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Standard Practice for
Applying Non-Electrolytic Conversion Coatings on Magnesium and Magnesium Alloys[1]

This standard is issued under the fixed designation B879; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a guide for metal finishers to clean and then provide a paint base for the finishing of magnesium and magnesium alloys using chemical conversion coatings. Where applicable (for example, aerospace) secondary supplementary coatings (for example, surface sealing) can be used (see Appendix X1).

1.2 Although primarily intended as a base for paint, chemical conversion coatings provide varying degrees of surface protection for magnesium parts exposed to indoor atmosphere either in storage or in service under mild exposure conditions. An example is the extensive use of the dichromate treatment (see 5.2) as a final coating for machined surfaces of die cast magnesium components in the computer industry.

1.3 The traditional numbering of the coating is used throughout.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 The following documents form a part of this practice to the extent referenced herein.

2.2 *ASTM Standards:*[2]

D1732 Practices for Preparation of Magnesium Alloy Surfaces for Painting

2.3 *SAE Standard:*[3]

AMS 2475 Protective Treatments—Magnesium Alloys

2.4 *Military Specifications:*[4]

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

DTD 911 (British), Protection of Magnesium-Rich Alloys Against Corrosion

DTD 5562 (British), Clear Baking Resin for Surface Sealing Magnesium

DTD 935 (British), Surface Sealing of Magnesium Rich Alloys

3. Significance and Use

3.1 The processes described in this practice clean and provide a paint base for the finishing of magnesium and magnesium alloys. Service conditions will determine, to some degree, the specific process to be applied.

4. Reagents

4.1 The chemicals that are used to formulate and control the processing solutions are listed in Table 1. Commercial grade chemicals are satisfactory. The concentrations stated for chemicals that are normally supplied at less than a nominal 100 % strength are those typically available. Other strengths may be used in the proportions that yield the specified processing concentrations. Unless otherwise stated all solutions are made up using water.

5. Types of Coating

5.1 *Chrome Pickle (Traditional Number 1) Treatment (See Practices D1732):*

5.1.1 With slight variations this treatment can be applied to all alloys and forms of magnesium. The treatment removes up to 15 µm of metal per surface, 30 µm per diameter. Therefore, it may not be applicable to machined surfaces with close tolerances. Parts with steel inserts may be processed, but some slight etching of the steel surface may occur.

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[1] This practice is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.07 on Conversion Coatings.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

[4] Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://dodssp.daps.dla.mil.

TABLE 1 Processing Chemicals

Acetic acid glacial, (CH_3COOH)
Aluminum sulfate ($Al_2 [SO^3]_3 \cdot 14H_2O$)
Ammonium bifluoride (NH_4HF_2)
Ammonium hydroxide (NH_4OH), 30 %
Ammonium phosphate monobasic ($NH_4H_2PO_4$)
Ammonium sulfate ($[NH_4]_2SO_4$)
Ammonium sulfite ($[NH_4]_2SO_3 \cdot H_2O$)
Calcium chromate ($CaCrO_4$)
Calcium fluoride (CaF_2)
Calcium sulfate ($CaSO_4 \cdot 2H_2O$)
Chromic acid (CrO_3)
Ferric nitrate ($Fe[NO_3]_3 \cdot 9H_2O$)
Glycolic acid ($HOCH_2COOH$), 70 %
Hydrofluoric acid (HF), 60 %
Magnesium fluoride (MgF_2)
Magnesium nitrate ($Mg[NO_3]_2 \cdot 6H_2O$)
Magnesium sulfate ($MgSO_4 \cdot 7H_2O$)
Manganese sulfate ($MnSO_4 \cdot 5H_2O$)
Nitric acid (HNO_3), sp gr 1.42
Phosphoric acid (H_3PO_4), 85 %
Potassium fluoride (KF)
Potassium bifluoride (KHF_2)
Sodium bifluoride ($NaHF_2$)
Sodium bisulfate ($NaHSO_4$)
Sodium carbonate (Na_2CO_3)
Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$)
Sodium hydroxide (NaOH)
Sodium metasilicate (Na_2SiO_3, or $Na_2SiO_3 \cdot 4H_2O$)
Sodium nitrate ($NaNO_3$)
Sulfuric acid (H_2SO_4), sp gr 1.84

5.1.2 The color, luster, and etch produced by the treatment will vary with the age and usage of the solution, alloy composition, and heat treatment of the alloy. The most desirable paint base is a matte grey to yellow-red, iridescent coating which exhibits a pebbled etch finish when viewed under low magnification (5 to 10×). Bright brassy coatings, showing a relatively smooth surface with only occasional rounded pits under low magnification are unsatisfactory as a paint base but are acceptable for protection during shipping and storage.

5.2 *Dichromate (Traditional Number 7) Treatment (see Practices D1732):*

5.2.1 This treatment provides an improved paint base compared with the chrome pickle treatment, and for temporary protection on all standard alloys except, EK41A, HM31A, HM21A, HK31A, WE54, WE43, and M1A on which the coating does not form. The treatment causes no appreciable dimensional changes, is normally applied after machining, and is suitable for close clearance parts. Parts containing inserts of bronze, brass, steel, or cadmium plated steel should not be treated unless the dissimilar metals are masked or it is demonstrated that the treatment will not adversely affect them. For assemblies containing aluminum inserts or rivets, the acid fluoride treatment (see 7.2.3) should replace the hydrofluoric acid treatment in part preparation.

5.2.2 Coatings vary from light to dark brown depending upon the alloy. On AZ91C-T6 and AZ92A-T6 castings the coating is grey.

5.3 *Galvanic Chromate (Traditional Number 9) Treatment (see Practices D1732):*

5.3.1 This treatment can be used for all alloys and is specifically used for those alloys which do not react or form satisfactory conversion coatings in other baths. The treatment requires no external current but utilizes the relatively high potential difference between suitably racked magnesium components and steel tank walls or other cathodes. As with the dichromate treatment, a prior immersion in acid fluoride solution is required to condition the magnesium surface. The galvanic chromate treatment causes no appreciable dimensional change and is normally applied after machining.

5.3.2 Properly applied coatings vary from dark brown to a dense black color depending on the alloy. The treatment is particularly useful for application to optical equipment requiring a nonreflective black coating.

5.4 *Chromic Acid Brush-On (Traditional Number 19) Treatment:*

5.4.1 This treatment can be applied to parts that require touch up. It is generally used in refinishing procedures or where parts or assemblies are too large to be immersed. It is effective on most alloys and causes negligible dimensional changes.

