

INCH-POUND

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FEDERAL SPECIFICATION

CHEMICAL CONVERSION COATINGS AND PRETREATMENTS FOR METALLIC SUBSTRATES (BASE FOR ORGANIC COATINGS)

The General Services Administration has authorized the use of this federal specification by all federal agencies.

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers processes, pretreatments, and pre-primer surface preparations of metallic substrates for coating applicators. It covers metal surface preparation for delaying corrosion initiation and promoting primer adhesion. In addition, this specification covers the testing requirements for qualification of conversion coatings, the pretreatments and pre-primer coatings. Additional qualification requirements may be required based on each specific application.

1.2 Classification. This specification covers the following cleaning methods, surface preparation processes, and substrate classes (see 6.2).

Beneficial comments, recommendations, additions, deletions, clarifications, etc. and any data that may improve this document should be sent to: Director, CCDC Army Research Laboratory, Weapons and Materials Research Directorate, CCDC ARL Specifications and Standards Office, ATTN: FCDD-RLW-MC, Aberdeen Proving Ground, MD 21005-5069. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <https://assist.dla.mil/>.

AMSC N/A

AREA MFFP

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1.2.1 Surface cleaning. Surface cleaning may include one or more of the following methods to meet surface cleanliness requirements (see 6.1.1 and 6.1.2). Not all methods of surface cleaning are listed below. Other methods if not prohibited by drawing or contract, may be used, provided the requirements in this specification are met.

Method I	Mechanical or abrasive blast cleaning, sanding, grinding, in accordance with The Society For Protective Coatings (SSPC) standards.
Method II	Solvent cleaning by immersion, spray, vapor, or hand wiping.
Method III	Detergent cleaning by immersion, spray, ultrasonic, hot alkaline, or electrolytic methods.
Method IV	Emulsion cleaning, with or without added water.
Method V	Derusting by chemical means.
Method VI	Phosphoric acid cleaner (detergent or solvent-type with detergent).
Method VII	Steam cleaning, with or without assisted pressure washing.

1.2.1.1 Ozone depleting chemicals. To comply with the Clean Air Act regulations, materials can not contain class I or class II ozone depleting substances.

1.2.2 Conversion coatings, pretreatments, and pre-primer coatings. Chemical conversion and pretreatment coatings will be furnished in the following types (see 6.2).

Type I	Zinc phosphate.
Type II	Aqueous iron phosphate.
Type III	Organic pretreatment.
Type IV	Inorganic pretreatment.
Type V	Medium weight zinc phosphate.
Type VI	MIL-DTL-5541, Chemical Conversion Coatings on Aluminum and Aluminum Alloys.
Type VII	Anodic coating and electrolytic passivation.
Type VIII	MIL-PRF-32550, Metal-rich Primers.

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.

1.2.3 Substrate classes. Each of the aforementioned types may have materials that are appropriate for multiple metals qualification. In order to systematize the listing of products qualified to this specification found in the Qualified Products Database (QPD), a class will be assigned to each metal type.

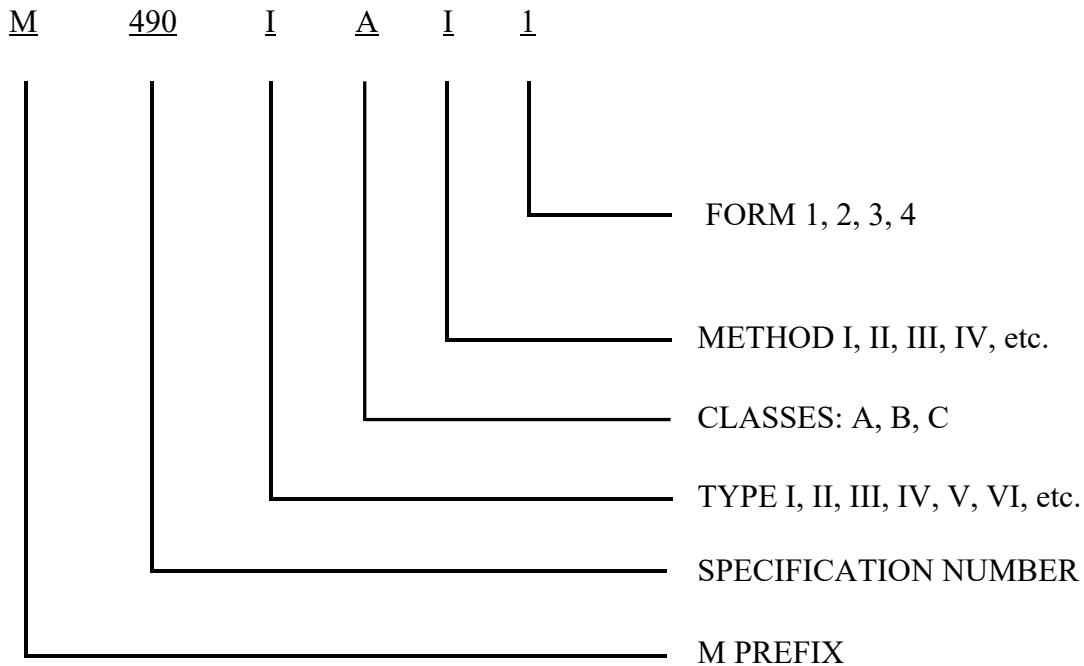
Class A	Steel alloys consisting mostly of iron other than corrosion resistant steel alloys which would be classified under class C.
Class B	Aluminum alloys.
Class C	Other alloys, multi-metal combinations of steel and aluminum or applications for other metallic substrates as required by the manufacturer and where approved by the CCDC Army Research Laboratory (ARL) during the qualification process.

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1.2.4 Forms. Chemical conversion and pretreatment coatings are differentiated by process through the following forms.

Form 1	Spray application.
Form 2	Spray application and rinse.
Form 3	Immersion application.
Form 4	Immersion application and rinse.

