with the Type I treatment may prefer to use this treatment for touchup application. However, it should be noted in comparing the Types I and VI treatments for brush application, that the Type I solution is more costly to prepare as well as presenting greater toxicity hazards, and requires careful rinsing of the retouched part as an essential control factor. Coatings by the Type VI treatment are not adequate for interfaces used in electrical bonding but are satisfactory for use on entrapment areas of all magnesium assemblies. Type VI processed coatings require painting or sealing before exposure to overnight, outdoor atmospheric conditions. Coatings by the Type VI treatment can be varied from a brassy iridescence to a dark brown depending upon treatment time. Up to 1 minute of treatment produces a brassy film and from 2 to 3 minutes a dark brown coating. The brush-on treatment shall not be less than 30 seconds nor longer than 3 minutes. Prolonged treatment produces loose powdery coatings similar to those listed in 3.5.5.4. For best paint adhesion, the dark brown coatings are preferred.
- # 3.9 Type VII treatment - The Type VII treatment or the fluoride anodizing treatment is essentially an anodic treatment followed by a stripping operation to remove the fluoride coating and then post-treated with a corrosion preventative treatment to provide the optimum protection. The anodizing and stripping portions of the treatment may be applied to all alloys and forms. The anodizing portion of the treatment is suitable to take the place of shot or grit blasting as a means of cleaning magnesium alloys from adherent foundry sand (see 3.2.2 and 3.2.2.1). Subsequent pickling operations such as specified in 3.2.4.2 will therefore not be necessary. The anodizing portion of the treatment system removes cathodic impurities at the surface.
- # 3.9.1 Anodic treatment - The anodizing process is one step treatment employing an aqueous ammonium bifluoride bath in which alternating high voltage current generally produces a uniform clean white or pearly-gray coating. With QE22A alloy, the coating will have a creamy or yellowish appearance because of the silver in alloy composition.
- # 3.9.1.1 Application - The anodizing treatment may be applied with alternating current. Magnesium parts should be anodized by the fluoride treatment prior to attaching dissimilar metal parts. Where inserts and attachments must be in place before treatment, they must be adequately shielded. Suitable masking material such as polyvinyl-butyril may be used for this purpose. Steel studs may be fitted with rubber or plastic caps. Cast-in steel or copper tubes may be plugged with rubber bungs. In suitable positions, magnesium plugs may be fitted or magnesium blanking off plates may be applied to prevent access of the bath solution to areas where it is required to prevent the process from taking place. Certain chloride containing plastics are known to be harmful, so tests shall be performed prior to adopting any material for masking purposes.

3.9.1.2 Treatment - Parts to be given the anodic treatment need not usually require cleaning in accordance with other requirements of this specification. Loosely held sand should be removed by knocking or brushing from castings. Any thick layers of grease should be removed by solvent cleaning (see 3.2.1) to prevent contamination of the anodizing bath. The magnesium parts to be treated are suspended in the anodizing bath. The work is fixed in pairs in good electrical contact on electrode bars across the insulated tank and suspended not less than 9 inches below the surface of the bath. The work shall be arranged so that approximately equal surface areas are present at each electrode. All parts of the fixed clamps below the bath surface shall be of magnesium-rich alloy, such as AZ31, AZ63A, AZ91, or EZ33A. Because of the relatively high voltage used to obtain the 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. This further removes traces of foreign matter, graphite, corrosion products and other non-metallic films. Good electric contact with the work is essential. Anodizing is carried out at constant current in the bath whose composition is detailed in Table VII. The voltage is continuously raised to maintain the desired current density as the resistance increases. The voltage is increased progressively until 120 volts are reached. Current flow is usually heavy at first, but diminishes rapidly as impurities are removed and a coating of unbroken magnesium fluoride is formed in their place. When this occurs, the treatment may be regarded as complete. After the indicated quantity of electricity has been applied, the voltage is maintained for 10 to 15 minutes or until the current falls to less than 5 amperes per square foot. Parts shall then be removed and rinsed in hot running water. Parts are then quickly dried by exposure to heated air.

3.9.1.3 Coating characteristics - A thin coating, magnesium fluoride, less than 0.0001 inch is produced. The film itself has no measurable thickness. The treatment leads to no detectable dimensional loss likely to cause difficulty even when working to definite fine limits. The fluoride film, after stripping does not prevent application of other chromate treatments such as Types I, III, or IV.

3.9.2 Post anodic treatments -

3.9.2.1 Chromic acid pickle - After the parts have been given the anodizing treatment as specified in 3.9.1, they shall be boiled for 1 to 15 minutes in the chromic acid pickle bath listed in Table II (see 3.4.1) or that given in Table VII. After the immersion period, the parts shall be rinsed in cold water.

