

PHOSPHATE COATING PROCEDURE PER MIL-DTL-16232

1. Scope: This procedure covers the application of zinc/manganese phosphate coatings on ferrous alloys, meeting the requirements of MIL-DTL-16232.

a. A record will be kept of the history of each processing bath showing all additions of chemicals and results of the analysis performed for no less than one year.

b. Records of bath controls shall be maintained and made available to the DCMAO representative or command upon request.

2. Material Used:

a. *Precleaning: (options)*

(1a) Degreasing solvent: Fast Solvent blend A2360, mfr. Barton Solvents

(1b) Degreasing solvent: Metal Prep 5-gallon pails 3-674, 55-gallon drums 3-677, Klinger

(2) Alkaline cleaner: Uni Kleen 88, Heatbath Corporation

b. *Abrasive blast media:*

100 grit & 180 grit aluminum oxide, 50-140 grit Staurolite (mixed media), silicone carbide, #8-#13 glass bead and SAE-J444 steel grit all purchased from Spesco. Crystalline silica is not used.

c. *Phosphate chemicals:*

(1) Zinc phosphate: Parkerizing 210 purchased from Henkel Surface Technologies

(2) Manganese phosphate:

2.1 Parco-Lubrite 2 purchased from Henkel Surface Technologies

2.2 Key Kote 40 purchased from MacDermid

d. *Final rinse:*

(1) Chromic acid: Chromium trioxide purchased as chromic acid flake from manufacturer Elementis Chromium LP. Bath concentration: 0.3 grams per liter (0.03%). Phosphoric acid added as needed to extend control of TA/FA ratio. Phosphoric acid and chromic acid flake purchased through distributor Interstate Chemical.

e. *Supplemental treatment from the Qualified Products List (QPL):*

(1) Oil preservatives (All Tectyl products are manufactured by Daubert Chemical):

a. MIL-C-16173 Grade III, Tectyl 894, QPL 16173-94-1

b. MIL-PRF-32033 (formerly known as VV-L-800) Tectyl 900, QPL N/A

c. MIL-PRF-3150, Tectyl 802A, QPL L-6568

(2) Dry film lubricant (all dry film lube products are purchased from Sandstrom Products):

a. MIL-L-46010D type III, water base product 099 (400F cure) MIL-PRF-46010F

b. MIL-PRF-46147 air-dry, form I (bulk) product 27A

c. MIL-PRF-46147 air dry, form II (aerosol) product 27A

f. *Paint from the Qualified Products List (QPL). See also "k" below.*

(1) CARC systems purchased from Niles Chemical Paint (now known as NCP).

a. PRIMER: MIL-P-53022 1.0-1.5 mils thick, NCP QPL Q475, product N1088.

b. TOPCOAT: MIL-DTL-53039 OR MIL-DTL-64149 applied 1.8 to 2.5 mils thick, color as specified [Example: color 383 (color 34094 per federal standard 595), QPL Q1684, product N-8045]

3. Process Sequence:

a. Inspect incoming parts for hardness (Rockwell C) and alloy type in addition to inspection for defects and damage. Dimensions and surface finish apply before phosphating unless otherwise specified. Phosphating will be applied after all machining, welding, forming, heat treatment, etc. have been completed. If production parts are not capable of being tested due to physical size restraints, sections of parts will be utilized.

b. Unless otherwise specified, parts (including carburized parts) which are Rockwell C 39 or harder, or parts of any hardness which are ground, cold formed or cold straightened, shall be given a stress relief heat treatment 350-400 degrees F for a minimum of one hour for every inch of thickness, but not less than one-half hour for a thickness less than one-half inch. Optional heat treatment for carburized parts, 107 to 135 degrees C (225 to 275 degrees F) for 8 hours.

- c. (1) Degrease using solvent identified in 2a(1) or;
(2) Hot alkaline clean using material identified in 2a(2), rinse and dry.
(a) *Normal Concentration:* 2 to 6 ounces/gallon
(b) *Normal Temperature:* 180 to 210 degrees Fahrenheit
(c) *Normal Immersion time:* 10-55 minutes

d. Mask or plug appropriate areas if required.

e. Abrasive blast using material identified in 2b. Any abrasive residue will be removed with a blast of clean, dry air. Inspect to ensure a uniform blast.

f. Zinc/manganese phosphate coat in the solution identified in 2c under the following conditions:

(1) **Concentration (control ranges):**

- (a) Zinc: TA: 5-12 mL, 2mL sample, 0.1 N NaOH, phenolphthalein end point
FA: 0.8-1.2 mL, 2-mL sample, 0.1 NaOH, brom cresol green end point
Fe: 2-5 mL, 10 mL sample, 0.18 N KMNO₄

(b) Manganese:

- TA: 11-24 mL, 2mL sample, 0.1 N NaOH, phenolphthalein end point
FA: 1.5-2.2 mL, 2-mL sample, 0.1 N NaOH, brom cresol green end point
Fe: 2-5 mL, 10-mL sample, 0.18 N KMNO₄

(2) **Frequency of above tests:** Prior to starting production and every 2 to 4 hours.