1.3 Part or Identifying Number (PIN). PINs to be used for pretreatments acquired by this specification are created as follows:



2. APPLICABLE DOCUMENTS

2.1 Government publications. The issues of the following documents, in effect on the date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

FEDERAL STANDARDS

FED-STD-313 - Material Safety Data, Transportation Data, And Disposal Data For Hazardous Materials Furnished To Government Activities

(Single copies of this specification, and other federal specifications required by activities outside the Federal Government for bidding purposes are available without charge from the General Services Administration, Federal Supply Service, <https://www.gsa.gov/>).

(Copies of this document are available online at <https://quicksearch.dla.mil/>.)

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DEPARTMENT OF DEFENSE SPECIFICATIONS

- MIL-DTL-5541 - Chemical Conversion Coatings on Aluminum and Aluminum Alloys
- MIL-C-8514 - Coating Compound, Metal Pretreatment, Resin-Acid.
- MIL-A-8625 - Anodic Coatings for Aluminum and Aluminum Alloys
- MIL-PRF-16173 - Corrosion Preventive Compound, Solvent Cutback, Cold-Application
- MIL-PRF-23377 - Primer Coatings: Epoxy, High-Solids.
- MIL-PRF-32348 - Powder Coating, Camouflage Chemical Agent Resistant Systems
- MIL-PRF-32550 - Metal-rich Primers
- MIL-DTL-53022 - Primer, Epoxy Coating, Corrosion Inhibiting Lead and Chromate Free
- MIL-DTL-53030 - Primer Coating, Epoxy, Water Based, Lead and Chromate Free
- MIL-DTL-53072 - Chemical Agent Resistant Coating (CARC) System Application Procedures and Quality Control Inspection
- MIL-DTL-53084 - Primer, Cathodic Electrodeposition, Chemical Agent Resistant
- MIL-PRF-85582 - Primer Coatings: Epoxy, Waterborne

DEPARTMENT OF DEFENSE STANDARDS

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

(Copies of these documents are available online at <https://quicksearch.dla.mil/>.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless a specific issue is identified, the issue in effect on the date of invitation for bids or request for proposal shall apply.

ASTM INTERNATIONAL

- ASTM A109/A109M - Standard Specification for Steel, Strip, Carbon (0.25 Maximum Percent), Cold Rolled
- ASTM A1008/A1008M - Standard Specification for Steel, Sheet, Cold Rolled, Carbon, Structural, High Strength Low Alloy, High Strength Low Alloy with Improved Formability, Solution Hardened, and Bake Hardenable
- ASTM B117 - Standard Practice for Operating Salt Spray (Fog) Apparatus
- ASTM D522 - Standard Test Methods for Mandrel Bend Test of Attached Organic Coatings
- ASTM D610 - Standard Practice for Evaluating Degree of Rusting on Painted Steel Surfaces

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- ASTM D1654 - Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments
- ASTM D1849 - Standard Test Method for Package Stability of Paint
- ASTM D3330/D3330M - Standard Test Method for Peel Adhesion of Pressure Sensitive Tape
- ASTM D3359 - Standard Test Methods for Measuring Adhesion by Tape Test
- ASTM D3363 - Standard Test Method for Film Hardness by Pencil Test
- ASTM D3924 - Standard Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials
- ASTM D5486/D5486M - Standard Specification for Pressure-Sensitive Tape for Packaging, Box Closure, and Sealing
- ASTM F21 - Standard Test Method for Hydrophobic Surface Films by the Atomizer Test
- ASTM F22 - Standard Test Method for Hydrophobic Surface Films by the Water-Break Test
- ASTM F519 - Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments
- ASTM G50 - Standard Practice for Conducting Atmospheric Corrosion Tests on Metals

(Copies of these documents are available from <https://astm.org/>.)

GENERAL MOTORS ENGINEERING STANDARDS

- GMW 14872 - Cyclic Corrosion Laboratory Test.

(Copies of this document are available from <https://ihsmarkit.com/>.)

SSPC: THE SOCIETY FOR PROTECTIVE COATINGS

NACE International Standard Practices

- SSPC-Guide 15 - Field Methods for Retrieval and Analysis of Soluble Salts on Steel and Other Nonporous Substrates.
- SSPC-SP5/ NACE No. 1 - White Metal Blast Cleaning
- SSPC-SP10/ NACE No. 2. - Near-White Blast Cleaning
- SSPC-VIS 3 - Guide and Reference Photographs for Steel Surfaces Prepared by Hand and Power Tool Cleaning

(Copies of these documents are available from <https://sspc.org/>.)

and/or

(Copies of these documents are available at <https://store.nace.org/>.)

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

- ISO 9000 standards - Quality Management Systems - Fundamentals and Vocabulary
- ISO/IEC 17025 directives - General Requirements for the Competence of Testing and Calibration Laboratories

(Copies of this document are available from <https://www.iso.org/home.html>.)

NATIONAL AEROSPACE AND DEFENSE CONTRACTORS ACCREDITATION PROGRAM (NADCAP)

- AS7003E - NADCAP Program Requirements
- AC7004F - Quality Polies Handbook

(Copies of these documents are available from <https://p-r-i.org/>.)

OSHA Regulations - Hazardous Communication Standards

- 29 CFR 1910.1200 - Hazard Communication

(Copies of these documents are available from <https://www.osha.gov/>.)

2.3 Order of precedence. Unless otherwise noted herein or in the contract, 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. This base document has precedence over Supplementary Requirements as specified for Non-CARC coatings for types I, II, and V.

3. REQUIREMENTS

3.1 Material. All material used shall be as specified herein, on the drawings, or in the contract. Materials not specified shall be selected by the contractor and shall be subject to all provisions of this specification. The prime and sub-tier contractor shall meet all requirements unless directed otherwise by the contract. In order to make more flexible use of hexavalent chromium-free pretreatments and to ease the burden of replacing hexavalent chromium based pretreatments, pretreatments free of hexavalent chromium shall be used unless contraindicated by contract or purchase order.

3.2 Preproduction approval. Preproduction approval for types I, II, and V as required by contract. For Non-CARC requirements for types I, II, and V, see Supplementary Requirements.