3.9.2.2 Dichromating treatment - The stripped and rinsed parts shall then be immersed in a bath for the acid fluoride treatment (see 3.6.2) for 5 minutes at room temperature. The parts shall then be rinsed and immersed

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TABLE VII
SOLUTIONS FOR TREATING MAGNESIUM ALLOYS BY TYPE VII TREATMENT

Bath	Composition ^{1/}		Immersion time	Operating temperature °F	Current density min.	Voltage	Tank construction
	Material	Oz.					
Anodizing	Ammonium acid fluoride (NH ₄ HF ₂)	19 to 38	10 to 15 min	60 to 86°	5 amp/sq. ft.	0 to 120 AC	Steel, ceramic, rubber lined, or tanks lined with vinyl based materials.
Chromic (also see 3.4.1)	Chromic acid (CrO ₃)	19-1/2	1 to 15 min.	190 to 210°	---	---	Lead lined steel, stainless steel, or 1100 aluminum
Dichromate-nitric (also see 3.5.1)	Sodium dichromate (Na ₂ Cr ₂ O ₇ 2H ₂ O) Nitric acid (sp gr 1.42)	19-1/2 25-1/2 to 32	2 to 30 min. ^{2/}	60 to 90°	---	---	Stainless steel, ceramic, synthetic rubber lined tanks, or tanks lined with vinyl based materials.
Dichromate	Sodium dichromate (Na ₂ Cr ₂ O ₇ 2H ₂ O)	13 to 15	40 to 60 min	Boiling	---	---	Steel

^{1/} Avirdupois or fluid ounces per gallon Water is added to make one gallon. When available, water from steam condensate or treated by ion exchange should be used in preference to well or tap water.

^{2/} Sufficient to remove 0.002 inch of surface.

in the boiling solution specified for the Type III dichromate treatment for 30 minutes (see 3.6.3) After this immersion period, the parts shall be rinsed in cold running water followed by a hot water dip to facilitate drying, or by exposure to heated air. Controls for these dichromating treatments shall be as detailed in 3.6.4.2 and 3.6.4.3. Operational difficulties and possible explanations are contained in 3.6.4.4.

3.9.2.3 Other dichromate treatments - Where so specified in the contract, purchase order or applicable drawing, other dichromate treatments may be used than those detailed in 3.9.2.1 and 3.9.2.2. The dichromate-nitric acid bath given in Table VII or that of Table III (see 3.5.1) may be used in place of the boiling chromic acid bath (see 3.9.2.1) to strip the fluoride film. The dichromate-nitric bath, used cold, leads to some dimensional loss and should not be used on machined parts where permitted tolerances are small. Controls and operational difficulties with this bath are detailed in 3.5.5. A very satisfactory chromate film may also be produced by boiling the part or component directly after the anodizing treatment (see 3.9.1) in the dichromate bath specified in Table VII for 40 to 60 minutes. After the immersion period by either of the above two treatments the parts shall be rinsed in cold running water, followed by a hot water dip to facilitate drying or by exposure to heated air. The specified paint coating may be applied as soon as practical after the parts treated in accordance to 3.9.2.2 or 3.9.2.3 are dried, or the chromated parts may be sealed as specified in 3.9.3 prior to the painting operation

3.9.2.4 Dichromate treatment effectiveness - The presence of the fluoride film on the anodized magnesium alloys does not prevent the application of the dichromate treatment. The fluoride layer is slowly displaced by the chromate film unless the fluoride anodized film is first removed by boiling in chromic acid solution (see 3.9.2) It often happens that after chromating is complete, the displaced fluoride coating remains on the chromate surface as a fine impalpable powder or bloom. This is not harmful. It does not interfere in any way with adhesion of subsequent paint systems. If desired, however, it can be removed by gently brushing or wiping with a soft cloth. However for optimum results in the dichromating step, the fluoride should be removed as there may be conditions where the loose anodized film would be harmful by retaining moisture.

3.9.3 Surface sealing treatments - Parts receiving the Type VII treatment should be given a surface sealing treatment to improve corrosion resistance even if followed by a paint coating system. This is especially applicable to castings. The work, as soon as possible, after the dichromate treatment (see 3.9.2.2 and 3.9.2.3) should be heated to approximately 392° F for about 1/2 hour. This effectively dries the surface and furthermore drives moisture from surface flaws. The casting or work is cooled to about 140° F and dipped into a surface coating resin. After evaporation of the solvent,

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the casting is reheated to approximately 392° F for 15 minutes and the treatment of cooling, dipping and reheating repeated until three coats of the resin have been applied. Three such coats, suitably applied should total less than 0.001 inch in thickness with a weight increase of less than 1-1/2 ounces per square yard of surface.

- # 3.9.3.1 The surface sealing treatment may also be applied as detailed in MIL-M-46080 and the surface coating resin may be in accordance with the requirements of MIL-C-46079 for a baking type epoxy coating for castings.

- # 3.9.3.2 The surface sealing can be applied equally to any of the processes, Type I (see 3.5), Type III (see 3.6), Type IV (see 3.7), Type VI (see 3.8), or Type VIII (see 3.10), after the final drying operation and prior to painting.

