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Standard Practices for Preparation of Magnesium Alloy Surfaces for Painting¹

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These practices cover two classes of treatment for preparation of magnesium alloy surfaces for painting, as follows:

Class I—Chemical Treatments. Class II—Anodic Treatments.

In general, the latter treatments are the more protective of the two classes. Mechanical (abrasive) treatments, solvent cleaning, alkaline solution treatments, and acid pickles not resulting in protective conversion coatings are suitable preliminary treatments only for metal to be exposed under mildly corrosive (indoor) exposures. When a high degree of corrosion protection and paint adhesion are desired, as in many outdoor environments, surface preparation by one of the above conversion-coat classes is necessary.

NOTE 1—*Testing of Coatings*—Quality control tests of coatings are frequently desirable, and these generally consist of exposures, with or without paint, to salt spray, humidity, or natural environments, with suitable procedures for assessing the degree of breakdown suffered after fixed time intervals. It is recommended that quality control tests of coatings shall be made as far as possible with high-purity material (for example AZ31A alloy),² the inherent corrosion rate of which is relatively consistent from batch to batch) and that precautions shall be taken to remove surface contamination before coatings are applied. Such contamination shall be removed by acid pickling to a depth of at least 0.001 in. (25 μm) per side.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. (See Note 11.)*

¹ These practices are under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.07 on Chemical Conversion Coatings.

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² For information concerning magnesium and aluminum alloys, see ASTM Specification B 80, B 90, B 91, B 93, B 107, and B 209 covering these alloys, in the section on Aluminum and Magnesium and Their Alloys, *Annual Book of ASTM Standards*, Vol 02.02. See also ASTM Practice B 275, for Codification of Certain Nonferrous Metals and Alloys, Cast and Wrought.

PRELIMINARY TREATMENT OF SURFACES

2. Procedure

2.1 Certain anodic treatments simultaneously produce conversion coatings on, and remove contamination from, magnesium alloy surfaces. In general, however, apply conversion coatings only to surfaces previously freed from all contamination, including oxide, rolling-scale, corrosion product, burned-on drawing and forming lubricant, and the contamination introduced by blast cleaning and fabrication operations. Contamination in or under surface conversion coatings seriously reduces their protective values (Note 2). For the removal of tenacious surface contamination, such as rolling-scale or casting skin, an acid pickle to dissolve some of the actual surface is essential. When organic contamination, such as grease or oil, is also present, an initial degreasing operation in solvent or in an alkaline degreasing solution is usually necessary to allow the subsequent acid to wet the surface. These matters are discussed in more detail under the headings of the specific cleaners or treatments (Note 3), as follows:

2.2 *Alkaline Cleaners*—Oil, grease, and old (but not baked) chrome-pickle coatings are readily removed by most commercially available heavy-duty alkaline cleaners; but such cleaners are not suitable for removing oxide and the like, for which purpose use acid pickles, preceded by alkaline cleaners. Remove graphite lubricant and also baked chrome-pickle coatings by a solution conforming to the following composition:

Caustic soda (NaOH)	12 oz (90 g)
Wetting agent	0.1 oz (0.74 g)
Water (Note 4)	1 gal (1 litre)

Soak the parts in the above cleaner for 10 to 20 min at boiling-point, and a treatment shall follow either in the chromic acid-nitrate pickle described under 2.3.3 or, for parts machined to fine tolerances, in the chromium trioxide solution described under 2.3.1. After alkaline cleaning, rinse in water very thoroughly. Alkaline cleaners may be held in plain steel tanks.

2.3 *Acid Cleaners* (Note 1)—Acid pickling removes mill-scale, oxide, corrosion product, and the like. Use as a preliminary treatment for surface conversion coatings when the highest degrees of surface uniformity and protective values are required. Acid cleaners are as follows:

2.3.1 *For Sand and Permanent Mold Castings:*

2.3.1.1 *Nitric-Sulfuric Acid Solution*— Use a solution of 8 volume % of concentrated nitric acid (HNO_3) plus 2 volume % of concentrated sulfuric acid (H_2SO_4) in water (see Note 1(a)) at 70 to 90°F (21 to 32°C) as a preliminary treatment for new sand castings and to remove the surface-contaminating effects of blast cleaning. Immerse for about 10 to 15 s, or until 0.002 in. (51 μm) per surface is removed. The solution may be held in ceramic, rubber, synthetic rubber, or vinyl-lined tanks.