3.2.1 Procedure approval (types I, II, and V). Unless otherwise specified in the contract (see 6.2h), 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

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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 (see Supplementary Requirements for Non-CARC).

3.2.2 Preproduction validation panels (types I, II, and V). Unless otherwise specified in the contract (see 6.2h), the contractor shall coat fifteen (4 X 6 inch) panels (see 4.7.2) by the proposed zinc or iron 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. Three of the coupons shall be coated with the primer and topcoat. The primer dry film thickness shall be 1.5 (+/- 0.2) mils unless otherwise specified in the contract. Three of the non-primed coupons shall be checked for phosphate coating weight, 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 for accelerated corrosion resistance unless superseded by an accelerated corrosion test specified in the contract. Ferrous surface specimens of zinc or zinc alloy coated surfaces shall be exposed as specified in ASTM B117. The remaining nine panels (three phosphated only; three phosphate plus primer; three phosphate, primer, and topcoat) shall be forwarded to the procuring activity for testing and evaluation (see Supplementary Requirements for Non-CARC).

3.2.3 Stress Relief (types I, II, and V). Unless otherwise specified in acquisition documents or contract, parts with a surface or through hardness of Rockwell C 39 or greater shall be given a stress relief treatment. This includes carburized, induction hardened, flame hardened, etc. treatments. Also, any part that is ground, cold formed, cold straightened, etc., that may induce residual tensile stresses after machining or heat treatment shall be given a stress relief treatment. The stress relief treatment shall consist of a heat treatment at 350 to 400 °F (177 to 204 °C), or 50 °F (28 °C) below the tempering temperature, whichever is lower, for a minimum of one hour for every inch of thickness but not less than one half hour for thicknesses less than one-half inch. Optional heat treatment for carburized parts is 225 to 275°F (104 to 155°C) for 8 hours (see Supplementary Requirements for Non-CARC).

3.2.4 Qualification (types III, IV, and VIII). The conversion coatings, pretreatments, and pre-primer coatings furnished under this specification shall be products which are qualified for listing on the applicable QPD before contract award (see 4.3, 6.3). Any change in the formulation of a qualified product shall necessitate its requalification. The material supplied under contract shall be identical, within manufacturing tolerances, to the product receiving qualification.

3.2.5 Prior to introduction for production or use. Materials as described for each coating type and substrate class shall be validated for performance by the applicator in accordance with the requirements of this specification and by the individual performance requirements of each primer or coating system. Test specimens/coupons representing types III, IV, and VIII materials and process shall be submitted to the ARL for testing, and possible consideration for inclusion in the QPD for this specification. One exception to this is the type VI coating. This coating shall be validated for performance in accordance with MIL-DTL-5541 materials and processes.

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3.2.6 Contractors prior to using approved materials in production. Contractors shall have Objective Quality Evidence (OQE) (see 6.13) of performance in accordance with this document from an ISO 9000/IEC 17025 or NADCAP certified laboratory. The minimum corrosion resistance and adhesion requirements for the types and corresponding classes described herein shall be driven by this specification, the referenced specifications cited herein, and the performance requirements of the selected primer/topcoat system. The requirements that are greater, that is, more stringent, shall apply. Verification of the contractor's process shall be performed on a monthly basis using a certified lab or test facility with substantiating OQE or as required by contract (see 4.2).

3.2.7 Embrittlement testing for steel alloys at Rockwell C hardness (HRC) 39 or greater. Materials and corresponding application processes for conversion coating or other coatings described within this specification shall not contribute to hydrogen embrittlement issues for high strength steels or other materials susceptible to embrittlement during the application and service life of the coatings or pretreatments. Testing to validate that the materials to be processed or procedures do not contribute to hydrogen embrittlement shall be performed for types III, IV, and VIII during the process for qualification of the supplier's coating materials by ARL (see 4.8.11). Procedures for each contractor's process shall be tested prior to the production start in accordance with ASTM F519 and verified, at a minimum of every 120 days using an ISO 9000/IEC 17025 or NADCAP certified laboratory for OQE, that the treated substrates are not compromised. Any application to steel alloys at HRC 39 or greater shall require this testing. The effects of cleaning or processing shall not contribute to hydrogen embrittlement. If the process is demonstrated to contribute to hydrogen embrittlement, then relief shall be performed in accordance with MIL-STD-171.

3.3 Cleaning.

3.3.1 Removal of soils, corrosion, and salts. The parts shall be thoroughly free of oil, grease, wax, dirt, scale, and other foreign matter and shall not show visible signs of corrosion products. With all cleaning methods the water break test specified in 4.7.3.1 shall be used to check for freedom from organic contaminants. Inorganic contamination may still be present even if the water break test is acceptable and tests for those materials are necessary to ensure performance of the coating system. To ensure process robustness, a test for salts shall be performed periodically. The frequency for this testing shall be agreed upon between the applicator and the procuring authority to provide OQE for the process. Use SSPC-Guide 15 to determine salt contamination. The maximum allowable levels of chloride shall be measured by the adhesive patch/conductivity meter method. Currently the adhesive patch requirements are 3 $\mu\text{g}/\text{cm}^2$ for tanks and immersed surfaces and 5 $\mu\text{g}/\text{cm}^2$ for topside and non-immersed surfaces. Similarly, the conductivity requirements are 30 $\mu\text{S}/\text{cm}$ for immersed surfaces and 70 $\mu\text{S}/\text{cm}$ for non-immersed applications.

3.3.1.1 Removal of weld by-products. 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 functional or 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. Surface cleaning methods V or VI shall not be used on steels with HRC 39 or greater unless test data can substantiate that the combined chemical cleaning or

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phosphoric acid cleaning and subsequent coating pretreatments are not detrimental to the steels being treated. Ferrous materials HRC 39 or greater shall not be chemically etched or chemically derusted unless the coating material and process performed has been preapproved during the approval process described in 3.2 and are tested in accordance with ASTM F519 at an ISO 9000/IEC 17025 or NADCAP certified laboratory a minimum of every 120 days for OQE that the treated substrates are not compromised.

