INCH-POUND

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FEDERAL SPECIFICATION

CHEMICAL CONVERSION COATINGS AND PRETREATMENTS FOR FERROUS SURFACES (BASE FOR ORGANIC COATINGS)

This specification was approved by the Assistant Administrator Office of Federal Supply and Services, General Services Administration, for the use by all Federal Agencies.

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers cleaning, surface conditioning for crystal refinement and pretreatment by chemical conversion of ferrous metals and zinc/zinc alloy coated metals. The application of chemical conversion and pretreatment coatings provides uniformly textured substrates for receiving and retaining paint, lacquer, etc. In addition, this specification covers suitable cleaning processes for nonferrous surfaces (see 6.1, 6.1.2).

1.2 Classification. This specification covers the following cleaning methods and surface preparation processes:

1.2.1 Surface cleaning. Surface cleaning should be by any of the following methods as specified (see 6.4):

Method I	Mechanical or abrasive cleaning.
Method II	Solvent cleaning by immersion, spray or vapor.
Method III	Hot alkaline cleaning by immersion, spray or electrolytic methods.
Method IV	Emulsion with or without added water.
Method V	Alkaline derusting.
Method VI	Phosphoric acid (alcohol, detergent or solvent type with detergent).

1.2.1.1 Ozone depleting chemicals. The cleaning material should not contain any chemical, which is classified in the Clean Air Act Amendments of 1990 as a Class I or Class II ozone depleting substance.

1.2.2 Conversion coatings and pretreatments. Chemical conversion and pretreatment coatings should be of the following types as specified (see 6.4).

Type I Zinc phosphate spray application (150 mg/ft^2 min - 500 mg/ft^2 max)

Zinc phosphate immersion or dip application (300 mg/ft^2 min - 500 mg/ft^2 max)

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Director, U.S. Army Research Laboratory, Weapons and Materials Research Directorate, ATTN: AMSRL-WM-M, Aberdeen Proving Ground, MD 21005-5069 by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

Type II Aqueous iron phosphate (35 mg/ft^2 min)

Type III Organic pretreatment coating (unless otherwise specified, MIL-C-8514 or DOD-P-15328)

Type IV Discontinued

Type V Zinc phosphate (500 mg/ft^2 min - 1100 mg/ft^2 max)

1.2.2.1 Method of application. New design documents, engineering drawings and ordering data should indicate the type of coating required and method of application when applicable.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1..Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation.

SPECIFICATIONS

MILITARY

MIL-C-8514 Coating Compound, Metal Pretreatment, Resin-Acid
DOD-P-15328 Primer, Pretreatment (Formula 117 for Metals)

STANDARDS

FEDERAL

FED-STD-313 – Material Safety Data Sheets, Preparation and Submission of

MILITARY

MIL-STD-171 - Finishing of Metal and Wood Surfaces

(Activities outside the Federal Government may obtain copies of Federal specifications, standards, and handbooks as outlined under General Information in the Index. The Index, which includes cumulative monthly supplements, as issued, is for sale on a subscription basis by the Superintendent of Documents, US Government Printing Office, Washington, DC 20402.

Single copies of this specification and other product specifications required by activities outside the Federal Government for bidding purposes are available without charge from General Services Administration Business Office Centers in Boston; New York; Philadelphia; Washington, DC; Atlanta; Chicago; Kansas City, MO; Ft. Worth; Houston; Denver; San Francisco; Los Angeles; and Seattle, WA.

Unless otherwise indicated, copies of federal and military specifications, standards, and handbooks are available from the Standardization Documents Order Desk, Bldg. 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.)

2.1.2 Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the solicitation.

ENVIRONMENTAL PROTECTION AGENCY (EPA)

CODE OF FEDERAL REGULATIONS
29 CFR 1910.1200: Toxic Chemical Release Reporting

(Application for copies should be addressed to the superintendent of documents, Government Printing Office, Washington, DC 20402.)

2.2 Non-Government Publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the DoD adopted documents are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM A 109	Steel, Strip, Carbon (0.25 Maximum Percent) Cold-Rolled
ASTM A 366	Commercial Steel (CS) Sheet, Carbon, (0.15 Maximum Percent) Cold-Rolled
ASTM B 117	Salt Spray (Fog) Testing
ASTM D 610	Evaluating Degree of Rusting and Painted Steel Surfaces
ASTM D 1654	Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments
ASTM D 3359	Measuring Adhesion by Tape Test
ASTM D 5486	Pressure-Sensitive Tape for Packaging, Box Closure, and Sealing
ASTM F 519	Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103-1187.)

GENERAL MOTORS ENGINEERING STANDARDS

GM 9540P-97 Accelerated Corrosion Test

(Non-Government standards and other publications are normally available from the organizations that prepare or distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.3 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Material. All material used shall be as specified herein, on the drawings, or in the contract.