5.4.2 Coatings produced by this treatment can vary from a brassy iridescence to a dark brown depending upon treatment time. Prolonged treatment produces powdery coatings. For best adhesion, dark brown coatings are preferred.

5.5 *Chromate Treatment (see DTD 911):*

5.5.1 This treatment is suitable for all magnesium alloys. The treatment causes no dimensional change and is normally applied after machining. The pickling procedures and the composition of the treating solution generally vary with the alloy being processed.

5.5.2 The coating will vary from dark brown to light reddish-brown depending on the alloy.

5.6 *Chrome-Manganese Treatment:*

5.6.1 This treatment provides an improved paint base compared with the chrome pickle treatment and protection on all standard alloys except EK41A, HM31A, HM21A, HK31A, and M1A on which the coating does not form. The treatment causes no appreciable dimensional change, and normally is applied after machining. It is suitable for close clearance parts. Parts containing inserts of bronze, brass, steel, or cadmium plated steel should not be treated unless the dissimilar metals are masked or it is demonstrated that the treatment will not adversely affect them.

5.6.2 The bath generally gives dark brown to black films on both cast and wrought magnesium alloys. Treatment of aluminum containing alloys may require bath temperatures above 50°C.

5.7 *SemiBright Pickle (Traditional Number 21) Treatment*—This treatment provides a semibright silvery surface on magnesium parts that prevents tarnishing and corrosion for indoor storage up to six months in non-air-conditioned environments. Extended storage times can be obtained by using air conditioning. This process causes negligible dimensional change. It is a simple, economical way to apply an attractive shelf-life finish and is a good base for clear lacquers. The treatment greatly reduces or eliminates "filiform or worm-tracking" corrosion usually experienced when clear paints are used directly over polished metal surfaces.

5.8 *Phosphate Treatment:*

5.8.1 Phosphate treatments can provide a satisfactory paint base on magnesium for many applications when it is necessary to avoid the use of chromates. Commercial iron phosphate treatments applied by spray or dipping have been successfully used on magnesium die castings for automotive and other consumer product applications. The suitability of a particular phosphatizing process for magnesium should be verified by testing. Iron phosphate treatments containing nickel or copper salts as accelerators are detrimental to the corrosion resistance of magnesium and should not be used.

5.8.2 Phosphate treatments do not provide interim stand-alone protection against atmospheric oxidation and tarnish equal to that provided by some chromate conversion coatings.

6. Part Preparation

6.1 *Cleaning—General:*

6.1.1 Before considering the use of solvent degreasing, consult federal and state safety and environmental laws and regulations. Many of the commonly used solvents are now being banned from use. Exposure to their vapor (VOC) is being strictly regulated for health, safety, and environmental reasons. Obtain current safe exposure levels for various solvents before use. Follow all federal, state, and local regulations for the disposal of solvents.

6.1.2 *Solvent Cleaning*—Grease or oil may be removed by means of vapor degreasing, ultrasonic cleaning, solvent washing, or an emulsion cleaning process that utilizes a mineral oil distillate and an emulsifying agent. Chlorinated solvents, petroleum spirits, naphths, lacquer thinner, and similar solvents that do not attack magnesium may be used. Methyl alcohol (CH_3OH) should not be used because it may react with the magnesium surface.

6.1.3 *Mechanical Cleaning*—Mechanical cleaning may consist of sand, shot, pumice, grit or vapor blasting, sodium carbonate slurry, sanding, hard bristle brushing, grinding and rough polishing. Sand, shot, or grit blasting leaves surface contamination that will greatly increase the corrosion rate of the magnesium on exposure to salt water or humid environment. If these methods are used, specific pickling procedures must be employed after blasting (see 6.4.2).

6.1.4 *Alkaline Cleaning*—Cleaning prior to application of treatments other than the chrome pickle treatment (see 5.1), when used for protection during shipment or storage, should be done in an alkaline cleaner recommended for steel or in a cleaning solution as specified in 6.1.4.1. Maintain the solution pH above 8.0. Alkaline cleaning prior to the application of the chrome pickle treatment (see 5.1), when used for protection during shipment and storage only, may be omitted provided the parts are free of grease, oil, and other deleterious deposits at the time of application. Alkaline cleaning solutions containing more than 2 % sodium hydroxide will etch ZK60A, ZK60B, and some other magnesium alloys producing a change in dimensions. If such a dimensional change is undesirable, use cleaners with lower alkali content.

6.1.4.1 Alkaline cleaning may be carried out in solutions of proprietary cleaners. In this case the operating conditions should be as specified by the supplier. In no case should a cleaner having pH lower than 8.0 be used. Most recommended cleaners are used by simple immersion. After alkaline cleaning, rise parts thoroughly in cold running water. No water breaks should be observed in the rinse.

6.1.5 *Electrolytic Cleaning*—Use of anodic current for cleaning is not generally recommended because of the possible formation of oxide films, pitting of the magnesium surface, or both. However, electrolytic cleaning using cathodic current at 1 to 4 A/dm^2 may be carried out in properly formulated cleaners.

6.2 *Graphite Lubricant Removal:*

6.2.1 Remove graphite-based lubricants from hot formed magnesium sheet parts by soaking the parts for 10 to 20 min in 100 g/L sodium hydroxide maintained at 88 to 100°C. The pH should be above 13.0. Add wetting agent (0.75 g/L), if needed, for the removal of heavy films of mineral oil. Then rinse parts thoroughly in cold water and immerse for 3 min in a chromic-nitrate pickle as specified in 6.5.2. Repeat the cycle until all parts are clean.

6.2.2 Because of the difficulty of removing graphite from chrome pickled sheet, such sheet should not be used for forming unless the chrome pickle is removed as outlined in 6.3 before forming.

6.3 *Previously Applied Chemical Finishes:*

6.3.1 Magnesium base alloys are often supplied with a chrome pickle treatment to protect them during shipment, storage, and machining. The coating from this treatment remaining on unmachined areas will impair the film produced by any subsequent chromate treatment and therefore must be removed.

6.3.2 Previously applied coatings may be removed with the alkaline cleaners recommended in 6.1.4.

6.3.3 If the finish is difficult to remove, immerse the part in the chromic acid pickle given in 6.5.1. Alternate immersion in the alkaline cleaner and the chromic acid pickle may be required to remove aged finishes. Rinse well in water between acid and alkaline pickling.

6.3.4 The chromic acid brush-on treatment (see 5.4) may be applied over the chrome pickle finish or over previously applied brush-on coatings without removing the previously applied coating.

6.4 *Acid Pickling:*

6.4.1 General pickling to remove oxide layers, old chemical finishes, burned-on drawing and forming lubricants, and other water insoluble or non-emulsifiable substances is preferably carried out using a chromic acid type pickle as described in 6.5.1 or 6.5.2.