(3) **Temperature:** 200+/-10 degrees F.

(4) **Immersion time:** Zinc 25-30 minutes; Manganese 20-45 minutes.

Equipment is constructed of materials resistant to the action of the phosphating solution and shall be free of copper alloy fittings and brazing materials. Fog sprays are provided and used as necessary to prevent the phosphating solution from drying on the parts prior to water rinsing.

g. Rinse in clear cold running water for a minimum of at least one minute. Water flow will be regulated to prevent contamination, i.e., 3.0 ml of 0.1 N NaOH per 100 ml sample of rinse water.

h. Chromic acid rinse using the material identified in 2d. The chromic acid rinse will be applied at 150-200 degrees F on all parts except when Class IV coatings are specified. This rinse will be maintained at a pH of 2 to 4 by the addition of flake chromic acid or chromic/phosphoric acid. The rinse will be analyzed prior to starting production and at least every 8 hours thereafter by a standard free and total acid titration along with a pH reading. The rinse will be discarded when the free acid to total acid ratio exceeds a value of 7. The parts will remain in the rinse for a minimum of one minute and be removed. Following the chromic acid rinse, the parts are thoroughly dried before application of supplementary treatment. A blast of dry air may be used to facilitate drying.

i. Parts Rockwell C 39 or harder will be given the specified supplementary treatment and allowed to age at room temperature for 120 hours to relieve any embrittlement due to uptake of hydrogen. If necessary, parts will be baked at 220+/-5 degrees F for eight hours to relieve any embrittlement due to hydrogen uptake and then will be given the specified supplementary treatment.

j. Parts/specimens required for testing or examination will be removed at this time. See applicable testing procedures.

k. Parts will be painted: See "f" above.

4. Testing Procedures:

- a. Workmanship shall comply with paragraph 3.10 on all production.
- b. Salt spray tests will be performed per ASTM-B117 on coatings prior to application of supplementary treatments. Parts shall show no signs of corrosion visible to the unaided eye after exposure times of 1.5 hours for manganese and 2.0 hours for zinc phosphate coatings.
- c. Coating weight tests will be performed per paragraph 4.7.3.2 for zinc coatings and recorded. A minimum of 11 grams/meter squared is required. Coating weight tests will be performed per paragraph 4.7.3.1 for manganese coatings and recorded. A minimum of 16 grams/meter squared is required.
- d. Adequacy of the hydrogen embrittlement relief treatment will be demonstrated on a 120 day interval by testing per paragraph 4.3.5 using *ASTM-F519, Type 1a* specimens of AISI 4340 steel and 51-54 Rockwell C hardness. Three uncoated, notched tensile specimens will be pulled to determine the ultimate notched tensile strength. Specimens subjected to the phosphate coating procedure and given the embrittlement relief treatment will be subjected to a load of 75 percent of the ultimate notched tensile strength for 200 hours. Specimens will be examined for cracks after the test.
- e. Testing for salt spray and coating weight will be accomplished at least per lot, once every eight (8) hours or less.
- f. Parts requiring a specific thickness of paint or dry film lubricant will be measured using an approved thickness tester after the paint or the dry film lubricant has been sufficiently dried or baked. Film thickness of the paint or the dry film lubricant will include any thickness that may result from the phosphate coating.
- g. Salt spray testing of the phosphate with primer per ASTM B117 shall be performed for 336-hours. The frequency for salt spray testing is "per lot or every two weeks." Parts shall show no rusting visible to the unaided eye, no more than 5-blisters with non larger than 3/64 inches in diameter. Scribe attack shall not exceed 1/8 inch. Salt spray testing of the phosphate with dry film lubricant per ASTM B117 shall be performed for 100 hours. The frequency for salt spray testing is "per lot or weekly." Parts shall show no rusting visible to the unaided eye, no more than 3 blisters with none larger than 1.0 mm in diameter.
- h. Painted parts shall be checked daily for thickness and for adhesion per ASTM-3359, method B. Parts with dry film lubricant shall be checked daily for thickness and for adhesion per ASTM D2510, procedure A.
- i. When applicable, provisions of QAP paragraphs 502, 503 and 504 of "General Requirements for Quality Assurance Provisions (Small Caliber Weapon Systems)", drawing number 12993884, will be performed and reported.

Date: October 3, 2013

Company Name: *Triple E Manufacturing*

Signature _____

Dennis M. Johnson, President