3.2 Preproduction approval (types I and V).

3.2.1 Procedure approval (type 1 and V). Prior to production, a detailed written procedure identifying cleaning, pretreatment and painting processes shall be submitted to the procuring agency for approval. Process time, temperature, chemical concentrations, process controls and acceptance criteria and any other pertinent details shall be listed for each step of the application process. The procedure shall include the manufacturer and exact proprietary designation of any material used as well as any equipment used in the application of the procedure. The complete procedure which demonstrates ability to pass the performance

tests contained herein is to be signed by a company official prior to its submittal for approval. Deviation from the approved procedure is not permitted without written approval from the procuring agency.

3.2.2 Preproduction validation panels. The contractor shall coat twelve (4 X 6 inch) panels (see 4.2.2) by the proposed zinc phosphate coating procedure outlined for use in the contract. The test panels shall be the same substrate as will be used in production. Standard test coupons may be used for processing low carbon steels only. Six of the coupons shall be coated with the primer (or paint for single coat systems) to be used in production. The primer dry film thickness shall be no greater than 1.5 mils unless otherwise specified in the contract. Three of the nonprimed coupons shall be checked for coating weight (see 4.2.6.1), and the resultant coating weight test analysis report shall be provided to the procuring agency. Three of the primed coupons shall be performance tested by the contractor in neutral salt spray (see 4.2.8) unless superceded by an accelerated corrosion test specified in the contract. See 4.2.8.1 for zinc or zinc alloy coated surfaces. The remaining six panels (three phosphated only; three phosphate plus primer) shall be forwarded to the procuring activity for evaluation and testing.

3.2.3 Stress relief. Unless otherwise specified in contract, parts cold worked after heat treatment and parts having a hardness of Rockwell C40 or greater shall be stress relieved prior to phosphating.

3.3 Cleaning methods.

3.3.1 Removal of soils, corrosion and weld by-products. The parts shall be thoroughly free of oil, grease, wax, dirt, scale, and other foreign matter and shall not show visible signs of red or white corrosion products. The water break test specified in 4.2.3.1 shall be used to check for freedom from organic contaminants. Inorganic contamination may still be present even if the water break test is acceptable. All weld slag/flux residues shall be removed. Weld spatter shall be removed from all surfaces exposed to normal view in the end item configuration. If the weld spatter has been overcoated with a zinc or zinc alloy deposit, removal of the spatter is not required unless there are cosmetic concerns. The method or combination of methods used shall be selected to suit the properties of the metal, the type of soil and the degree of contamination present. Method VI shall not be used on steels with hardness greater than Rockwell C40 unless test data can substantiate that the combined phosphoric acid cleaning and phosphate-coating treatment is not too severe for the steels being treated or that an adequate hydrogen embrittlement relief bake has been performed. Acid pickling shall not be used unless specifically approved or authorized by the drawing or specification for the item being processed.

3.3.2 Rinsing. Adequate rinsing shall be performed following any chemical process to remove residual material remaining from the cleaning operation. Special care shall be exercised in rinsing complex shapes to prevent contamination of any subsequent cleaning or coating process. The water rinse, after the cleaning operation, shall be checked for contamination as measured by total alkali or total acid. The rinse shall be regulated so that the total alkali contamination does not exceed 0.5 ml plus any total alkalinity correction factor in the water, or the total acid contamination does not exceed 0.5 ml (see 4.2.4). With the approval of the contracting office, conductivity devices may be used to automatically regulate the flow of rinse water according to rate of production.

3.3.3 Drying. Care shall be exercised to ensure complete drying in crevices, seams, or other difficult to dry places prior to painting.

3.4 Processing requirements (types I, II, and V).

3.4.1 Cleaning. Cleaning shall be in accordance with 3.3.1.

3.4.2 Rinse after cleaning. Rinsing after cleaning shall be in accordance with 3.3.2.

3.4.3 Chemical conversion coating.

3.4.3.1 Type 1 and type V. The properly cleaned articles shall be subjected to a balanced aqueous solution containing phosphoric acid, zinc, and accelerating agents until a uniform, insoluble, phosphate coating is produced. Dated records should be maintained for the chemical analyses and additions made to the solutions.

3.4.3.1.1 For type I only. Unless otherwise specified, type I zinc phosphate processes shall incorporate a crystal modifier or grain refining accelerator in the phosphating solution, a separate grain refiner as a pretreatment or a grain refiner addition to the cleaner.

3.4.3.2 Type II. The properly cleaned articles shall be subjected to a balanced aqueous solution containing phosphoric acid or acid phosphate salts with or without the addition of accelerating agents until a uniform insoluble phosphate coating is produced.

3.4.4 Rinse after phosphating (types 1, II, and V). Phosphating shall be followed by a clean water rinse, less than 100ºF or 38ºC, in order to remove residual salts and unreacted phosphating material.

3.4.5 Final rinse (types I, II, and V). Proprietary final acid/alkaline rinses are available and permissible for use over phosphate coatings provided they are specifically approved by the procuring agency. The materials, temperature, concentrations, and other process controls (including replenishment and discard criteria) shall be stated in the procedure. Phosphatized coatings must meet the requirements specified for paint adhesion and salt spray resistance or must meet those requirements specified for the designated coating system (see 6.8).