3.3.2 Rinsing. Adequate rinsing shall be performed following any chemical process to remove residual material remaining from the cleaning operation in accordance with the chemical supplier's recommendations to prevent interference with subsequent process steps. Special care shall be exercised in rinsing complex shapes to prevent contamination of any subsequent cleaning or coating process. Rinse water in a cleaning or pretreatment system that is reused or recycled shall be monitored for contaminants and controlled in accordance with the technical requirements of the material supplier (see 4.7.4 and 4.7.4.1).

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

3.3.3 Cleaning methods.

3.3.3.1 Method I - mechanical or abrasive cleaning; sanding, grinding, in accordance with SSPC standards. Abrasive blast cleaning shall meet or exceed the minimum requirements for SSPC-SP 10/NACE No. 2 near-white metal cleanliness unless otherwise specified by contract or the technical data package. Method I shall always be preceded by cleaning methods II, III, IV, VI, or VII, whichever is most appropriate for the application, to assure the substrate is water-break free clean. Using clean water, for example, distilled, deionized, Reverse Osmosis (RO) purified or filtered water in an atomizing sprayer works well in this method and method II for determining the results as described in 4.7.3.1. The blast media and maintenance of the abrasive blasting system shall be such that a consistent surface profile is maintained throughout the process and subsequent abrasive blast cleaning. Surface profile measurements shall not exceed the recommended range for the coating system applied and be recorded daily for purposes of OQE. If not specifically regulated by the coating system specification, the surface profile requirement shall be specified by the coating manufacturer so as to maintain the performance for coating adhesion and corrosion resistance. Ferrous media or media contaminated with ferrous spoils from previous abrasive blasting shall not be used on non-ferrous metallic substrates. Sanding and grinding to remove scale and rust shall also be preceded by the aforementioned cleaning methods contained herein. Cleanliness shall be in accordance with SSPC-VIS 3, with any of the appropriate SSPC cleaning standards to achieve visual cleanliness condition A, SP3/SD minimum unless otherwise specified by drawing or design engineering. Use the visual standards for cleanliness for steel as a guide for other metal substrates as well. Water break testing for surface cleanliness shall precede and follow method I cleaning to ensure that the cleaning tools or media do not become contaminated and the substrate is meticulously clean.

3.3.3.2 Method II - solvent cleaning by immersion, spray, vapor, or hand wiping. Solvent cleaning often leaves a hydrophobic surface even though organic soils have been removed. Failure of the water break test shall require repeating method II or the use of one or more of the other cleaning methods cited. Do not use solvents that would damage any existing coatings, sealants, adhesives or other materials that are to remain in place. The wipers or cloths used and

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the cleaning solvent shall be kept clean so as not to spread the contaminant over the substrate. Do not use halide solvents to clean titanium, magnesium or aluminum. Unless in a vapor controlled system whereby the solvent is distilled and reused, cleaning with solvent shall not be the first choice for cleaning. Substrates that are cleaned by method II shall pass the water break test as described in 4.7.3.1.

3.3.3.3 Method III - detergent cleaning by immersion, spray or electrolytic methods.

Detergent cleaning shall be used to remove unwanted substances, contamination and soils. Do not use detergents that are so aggressive as to damage the metal substrate or any coatings or other materials that are to remain in place. Use guidance that is supplied by the detergent supplier's technical data sheet to avoid excessive etching or other deleterious effects. Substrates that are cleaned by method III shall pass the water break test as described in 4.7.3.1.

3.3.3.4 Method IV- emulsion cleaning. With this method of cleaning three distinct steps shall be performed. Step 1 is pre-cleaning whereby the heavy soils are removed using the cleaning solutions either by immersion or delivered with pressurized nozzles. This pre-cleaning keeps the heavy soils from fouling the secondary cleaning system. Step 2 is a secondary cleaning cycle utilizing the same or similar cleaning solutions delivered by an array of pressurized nozzles to remove the soils. Step 3 is a rinse cycle utilizing clean water to thoroughly rinse the cleaning solution from the parts. Do not use cleaning materials or temperatures that could damage the metallic substrate or any previously applied coating. Use guidance provided by the chemical supplier's technical data sheet to avoid etching or other deleterious effects. Substrates that are cleaned by method IV shall pass the water break test as described in 4.7.3.1.

3.3.3.5 Method V - derusting/deoxidizing by chemical means. Deoxidize or derust metals by chemical means or enhance the process by using electrolytic systems for faster removal. Extreme caution shall be exercised to avoid excessive material removal especially on critical surfaces with close tolerances and materials subject to hydrogen embrittlement. Ferrous materials, HRC 39 or greater, shall not be chemically etched or derusted unless the materials used and the process has been preapproved (see 3.2.7). Substrates that are cleaned by method V shall pass the water break test as described in 4.7.3.1.

3.3.3.6 Method VI - phosphoric acid cleaning (detergent or solvent type with detergent). Ferrous materials, HRC 39 or greater, shall not be cleaned or derusted with phosphoric acid containing compounds unless the materials and process used has been pre-approved. Substrates that are cleaned by method VI shall pass the water break test as described in 4.7.3.1.

3.3.3.7 Method VII - steam cleaning. Steam cleaning shall be followed with methods II, III, or IV to achieve a water-break free surface. Substrates that are cleaned by method VII shall pass the water break test as described in 4.7.3.1.

3.4 Types of conversion coatings, pretreatments and pre-primer coatings. Types III and IV are interchangeable where appropriate, provided the performance in this specification and contract are met. Other types may also be interchangeable where appropriate provided the performance in this specification and contract are met and the procuring authority concurs.

3.4.1 Type I. Type I coatings shall be applied by spray or immersion and have coating weights in the range of 150 - 500 mg/ft² (1.6 - 5.4 g/m²). When painted, all corrosion, flexibility

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and adhesion testing within this document applies (see Supplementary Requirements for Non-CARC).

3.4.2 Type II. Type II coatings shall be applied by spray, steam, immersion or hand wiping, and have a minimum coating weight of 35 mg/ft² (0.4 g/m²). When painted, all corrosion, flexibility and adhesion testing within this document applies.