2.3.1.2 *Chromic Acid Solution*—Use a boiling 20 weight % solution of chromic anhydride (CrO_3) in water to remove old chemical and anodic treatments, corrosion product, and oxide layers, without significant dissolution of metal and hence without changing the dimensions of machined parts. Immersion time varies from 1 to 5 min, depending upon the condition of the surface. The solution may be held in lead-lined steel or ASTM alloy No. 990A or its Aluminum Association equivalent, alloy No. 1100 aluminum tanks.

2.3.2 For Die Castings:

2.3.2.1 *Chromium Trioxide-Nitric-Hydrofluoric Acid Solution*—This solution is used to produce a smut-free surface on die castings, without violent attack of the metal. The solution shall conform to the following composition:

Chromium trioxide (CrO_3)	37.5 oz (280 g)
Hydrofluoric acid (60% HF)	1 fl oz (8 ml)
Nitric acid (70% HNO_3)	3.25 fl oz (25 ml)
Water (Note 4)	to 1 gal (1 litre)

Immerse the parts in the above solution at 70 to 90°F (21 to 32°C) for 30 s to 2 min, or until a bright, clean surface is obtained. The solution may be held in tanks lined with synthetic rubber or vinyl-base materials.

2.3.3 For Wrought Products:

2.3.3.1 *Acetic Acid-Nitrate Solution*—This solution rapidly removes surface contamination to 0.001 in. (25.4 μm). Use for wrought parts subsequently to be finished for the maximum protective value. The solution shall conform to the following composition:

Glacial acetic acid	25.5 fl oz (199 ml)
Sodium (NaNO_3)	6.6 oz (49.5 g)
Water (Note 4)	to 1 gal (1 litre)

Immerse the parts in the above solution at 70 to 90°F (21 to 32°C) for 30 s upwards, or until a bright, clean surface is obtained. When heavy surface contamination, such as hot-rolled mill-scale is to be removed, immersion times shall be sufficient to remove at least 0.001 in. (25 μm) per surface. The solution may be held in No. 990A aluminum, ceramic, or rubber-lined tanks.

2.3.3.2 *Chromium Trioxide-Nitrate Solution*— Use this solution following the use of the method described in 2.2 for the removal of burned-on graphite lubricants from hot-formed parts. The solution shall conform to the following composition:

Chromium trioxide (CrO_3)	1.5 lb (180 g)
Sodium nitrate (NaNO_3)	2 oz (15 g)
Water (Note 4)	to 1 gal (1 litre)

Immerse the parts in the above solution at 70 to 90°F (21 to 32°C) for approximately 5 min, for the removal of graphite.

2.4 Abnormally slow reaction in the above solution indicates that it is depleted and that the pH has risen to 1.7 or higher. Revivify the solution by the addition of chromium trioxide to bring the pH back to 0.5 to 0.7. Attempt no more

than four revivifications. The solution may be held in ceramic, No. 990A aluminum, 18-8 stainless steel, or synthetic rubber-lined tanks.

NOTE 2—*Removal of Contamination by Welding Fluxes*—When a part to be painted has been welded by an operation involving the use of flux, such flux shall be removed before the subjection of the part to any surface preparation process. Such removal shall be made immediately by immersing the part in hot water with scrubbing, and finally by immersing it for 1 h in a boiling 5% solution of sodium dichromate, after which the part shall be well rinsed.

NOTE 3—*Suspension of Articles for Treatment*—The use of magnesium alloy suspension wires is preferred for use in acid pickles in order to avoid objectionable contamination of the solutions through dissolution of the wire materials. Heavy metal contamination, particularly of copper, may deposit on the magnesium surface and lead to seriously reduced corrosion resistance. Copper suspension wires in the hot dichromate solutions are not objectionable.

NOTE 4—*Quality of Water*—In the preparation and make-up of acid pickles, dichromate solutions, and hot-water rinses, precautions shall be taken against the use of water contaminated with heavy-metal impurities, or excessive chlorides or sulfates. No upper limits can be specified at this stage for soluble impurities in the water, but powdering of coatings and poor resistance to corrosion are known to result from the use of contaminated water. Thus, when a choice exists, water from steam condensate or ion-exchange-treated water shall be employed in preference to well water or hard tap water.

SURFACE PREPARATION PROCEDURES— CHEMICAL

3. Class I, Type I (Chrome Pickle)³

3.1 *Scope*—Class I, Type I treatment is applicable to all forms and alloys of magnesium except certain special alloys containing silver, but since it may remove as much as 0.0006 in. (15 μm) of metal per surface, it shall not be used on parts machined to fine tolerances. When properly applied, the process constitutes a good paint base, but rigid control is required at each step. The treatment is applicable to magnesium alloy containing inserts of, or attached to, other metals.