3.5 General requirements.

3.5.1 Appearance (type 1, II, and V). All coatings shall be continuous, uniform in texture, and evenly distributed.

3.5.1.1 Type I, zinc phosphate. Type I coating deposits shall be gray to black in color. The coating shall be uniformly colored and be free of smut, powder, corrosion products, or white stains. Non-uniformity of color due to heat treatment, composition of the base metal, the degree of cold work performed on the base metal or presence of brown or orange stains inherent from the acidified final rinsing process shall not be cause for rejection.

3.5.2 Lighting conditions. A minimum light intensity of 50 lumens per square ft or 538 lux shall be provided at the working surface where manually controlled paint finish operations are performed. Minimum lighting applies to the manufacturing operations as well as in-process and final inspection areas (see 4.2.1.1).

3.5.3 Phosphate coating weight (types I, II, and V). Coating weight shall be controlled or tested as in paragraphs 4.2.6.1.1, 4.2.6.1.2, and 4.2.6.1.3. Coating weights as defined below are applicable.

3.5.3.1 Type I zinc phosphate coatings. Type I coatings may be applied by spray, dip or immersion and permit a minimum coating weight of 150 mg/ft^2 for spray applications and 300 mg/ft^2 for immersion applications. A maximum coating weight of 500 mg/ft^2 is specified for all application methods.

3.5.3.2 Type II aqueous iron phosphate coating. Type II has a minimum coating weight of 35 mg/ft^2.

3.5.3.3 Type V zinc phosphate coatings. Type V zinc phosphate requires a coating weight between 500 - 1100 mg/ft^2 inclusively.

3.5.4 Film thickness (type III only). The dry film thickness shall be 0.0003 to 0.0005 inch when tested as in 4.2.7.1.

3.5.5 Application of organic coating. The organic coating shall be applied to thoroughly dried surfaces within 24 hours after pretreatment. The dried surface shall not show any rusting or soiling prior to painting. The temperature of the metal surface shall be controlled to eliminate blistering, poor adhesion or unsightly film results.

3.5.6 Paint thickness. For all tests requiring painted test specimens, the dry film thickness (see 4.2.7) on all surfaces shall be as specified for the end item. When the paint thickness is not covered in the end item specification, the drawing, or paint specification requirement, the precedence shall be in the order cited in the contract.

3.5.7 Paint adhesion (all methods and types). The CARC or non-CARC (e.g., alkyd, enamels, lacquers, etc.) painted items or specimens shall show the following satisfactory paint adhesion when tested as in 4.2.7.2 and 4.2.7.3.

3.5.7.1 CARC painted items. An ASTM D3359 rating lower than 3B or the removal of two or more complete squares of topcoat or top coat-primer-pretreatment coating from any test unit constitutes failure of this test (see 4.2.7.2).

3.5.7.2 Non-CARC painted items. Unsatisfactory adhesion shall be indicated by exposure of bare metal or underlying phosphate pretreatment by any of the following conditions:

(a) Any area exceeding 1/8 inch average diameter.
(b) More than one area exceeding 1/16 inch average diameter.
(c) More than five areas less than 1/16 inch diameter.

3.5.8. Accelerated corrosion resistance (all types). After pretreatment and primer (or paint for single coat systems) application, the specimens subjected to the salt fog test as in 4.2.8 for the number of hours prescribed shall show no more than 1/8 inch creep, blistering, or loss of adhesion of the paint from the scribe mark. There shall be no more than a trace of film failure (rust grade no. 9, ASTM D610), and not more than 5 scattered blisters, none larger than 3/64 inch in diameter on a 4 by 6 inch test panel or equivalent area of test specimen or item. For items or specimens having an area less than 24 square inches, a proportionately smaller number of failed areas will be permitted. Neutral salt spray testing shall not be used to qualify zinc or zinc alloy coated substrates (see 4.2.8.2 and 6.1.3).

3.5.9 Relief of hydrogen embrittlement. After cleaning by method VI or phosphating, parts having a hardness of Rockwell C40 or greater shall be heat treated for 8 hours at 210-225ºF (99-107ºC) or must be held for 240 hours at room temperature, prior to release, to relieve any embrittlement due to hydrogen.

3.5.10 Hydrogen embrittlement relief test. Adequate verification tests must be performed for all parts having a hardness of Rockwell C40 or greater. Treated parts shall not reveal any developing cracks upon subsequent inspection (see 4.2.9).

3.5.11 Toxicity. The solvents used in methods II – VI shall have no adverse effects on human health when used as intended (see 6.1). Material safety Data sheets (MSDS) must be prepared, submitted and made available in accordance with FED-STD-313; additionally, they must conform to 29 CFR 1910.1200 (see 6.3).

4. QUALITY ASSURANCE PROVISIONS

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

4.1.1 Responsibility for compliance. All items shall meet all requirements of section 3. The inspections set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of ensuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling inspection, as part of manufacturing operations, is an acceptable practice to ascertain conformance to requirements, however, this does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to accept defective material.

4.2 Test methods.

4.2.1 Tests. Tests shall be conducted as required in this specification. The right is reserved to make any additional tests deemed necessary to determine that the process meets the requirements of this specification.

4.2.1.1 Lighting adequacy. Should adequate lighting appear suspect at the work/inspection surface, a light intensity meter shall be used to assure that 50 lumens per square ft or 538 lux minimum is provided.