3.4.3 Type III. Type III coatings shall contain greater than 50% organic compounds by weight in the dried film, such as wash primers, adhesion promoters and flash-rust inhibitors as defined by the manufacturer of the coating. When painted, all corrosion, flexibility and adhesion testing within this document applies. All products under this type shall be Cr⁶⁺ free and shall require product qualification.

3.4.4 Type IV. Type IV coatings shall contain greater than 50% inorganic compounds by weight in the dried film, such as conversion coatings, adhesion promoters, and flash-rust inhibitors, as defined by the manufacturer of the coating. When painted, all corrosion, flexibility, and adhesion testing within this document applies. Product qualification required for this coating type.

3.4.5 Type V. Type V coatings are the same as type I coatings except that the coating weight is in the range of 500 - 1100 mg/ft² (5.4 – 11.8 g/m²). When painted, all corrosion, flexibility and adhesion testing within this document applies.

3.4.6 Type VI. Type VI coatings shall be validated for performance using MIL-DTL-5541. Whenever one of the coating types is not cited in a relevant contractual document or drawing, select the type II coating in MIL-DTL-5541. When painted, all corrosion, flexibility and adhesion testing within MIL-DTL-5541 applies.

3.4.7 Type VII. Type VII coatings shall be validated for performance using MIL-A-8625, Anodic Coatings for Aluminum and Aluminum Alloys. When painted, all corrosion, flexibility and adhesion testing within MIL-A-8625 and TT-C-490 applies.

3.4.8 Type VIII. Type VIII coated surfaces shall be abrasive blast cleaned. These coatings are classified as metal-rich finely divided metal powder, including ethyl silicates, alkyl silicates, potassium silicates, and metal-rich polymer coatings. When painted, all corrosion, and adhesion testing within this document applies. Product qualification required for this coating type. In accordance with MIL-DTL-53072, a sealer coating of MIL-DTL-53022 or MIL-DTL-53030 shall be applied over the metal-rich primer.

3.5 End item requirements. The following requirements shall apply to the applicator of the coating systems. All test specimens/coupons shall be produced in the same manner as the end item.

3.5.1 Type I and type V, zinc phosphate. Type I and type V 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 residues. 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.

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3.5.2 Lighting conditions. A minimum light intensity of 50 lumens/ft² or 538 lux shall be provided at the working surface where manually controlled paint finish operations are performed. Minimum lighting shall apply to the final inspection areas (see 4.7.1.1).

3.5.3 Phosphate coating weight (types I, II, and V). Coating weight shall be controlled and tested every four hours of line operation.

3.5.4 Application of paint. The paint shall be applied to thoroughly dried surfaces within 24 hours after pretreatment to prevent contamination or rusting. Should 24 hours be exceeded, the applicator shall maintain OQE to determine if the substrate is suitable for coating. Use the manufacturer's technical data sheets for application of coatings. The dried surface shall not show any corrosion or soiling prior to painting and be meticulously cleaned (see 3.3). The temperature of the metal surface and the application environment shall be controlled to eliminate blistering, poor adhesion or other film irregularities.

3.5.5 Paint thickness. For all tests requiring painted test specimens/coupons, the dry film thickness (see 4.7.6.1) on all surfaces shall be in accordance with MIL-DTL-53072 or 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.

3.5.6 Paint adhesion (all methods and types). The CARC or non-CARC painted items or specimens / coupons shall show the following satisfactory paint adhesion when tested as in 4.7.6.2 and 4.7.6.3 (see Appendix A).

3.5.6.1 CARC painted items. The painted items, evaluated by using ASTM D3359 method B, shall have a minimum rating of 4B at 2 mm spacing. Removal of topcoat or topcoat-primer-pretreatment coating from any individual cross-hatch test on the unit with a rating of less than 4B constitutes failure of this test (see 4.7.6.2).

3.5.6.2 Non-CARC painted items. Unless otherwise specified by contract, coating system or engineering design when tested in accordance with ASTM D3359 method B, unsatisfactory adhesion shall be indicated by exposure of primer, bare metal, or underlying pretreatment by any of the following conditions:

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

3.5.7 Accelerated corrosion resistance (all types). After pretreatment and primer (or paint for single coat systems) application, the specimens/coupons shall be subjected to the accelerated corrosion test as in 4.7.7, 4.7.7.1, 4.7.7.2 and 4.7.7.3 for the required number of hours/cycles specified. Specimens/coupons shall show no blistering or loss of adhesion of the paint from the scribe mark (for steel, a minimum rating of 6, for aluminum, a minimum rating of 8, ASTM D1654 procedure A) (see 4.7.7). Blisters shall cover no more than 5% of the exposed area (rating of 7; see table I, Ratings for unscribed coatings), none larger than 1 mm in diameter on a standard test specimen/coupon or equivalent area of test specimen/coupon or item. Neutral salt

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spray testing shall not be used to qualify zinc or zinc alloy (see 4.8.10.1 and 6.1.3). GMW14872 cyclic corrosion testing shall be required in its place.

TABLE I. Ratings for unscribed coatings.

Area Failed, %	Rating Number
No Failure	10
0 to 1	9
2 to 3	8
4 to 6	7
7 to 10	6
11 to 20	5
21 to 30	4
31 to 40	3
41 to 55	2
56 to 75	1
over 75	0

3.6 Qualification requirements of pretreatments (types III, IV, and VIII). The following test requirements shall be for qualification testing of the pretreatment coatings, as specified in 4.8. All tests, other than storage stability, shall be conducted on specimens/coupons, as specified in 4.8.2, and coated with epoxy primer, as specified in 4.8.4. Pretreatments designed for aircraft and their assets application, and other than those qualified to type VI (see 1.2.2), shall include additional testing, as specified in MIL-DTL-5541.

3.6.1 Storage stability. The manufacturer of the pretreatment to be qualified shall provide details on the storage stability of their product, storage conditions required to maintain storage life and the containers required for storage (see 4.8.5). To verify the storage stability of the pretreatment, ARL has the option to perform additional testing.