6.4.2 Pickle sand and permanent mold castings that have been mechanically cleaned as described in 6.1.3 in the sulfuric acid pickle described in 6.5.4 or in the nitric-sulfuric acid pickle described in 6.5.5. The pickling operations should be of such duration that 50 µm of surface is removed whenever dimensional tolerances permit.

6.4.3 Magnesium sheet and plate up through 12.5 mm thick should have the mill scale removed to ensure a clean surface for the subsequent treatments. Where dimensional tolerances permit, remove 12 to 25 µm per metal surface. The acetic-nitrate pickle described in 6.5.8, or an equivalent solution, should be used.

6.4.4 Pickle die castings that are to be pickled for maximum corrosion resistance and a more uniform surface for applying subsequent coatings in the chromic-nitric-hydrofluoric acid pickle described in 6.5.6 or the phosphoric acid pickle described in 6.5.7.

6.5 *Acid Pickling Solutions:*

6.5.1 *Chromic Acid Pickling*—Chromic acid pickling causes no dimensional change and therefore may be used on parts with close tolerances. It may be used for removal of previously applied chemical finishes (see 6.3.3). It is satisfactory for the removal of surface oxidation and corrosion products and for general cleaning. It is *not* satisfactory for the removal of sand or effects of blasting and should *not* be used on parts that contain copper inserts unless the copper can be completely masked off. Excessive concentrations of chloride, sulfate, and fluoride must be avoided in the solution since these will cause etching or film formation rather than cleaning. The solution contains 180 g/L of chromic acid. Treatment time is 1 to 15 min at 88 to 95°C. Lower temperatures are permissible provided the treatment time is suitably increased.

6.5.2 *Chromic-Nitrate Pickle*—The chromic-nitrate pickle is not generally used for the removal of corrosion products of surface oxidation but may be substituted for the chromic acid pickle described in 6.5.1. It is mainly used for the removal of burned on graphite lubricants as described in 6.2. It is *not* suitable for the removal of sand from castings or for correcting the effects of blasting. It should *not* be used for parts containing copper inserts unless the inserts can be completely masked off. The solution contains 180 g/L chromic acid and 30 g/L sodium nitrate. Immersion time is 2 to 20 min at 15 to 30°C which normally removes 12.5 µm of metal/surface. Lack of chemical action and a pH of 1.7 or higher indicates depletion of the bath. The addition of sufficient chromic acid to restore a pH of 0.5 to 0.7 will rejuvenate the solution. After three to four replenishments, the additions of chromic acid required to rejuvenate become excessive, and the bath should be discarded and replaced.

6.5.3 *Nitric Acid Pickle*—Nitric acid pickle is used to clean and brighten alloys on which chromate films are *not* desired. The solution contains 70 mL/L nitric acid. Immersion time is 30 to 60 s at 20 to 27°C.

NOTE 1—This pickle will remove 12.5 to 25 µm per metal surface. Use ceramic, rubber-lined, or aluminum containers.

6.5.4 *Sulfuric Acid Pickle*—The sulfuric acid pickle is used on magnesium sand castings to remove the effects of blasting operations. Use the pickle before any machining operations since the amount of metal removed is likely to exceed permissible tolerances. The solution contains 32 mL/L sulfuric acid in water. Immersion time is 10 to 15 s at 20 to 32°C or as required to remove approximately 50 µm of metal per surface.

6.5.5 *Nitric-Sulfuric Acid Pickle*—As an alternative for the sulfuric acid pickle (see 6.5.4) the nitric-sulfuric acid pickle may be used. The solution contains 80 mL/L nitric acid and 20 mL/L sulfuric acid. Conditions of operation and time of immersion are the same as for the sulfuric acid pickle.

6.5.6 *Chromic-Nitric-Hydrofluoric Acid Pickle*—This pickle may be used on any casting, but is especially effective on die castings. It will remove metal at approximately 12.5 µm per minute per surface. The solution contains 280 g/L chromic acid, 25 mL/L nitric acid, and either 7.5 mL/L HF or 6.5 g/L ammonium bifluoride. Immersion time is 0.5 to 2 min at 21 to 32°C.

6.5.7 *Phosphoric Acid Pickle*—The phosphoric acid pickle may be used for pickling all castings, particularly die castings. It is especially effective at removing segregated aluminum from the surface of AZ91A, AZ91B, and AZ91D alloys. It has been used for some wrought alloys, such as HK31A. Dip the parts for 10 to 15 s in 50 to 85 v/o phosphoric acid and allow to drain and react in air for 30 to 60 s. *Without rinsing*, dip in sodium hydroxide 80 to 120 g/L for 30 s at 20 to 25°C. Follow with a cold-water rinse. Sodium hydroxide corrects the powdering effect of the phosphoric acid. Metal loss is 12.5 to 25 µm. Contain the phosphoric acid solution in tanks with rubber, glass, ceramic or lead lining.

6.5.8 *Acetic-Nitrate Pickle*—The acetic-nitrate pickle is suitable for the removal of mill scale and other surface contamination from sheet to ensure maximum effectiveness of the protective coating. This solution can be used on wrought forms and on solution heat treated castings. Castings in the as-cast condition (-F) or in the solution heat treated and aged condition (-T6) should not be pickled in this solution because a loose grey smut forms. Treat castings in the -F or -T6 conditions in the chromic-nitric-hydrofluoric acid pickle (see 6.5.6). For best results, the pickle should be allowed to remove 10 to 25 µm of metal per surface. The treatment may not therefore be suitable for the treatment of parts with close tolerances. The solution contains 200 mL/L acetic acid and 50 g/L sodium nitrate. Immersion time at 20 to 30°C is 0.5 to 1 min.

6.5.9 *Glycolic-Nitrate Pickle*—In cases where spray pickling is used or fumes are a problem, the glycolic-nitrate pickle may be substituted for the acetic-nitrate pickle. This will also reduce acid loss from vaporization. The solution contains 250 mL/L commercial glycolic acid, 200 g/L magnesium nitrate and 37.5 mL/L nitric acid. Immersion time at 20 to 30°C is 3 to 4 min.

6.5.10 *Pickling Prior to Spot Welding*—A special cleaning and pickling sequence is used to prepare parts for spot welding. The treatment results in a surface that has a very low surface resistance and that will retain this low value for some time. The sequence consists of: alkaline cleaning and rinsing as in 6.1.4; alkali neutralization in a dilute acid solution such as 0.5 to 1.0 volume % sulfuric acid or a 7.5 to 15 g/L sodium acid sulfate solution; treatment in a chromic-sulfuric acid pickle containing 180 g/L chromic acid and 0.5 mL/L sulfuric acid. Immersion time is 3 min at 20 to 30°C.