- # 3.9.4 Racks for the Type VII treatment - Racks made of magnesium alloys such as AZ31B, AZ63A, AZ91C, shall be used in applying the anodizing portion, Type VII treatment. Rubber, synthetic rubber, or polyvinylchloride covered, steel racks may be used. Design of the rack shall be such as to ensure very firm contacts at the bus bar and to the part. The portion of the rack connecting the bus bar shall be made of copper, bronze, or brass which can be bolted to the lower part of the rack. The rack shall make good contact with the magnesium parts. Steel racks shall be exposed only at the point where contact is made to the parts. The legs of the racks may be wrapped with a suitable tape such as polyvinylchloride or to fit polyethylene or rubber sleeves, especially at the "wind and water" line. Since the coating acts as an insulator, magnesium racks shall be stripped of the coating at the contact point by sanding prior to each use or by the use of chromic acid pickle, specified in 3.4.1, or the chrome-pickle, specified in Table III for wrought materials.

- # 3.9.5 Control for Type VII treatment - The Type VII anodizing treatment bath (see 3.9.1) depletes slowly for all practical purposes. Depletion is indicated by roughness or an etched appearance in the coating. Replenishment of the bath can be accomplished by adding ammonium bifluoride solution. Standard analytical methods for testing for fluoride may be used for controlling the bath. The fluoride concentrate is not critical provided it exceeds the minimum concentration.

- # 3.9.6 Operational difficulties and possible explanation - The following operational difficulties may be encountered during application of the anodizing portion of Type VII treatment.

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- (a) Castings which have received too short a treatment will show some areas with a characteristic whitish or pearl-gray film while other areas may appear not to have responded.
- (b) Castings which are not free from attached foundry sands will have some areas darker than others. If obstinate areas of foundry sands still remain after treatment, or blemishes which are not being cleaned up quickly as the rest of the observed surface of the work, mechanical treatment with a stiff wire or bristle brush may be given at this stage and the work returned for a further short period of fluoride anodizing.
- (c) Painted parts or components should not be normally treated until the bulk of the paint has been removed. Paint films themselves are softened by reaction in the bath. Nickel and copper electroplates can also be removed by the treatment. Unless these precautions are taken, contamination of the bath will result, reducing bath efficiency.
- (d) Too low a voltage or too short a time in the bath will be usually indicated by a very thin semitransparent film. On machined surfaces or wrought material, which is initially in good condition, such a thin film may be acceptable.
- (e) In hollow parts or components with re-entrant angles, dark areas may remain in such recesses and indicate gas entrapment. In such cases the items should be turned about once or twice in the bath to ensure complete contact with the liquid.
- (f) An etched appearance may indicate too hot a bath, possibly operation at too high a voltage, or a bath too low in ammonium bifluoride. The strength of the bath must not be allowed to fall below 10 percent. A stronger solution will do no harm but is wasteful.
- (g) A dense film may indicate the presence of acid radicals other than fluoride. No foreign metals, organic matter, salts or acid radicals other than fluoride should be introduced into the bath. Pitting of the work surface may also be caused by the presence of the chloride ion.

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- (h) The bath, during continuous operation must be stirred well between batches of items with a wooden or plastic pole to ensure that the upper layers of the liquid are not depleted of the fluoride or become warmer than the bulk of the bath. It may be advantageous to arrange for continuous agitation of the solution by air stream. A large volume of air should not be blown through the bath as it promotes heavy current flow which in turn creates undue heating. Only the merest trickle, sufficient to cause a gentle circulation of the liquid in the bath, shall be permitted.
- (i) In the progress of operation, if the tendency of the current to fall away to low values at any voltage up to about 120 volts does not occur, observations should be made to see if the anodizing solution, as specified in Table VII is too weak; a foreign metal insert, rivet or other attachment is present; part of the clamp that is not made of magnesium is below the surface of the bath solution; the clamp, as specified in 3.9.1.2 is not of one of the materials detailed, or the work is too severely contaminated. In the latter case, this may arise from having been heavily shot-blasted (see 3.2.2) or from the presence in the casting of one or more flux inclusions. It will be found that when the last of the contaminants, iron particles, have been removed, the current during anodizing will fall normally.

3.10 Type VIII treatment - The Type VIII or chromate treatment provides a satisfactory paint base and protective coating on all magnesium alloys, unlike the restrictive Type III treatment (see 3.6) The coating may be applied to the magnesium alloys by dip or brush. Type VIII treatment may be substituted, when approved by the procuring activity, for the Types I, III or VI treatments as well as the corrosion preventative treatment of the Type VII. The coating deposited is somewhat like that of the Type III, being dark brown to light reddish brown in appearance. The Type VIII treatment causes no appreciable dimension changes and normally is applied after machining.