3.6.2 Adhesion. The assessment of the adhesion of the coating film shall be determined by its ability to not peel from the substrate when tested in accordance with ASTM D3359, as specified in 4.8.6. The resultant test rating shall be classified as scale 4B or better.

3.6.3 Flexibility. A pretreatment with a film of primer, tested as specified in 4.8.7, shall withstand bending without cracking or flaking (see ASTM D522). Type VIII coatings are exempted from this requirement.

3.6.4 Water resistance. A pretreatment with a film of primer, tested as specified in 4.8.8, shall show no wrinkling or blistering immediately after removal of the specimen/coupon from the water. The primer shall be no more than slightly affected when examined 2 hours after removal. After 24 hours air drying, the portion of the specimen/coupon which was immersed shall be the same with regard to hardness, adhesion, color and gloss as compared to the portion which was not immersed. Film softening shall not exceed a difference in magnitude of 2 pencil hardness values (see ASTM D3363) from an unexposed film with identical cure history prior to water exposure.

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3.6.5 Hydrocarbon fluid resistance. A pretreatment with a film of primer, tested as specified in 4.8.9, shall show no blistering or wrinkling and after drying, there shall be a maximum Delta (Δ) E color change of 2.0 L a b values when comparing a portion of the untested panel to that of the tested area. This is commonly observed as a slight yellow to beige color change. After 2 hours air drying, the portion of the panel which was immersed shall be the same with regard to hardness, adhesion, color and gloss as compared to the portion which was not immersed. Film softening shall not exceed a difference in magnitude of 2 pencil hardness values (see ASTM D3363) from an unexposed film with identical cure history prior to hydrocarbon fluid exposure.

3.6.6 Corrosion resistance.

3.6.6.1 Salt spray resistance. A pretreatment with a film of primer tested as specified in 4.8.10.1 and examined immediately after removal from the salt spray test shall show minimal blistering or loss of adhesion of the paint from the scribe mark (for steel, a minimum rating of 6, for aluminum, a minimum rating of 8, ASTM D1654 Procedure A) (see 4.7.7). Blisters on the non-scribed areas shall cover no more than 5% of the exposed area, none of which shall be larger than 1mm in diameter (rating of 7; see table I, Ratings for un-scribed coatings).

3.6.6.2 Cyclic corrosion resistance. A pretreatment with a film of the primer tested as specified in 4.8.10.2 and 4.7.7 and evaluated using ASTM D1654 Procedure A shall have a minimum rating of seven (7). Blisters shall cover no more than 5% of the exposed area, none of which shall be larger than 1mm in diameter (rating of 7; see table I, Ratings for un-scribed coatings).

3.6.6.3 Atmospheric corrosion resistance. A pretreatment with a film of the primer and top-coat tested as specified in 4.8.10.3 shall show minimal blistering or loss of adhesion of the paint from the scribe mark (for steel, a minimum rating of 6, for aluminum, a minimum rating of 8, ASTM D1654 Procedure A). Blisters on the non-scribed areas shall cover no more than 5% of the exposed area, none of which shall be larger than 1mm in diameter (rating of 7; see table I, Ratings for un-scribed coatings).

3.6.7 Hydrogen Embrittlement. Testing for hydrogen embrittlement shall be performed on a regular basis on ferrous substrates in excess of HRC 39, not to exceed 120 calendar days, to maintain process integrity and OQE. To represent the pretreatment process or parts, a minimum lot of 3 notched round bars Type 1a.1 as described in (FIG. A1.1) ASTM F519 shall be used. The lot shall be rejected if any coated specimen develops any crack or fails by fracture as a result of the test.

3.7 Toxic ingredients. Other than parachlorobenzotrifluoride (PCBTF), the pretreatment material being supplied by the applicator or chemical/coating manufacturer for qualification to ARL shall contain no benzene (benzol), chlorinated solvents or ethylene based glycol ethers and their acetates (see 4.8.12). The solvents used in the cleaning methods cited herein shall have no adverse effects on human health when used as intended. With the exception of type VI, compounds of antimony, arsenic, beryllium, cadmium, chromium, cobalt, cyanide, lead, manganese, mercury, nickel, and selenium shall also be absent from all pigments in the products being submitted for qualification. Pigments contained in these materials shall be HAP-free.

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3.8 User instruction markings. All primary containers shall be legibly marked or labeled, as applicable, with the manufacturer's mixing instructions, the VOC content (in pounds/gallon or grams/liter), hazardous air pollutant (HAP) content for the applicator and the following:

PRECAUTION: Approved respirators must be used unless air sampling shows exposure to be below occupational exposure limits. Avoid contact with skin and eyes. Use with adequate ventilation. For other safety recommendations refer to the Safety Data Sheet (SDS). Keep containers closed.

3.9 SDS. The manufacturer shall comply with the requirements as specified in the OSHA Brief (see: <https://www.osha.gov/dsg/hazcom/>) and as specified in Appendix D of 29 CFR 1910.1200. (see: <https://www.osha.gov/laws-regs/regulations/standardnumber/1910/1910.1200AppD>). A SDS shall be prepared for the material in accordance with FED-STD-313 and forwarded to the qualifying activity (see 6.3). The SDS shall be included with each shipment of the material covered by this specification and submitted to pertinent Government agencies as stated in FED-STD-313.

3.10 Toxicity clearance. All new chemicals and materials being added to the Government supply system shall have a toxicity clearance. A toxicity clearance involves a toxicological evaluation of materials prior to introduction into the Government supply system. The federal program manager shall be responsible for identifying technically feasible materials and requesting a toxicity clearance for use of that material within their program (see 6.5).

4. QUALITY ASSURANCE PROVISIONS

4.1 Classification of inspections. The inspection requirements specified herein are classified as follows:

- a. Responsibility for inspection (see 4.2).
- b. Qualification inspection (see 4.3).
- c. Quality conformance inspection (see 4.4).