6.5.11 *Hydrofluoric-Sulfuric Acid Pickle*—This pickle is used (particularly in brightening die castings) when the 12.5 µm metal loss caused by the phosphoric acid pickle cannot be tolerated. This solution contains 15 to 20 volume % hydrofluoric acid and 5 volume % sulfuric acid. Immersion time at room temperature is 2 to 5 min. After the dip, rinse the parts in cold water.

6.6 *Fluoride Anodizing Cleaning (see MIL-M-3171 and DTD 911):*

6.6.1 *Fluoride Anodizing Cleaning*—This electrolytic process was developed for the cleaning of magnesium parts prior to conversion coating. While the process is normally used to clean raw sand castings, it is also particularly useful for the cleaning of components because the process does not significantly alter dimensions.

6.6.2 The electrolytic bath contains 150 to 250 g/L ammonium bifluoride in a tank lined with non-conducting hard rubber or suitable plastic material. A power source is required capable of delivering a progressively increasing ac output up to a maximum of 120 V. Current requirements will vary depending upon the size of the installation, but a minimum of 150 A/m^2 on one electrode, when the bath is being used to capacity is recommended. The power is applied via normal bus bars and clamps. All sections of fixing clamps immersed in the electrolyte must be of magnesium alloy, and parts to be cleaned should not contain any dissimilar metal inserts.

6.6.3 Parts are fixed in good electrical contact with the bus bars and located in the tank so that they are at least 0.25 m below the electrolyte surface and there is approximately equal surface area on each electrode. Alternating current is applied and the voltage progressively increased until 120 V is reached. Current flow is heavy at first but rapidly diminishes as the surface impurities are removed and an insulating coating of magnesium fluoride is formed. The treatment is complete when the current density falls below 50 A/m^2.

6.6.4 Certain alloys, however, may be etched at 120 V, particularly if the ammonium bifluoride concentration is towards the lower end of the range. For these alloys the process should be terminated at about 90 V. The magnesium surface, when satisfactorily anodized, should be a uniform gray-white color.

6.6.5 Thoroughly rinse parts in cold water after treatment, and strip the fluoride film in hot chromic acid as detailed in 6.5.1. If fluoride anodizing is followed by an hard anodizing process, such as the HAE treatment, removal of the fluoride film is not necessary.

7. Application of Coatings

7.1 *Chrome Pickle (Traditional Number 1) Treatment (see Practices D1732):*

7.1.1 Cleaning of parts to receive this treatment should be in accordance with the procedures outlined in Section 6.

7.1.2 *Procedures for Wrought Parts*—The solution for treating wrought parts contains 180 g/L sodium dichromate and 190 mL/L nitric acid. The bath is operated at 20 to 45°C and immersion times can range from 30 s to 2 min depending on the activity of the solution. Immerse the part and agitate in the solution under the conditions indicated above. Following treatment, drain the parts for at least 5 s and then rinse thoroughly in cold running water. Drying may be facilitated by a dip in hot water or with a hot air blast. Avoid heating above 200°C. Under some conditions, the coating may be applied by spraying the solution over the part rather than immersing the part in the solution.

7.1.3 *Procedures for Sand, Permanent Hold and Die Castings*—Chrome pickling of the widely used sand, permanent mold and die castings of high aluminum content (AZ91 types) requires modified solutions and procedures. The aluminum-rich phase present in these alloys can cause problems ranging from failure to react to selective reaction leaving a loose dark aluminum-rich smut deposit on the surface. Preventive action includes the addition of fluoride and sulfate to the solution, adjustment of temperature and treatment times, and the use of pretreatment pickles (chromic-nitric-hydrofluoric, phosphoric, or hydrofluoric-sulfuric) where necessary.

7.1.3.1 The solution for treating these types of castings contains of sodium bifluoride 15 g/L, sodium dichromate 120 g/L, aluminum sulfate 10 g/L, nitric acid 90 mL/L. Operating temperatures can range from 21 to 60°C. Preheat die castings, permanent mold, and aged sand castings by dipping in hot water (80 to 95°C) followed by immediate immersion in the treating solution. A 10 s immersion is sufficient if the bath is operated at 50 to 60°C. Lower temperatures require longer treating times.

7.1.3.2 Excessive treatment time will produce a powdery coating. Failure to preheat the part in hot water may result in failure of the coating to form. If a separate solution for die cast, permanent mold, and aged sand castings is not available, the bath used for wrought parts may be used. Sand castings in the solution heat treated condition may be chrome pickled in this solution at room temperature with no preheating. After treatment, drain the parts and rinse for at least 5 s and then rinse thoroughly in cold water. Drying may be facilitated by a dip in hot water.

7.1.3.3 *Optional Hydrofluoric Sulfuric Pre-Pickle*—Hydrofluoric acid 30 to 40 volume %, sulfuric acid 5 volume %, 2 to 5 min, at room temperature, followed by a cold water rinse.

7.1.3.4 *Dilute Chrome Pickle Treatment*—The hexavalent chromium content of this bath is about 20 % lower than the commonly used chrome pickle baths. This results in lower initial cost, less dragout loss, and lower effluent treatment costs. The paint base created is equivalent to that of a standard chrome pickle. The dimensional change is about 2 µm per surface for a treatment time of 20 to 30 s at 20 to 30°C. The bath contains: sodium bifluoride 2.5 g/L, sodium dichromate 35.0 g/L, magnesium sulfate 3.0 g/L, and nitric acid 30 mL/L.

7.1.4 *Precautions*—Chrome pickle baths may remove as much as 15 µm of metal per surface during treatment. Therefore, they can not be used on machined surfaces unless tolerances will permit or allowances are made. Magnesium parts containing steel inserts can be given the chrome pickle treatment. An excess of nitric acid or buildup of nitrate salts in the solution can result in the formation of bright brassy coatings as described in 5.1.2.

7.1.5 *Brush Application*—Parts too large to be immersed may be carefully brushed with a generous amount of fresh pickle solution made as specified in 7.1.2 or 7.1.3. The solution should remain on the surface for at least one minute while brushing is continued and washed off immediately thereafter with cold running water. The coating formed by this procedure is less uniform in color than that produced by immersion but is an equally good paint base.

7.1.5.1 Powdery coatings are not good paint bases and indicate poor rinsing or failure to keep the surface wet with solution during the one minute treatment time. The area being treated must be continually brushed with solution so that the surface never dries.