4.2.2 Test specimens. Test specimens shall be prepared from actual production items or parts thereof, or if size is prohibitive, from scrap parts of the same kind and finish (from the same manufacturing lot if possible) which have been rejected for causes other than phosphating, material composition, heat treatment or any combination thereof. Specimens need not be identical in shape or size but shall be stamped, etched, or otherwise indelibly marked for identification as a test specimen. When used, standard panels shall be not less than 4 x 6 x 1/32 inch in size. Standard panels (e.g. SAE 1010) may be used when authorized by the contracting officer and shall conform to ASTM A109 or ASTM A366 having a Rockwell B hardness of 55 to 75, and a surface roughness of 30 to 45 microinches (arithmetic average) as rolled. For zinc coated surfaces, the panels shall be made of the same coated material as specified in the contract. When coated steels are used, the standard test panels shall be made of the same material as that specified in the contract. All test specimens of standard panels shall be processed through all the cleaning, phosphating, painting, and drying steps along with the items being processed. Test specimens shall not be reused.

4.2.3 Removal of soils, corrosion, and weld by-products. Care should be taken when identifying the type of soil that is to be removed, as cleaning materials which may be effective against one type of contaminant may be ineffective against another. Multiple cleaning procedures may be required to provide the required water break free surface. After cleaning and rinsing, and prior to phosphating, at least two test specimens shall be subjected to a water break test at the conclusion of a maximum of every four hours of production. These specimens shall be dried and examined visually for rust, corrosion products, and soils. If specimens fail the water break test or if the surface shows signs of soils or corrosion products, all items processed since last acceptance shall be rejected and corrective action taken. After corrective action, testing shall be continued at least once every hour until indications of soils or corrosion products are eliminated. Testing frequency shall then revert to at least two test specimens at the conclusion of a maximum of every four hours of production.

4.2.3.1 Water break test. The water break test shall be conducted by subjecting the surface to a mist of distilled water by means of a convenient small atomizing device. If the water droplets tend to coalesce into large lenses lasting for 25 seconds (without a sudden flashout), the surface shall be considered as having satisfactorily passed the water break test. If the water gathers into droplets within 25 seconds (if the surface shows a "water break" within that time), the surface shall be considered as having failed the test. If the water forms a continuous film by flashing out suddenly over a large area, this shall be considered as evidence of the presence of an impurity on the surface such as free alkali, residual detergent, etc., and the surface shall be considered as having failed the test. Failure to support an unbroken water film shall be sufficient cause to do additional cleaning. If more than four hours have passed since performing the water break test, re-examine the surface for corrosion, foreign matter or oily residues and repeat the water break test prior to pretreatment. Cleaning materials which may be effective against one type of contaminant may be ineffective against others. Multiple cleaning procedures may be required to provide the required water break free surface.

4.2.4 Rinsing. The water rinses after the cleaning and phosphate operation shall be tested for contamination after every four hours of production (see 3.3.2).

4.2.4.1 Total alkali contamination test. Take a 10 ml sample of the rinse solution, dilute to 50 ml with distilled water and add 5 drops of Bromcresol Green Indicator. Slowly add testing solution (0.1N HCl) from a burette until the color of sample changes from blue-green to pale yellow. This is the endpoint and the number of ml of acid testing solution used is the total alkali contamination.

4.2.4.2 Total acid contamination test. Take a 10 ml sample of the rinse solution, dilute to 50 ml with distilled water and add 5 drops of phenolphthalein indicator. Slowly add alkaline testing solution (0.1N NaOH) from burette until the first permanent pink color is produced. This is the endpoint and the number of ml of alkaline testing solution used is the total acid contamination.

4.2.5 Final rinse. The final rinse shall be tested, replenished and discarded in accordance with procedures designated by the suppliers of the final rinse chemicals; these quality assurance provisions shall be documented in the approved procedure. Solution analysis shall be performed every four hours of production.

4.2.6 Phosphating process controls. All process control tests shall be conducted every four hours of production or as specified in the contract.

4.2.6.1 Phosphate coating weight (types I, II, and V). Three test specimens (see 4.2.2), at the conclusion of a maximum of every four hours of phosphate processing, shall be selected by the inspector for the test which is to be performed within one hour of processing to assure uniform coating weights are maintained. The clean, dry specimens shall be accurately weighed and the surface area of each calculated. The coating weight shall be determined according to the formula in 4.2.6.2. Acceptance criteria specified in 3.5.3 are determined as outlined in 4.2.6.2. Unless otherwise approved by the procuring agency, the following stripping procedures shall be used.

4.2.6.1.1 Zinc phosphate stripping (types I and V) for coating weight determination on steel. The phosphate coating shall be completely removed by immersion in a sodium hydroxide/tetrasodium ethylenediamine-tetraacetic acid (EDTA) solution for 15 minutes at 70ºF (21ºC). The solution can be formulated by adding 125 grams of sodium hydroxide and 125 grams of tetrasodium EDTA to 750 ml of deionized water. Upon removal from the stripping solution, rinse the samples in water, dry the test specimens and reweigh to the nearest milligram. Repeat this process, in five minute increments, until a constant weight is obtained.