4.2 Responsibility for inspection. Unless otherwise specified in the contract (see 6.2h), the contractor is responsible for the performance of all inspection requirements (examinations and tests) as specified herein. Except as otherwise specified in the contract, the contractor may use his own ISO 9000/IEC 17025 or NADCAP certified facility or any other facilities certified to ISO 9000/IEC 17025 or NADCAP for the inspection requirements or for verification of those monthly inspection requirements specified herein. Daily inspection events which contribute to the overall monthly OQE need not to be performed by an ISO 9000/IEC 17025 or NADCAP certified facility. Specifically, corrosion testing must be performed and verified by an ISO 9000/IEC 17025 or NADCAP certified facility. 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.

4.3 Qualification inspection. Qualification of submitted pretreatments for types III, IV and VIII shall be conducted by the qualifying activity (see 6.3). The qualification test sample size shall be

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designated by the qualifying activity and shall be determined based upon the type of pretreatment (see 1.2.2). Pre-coated test specimens/coupons shall also be required, as designated by the qualifying activity. The samples shall be legibly identified (see 6.3.2). Qualification inspection shall consist of tests for all requirements specified in section 3.6 and table III. These test methods described in 3.6 and table III are for chemical and coating suppliers submitting their products to ARL for qualification for types III, IV and VIII. Qualification inspection shall examine for user instruction markings (see 3.8). The results of each test shall be compared with the applicable requirement in section 3.6 and table III. Failure to conform to any requirement shall be counted as a defect and the material represented by the sample test shall not be approved for inclusion in the QPD under this specification. Coated specimens/coupons in accordance with this document shall be forwarded to CCDC Army Research Laboratory, ATTN: FCDD-RLW-MC, Camouflage, Coatings and Corrosion Team, 6300 Rodman Road, Building 4600, Aberdeen Proving Ground (APG), MD 21005-5066.

4.3.1 Touch-up kits. Pretreatments will, at times, need to be re-applied due to repair or damage to the substrate. Due to this, touch-up kits shall also be qualified for the types requiring original qualification (see 4.3). Touchup shall be in accordance with the coating manufacturer's specifications and inspected, tested, and verified in the same manner as the base pretreatment. Touchup procedures using "Touchup kits or Touchup pens" are permitted provided the applications for such are used in accordance with the coating manufacturer's specifications and inspected, tested, and verified in the same manner as the base pretreatment. Furthermore, Touchup kits or Touchup pens and specialized procedures must be approved by ARL and qualified in the TT-C-490 QPD. Use of Touchup kits or Touchup pens for repairing aluminum pretreatments in accordance with MIL-DTL-5541 shall follow the directives of the MIL-DTL-5541 specification.

4.4 Quality conformance inspection. 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.

4.5 Inspection conditions. Where possible, coating verification tests will be performed IAW ASTM D3924. The laboratory temperature shall be 70-77 °F (21-25 °C) and the relative humidity 45-55 %. It is understood, in a production facility, that laboratory conditions may not be practical for coating verifications due to size and location of coated items. Inspections performed in a production environment may be limited to prevailing conditions. In no case shall a coating inspection be performed outside of the limits established by the coating manufacturer's guidelines.

4.6 SDS. The SDS shall address all components of the pretreatment and be in compliance with FED-STD-313. Noncompliance to 3.9 shall be cause for rejection.

4.7 Quality conformance test methods. The tests specified in 4.7.1-4.7.8 pertain to the applicator of the pretreatments and coatings and does not apply to qualification of pretreatments.

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4.7.1 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.

4.7.1.1 Lighting adequacy. Light intensity at the work or inspection surface shall be 50 lumens/ft² or 538 lux minimum.

4.7.2 Test specimens/coupons. Where practical, test specimens/coupons 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/coupons need not be identical in shape or size but shall be stamped, etched, or otherwise indelibly marked for identification as a test specimen/coupon. When parts are not available, use standard specimens/coupons not less than 4 x 6 x 0.0312 inches in size. Standard steel specimens/coupons such as cold rolled SAE 1010 steel, shall be used when authorized by the contracting officer. These specimens/coupons shall conform to ASTM A109/A109M or ASTM A1008/A1008M. Standard aluminum specimens/coupons of 2024-T3 shall be used when authorized by the contracting officer. For zinc coated surfaces, the specimens/coupons shall be made of the same coated material as specified in the contract. When coated steels are used, the standard test specimens/coupons shall be made of the same material as that specified in the contract. All test specimens/coupons shall be processed through all the cleaning, pretreating, painting, and drying steps along with the items being processed. Test specimens/coupons shall not be reused.

4.7.3 Removal of soils, corrosion, and weld by-products. Multiple cleaning procedures may be required to provide the required water break free surface. After cleaning and rinsing, and prior to the application of a coating, at least two production test specimens/coupons shall be subjected to a water break test at the conclusion of a maximum of every four hours of production. These specimens/coupons shall be dried and examined visually for rust, corrosion products, and soils. If specimens/coupons 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 during production until indications of soils or corrosion products are eliminated. Testing frequency shall then revert to at least two test specimens/coupons at the conclusion of a maximum of every four hours of production. Periods of manufacturing or process cessation of more than 30 calendar days shall require start up procedures with corresponding OQE as those described in TT-C-490 for cleaning, pretreatment, and coatings application. Those interruptions shall be reflected in the OQE history. Unless required by contract or procurement authority, no government oversight is required to restart the process.

4.7.3.1 Water break test. As specified in MIL-DTL-53072, all properly cleaned and pretreated surfaces shall be examined just prior to product application to assure that the surface is dry and free from soil or contamination of any kind. Immediately prior to painting, the surface shall be subjected to a water break test (see exceptions 6.12). The water break test shall be conducted in accordance with either ASTM F21 or ASTM F22 (whichever procedure best befits the situation). 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

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test prior to pretreatment or coating (see exceptions 6.12). 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.

4.7.3.2 Metal-rich coatings. Cleaning and surface profile shall be performed and verified in accordance with SSPC-SP 5/NACE No. 1, SSPC-SP 10/NACE No. 2 or manufacturer's recommendations for application (see 3.4.8).