7.1.5.2 Brush application is suitable for touchup of all types of treatments that have been damaged. This type of coating is also best for touchup of bare areas before making an electrical bonding joint and where no primer can be applied.

7.1.6 Parts, such as tanks, that must be treated internally and that take a long time to fill and empty may be treated with the chrome pickle solution for wrought parts diluted with an equal quantity of water. The treatment time should be sufficient to produce a complete coating on all areas. The pickle bath may be diluted in a similar manner to increase treatment times to fit the time cycles of automated processing equipment.

7.1.7 *Control of the Chrome Pickle Treatment*—Control the process in accordance with the principles outlined in X1.1.

7.2 *Dichromate (Traditional Number 7) Treatment (see Practices D1732 and AMS 2475):*

7.2.1 Close control is required when applying this treatment to AZ31B-H24. Where maximum corrosion resistance is required on magnesium alloy sheet, use acid pickling as described in 6.5.2, 6.5.7, or 6.5.8.

7.2.2 *Hydrofluoric Acid Treatment*—After parts have been cleaned as described in Section 6, give them a hydrofluoric acid treatment. The solution contains 300 mL/L hydrofluoric acid operated at 20 to 30°C for 30 s to 5 min. This both cleans and activates the surface. Immerse parts made of AZ31B alloy for 30 s. Immerse all other wrought and cast alloys for 5 min. Following treatment, give the parts a thorough rinse in cold running water. Drag-over of fluoride ion will render the dichromate solution inoperative.

7.2.3 *Acid Fluoride Treatment*—Use this treatment for all parts containing aluminum inserts, rivets, etc. and as an alternative treatment to the hydrofluoric acid treatment (see 7.2.2). It is the preferred treatment for AZ31B and AZ31C alloy parts. It is generally more economical and safer than the hydrofluoric acid treatment, however, it will not remove the dark smudge that forms on some castings after sandblasting and pickling. If a dark smudge forms on the casting, use the hydrofluoric acid treatment in 7.2.2.

7.2.3.1 The acid fluoride solution contains 50 g/L of sodium, potassium, or ammonium bifluoride. It is operated at 20 to 30°C for a minimum of 5 min. After treatment, rinse the parts thoroughly in cold running water. See 7.2.2 on dragover of fluoride.

7.2.4 *Dichromate Treatment*—The solution consists of 120 to 180 g/L of sodium dichromate and 2.5 g/L of calcium or magnesium fluoride operated at boiling point for 30 to 45 min. After treatment, rinse the parts thoroughly in cold running water and dip in hot water or blow dry with hot air to facilitate drying. Apply required paint coatings as soon as practical after treated parts are thoroughly dry. Because ZK60A accepts the coatings more easily, a 15 min treatment is adequate for this alloy.

7.2.5 *Control of Dichromate Treatments*—Control the process as outlined in X2.2.

7.3 *Galvanic Chromate (Traditional Number 9) Treatment (see Practices D1732):*

7.3.1 *Acid Fluoride Treatment*—After parts have been cleaned as described in Section 4, immerse them for 5 min in a solution that contains 50 g/L sodium, potassium or ammonium bifluoride at 20 to 30°C. Alternatively, the hydrofluoric acid solution detailed in 7.2.2 may be used. After treatment rinse the parts thoroughly in cold running water.

7.3.2 *Galvanic Chromating*—The chromating solution, that is contained in a steel tank, contains 30 g/L ammonium sulphate, 30 g/L sodium dichromate, and 7.8 mL/L ammonium hydroxide, maintained at 50 to 60°C. The magnesium parts are electrically connected to the tank by an external circuit containing an Ammeter and rheostat. The magnesium parts are immersed in the solution and galvanically chromated at a current density of 21.5 to 105 A/m^2. A total of 750 to 1600 ampere minutes per square metre of magnesium is usually required to develop a uniform coating. After treatment rinse the parts thoroughly in cold running water followed by hot water to facilitate drying.

7.3.3 *Control of the Galvanic Chromate Treatment*—The process should be controlled as outlined in X2.5.

7.4 *Chrome-Manganese Treatment (see DTD 911):*

7.4.1 Cleaning of parts to receive this treatment should be in accordance with the procedures outlined in Section 6. It is however advantageous to precede the chromate treatment with an alkaline clean and, after rinsing, to immediately transfer parts, while still wet, to the chrome-manganese bath.

7.4.2 The solution contains of 100 g/L sodium dichromate, 50 g/L manganese sulphate, and 50 g/L magnesium sulphate. The bath is normally operated at room temperature (20 to 30°C) with immersion times of 1.5 to 2 h. The bath may be operated at higher temperatures to shorten treatment time, for example, 30 min at 50 to 60°C, 15 min at 70 to 80°C, and 10 min at 90 to 95°C. Satisfactory chromating of the magnesium-aluminum series alloys, for example, AZ91, AZ92, AZ61, and AZ31, may require elevated temperature bath operation. After treatment thoroughly rinse the parts in cold then warm water and dried.

7.4.3 *Control of the Chrome Manganese Treatment*—Control the process as outlined in X1.4.

7.5 *Semi-Bright Pickle (Traditional Number 21) Treatment:*

7.5.1 Any of the usual cleaning and degreasing processes used for magnesium alloys can be used. However, the bright appearance given by this treatment can be enhanced by giving the part a nitric acid pickle (see 6.5.3) or mechanical cleaning.

7.5.2 The solution consists of 180 g/L of chromic acid, 40 g/L ferric nitrate, and 3.5 g/L potassium fluoride. The bath is operated at 18 to 38°C. Lower bath temperature reduce staining in air during transfer from pickle to rinse. Longer pickling times yield maximum brightness. Immersion times are 15 s to 3 min. The solution, castings, or both, must be agitated during treatment. After removal from the pickling solution, rinse immediately in cold running water followed by a hot water rinse to speed drying.

7.5.3 If stains develop due to the length of time between pickling and rinsing, go directly from the pickling tank to a tank containing the following solution: 50 g/L of sodium metasilicate, sodium carbonate, or sodium hydroxide. Immerse the parts for 30 s to 1 min in the hot solution (70 to 80°). After removing the parts from the dip solution, rinse thoroughly in cold running water followed by a hot water rinse to aid in drying.

7.6 *Phosphate Treatment:*

7.6.1 Any of the de-greasing processes used for magnesium alloys are satisfactory, followed by an alkaline cleaning.

7.6.2 The solution consists of 120 g/L ammonium phosphate monobasic, 30 g/L ammonium sulfite, and 16 mL/L ammonium hydroxide. The bath is operated at room temperature with mild mechanical agitation. Immersion time is 1.5 to 2 min or until gassing stops. Immediately rinse the treated parts in cold water after removal from the bath and before the surface dries.