3.2 *Procedure*—For wrought parts the bath shall conform to the following composition:

Sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$)	1.5 lb (180 g)
Nitric acid (HNO_3) (sp gr 1.42)	1.5 pt (187 ml)
Water (Note 4)	to 1 gal (1 litre)

3.2.1 For die-, sand- and permanent-mold castings the solution shall conform to the following composition:

Sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$)	1.5 lb (180 g)
Nitric acid (HNO_3) (sp gr 1.42)	1.5 pt (187 ml)
Sodium potassium, or ammonium acid fluoride (NaHF_2 , KHF_2 , or NH_4HF_2)	2 oz (15 g)
Water (Note 4)	to 1 gal (1.0 litres)

For wrought products, sand, and permanent-mold castings the above solutions operate at 70 to 90°F (21 to 32°C). The immersion times shall be from 1 to 2 min, the necessary time increasing with use of the solution. For die-castings give the parts a 15 to 30 s dip in water at 160 to 180°F (71 to 82°C), followed immediately by a 10-s dip in the second of the above baths, operated at 120 to 140°F (49 to 60°C). Failure to preheat the castings results in no coating in 10 s.

³ Conforming to Class I, Type I treatments are the Dow No. 1 process, the AMC "A" process, and the Type I process of Military Specification MIL-M-3171A.

3.2.2 Following immersion remove the parts, allow to drain for not less than 5 s nor more than 30 s, then wash thoroughly in cold running water, followed by a dip in hot water at 160 to 180°F (71 to 82°C) to facilitate drying. Do not allow the parts to drain following the chromate treatment for more than the specified 30 s; excessive drainage times result in powdery coatings of poor value as paint bases. Such coatings also result from the use of hot-water rinses, the temperatures of which are in excess of 180°F (82°C). Paint the parts preferably immediately after they are dry. The solution may be held in Type 316 stainless steel, or ceramic tanks or in steel tanks lined with synthetic rubber or vinyl-base materials. Tanks of ASTM alloy No. 990 A, or its Aluminum Association equivalent, Alloy No. 1100 aluminum are satisfactory for the nonfluoride-containing pickle.

3.3 *Revivification of Solutions*—Sluggish reaction with the metal, associated with pale yellow, lustrous coatings, indicates that the solution is depleted. Revivification is accomplished by the addition of dichromate and nitric acid, to raise the dichromate again to 1.5 lb (180 g)/gal (1 litre) and the free nitric acid to levels indicated in the table below. Revivify when the free nitric acid content of the solution is depleted to 0.5 pt/gal (62.3 ml/1 litre) (see Section 11 for analytical procedure) and shall take place once only for M1 and ZK60A alloys,³ and not more than six times for other alloys if good paint-base properties are desired. Excessive use of the solution or too many revivifications result in smooth, lustrous coatings not possessing the degree of etch necessary for the best paint adhesion with conventional primers. Revivify in accordance with the following table:

Revivification Number	Adjust Concentration of HNO ₃ to:
1	1.3 pt/gal (162 ml/1 litre)
2	1.1 pt/gal (137 ml/1 litre)
3 to 6	0.9 pt/gal (113 ml/1 litre)

4. Class I, Type II (Sealed Chrome Pickle)⁴

4.1 *Scope*—Class I, Type II treatment is applicable to all types and forms of magnesium-base alloys, subject only to the limitations of the Class I, Type I treatment, since it is essentially the same process as the latter, followed by sealing. In protective qualities and as a paint base, this treatment is somewhat superior to that of Class I, Type I treatment under severe exposure conditions (Note 5).

4.2 *Procedure*—Following chrome pickling as specified under Section 3 and rinsing in cold water, transfer the parts immediately to a boiling solution conforming to the following composition:

Sodium dichromate (Na ₂ Cr ₂ O ₇ ·2H ₂ O)	1.5 lb (180 g)
Calcium or magnesium fluoride (CaF ₂ or MgF ₂)	½ oz (2.5 g)
Water (Note 4)	to 1 gal (1.0 litre)

Boil the parts in the above solution for 30 min, after which rinse them in cold running water, followed by a rinse in hot water at a temperature of not less than 160°F (71°C) nor more than 180°F (82°C) to facilitate drying. Preferably apply the paint coating immediately after the parts are dry. The solution

may be held in a steel tank.