3.10.1 For alloy containing more than one percent aluminum - Parts of magnesium alloys containing more than one percent aluminum shall be cleaned in accordance with other requirements of this specification. For parts of magnesium alloys with 3.5 percent aluminum or less, such as AZ-31, the chromic-nitrate pickle (see 3.4.2) shall be used, whereas with those alloys having an aluminum content over 3.5 percent, such as AZ-61, AZ-81, and

AZ-91, the chromic-nitric-HF pickle shall be used (see 3.4.6) following the alkaline cleaning. If mill pickled stock is to be treated, parts shall be immersed in the acid pickle for 15 to 30 seconds, but if unpickled or cast materials are used for parts, the immersion time in the acid pickle solution shall be from 2 to 3 minutes. The parts shall then be rinsed prior to treatment in the chromate bath, Solution No. 1. The Solution No. 1 composition and operation are contained in Table VIII. After the parts have been treated with the chromate bath, Solution No. 1 for 15 to 30 seconds, the parts shall be double rinsed in cold running water followed by a hot water dip to facilitate drying, or by exposure to heated air. Drying temperatures in the range of 160 to 200° F will produce a dark brown coating of increased hardness and reduced solubility.

- # 3.10.2 For alloys containing less than one percent aluminum -
Parts of magnesium alloy without or containing less than one percent aluminum shall be cleaned in accordance with other requirements of this specification. The chromic-nitrate solution (see 3.4.2) may be used for acid pickle under the same conditions for stock as recommended in 3.10.1. After the rinsing, following the pickling treatment, the parts shall be treated in the chromate bath, Solution No. 2. The Solution No. 2 composition and operation are contained in Table VIII. Further treatment is as detailed in 3.10.1
- # 3.10.3 Control of the Type VIII treatment - The solution baths used for the chromate treatment shall be controlled by maintaining the pH between 0.2 and 0.6 for Solution No. 1 and between 0.6 and 1.0 for Solution No. 2 by adding the chromate salts and hydrochloric acid in the original proportions. The wetter detergent should be controlled by adding 0.034 ounce of the detergent for every 5 ounces of the chromate salts when the solution bath is in constant use. When the solution bath has been allowed to stand for a week or more, and a thin foam layer does not form during treatment, 0.034 ounce of the wetter detergent shall be added for every gallon of the bath so as to provide a thin blanket of foam during the processing. Scratch Type VIII treatment coatings can easily be touched up by swabbing with the solution used for working the part and rinsed with flowing water.
- # 3.10.4 Bath life - Repeated replenishment additions may be made until satisfactory coatings are no longer obtained (even though the chromate solution is within its correct operating limits). The solution should not require dumping until the total replenishment additions are approximately one and a half to double the original make-up amounts, and the amount of work treated is equivalent to approximately 175 square feet of alloy surface per gallon of the working bath.
- # 3.10.5 Operational difficulties and possible explanations - The following operational difficulties may be encountered during chromate treatment:

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TABLE VIII
TYPE VIII CHROMATE SOLUTIONS FOR TREATING MAGNESIUM ALLOYS

Solutions	Composition 1/ Material		Metal removed (mils) 2/ Oz	Immersion time	Operating temperature °F	Tank construction	Rack and basket construction	Heating coils
Chromate Solution No 1	Chromate Salts (prop)	5	0 05	15 to 30 sec 3/	75 to 100 ° F	Polyvinyl chloride, polyethylene, polypropylene	PVC coated steel	Tantalum, quartz or graphite immersion heaters
	Hydrochloric Acid 20 °Be 4/ Wetter (prop)	7-1/2 0 034						
Chromate Solution No 2	Chromate Salts (prop)	5	0.05	15 to 30 sec 3/	75 to 100 ° F	Polyvinyl chloride, polyethylene, polypropylene	PVC coated steel	Tantalum, quartz or graphite immersion heaters
	Hydrochloric Acid 20 °Be 4/ Wetter (prop)	2 0.034						

1/ Avoidupis or fluid ounces per gallon Water is added to make 1 gallon When available, water from steam condensate or treated by ion exchange should be used in preference to well or tap water

2/ Per surface for 15 seconds treating time

3/ For machined surfaces or if preceded by acid pickle. On unmachined surfaces, not preceded by acid pickle, up to 2 minutes in solution will be allowed

4/ For hydrochloric acid other than 20 ° Be use the following amounts

Gravity (° Be)	Solution No 1 - Oz		Solution No 2 - Oz.	
16°	9.6		2 6	
18°	8.5		2 3	
22°	6 75		1 8	

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(a) Failure to form coatings:

1. The pH of the solution is too high. Solutions should be as specified in 3.10.3.
2. The solution may be too cold. The solution temperature shall be as specified in Table VIII.
3. The metal is not properly degreased and cleaned. The part was not pickled prior to treatment in the chromate solution.
4. The ratio of the acid concentration to the chromate salts may be too low by using the improper amount of acid or strength.

(b) Non-adherent powdery coatings:

1. The part may have been of magnesium alloys containing less than 1 percent aluminum and the improper chromate Solution No. 1 used on the part. This can be eliminated by treatment with the bath as specified in 3.10.2 and Table VIII
2. The pH of the solution is too low. Solutions should be as specified in 3.10.3.
3. The metal is not properly degreased and cleaned. The part was not pickled prior to treatment in the chromate solution.
4. The ratio of the acid concentration to the chromate salts may be too high by using improper amount of acid or strength

(c) Excessive smut on parts:

1. The parts may have excessive aluminum smut on aluminum-bearing alloys by being kept in the bath too long. This may be minimized or virtually eliminated by proper treatment time as specified in Table VIII

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3.11 Other treatments - Other treatments may be substituted for the specified treatments when approved by the procuring agency. (See 6.8.)