4.2.6.1.2 Iron phosphate stripping (type II) for coating weight determination on steel. The phosphate coating shall be completely removed by immersion in a sodium hydroxide/sodium tartrate solution for 15 minutes at 70ºF (21ºC). The solution can be formulated by adding 50 grams of sodium hydroxide and 10 grams of sodium tartrate to 940 ml of deionized water. Upon removal from the stripping solution, rinse the samples in water, dry the test specimens and reweigh to the nearest milligram. Repeat this process, in five minute increments, until a constant weight is obtained.

4.2.6.1.3 Phosphate stripping for coating weight determination on zinc and zinc alloy coated steels (types I and V). The phosphate coating shall be completely removed by immersion in ammonium dichromate stripping solution for 5 minutes at approximately 70ºF (21ºC). The solution can be formulated by adding 40 grams of ammonium dichromate to 2.5 liters of ammonium hydroxide. Adequate ventilation is required when using these chemicals. Upon removal from the stripping solution, use a white plastic policeman to gently scrape the surfaces to remove any residual coating and rinse the samples in water. Dry the test specimens and reweigh to the nearest milligram. Repeat this process until a constant weight is obtained.

4.2.6.2 Coating weight formula. The coating weight is determined by the formula below:

$$\text{Coating wt } (\text{mg/ft}^2) = \frac{(\text{Initial wt in g - Final wt in g})\ (144{,}000\ \text{inch}^2\ \text{mg/g ft}^2)}{\text{Total surface area in square inches}}$$

where the initial weight in grams represents the weight of the phosphate coated specimen
and
where the final weight in grams is the weight of the stripped specimen

or

$$\text{Coating wt } (\text{mg/m}^2) = \frac{(\text{Initial weight in mg - Final weight in mg})}{\text{Total surface area in square meters}}$$

Intervals of four hours production shall be considered acceptable for testing, provided that the average coating weight of the 3 specimens equals or exceeds the minimum coating weight required (see 3.5.3) for the applicable type and not more than one sample falls below the minimum. The sample falling below the minimum shall be within 10 percent of the minimum requirement. The production lot represented by the failed test shall be reworked and corrective action taken until production is again acceptable. When determining the surface area of irregularly shaped objects, consideration and care should be taken to correctly determine the surface area of both the inner and outer surfaces of the test piece.

4.2.6.3 Phosphate coating process controls. Discard (dump) criteria shall be established for each chemical phase of the process. A free acid, total acid and accelerator titration as specified or recommended by the supplier's maintenance procedure for control. If additional process control tests such as titanium concentration or ferrous iron are required by the chemical suppliers product technical bulletin, these additional tests shall also be performed by the contractor.

4.2.7 Organic coating controls. Organic coatings must be applied in accordance with applicable coating application, system, or item specification.

4.2.7.1 Thickness of coatings. All applicable surfaces shall be coated and the basis for acceptance (including the number of specimens tested) shall be as specified in the contract. All coatings, inorganic and organic shall be checked daily for dry film thickness, as required by the applicable specification or drawing. Film thickness gages shall be used for determining organic coating thickness (see 6.9) after the gage has been standardized using the same surface as that over which the organic finish has been applied.

4.2.7.2 Adhesion test for CARC. After the complete (specified) paint finish has been applied and cured, three samples from each lot shall be selected for paint adhesion testing (see 4.2.2). Adhesion testing on the painted test specimens shall be performed after the complete paint finish has fully cured. A two coat paint

system should be tested after drying in accordance with applicable paint specifications. Each sample shall be tested using ASTM D3359, method B. Nonconformance to 3.5.7.1 shall constitute failure of this test. All items processed since last acceptance shall be rejected and corrective action shall be taken.

4.2.7.3 Paint adhesion test for non-CARC. Unless otherwise specified, a minimum of two test specimens (see 4.2.2) from each day's production shall be run through all steps of the regular production process including painting. Adhesion testing on the painted test specimens should be performed after the coating has dried in accordance with the applicable paint specification. Press a 2 inch length of a somewhat longer piece of pressure-sensitive adhesive tape (3/4 inch width) conforming to ASTM-D5486 (type I or type II), firmly onto a flat or cylindrical surface of the item, rubbing out all air bubbles under the tape. Allow approximately 10 seconds for the test area to return to room temperature. Grasp a free end of the tape and at a rapid speed strip it from the item by pulling the tape back upon itself at 180 degrees (in such a manner that the tape is folded back to back during the procedure). Observe for bared areas where the paint is removed. Disregard flecks of paint on tape where the underlying metal or phosphate coating is not visibly exposed. Nonconformance to 3.5.7.2 shall constitute failure of this test, and all item processed since last acceptance shall be rejected and corrective action shall be taken.