NOTE 5—*Causes of Defective Coatings*—The following information is intended to provide guidance on the causes of the most usual defects arising in the application of either Class I, Type I or Class I, Type II coatings:

- (a) *Spotted Coatings* are caused by ineffective preliminary degreasing or by the presence of excessive surface contamination not removed prior to or during the chrome pickling treatment, or both.
- (b) *Nonadherent Powdery Coatings* are caused by:
 - (1) Too long an interval between removal from the chrome pickle and rinsing,
 - (2) Ratio of acid to sodium dichromate too high,
 - (3) Temperature of the solution or of the hot-water rinse too high.
 - (4) Metal improperly degreased, or
 - (5) Solution revived too many times.

5. Class I, Type III⁵

5.1 *Scope*—Class I, Type III treatment is applicable to all types and forms of magnesium-base alloys except M1 alloy² and certain rare-earth alloys similar to EK30A.² It produces in itself no appreciable dimensional change and is, therefore, applicable to parts machined to fine tolerances. The treatment is applicable to magnesium containing inserts of, or attached to, other metals, but in such cases the preliminary treatment (5.2) shall be in the bifluoride bath (Solution No. 2) described in 5.2.2.

NOTE 6—*Causes of Defective Coatings*—The following information is intended to provide guidance on the causes of the more usual defects arising in the application of coatings from the Class I, Type III treatment.

- (a) *Nonadherent Powdery Coatings* are caused by:
 - (1) Over-dilution of the hydrofluoric acid or acid fluoride solution,
 - (2) Low pH (less than 4.0) of the dichromate solution,
 - (3) Insufficient precleaning of the metal surface, and
 - (4) Direct contact between the steel tank containing the dichromate solution and the article being treated.
- (b) *Failure to Coat and Nonuniform Coatings* are caused by:
 - (1) High pH of the dichromate solution,
 - (2) Low concentration of the dichromate,
 - (3) Insufficient precleaning of the metal surface,
 - (4) Omission of fluoride treatment,
 - (5) Use of an unsuitable alloy for the treatment, for instance, M1 alloy,
 - (6) Excessive immersion time in the hydrofluoric acid solution or use of an H₂F₂ concentration in the dichromate solution in excess of 0.2%,
 - (7) Insufficient rinsing after the hydrofluoric acid dip, and
 - (8) Insufficient heating of the dichromate solution (minimum temperature should be 200°F (93°C)).

5.2 *Procedure*—Following cleaning as prescribed in Section 2, treat the parts first by immersion at 70 to 90°F (21 to 32°C) in one or other of the following solutions:

5.2.1 Solution No. 1:

Hydrofluoric acid (60% H ₂ F ₂)	24 fl oz (187 ml)
Water (Note 4)	to 1 gal (1 litre)

5.2.2 Solution No. 2:

Sodium, potassium, or ammonium acid fluoride (NaHF ₂ , KHF ₂ , or NH ₄ HF ₂)	6⅔ oz (50 g)
Water (Note 4)	to 1 gal (1 litre)

⁴ Conforming to Class I, Type II treatments are the AMC "L" process, the Dow No. 10 process, and Type II treatment of Military Specification MIL-M-3171A.

⁵ Conforming to Class I, Type III treatments are the AMC "G" process, the Dow No. 7 process, and the Type III treatment of Military Specification MIL-M-3171A.

Solutions No. 1 and No. 2 may be held in tanks consisting of steel lined with lead or rubber. In solution No. 1, immerse AZ31A and AZ31B alloy parts³ for from 30 s to 1 min; immerse all other alloys for 5 min. In solution No. 2, immerse all the alloys for 5 min. Solution No. 2 is suitable for use with all forms of magnesium alloys except those castings which have *not* been acid-pickled after blasting; these castings shall be treated in the hydrofluoric acid solution (solution No. 1). Following one or other of the above treatments, the parts shall be rinsed thoroughly in cold running water and transferred to a solution conforming to the following composition:

Sodium dichromate (Na ₂ Cr ₂ O ₇ ·H ₂ O)	1 to 1.5 lb (120 to 180 g)
Calcium or magnesium fluoride (CaF ₂ or MgF ₂)	½ oz (2.5 g)
Water (Note 4)	to 1 gal (1 litre)

Operate the above solution at boiling point and immerse the parts therein for 30 min, following which thoroughly rinse them in cold running water, followed by a hot-water dip to facilitate drying. Preferably, apply the paint coating as soon as possible thereafter. A mild steel tank is suitable for holding the above solution.

5.3 Maintenance of Solutions:

5.3.1 *Fluoride Solutions*—Keep the concentration of free hydrofluoric acid in the solutions of 5.2.1 and 5.2.2 constant by the addition, as required, of either hydrofluoric acid or acid fluoride, respectively. See Section 9 for the analytical procedure.