3.12 General - The sequence of the operations to be used for the application of treatments specified herein shall be in accordance with Figures 1 and 2. Treated articles shall be primed as soon as practical after thorough drying. Parts which are handled or processed after sealing or remain unprimed for extended periods shall be cleaned prior to painting. Other methods of exposing the parts to the treatment solution than by immersion may be used provided the other requirements of the specification are met. Other types of tank construction other than those specified may be used provided they do not have an adverse effect on the solutions to be contained.

3.13 Dimensional changes - Unless otherwise specified, the dimensional changes resulting from treatment shall comply with the requirements stated in Table IX when tested in accordance with 4.4.5. The indicated dimensional changes do not include those resulting from the various cleaning processes. It should be noted that the 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 IX

DIMENSIONAL CHANGE REQUIREMENTS OF COATINGS IN MILS

Process	Min.	Max.	Typical <u>1/</u>
Type I - Chrome pickle	0.6	1.0	0.8
Type III - Dichromate	-	-	<u>2/</u>
Type IV - Galvanic	-	-	<u>2/</u>
Type VI - Chromic acid brush-on	-	-	<u>2/</u>
Type VII - Fluoride anodizing	-	-	<u>3/</u>
Type VIII - Chromate	-	-	<u>2/</u>

1/ On AZ31B magnesium alloy.

2/ No appreciable dimension changes.

3/ No appreciable dimension changes with chromic acid pickle and Type III process

3.14 Special requirements In addition to the requirements specified above, coating shall conform to any special requirements specified in the contract or purchase order