4.2.8 Accelerated corrosion resistance. Prior to initiation of production, or whenever a change in production or paint occurs, or when required by the procuring activity, a minimum of three test specimens (see 4.2.2), shall be run through all steps of the regular production process including primer application. Specimens having a hardness of Rockwell C40 or greater shall be treated for relief of hydrogen embrittlement prior to painting. The specimens shall be coated and cured for the time and temperature recommended for use in production. When approved by the procuring activity, an alternate test may be used (see 6.4), which allows one of the organic coatings listed in table II, paragraph 6.8.2 to be substituted for that used in production. The painted specimens shall be scribed in a vertical direction in a manner described in ASTM D1654. In instances where more than one metal is used, each metal shall be rated.
During production, one specimen (see 4.2.2) from each day's production shall be subjected to the accelerated corrosion test specified below (4.2.8.1, 4.2.8.2), until five consecutive days' production have successfully passed the test. Sampling may then be reduced to one sample twice per week. If failure occurs, all items processed since last acceptance shall be rejected and corrective action taken. Sampling shall revert to one specimen from each day's production until five consecutive days' production have again successfully passed the test. For the purpose of this test, the significant surface on cylindrical items shall be an area 60º on either side of a scribe mark. Production shall not be initiated until results of the salt spray test are received, except at the contractor's risk. When specified, the painted specimens may be scribed with two intersecting lines using the scribe tool described in ASTM D1654, (see 6.4 h).

4.2.8.1 Ferrous surfaces. The specimens, prepared above (4.2.8), shall then be exposed to the 5 percent salt spray specified in ASTM B117. Unless otherwise specified, epoxies shall be tested for 336 hours. Electrocoat primers shall be tested for 1000 hours. Within 24 to 48 hours after removal from the salt fog cabinet, the test coupon will be scraped with a metal putty knife at a 30 degree contact angle to the scribed surface and evaluation made for compliance to paragraph 3.5.8 for CARC systems.

4.2.8.2 Zinc or zinc alloy coated surfaces. All pretreatments and primers used on zinc or zinc alloy coated surfaces shall be qualified for 40 cycles by the GM 9540P accelerated cyclic corrosion test. The test protocol and calibration tests for mass loss shall be conducted as noted in the test specification. The test coupons will be subjected to 40 cycles of testing, and upon removal from the cabinet, the coupons will be scraped with a metal putty knife at a 30 degree contact angle to the scribed surface, and coupons shall meet the acceptance criteria as specified in paragraph 3.5.8 for CARC systems.

4.2.9 Hydrogen embrittlement relief test. Unless otherwise specified, the test for the effectiveness of the procedures used to control hydrogen embrittlement is performed using procedures outlined in ASTM F519 using a type 1 a specimen at a frequency of every 90 to 120 days.

5. PACKAGING

5.1 Packaging is not applicable to this specification.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Cleaning methods. The cleaning methods and chemical conversion coatings covered in this specification are suitable for use as rust removers, rust preventatives, and as metal conditioners for ferrous metals prior to application of paints.

6.1.1 Ferrous surfaces. Cleaning methods are intended primarily for ferrous metal cleaning, rust removing, descaling, or surface etching purposes in conjunction with chemical pretreatment processes. Method I (abrasive blasting) is generally recommended to remove heavy rust and mill scale on metals with thicknesses greater than 1/8 inch and, when specified, may be coated without being given a conversion coating. Method VI (phosphoric acid) may be used to remove light to moderate rusting but is not considered an acceptable substitute for a chemical pretreatment process. The selection of the cleaning process is dependent on the type and amount of soil and corrosion products on the items.

6.1.2 Nonferrous surfaces. Certain cleaning processes in this specification may also be used on nonferrous surfaces, for example: mechanical cleaning, solvent cleaning, vapor degreasing, and emulsion cleaning. Care should be exercised to ensure that cleaning materials do not have detrimental affects on the items being cleaned. In addition, this specification covers ferrous metal items containing small areas of zinc or aluminum surfaces or accessory fabricated parts of zinc or aluminum requiring treatment.

6.1.3 Nonferrous substrate testing variations. Zinc or zinc alloy coated substrates are tested in accordance with the GM 9540P accelerated cyclic corrosion test for 40 cycles, (see 4.2.8.2), then scraped after exposure and examined using the same acceptance criteria noted for the accelerated corrosion test in 3.5.8 of this specification, unless an accelerated corrosion test such as the sulfur dioxide salt spray test specified in ASTM G85 is specified in the contract. In that case, that test protocol rather than neutral salt spray should be used to qualify the pretreatment/primer.

6.2 Chemical conversion and pretreatment coatings.

6.2.1 Type I. Type I process is intended primarily for use as a general all purpose pretreatment prior to painting on tanks, trucks, sedans, ammunition and other items, as specified and is generally recommended where climatic exposure is anticipated. Type I is the preferred pretreatment for zinc plated (ferrous substrate) parts, prior to paint application.

6.2.2 Type II. Type II process is intended primarily for use where metal parts are to be formed (assembled) after painting and for other end items as specified.

6.2.3 Type III. Type III is intended for use where size and shape preclude the use of types I, II, or V, and where items containing mixed metal components are assembled prior to treatment.

6.3 Safety precautions. All safety requirements stated herein apply in addition to any applicable federal, state, and local rules or requirements. Chemical suppliers or manufacturers instructions and insurance underwriters instructions must be followed to insure safe handling practices and appropriate regulations governing chemicals. In accordance with 29 CFR 1910.1200, Material Safety Data Sheets (MSDS) are

required for each hazardous chemical used in an operation, and must be readily available to personnel using the material. Contracting officers will identify those activities requiring copies of the MSDS.