5.3.2 *Dichromate Solution*—Keep the pH of the dichromate solution within the limits 4.0 to 5.5, or 4.0 to 4.8 in the case of AZ31A or AZ31B alloy,² by addition of chromium trioxide as required. Maintain the level by the addition of water. Maintain the solution saturated with respect to either magnesium or calcium fluoride by continuous immersion of a cloth bag containing excess of one or other of these compounds.

SURFACE PREPARATION PROCEDURES—ANODIC

6. Class II, Type I (Galvanic Dichromate Treatment)⁶

6.1 *Scope*—This treatment produces black coatings of good protective and paint-base qualities, and is applicable to all alloys and forms of magnesium, including M1 alloy,² with or without attachments of other metals. No appreciable dimensional change results, and hence the treatment is also applicable to articles machined to fine tolerances.

6.2 *Procedure*—Following cleaning as prescribed in Section 2, treat the articles in fluoride solution No. 1 or No. 2 as prescribed in 5.2. Use the latter solution when other metals are attached to the magnesium. After rinsing, immerse the articles in a solution conforming to the following composition:

Ammonium sulfate ((NH ₄) ₂ SO ₄)	4 oz (30 g)
Sodium dichromate (Na ₂ Cr ₂ O ₇ ·2H ₂ O)	4 oz (30 g)
Ammonia (NH ₄ OH) (sp gr 0.880)	½ fl oz (2.6 ml)
Water (Note 4)	1 gal (1 litre)

Operate the above solution at 120 to 140°F (49 to 60°C). The articles shall be made the anodes in the solution, with the tank, if of mild steel, acting as cathode, or with separate steel

cathodes if the tank is lined with nonmetallic materials. No separate generator is necessary, but the magnesium parts must be electrically connected with the tank, or with the separate cathode plates through an external connection, taking care that the parts do not make direct contact with the cathode material.

6.2.1 The time of treatment shall be such that a uniform black coating is obtained on the articles. This takes from 10 to 30 min, and the consumption of from 70 to 150 A·min/ft² (929 cm²). An anodic current density of not more than 10 A/ft² (929 cm²) is desirable.

6.2.2 Following the treatment, rinse the parts thoroughly in cold running water, followed by a hot-water dip to facilitate drying.

6.3 Maintenance of Solutions:

6.3.1 *Fluoride Solutions*—Maintain the fluoride solutions as under 5.3.1. See Section 9 for the analytical procedure.

6.3.2 *Sulfate-Dichromate Solution*—Maintain the pH of this solution between 5.6 and 6.2 by periodic additions of a solution containing 5 weight % of both chromic anhydride (CrO₃) and concentrated sulfuric acid (H₂SO₄, sp gr 1.84).

7. Class II, Type II⁷

7.1 *Scope*—This treatment is applicable to all forms and alloys of magnesium. Many aluminum alloys will anodically polarize in the treatment solution, and hence magnesium parts with aluminum attachments or inserts can be anodized. With aluminum alloys containing copper, however, the coating of the magnesium will proceed only if the area of the aluminum alloy is small compared to that of the magnesium. Metals other than aluminum or its base alloys shall not be in contact with the magnesium.

7.2 *Coating Properties*—The anodic coating consists of two phases: the first-formed phase is greenish-tan in color and about 0.0002 in. (5 μm) thick; whereas the second phase, formed only at higher voltages following the production of the tan coating, is fused and vitreous in nature, dark-green in color, and of thickness about 0.0012 in. (30 μm), causing a dimensional increase of about 0.001 in. (25 μm) per surface. The corrosion-resistance and paint-base characteristics are similar, and excellent for both the above phases. The thicker coating possesses high abrasion resistance, but spalls under compression deformation. The dark-green coating can be partially impregnated with low-viscosity, penetrating, organic coating materials, a procedure that considerably reduces its spalling tendency.

NOTE 7—The dark-green coating of Class II, Type II treatments is recommended in preference to the thin tan coating only when:

- Preliminary removal of surface contamination is not convenient.
- The highest degree of abrasion-resistance is required from the coating.
- A dimensional increase (see above) can be tolerated.
- The article will not be subjected in service to impact, deformation, or flexing likely to cause spalling of the coating.

7.3 *Procedure*—The thin tan coating is formed with a current consumption and in a time approximately one quarter of that required to form the dark-green coating. The latter

⁶ Conforming to Class II, Type I treatment are the AMC "K" process, the Dow No. 9 process, and Type IV treatment of Military Specification MIL-M-3171A.

⁷ Conforming to Class II, Type II treatment is the Dow No. 17 process.