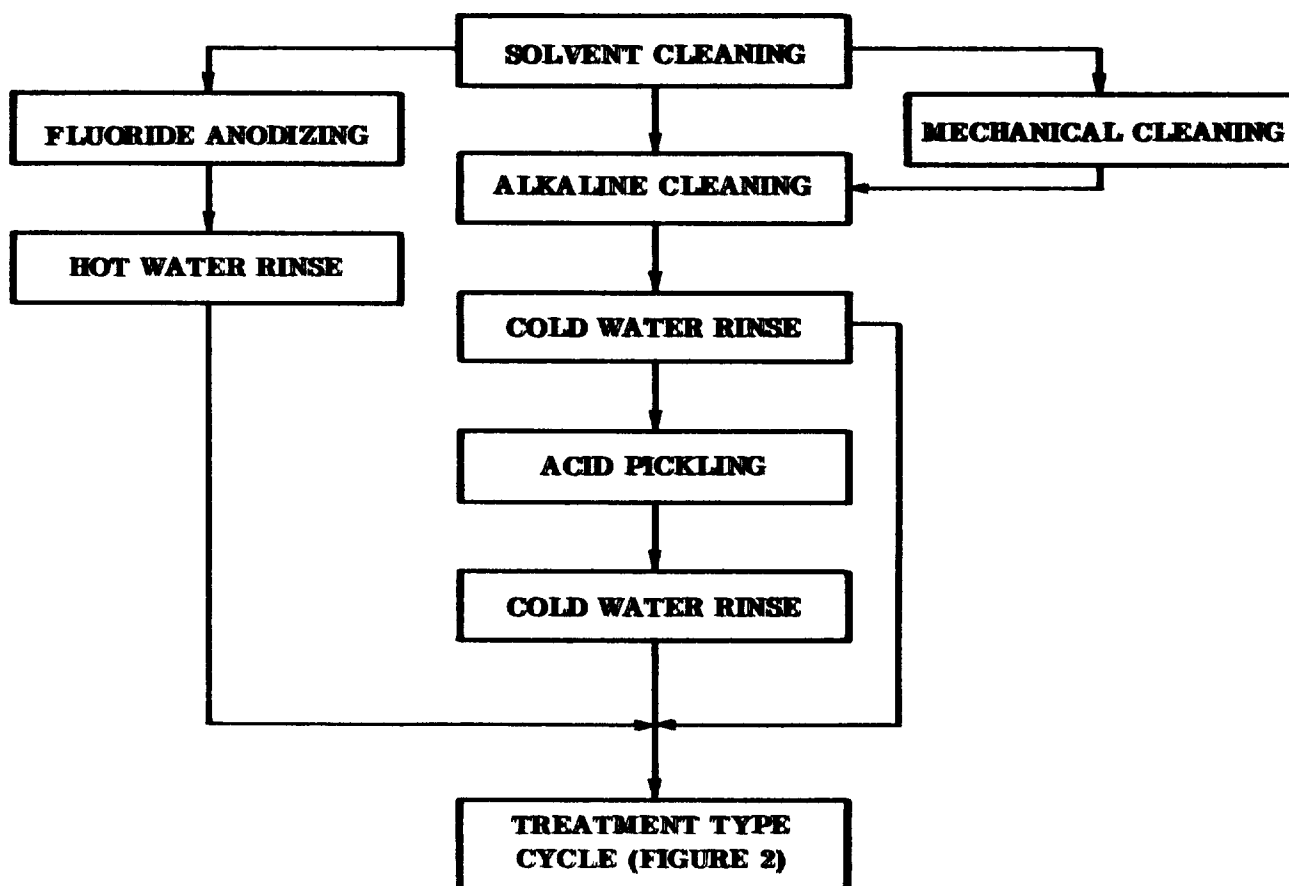


FIGURE 1. FLOW SHEET FOR CLEANING CYCLES

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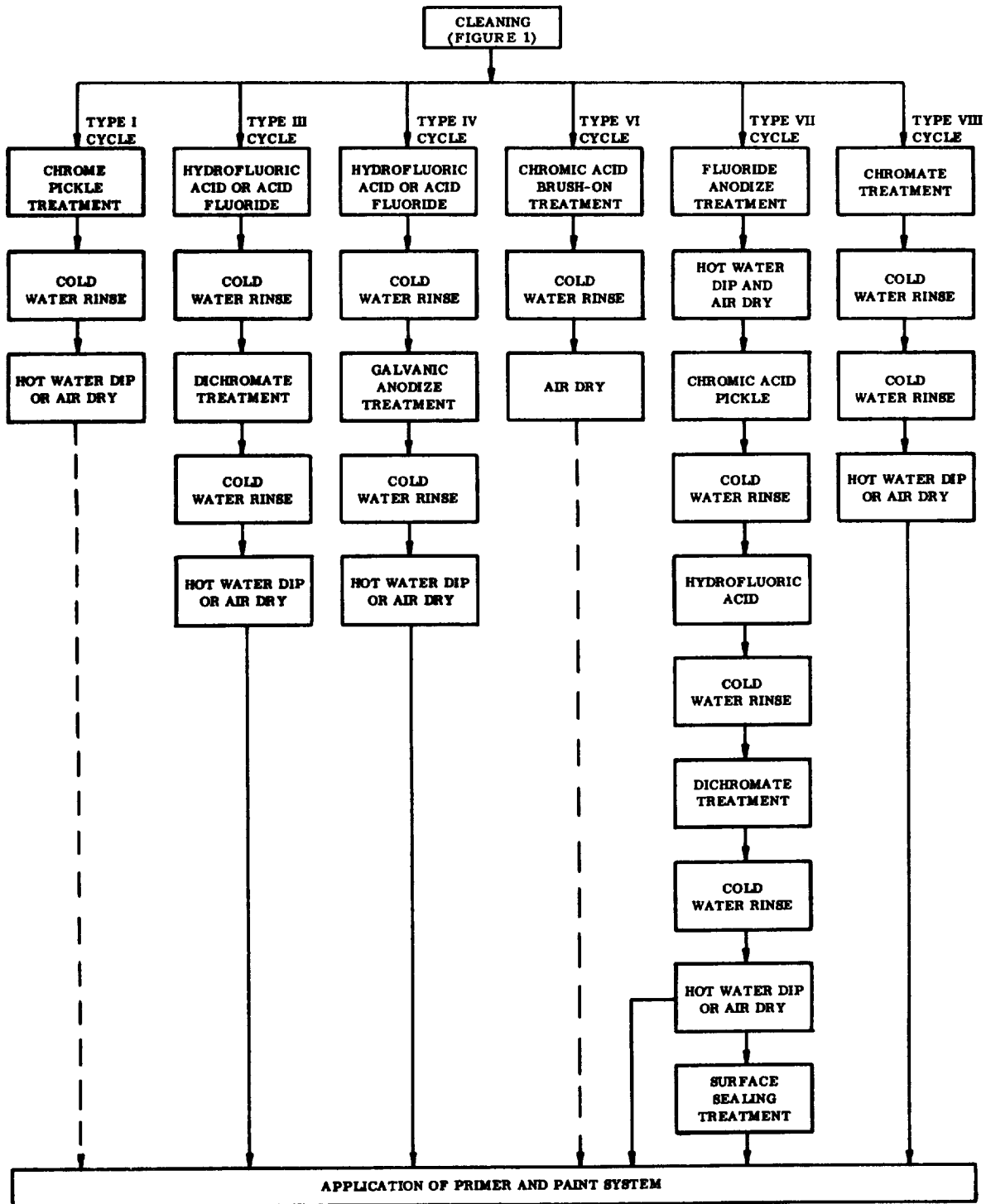


FIGURE 2. FLOW SHEET FOR TREATMENT CYCLES

4. QUALITY ASSURANCE PROVISIONS

4.1 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, the supplier may utilize his own facilities or any commercial laboratory acceptable to 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 Lot - A lot shall consist of all processed and treated parts of the same type produced or completed within a period of time specified by the procuring activity with no change in process. Lot size may also be determined by the procuring activity as a number of square feet of material surface processed per unit volume of the treatment bath.

4.3 Sampling -

4.3.1 Visual examination - Random sampling for visual examination and workmanship shall be conducted in accordance with MIL-STD-105, Inspection Level II, and with an AQL of 1.5 percent defective.