6.4 Ordering data. Procurement contracts should ensure that specific requirements/quality assurance provisions are specified as indicated in the following options:

(a) Specification identification includes: title, revision letter, and date of specification and any existing amendment. Type of conversion pretreatment and coating thickness classification must also be specified.
(b) Cleaning method and type coating required (see 1.2).
(c) Approval (see 3.2).
(d) Process (see 3.4).
(e) List each Material Safety Data sheet (MSDS) required for any solvent used in method II – VI (see 3.5.11, 6.3)
(f) Sampling and inspection (see 4.1).
(g) Hydrogen embrittlement relief test (see 3.5.9, 4.2.9)
(h) Allowance of alternative method for testing coating resistance to salt fog (see 4.2.7.2, 4.2.8, 6.1.3)

6.5 Preproduction approval (type I and V).

6.5.1 Army. Phosphatized coatings prepared for subsequent paint application or other applications require detailed information of the proposed procedure, chemicals and equipment to be used. Preproduction submittal and approval will be as specified by the contracting officer.

6.5.1.1 Army preproduction procedure approval for types I or V coatings. See 3.2 – 3.2.2.

6.5.2 Activities other than Army. Preproduction approval will not be required for proposed processes for applying crystalline phosphate base coatings that correspond to the procedure outlined and described in 6.5.3. Unless otherwise specified, procedures, chemicals and equipment which do not correspond to the procedures in 6.5.3 will require preproduction approval of the procuring agency.

6.5.3 Procedure (method of application of phosphate coating, applicable to type I and type V). The phosphate coating must be applied in a minimum of five stages. Additional stages may be added at the option of the contractor provided that the five basic stages are retained. The minimum requirements for either spray or dip application are as follows:

Stage 1, cleaning. The cleaning method used should be in accordance with one of the methods of 1.2.1 or a combination thereof.

Stage 2, rinse. The final cleaning method should be followed by a clean hot water rinse 125º - 180ºF (52ºC - 82ºC), or at temperature specifically recommended by manufacturer's instruction, with a constant overflow maintained by the continuous addition of fresh water entering from the bottom and/or by spraying fresh water on the parts leaving the rinse.

Stage 3, phosphating. The properly cleaned articles or items should be subjected to the phosphating solution (see 3.4.3.1) for at least the time required to secure the specified coating weight in accordance with the supplier's recommendation.

The equipment used must be constructed of materials resistant to the action of the phosphating solution and will not contain copper alloy fittings or brazing where they may come in contact with the solution.

The phosphating bath should be operated at temperatures and concentrations designated by the suppliers. Dated records should be maintained noting the periodic analyses and additions to the solutions. Fog sprays should be provided on both dip tanks and spray equipment to prevent the solution from drying on the work surface prior to the subsequent water rinse.

Stage 4, water rinse. A clean water rinse with a constant overflow is maintained by the continuous addition of fresh water entering from the bottom.

Stage 5, acidified rinse. A final acidified rinse should be prepared and maintained in accordance with the suppliers' recommendations (see 3.4.5). Such treatment is beneficial to the removal of unreacted chemical residues and provides a protective coating or seal that improves corrosion resistance. Care should be taken to maintain rinse at strengths that will not stain or hinder the coating.

As a starting point, follow manufacturer's suggestions for acidified rinse. Experiment with conditions (time, temperature, and agitation) to achieve optimum seal.

Improper rinsing techniques have a detrimental affect on items requiring subsequent paint. Although the harmful effect on the paint coating may not be immediately apparent, it can result in early failure of the paint by blistering or the rapid spread of corrosion products and flaking from a scratch.

After treatment, the phosphated parts are not to be handled with bare hands, oily or contaminated gloves.

Organic finishing of prepared surfaces should not be delayed as contamination from ambient sources may occur causing a reduction in adhesion of the organic coating. Specification requires application of coating within 24 hours of pretreatment.

6.6 Type II. The properly cleaned articles or items should be treated (see 3.4.3.2) until a phosphate coating is produced which is insoluble in water and has a color varying from golden yellow to purple. The article or items should be exposed to the phosphating solution in a spray process for a minimum of one minute or 3 minutes in an immersion process.

6.7 Type III. Correct film thickness is important for MIL-C-8514, and DOD-P-15328 pretreatment coatings. Small steel panels prepared with films, too thick, too thin, and correct, may serve as visual color guides for wash primer pretreatment.

6.8 Paint information. The information in tables 1 and II below is for illustrative purposes only; it contains examples of test requirements, and inspection and acceptance criteria that may be useful.

TABLE I. Paint information for non-CARC coatings.

Test	MIL-E-52891	MIL-E-11195
Dry film thickness, mils	0.9 - 1.1	1.3 - 1.7
Salt spray – air dry	72 Hours	7 Days
Exposure time, hours	150	120

TABLE II. Paint information for CARC coatings.