7.7 *Chromic Acid Brush-On (Traditional Number 19) Treatment:*

7.7.1 The brush-on technique for the chromic acid treatment is less critical than for the chrome pickle treatment. While the treatment solution will not cause problems if trapped between faying surfaces, such traps should be avoided in design or properly sealed to prevent entrance of corrosive liquids in the service environment. The lower concentrations used reduce the toxicity and waste disposal hazards.

7.7.2 *Pretreatment*—Normal methods of removing oil, grease, etc. as given in Section 6 are used prior to treatment.

7.7.3 *Application*—The solution contains of 10 g/L of chromic acid and 7.5 g/L of calcium sulfate and is used at 20 to 30°C for the times indicated in 5.7.4. Add the chemicals in the order given above, and agitate the solution vigorously, mechanically, or by air agitation, for at least 15 min before use.

7.7.4 *Brush-On Application*—Proper application requires that the parts be kept wet with the brush-on solution for a sufficient length of time, usually 1 to 2 min, so that a brown film is produced. Then rinse the parts in cold running water and dry either in an oven or hot air blast. In no case should the parts be rinsed in hot water.

7.7.4.1 Unlike application of the chrome pickle treatment by brush-on techniques, the time between treatment and rinsing is not critical. In fact, if running water is not available, the rinsing step can be eliminated without affecting the coatings.

7.7.4.2 The paint base properties are substantially equal to those of the chrome pickle treatment. The color of the coating may vary from pale brown to almost black, depending on the alloy and treatment conditions. The coating may be applied over previously applied thin films of all the chromate treatments.

7.7.5 *Operating Cautions*—Coatings applied by this method are not adequate for interfaces used in electrical bonding but are satisfactory for use on entrapment areas of magnesium assemblies. Parts processed with this treatment require painting or sealing before exposure to outdoor, overnight atmospheric conditions. The color of the coatings can vary from brassy iridescent to dark brown by altering the treatment time. Up to 1 min of treatment time produces brassy coatings, 2 to 3 min produces a dark brown coating. Prolonged treatment produces powdery coatings. The dark brown coatings are preferred for best paint adhesion.

7.8 *Chromate Treatment—Proprietary Iridite 15:*

7.8.1 *Alloys Containing More Than 1 % Aluminum*—Clean parts as described in Section 6. For alloys containing 3.5 % aluminum or less, such as AZ31, the chromic-nitrate pickle (see 6.5.2) should follow alkaline cleaning. For those alloys having an aluminum content greater that 3.5 %, such as AZ61, AZ81, and AZ91, use the chromic-nitrichydrofluoric acid pickle (see 6.5.6). If mill pickled stock is to be treated, immerse parts in the pickle for 15 to 30 s. If unpickled or cast materials are to be processed, the immersion time should be 2 to 3 min.

7.8.1.1 The use of the bath should be strictly in accordance with the manufacturers instructions.

8. Keywords

8.1 chemical conversion coatings; chromates; cleaning magnesium; preparation for painting; protection; surface sealing

APPENDIXES

(Nonmandatory Information)

X1. SURFACE SEALING OF MAGNESIUM ALLOY COMPONENTS

X1.1 For severe environmental conditions such as near the ocean or in high humidity areas, the use of the surface sealing process greatly improves the resistance of conversion coated magnesium to corrosion and enhances the performance of the entire protection system.

X1.2 *Surface Sealing (see DTD 5562 and DTD 935)*—Surface sealing is a technique developed to produce a thin, flexible, extremely water-impermeable resin coating on all suitably treated magnesium alloy surfaces. This technique should not be confused with “impregnation” techniques used to seal metal porosity. The coating should be applied as soon as possible after the conversion coating is formed.

X1.3 Coating Procedure

X1.3.1 Prebake the components at 200 to 220°C for 30 min or until this temperature has been maintained for at least 10 min.

X1.3.2 Allow to cool to 60°C then spray with, or preferably dip in, surface coating resin to DTD 5562 specification. If dipping is used hold the part in the resin for the first coat until all bubbling.

X1.3.3 Allow to air dry for 15 to 30 min. Ensure uniform drainage and remove any runs or tears that form with a small brush.

X1.3.4 Bake at 200 to 220°C for 10 to 15 min.

X1.3.5 Remove any drips or tears that may have formed with a sharp knife or sand paper taking care not to damage the conversion coating.

X1.3.6 Repeat X1.3.2-X1.3.5 twice more making three coats in all.

X1.3.7 Bake the final coat at 200 to 220°C for 45 min, or if the part is large, until the temperature of the part has been maintained for 45 min.

NOTE X1.1—The complete resin coating is approximately 25 µm thick when applied over chromate chemical conversion coatings. Application onto electrolytic conversion coatings (see Practices D1732) produces significantly less build-up due to the very porous nature of the anodic film.

NOTE X1.2—Prebaking the component is essential in order to ensure that all surface moisture and moisture in surface porosity is boiled off. Dipping is preferred to spraying since it leads to a complete and more uniform resin coverage, and dipping the component while still warm assists the resin to permeate and fill the surface cracking or crazing that is present in the conversion coating.

X2. CONTROL OF TREATMENT PROCESSES

NOTE X2.1—Only A R grade chemicals should be used.

X2.1 Chrome Pickle Treatment

X2.1.1 *Sodium Dichromate Determination*—Determine the sodium dichromate concentration by the following procedure or other recognized analytical procedure:

X2.1.1.1 Pipet a 1-mL sample of the chrome pickle solution into a 250-mL beaker containing 150 mL of distilled/deionized water. Add 5 mL of concentrated hydrochloric acid and 5 g of potassium iodide. Mix well. Allow at least 2 min for the reaction to run to completion. Stir and titrate the free iodine with 0.1 *N* sodium thiosulfate solution until the yellow color is almost gone. Add several drops of starch indicator solution. Continue the titration until the purple indicator color disappears. (**Warning—**Do not add the starch indicator solution until the brown color is almost completely gone or inaccurate results will be obtained. The final color will range from pale green to blue in color.)

X2.1.1.2 *Calculation:*

$$\text{mL } 0.1\ N \text{ sodium thiosulfate} \times 4.9745 = \text{g/L sodium dichromate} \quad (X2.1)$$

X2.2 *Nitric Acid Determination*—Pipet a 1-mL sample 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 0.1 *N* sodium hydroxide to a pH of 4.00 to 4.05.