4.3.2 For determination of dimension change - A number of pieces corresponding to Inspection Level S-4 of MIL-STD-105 shall be selected from the lot prior to treatment; 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.

4.3.3 Concurrently anodized specimens - When the use of a sample of anodized items for the determination of the dimensional change 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 Examination -

4.4.1 General - Examination of equipment, process and treated parts shall be made to ascertain compliance with this specification. The continued effectiveness of equipment and procedures, after approval by the government, is the responsibility of the supplier.

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

MIL-M-3171C**4.4.3 Treatment examination -**

4.4.3.1 Treatment examination shall consist of the observation of time of treatment, operating temperature, current density, voltage, and all other pertinent treatment conditions.

4.4.3.2 The process shall be examined for compliance with the requirements of this specification at the beginning of each production run and at the beginning of the reuse 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 ensure compliance with the requirements of this specification.

4.4.3.3 The supplier shall maintain a permanent record of the history of each processing bath, showing all additions of chemicals to the bath, the results of all analyses performed and the quality of parts of each kind processed in the bath. This record shall be available to the Government at all times.

4.4.4 Product examination - The samples of treated parts selected in accordance with 4.3.1 shall be visually examined to determine conformance with the requirements of this specification in regards to workmanship, quality of the treated surface and any other requirements specified by the procuring activity. A visual examination of the processed surface shall be made for complete and uniform coverage. Surfaces shall be examined for the coloring and general characteristics, described herein, peculiar to the type treatment being examined, which shall be an indication of satisfactory time of treatment and condition of solution. The color shall be a reasonable close approximation to that of a sample consisting of a treated piece or pieces agreed upon as a standard by the supplier and the procuring activity, if so agreed upon or specified in the contract or order.

4.4.5 Dimension change determination -

4.4.5.1 Measurement - The thickness of each piece selected prior to treatment shall be determined by measuring with a micrometer which reads accurately to 0.0001 inch. 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 treatment by measuring the same location as before. The dimensional changes of each piece shall be determined from the difference of the two measurements.

4.4.6 Other tests - Treated pieces shall be tested for conformance with any special requirements specified in the contract or order. The tests shall be performed according to the methods specified therein.

4.5 Rejection -

4.5.1 For nonconformance of process - Failure to conform to any of the requirements of this specification pertaining to treatment conditions shall be cause for rejection of the process. In the event unsatisfactory quality is indicated by inspection, 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 demonstrates its suitability for aircraft use to the satisfaction of the procuring activity. In addition, no further work will be accepted until the contractor demonstrates to the satisfaction of the procuring activity that the necessary improvements in the processing have been accomplished.

4.5.2 For nonconformance of coating - If a sample of coated part or article 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 submitted 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 shall have been corrected by the contractor.

5. PREPARATION FOR DELIVERY

5.1 This section is not applicable to this specification.

6. NOTES

6.1 Intended use -

- # 6.1.1 Type I - Chrome pickle treatment - For temporary storage, domestic shipment, electrical bonding, touching up of previously treated work, and brush application when permitted. Applicable to all alloys when close dimensional tolerances are not required. Parts processed by this treatment are not to be subjected to temperatures above 450° F.
- # 6.1.2 Type III - Dichromate treatment - For general long time protection of all alloys except EK30A, EK41A, HM21A, HM31A, La141A, HK31A and M1A, including work for which close dimensional tolerances are required. Parts processed by this treatment are not to be subjected to temperatures above 550° F.

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- # 6.1.3 Type IV - Galvanic anodizing treatment - For general long time protection of all alloys when close dimensional tolerances are required. Parts processed by this treatment are not to be subjected to temperatures above 550° F
- # 6.1.4 Type VI - Chromic-acid brush-on treatment - For temporary storage, protective touchup of previously treated work, and brush application where parts and assemblies are too large to be immersed. Applicable to all alloys when close dimensional tolerances are not required. Parts processed by this treatment are not to be subjected to temperatures above 450° F.
- # 6.1.5 Type VII - Fluoride anodizing treatment - For cleaning of magnesium alloys as a substitute for shot or grit blasting, by providing a relatively inert film when close dimensional tolerances are required and a good base favorable for the other type treatments, such as the Type III.
- # 6.1.6 Type VIII - Chromate treatment - For general long time protection of all alloys where close dimensional tolerances are required. Parts processed by this treatment are not to be subjected to temperatures above 550° F.
- # 6.2 Ordering data - Invitation for bids, contract or orders should specify the following:
- a. Title, number, and date of this specification.
 - b. Treatment type required (see 1.2.1)
 - c. Special requirements, if applicable (see 3.13)
 - d. Availability of treatment records (see 4.4.3.3)
 - e. Other tests, if applicable (see 4.4.6)
 - f. Other treatments, if approved (see 6.4)
- # 6.3 Post treatment - The term "post-treatment" means any treatment used after anodizing. Because all anodic coatings are more or less porous, it is advisable to follow the anodize treatment with a post treatment of chromate and/or an impregnation of a suitable clear baked epoxy resin. Several specially developed epoxy resin formulations are commercially available for impregnation treatment (see 3.9.3)
- # 6.4 Other treatments -