Test	MIL-P-53022	MIL-P-53030	MIL-P-53084
Dry film thickness, Mils	Type I: 0.9 - 1.1 Type II: 1.0 - 1.5	1.0 - 1.5	0.9 – 1.1
Cure (Dry) conditions (Recommended)	air dry: 3 Days Type I 7 Days Type II	air dry, for 168 hr	20 min @ metal temp of 350ºF (177ºC), air cool to room temp of 66-79ºF (19-26ºC)
Salt Spray Exposure, Hours	336	336	1000

6.9 The following instruments for measuring paint thickness have been found to be satisfactory:

Elcometer - Distributed by - Gardner Laboratory, Inc., Bethesda, Maryland

Erichsen Paint Inspection Gage - Erichsen Instruments, Inc., Akron, Ohio

"G.E." gage - General Electric Company, Schenectady, New York

Lea gage - Lea Manufacturing Company, Waterbury, Connecticut

Positector 2000 - KTA-TATOR, Inc., Pittsburgh, Pennsylvania

6.10 Appearance. After application of chemical conversion coating or use of one of the cleaning methods, changes in appearance are to be expected. Conversion coatings of types I, II, and V leave a chemical deposit. Cleaning methods leave the metal surface substantially bare. Method VI, alcoholic phosphoric acid and phosphoric acid containing a solvent and detergent discolors the surface. Method VI, hot phosphoric acid with a detergent etches the surface.

Table III lists examples of specification materials that may be used for cleaning purposes.

TABLE III. Specification material applicable to each method.

Cleaning method	Specification
Method I	SSPC SF 5
	SSPC SF 10
Method II[1/]	ASTM D 4081
	ASTM D 4376
	ASTM D 4080
	A-A-2904
	MIL-C-15074
Method III	A-A-59146
	A-A-59133
	SAE AMS 1547
Method IV	MIL-C-11090
	MIL-C-43616
Method V	A-A-59260
Method VI	MIL-C-10578

[1/] Note: Ozone depleting solvents are no longer specified for degreasing or cleaning the surface of parts. Solvent selection is left to the discretion of the manufacturer.

6.11 Temperature of base metal. In general, metal temperatures should be lower than 130ºF (54ºC) for cellulose lacquers and 160ºF (71ºC) for enamels unless the coating material has been specially formulated for the purpose. Temperatures from 60ºF - 120ºF (16 - 49ºC) are the most satisfactory (see 3.5.5). The temperature of the surface must not exceed 225ºF (107ºC) prior to painting, because heating above this temperature tends to dehydrate the phosphate crystal and deteriorates the basis coating.

6.12 Pretreatment. Application of organic coatings over bare untreated ferrous surfaces is not a recommended practice. Organic coating should be applied to surfaces prepared with a conversion coating or pretreatment.

6.13 Phosphate coating weights. The Environmental Protection Agency (EPA) regulations are imposed by both Federal and State agencies which place a financial burden on the contractor. Therefore, accurate alternative stripping methods may be substituted for the stripping solution described in paragraph 4.2.6.1 provided the same accuracy could be maintained.

6.13.1 Scanning electron microscope. The scanning electron microscope (SEM) is often used to study the morphology of zinc phosphate crystals. It can be used to assure proper coverage on production parts and is useful for preproduction approval.

6.13.2 Coating weight conversions. Coating weights are given in mg/ft^2 throughout this specification. However, because there may be a need to express the coating weight in mg/m^2, the following conversion factor is noted for informational purposes:

$$mg/m^2 = (mg/ft^2)\ (10.764\ ft^2/m^2)$$

6.14 Coating weight test results. Quality assurance inspection tests performed in accordance with paragraphs 4.2.6.1 and 4.2.6.2 are mandated to ensure timeliness and availability of the coating weights obtained. Coating weight verification tests must be performed within one hour of sample preparation to

assure that process controls are effective and that out-of-tolerance conditions are rapidly corrected. Records, including reports of the test results, must be maintained at the phosphate application site and must be made available upon request of the acquisition activity (see 3.5.3 and 4.2.6.1).

6.15 Problem alloys. Alloys such as AISI 4340 or SAE 4340 may be difficult to process. It is important that the contractor consult with the processing chemicals supplier to work out a procedure that will achieve the desired phosphatized coating with respect to coverage and coating weight.

6.15.1 Type I, spray systems. Stopping of the conveyor line while parts are being processed is unacceptable. Parts subjected to a line stoppage must be reworked should they fail to meet visual acceptance criteria.

Custodians:
Army MR
Navy – YD
Air Force – 11
GSA - FSS

Review activities:
Army- MI, CR, AV, EA, AR, AT, MD1
Navy - MC, OS, SH
Air Force - 80

Preparing activity:
Army - MR

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I RECOMMEND A CHANGE:	1.DOCUMENT NUMBER TT-C-490E	2. DOCUMENT DATE(YYYYMMDD) 20020722

3. DOCUMENT TITLE
CHEMICAL CONVERSION COATINGS AND PRETREATMENTS FOR FERROUS SURFACES (BASE FOR ORGANIC COATINGS)

4. NATURE OF CHANGE *(Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed)*

5. REASON FOR RECOMMENDATION

6. SUBMITTER

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