X2.2.1 *Calculation:*

$$\text{mL } 0.1\ N \text{ sodium hydroxide} \times 4.281 = \text{g/L nitric acid} \quad (X2.2)$$

X2.3 *Control Limits of Chrome Pickle Bath*—Depletion of the solution is indicated by paleness of color, shallowness of etch, and the slowness of action on the metal. Since insufficient exposure to air between removal from the bath and subsequent rinsing can also cause paleness, the two causes should not be confused.

X2.3.1 If the alloys being processed do not contain aluminum, the bath should be regenerated no more than once. For other alloys, it may be generated up to seven times. Regeneration should be carried out when the nitric acid content has been reduced to or near 60 mL/L of nitric acid. The nitric acid content should be reduced each time the bath is regenerated according to the following schedule:

Run Number	Chemical Analysis of Bath	
	Sodium Dichromate, g/L	Nitric Acid, mL/L
1	180	190
2	180	165
3	180	140
4 to 7	180	110

X2.3.2 If the solution is used to produce a protective coating for shipment and storage only, it may be regenerated up to 30 to 40 times, as long as a readily visible protective coating is formed.

X2.4 *Problems and Explanations*—The following operational problems may be encountered:

X2.4.1 Brown, non-adherent, powdery coatings:

X2.4.1.1 The part was in the air too long before rinsing.

X2.4.1.2 The ratio of acid concentration to sodium dichromate may be too high.

X2.4.1.3 Solution may be too hot. This may occur as the result of rapidly processing a large number of parts through a small volume of solution. Cool the solution, reduce the rate of processing, or use a larger volume of solution.

X2.4.1.4 Parts are not properly cleaned. Residual oil on the surface will cause powdery coatings.

X2.4.1.5 The solution has been regenerated too often. Excess nitrate concentrations can cause powdery coatings.

X2.4.2 Grey, nonadherent coatings on castings (**Warning—**This powdery coating may flash violently on impact, abrasion, or friction.):

X2.4.2.1 Use bifluoride modified treatment outlined in 7.1.3.1 instead.

X2.4.2.2 Part may have been excessively treated by being kept in the bath too long.

X2.4.2.3 If parts are to be scrapped, they should be rinsed thoroughly in cold water, dried and dipped in machine or motor oil. (For additional safety, the coating may be removed as described below before coating with oil.) If dimensional changes are not too severe for salvage, the coating may be removed by immersing the parts in 10 to 20 volume % hydrofluoric acid solution for 5 to 10 min.

X2.5 Dichromate Treatment

X2.5.1 *Hydrofluoric Acid Determination*—The hydrofluoric acid is depleted very slowly in use. It should not be allowed to fall below 10 % HF as determined by titration for free acid with 1 *N* NaOH and phenolphthalein indicator. A solution weaker than 10 % will attack magnesium severely. A titer of 10 to 20 mL of 1 *N* NaOH for a 2-mL sample roughly corresponds to 10 to 20 % by weight of HF. Pipet a 2-mL sample into approximately 100 mL of distilled water and titrate immediately. Use plastic pipet and beakers to prevent inaccurate results because of reaction of the HF with glass.

X2.5.2 *Bifluoride Determination*—If the acid fluoride treatment is used instead of the hydrofluoric acid treatment, control the solution by titration with sodium hydroxide. Maintain the acid fluoride content such that a 100-mL sample of the bath requires 45 to 55 mL of 1 *N* NaOH to produce a pink color using phenolphthalein indicator.

X2.5.3 *Control of Dichromate Bath*—The concentration of sodium dichromate in the bath can be determined by the method outlined in X2.1. Maintain the dichromate concentration between 120 and 180 g/L. Maintain the pH of the solution between 4.1 to 5.5 by the addition of chromic acid. Make the pH adjustment before the dichromate analysis is done.

X2.5.3.1 When treating AZ31B alloy, the pH range must be narrowed to 4.1 to 5.2. Dissolve the chromic acid in warm water to form a 10 % solution. This solution can then be used in the pH adjustment described below for making additions to the tank.

X2.5.3.2 *To Make the pH Adjustment*—Standardize a pH meter equipped with a glass electrode using a buffer solution of approximately 4.0. Using a convenient size measured sample, adjust the pH of the sample to 4.1 by adding measured amount of the 10 % chromic acid solution described above:

$$(\text{mLs } 10\,\%\ \text{CrO}_3\ \text{sol/sample size}) \times \text{tank size} \qquad (\text{X2.3})$$

$$= \text{amount of } 10\,\%\ \text{CrO}_3 \text{ solution to be added to tank}$$

X2.5.3.3 Process alloys low in aluminum on the low side of the pH range to obtain a good coating.

X2.5.3.4 *Problems and Explanations*—The following problems might be encountered when applying the dichromate treatment:

X2.5.4 Abnormally heavy and loose powdery coatings:

X2.5.4.1 The hydrofluoric acid or bifluoride bath is too dilute.

X2.5.4.2 The pH of the dichromate bath is too low. It should not be allowed to fall below 4.1. The pH may be adjusted upward by the addition of 10 % sodium hydroxide solution. The method outlined in X2.3 may be used by substituting sodium hydroxide solution for chromic acid solution.

X2.5.4.3 Treatment of oxidized, corroded, or flux contaminated parts will result in the formation of gray to yellow coatings which are only loosely adherent. Clean parts as outlined in 6.1.

X2.5.4.4 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.

X2.5.4.5 Prolonged treatment in the dichromate bath.

X2.5.5 Failure to coat or nonuniform coatings:

X2.5.5.1 The pH of the dichromate bath is too high. This is very important when hydrofluoric acid is used as the pre-dip on low aluminum containing alloys such as AZ31B. Adjust the pH as described in X2.3. Frequent checks may be necessary depending on tank size and processing volume.

X2.5.5.2 The dichromate concentration in the bath is too low.

X2.5.5.3 Oily matter has not been completely removed resulting in a spotty coating where some areas are covered and others not. Insufficient cleaning alone may not be the fault. Properly cleaned parts may be recontaminated by an oily film floating on the fluoride or dichromate bath. This oily film could be caused by insufficient rinsing after alkaline cleaning, oil mist in the air, or oil dropping from overhead equipment or other causes.

X2.5.5.4 Previously applied chrome pickle may not have been completely removed.

X2.5.5.5 The part was fluoride treated.

X2.5.5.6 Alloy is one not suited for this type of treatment.

X2.5.5.7 Too long a hydrofluoric acid 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 s to 1 min.