6.4.1 Chemical conversion coatings - In addition to the chemical conversion coatings detailed in this specification (Types I, III, VI, and VIII) there are others such as the half-hour RAE, chrome manganese and the selenious acid. Coatings produced from these treatments range in color from brown to black. Each has its own advantages and disadvantages. For example, the RAE requires 30 minutes in the bath and the pH of the solution is critical (5.90 to 6.18). However it exhibits the maximum corrosion protection and is an excellent paint base. The chrome-manganese treatment has excellent abrasion resistance, but is improved by baking at 356° F for 30 minutes. The range of pH tolerance of the chrome-manganese bath (4.0 to 6.0) makes easy control. The selenate coatings are used to repair damaged chromate type coatings but are not suitable as a base for paint systems. In addition, selenates are more toxic than the chromates.

6.4.2 Anodic coatings - In addition to the anodic coatings detailed in this specification (Types IV and VII), MIL-M-45202 details several treatments most often used with magnesium, namely the Dow 17, the HAE and CR-22. Anodic coatings on magnesium are much softer, less dense and less protective than they are on aluminum. Usually, anodic coatings on magnesium require an additional treatment with an organic finish or an inorganic sealant. In addition to the coatings detailed in MIL-M-45202, the Manodysz process is often used for an anodic coating. The coating produced is one of excellent wear resistant with good dielectric properties.

6.4.3 Other coating systems - The stannate protective coating for magnesium consists partly of a magnesium-tin-hydroxide and partly of metallic tin. This type coating has some of the properties of a chemical conversion coating and some of the properties of a metallic coating. The film, formed from a stannate bath, provides good corrosion resistance and can be used as a base for paint systems.

6.5 Cross-references - The correlation between the types used in this specification, the previous designations and the common commercial designations for the magnesium alloy processes are indicated in Table X.

6.6 Caution - Due consideration should be given by contractors to the occupational health hazards associated with the processes covered by this specification.

6.6.1 Among the special precautions indicated for the more significant hazards are: the use of protective clothing; the avoidance of skin contact with organic solvents, as well as avoidance of the inhalation of their vapors; protection during sandblasting operations; avoidance of skin contact and protection against the inhalation of alkali fumes or mists; the avoidance of skin contact with nitric, sulfuric, phosphoric, and hydrofluoric acids,

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TABLE X

CORRELATION OF TREATMENT TYPES

Treatment	MIL-M-3171C	MIL-M-3171B	MIL-M-3171A	Commercial designations
Chrome Pickle	Type I	Type I	Type I	Dow No. 1, MACroMag 101
Sealed Chrome Pickle	deleted	deleted	Type II	Dow No. 10
Dichromate	Type III	Type III	Type III	Dow No. 7, MACroMag D-7
Galvanic Anodizing	Type IV	Type IV	Type IV	Dow No. 9, MACroMag D-9
Caustic Anodizing	deleted	deleted	Type V	Dow No. 12
Chromic Acid Brush-on	Type VI	Type VI	---	Dow No. 19, MACroMag D-19
Fluoride Anodizing	Type VII	---	---	Magnesium Elektron
Chromate	Type VIII	---	---	Iridite No. 15

protection of the eyes by proper safety goggles, and the avoidance of the inhalation of their vapors or fumes; and protection against skin contact and inhalation of chromium compounds.

6.6.2 Storage and treating tanks should be provided with such hoods and other ventilating equipment as is necessary to prevent atmospheric contamination.

6.6.3 In the handling of acids or alkalies, rubber gloves and protective clothing should be worn. Accidental contact should be followed by speedy removal of the excess acid or alkali by blotting with clean mechanics waste, or a similar agent, after removal of the soiled clothing and shoes. Profuse irrigation of the surfaces involved should then be done with large quantities of water. Special care should be taken to avoid all skin contact with hydrofluoric acid.

6.6.4 A physician should be consulted in all cases of contamination of the skin with any of these acids and also in cases where inhalation of vapors or fumes causes respiratory distress

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6.6.5 Acid pickling of castings sometimes results in formation of a gray powder layer which can spark or ignite easily thus presenting a definite safety hazard. Although this powder is usually associated with chromate type coating, particularly Type I, it may also occur on castings treated in such acid pickles as sulfuric (see 3.4.3), nitric-sulfuric (see 3.4.4), etc., that are free of chromates. This powder results from excessive treatment time and can be eliminated by good production practices (see 3.5.5.4).

6.7 Changes from previous issue - The outside margins of this specification have been marked "#" to indicate where changes (deletions, additions, etc) from the previous issue have been made. This has been done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in those notations. Bidders and contractors are cautioned to evaluate the requirements of this document based on the entire content as written irrespective of the marginal notations and relationship to the last previous issue.

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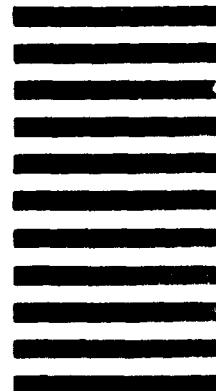
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