X2.5.5.8 The bath was not kept boiling during the treatment period. This is particularly important when processing AZ31B alloy. Minimum bath temperature is 93°C.

X2.5.5.9 Improper rinsing after the hydrofluoric acid or soluble fluoride carried over into the bath exceeds 0.2 %. Streaked coatings will be observed before this limit is reached. A 0.2 % solution of calcium chromate can be added to the bath to precipitate the excess fluoride as calcium fluoride. If the fluoride is reduced in this way, the bath need not be discarded.

X2.6 Chromate Treatment

X2.6.1 *Chromate Salts*—Control the concentration of the chromate salts as specified by the supplier. Alternatively, they may be controlled by the procedure outlined in X2.1.1. If this procedure is used however, a carefully prepared 37.5 g/L solution of the salts must first be titrated to determine the sodium dichromate content of the salts. The control point can then be corrected to this value.

X2.6.2 *pH*—Maintain the pH of the solution between 0.2 to 0.6 for the solution described in 7.3.1 and 0.6 to 1.0 for the solution described in 7.3.2.

X2.6.3 *Wetting Agent*—When the bath is in continuous operation, control the wetting agent content by adding 0.3 mL of agent for each 37.5 g of salts added to the bath. When the bath has been allowed to stand idle for a period of a week or more, and a thin foam layer does not form during processing, make a full addition of 0.3 mL/L of wetting agent.

X2.6.4 *Bath Life*—The bath may be replenished until satisfactory coatings are no longer obtained. Although the expected bath life will depend on the amount of dragout, the solution should not be dumped until the replenishment additions are at least equal to one and one-half times the original makeup. This is approximately equal to processing 4.2 m^2 of surface per litre of solution.

X2.6.4.1 *Operational Problems*—The following operational problems may be encountered during use of the treatment:

X2.6.5 Failure to form coatings:

X2.6.5.1 The pH of the solution is too high.

X2.6.5.2 Solution may be too cold.

X2.6.5.3 The metal is not properly cleaned. The part was not pickled before treatment in the chromate solution.

X2.6.5.4 The ratio of the acid concentration to the chromate may be too low by using the improper amount or strength of acid.

X2.6.6 Non-adherent powdery coatings:

X2.6.6.1 The part may be made of an alloy that requires a different treating solution, for example, solution specified as in 7.3.1 was used when the solution specified in 7.3.2 is required.

X2.6.6.2 The pH of the solution is too low.

X2.6.6.3 The part was not properly cleaned. The part was not pickled before treatment in the chromate solution.

X2.6.6.4 The ratio of the acid concentration to the chromate salts may be too high.

X2.6.7 Excessive smut on parts:

X2.6.7.1 Aluminum bearing alloys will develop aluminum smut if held in the treating solution for too long a period.

X2.7 Chrome-Manganese Treatment

X2.7.1 *Control*—The bath composition given provides for a long working life and yet is not so concentrated as to be expensive initially or wasteful in operation from "drag out" losses. Approximately 2.5 m^2 of surface can be treated per litre of solution before exhaustion. The pH of the solution varies from about 4 when freshly made to about 6 when nearing exhaustion. Within these limits no control of pH is necessary. Chromating times and the appearance of the films provide an adequate control mechanism. During use a small amount of black manganese dioxide sludge is produced; it should not be discarded. An exhausted bath can be revivified by the careful, controlled addition of sulphuric acid to obtain a pH of approximately 4.1 to 4.2. Alternatively the bath can be revivified by the addition of up to 5 % manganese sulphate. Sodium dichromate and magnesium sulphate deplete very slowly and further additions are rarely required except to replace "drag out" losses.

X2.7.2 *Operating Problems*—The bath may be contained in plastic or glass vessels, but since there is no free acid in the solution it is common to use tanks of aluminum, zinc, mild steel, or galvanized steel as desired. If metal tanks are used, it is important that the magnesium parts are not allowed to rest or otherwise be in contact with the tank. Failure to isolate the workpiece will result in the formation of thicker, powdery chromate films and a more rapid depletion of bath chemicals. The bath may however be intentionally used in a "galvanic" mode to produce a dense matte black surface for optical equipment parts, but slight dimensional losses may occur.

X2.8 Galvanic Chromate Treatment

X2.8.1 *Control*—The analytical procedures for hydrofluoric acid or acid fluoride determination are the same as those used for the dichromate treatment given in X2.1 and X2.2. The sulphate-dichromate-hydroxide bath pH should be maintained between 5.6 and 6.0 with careful additions of a solution containing 5 % by weight each of chromic acid and concentrated sulfuric acid.

X2.8.2 *Operational Problems*—The galvanic chromate treatment requires careful control during application. Treatment time, bath condition, and alloy composition affect the color of the coating. Gray, nonuniform coatings indicate unsatisfactory precleaning or baths that are depleted. A good, firm electrical contact must be made with the workpiece and care must be taken to ensure there is no contact with the tank other than by the external circuit. Racks made from monel, stainless steel, or phosphor bronze may be used to treat multiple parts. Parts must be firmly racked for the proper galvanic action to take place. If the tank is made of nonmetallic material, steel, nickel, or phosphor bronze cathode plates must be used. Nonadherent coatings are usually caused by too high a current density, too prolonged a treatment, or too low a bath pH. Poor coatings also result from the use of current densities below recommended levels.

X2.9 Semi-Bright Pickle Treatment

X2.9.1 A reduction in chemical reaction indicates depletion of the treating solution. The pH could be 1.7 or higher and small batches of the solution should be disposed of appropriately. Larger volumes of the solution can be revivified up to twelve times by one of the following methods:

X2.9.1.1 Add fresh stock solution to replace drag-out and evaporation loss if the bath is used continuously.

X2.9.1.2 Maintain original volume if the bath is used infrequently and losses are due mainly to evaporation by adding 25 % of the original amount of chemicals and sufficient water to bring the volume back to its original level.

X2.9.1.3 Analyze the solution and add chemicals as necessary to adjust the chemistry to the original bath composition.

X2.9.2 As noted under 7.5.2 lower operating temperatures reduce staining in air during transfer of the parts pickle to rinse, but longer pickling times result in maximum brightness. Short treatment times are best for polished surfaces.

X2.9.3 Brassy to dark brown stains can be caused by inadequate rinsing or by extended transfer times between pickling and cold water rinse. Should dark brown stains develop proceed as in 7.5.3.

X2.10 Phosphate Treatment

X2.10.1 The bath composition should be controlled to maintain to following ranges:

X2.10.1.1 Ammonium phosphate 105 to 120 g/L, ammonium sulfite 15 to 30 g/L, pH 5.9 to 6.1.

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