4.7.4 Rinsing. The water rinses in series systems such as pretreating shall be tested for contamination at practical intervals of production unless independent fresh water or suitably treated water is used as a secondary rinse (see 3.3.2). Technical data supplied by the material manufacturers should be used for guidance. The frequency for this testing shall be agreed upon between the applicator and the procuring authority to provide OQE for the process.

4.7.4.1 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 for CARC and non-CARC paints (see Supplementary Requirements).

4.7.5 Pretreatment coating process controls. All controls as designated by the coating supplier shall be followed. Titration, pH management, temperature, and discard criteria shall be established for each chemical phase of the process and maintained as OQE.

4.7.5.1 Phosphate coating process controls. Discard 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.

4.7.6 Organic coating controls. Organic coatings shall be put into practical use in accordance with the relevant coating application, system, or item specification.

4.7.6.1 Thickness of coatings. All applicable surfaces shall be coated and the basis for acceptance (including the number of specimens/coupons tested) shall be as specified in the contract. All coatings, inorganic and organic shall be verified daily for coating weight or dry film thickness, as required by the relevant specification or drawing. For types I, II, and V, coating weights shall be verified every four (4) hours of line operation. Film thickness gages shall be used for determining organic coating thickness (see 6.7) after the gage has been standardized using the same surface (indexing) as that over which the organic finish has been applied.

4.7.6.2 Adhesion test for CARC. After the complete specified paint finish has been applied and cured, two samples from each day's production shall be selected for paint adhesion testing (see 3.6.2 and 4.8.6). Adhesion testing on the painted test specimens/coupons shall be performed after the complete paint finish has fully cured for a minimum of 168 hours at ambient conditions or by force-curing. A two coat paint system shall be tested after drying in accordance with

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applicable paint specifications. Each sample shall be tested using ASTM D3359, method B using any commercially available tape (1 inch width) that will yield a minimum of 80 oz of adhesive resistance (and can be verified with OQE) over the tested coating when tested in accordance with ASTM D3330/D3330M. CARC adhesion shall not be less than scale 4B with any layer of the system. Where CARC dry film thickness has exceeded 5 mils (125 microns), method A of ASTM D3359 shall be used if permitted by the procuring authority. Nonconformance to 3.6.2 shall constitute failure of this test. All items processed since last acceptance shall be rejected and corrective action shall be taken. Contact the DoD CARC Commodity Item Manager at CCDC Army Research Laboratory, ATTN: FCDD-RLW-MC, Camouflage, Coatings and Corrosion Team, 6300 Rodman Road, Building 4600, Aberdeen Proving Ground, MD 21005-5069 for a current listing of approved adhesion test tapes.

4.7.6.3 Paint adhesion test for non CARC. Unless otherwise specified, a minimum of two test specimens/coupons (see 4.7.2) 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/coupons shall 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 (1 inch width) conforming to ASTM D5486/D5486M 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 3.5.6.2 shall constitute failure of this test, and all items processed since last acceptance shall be rejected and corrective action shall be taken (see Supplementary Requirements).

4.7.7 Accelerated corrosion resistance. Prior to initiation of production on a contract, or whenever a change in the production process or coating system occurs, or when required by the procuring activity, a minimum of three test specimens/coupons (see 4.7.2), shall be run through all steps of the regular production process including primer application. The specimens/coupons shall be coated and cured for the time and temperature recommended for use in production. When approved by the procuring activity, an alternate test shall be used, which allows one of the organic coatings listed in table II, CARC Approved Primers, to be substituted for that used in production. The painted specimen/coupons are to be "X" scribed using a tungsten carbide scribing tool as described in ASTM D1654-08 (paragraph 5.1.2). The "X" scribe is made by scribing two intersecting lines from one corner to its opposite corner across the face of the specimen/coupon. The scribe should initiate/terminate no closer than 1/2" to the edge of the specimen/coupon. In instances where more than one substrate is used, each substrate shall be rated for corrosion resistance pertaining to the requirements of the specification of the primer being used. During production, one specimen/coupon (see 4.7.2) from each day's production shall be subjected to the accelerated corrosion test specified below until five consecutive day's productions have successfully passed the test. The minimum test frequency for continued production validation shall be in accordance with the technical data package or every 60 days, if the test frequency is not specified in the technical data package. If failure occurs, all items processed since last acceptance shall be rejected and corrective action taken. Sampling shall revert to one specimen/coupon from each day's production until five consecutive day's productions have again successfully passed the test. For the purpose of this test, the significant

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surface on cylindrical items shall be an area 60° 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. Periods of manufacturing or process cessation of more than 30 calendar days shall require start up procedures with corresponding OQE as those described in TT-C-490 for cleaning, pretreatment, and coatings application. Those interruptions shall be reflected in the OQE history. Unless required by contract or procurement authority, no government oversight is required to restart the process.

TABLE II. CARC approved primers.

Specification	Dry film thickness	Substrate ^{1/}	ASTM B117 (hours)	GMW 14872 (cycles)
MIL-PRF-23377	0.6 - 0.9 mils	AL	2,016	N/A
MIL-PRF-32348, type I	1.8 - 2.2 mils	CRS and AL	1,512 (CRS) 3,024 (AL)	45 (CRS)
MIL-PRF-32348, type II	1.8 - 2.2 mils	CRS and AL	1,008 (CRS) 3,024 (AL)	45 (CRS)
MIL-DTL-53022, type II	1.3 - 1.7 mils	CRS and AL	336	N/A
MIL-DTL-53022, type III	1.3 - 1.7 mils	CRS and AL	336	N/A
MIL-DTL-53022, type IV	1.3 - 1.7 mils	CRS and AL	1,008	30
MIL-DTL-53030, type II	1.3 - 1.7 mils	CRS and AL	1,008	30
MIL-DTL-53084	0.8 - 1.2 mils	CRS and AL	1,008	N/A
MIL-PRF-32550	2.5 - 3.5 mils	CRS	-	80/100 ^{2/}
MIL-PRF-85582	0.6 - 0.9 mils	AL	2,016	N/A

^{1/} Aluminum (AL) and cold rolled steel (CRS).

^{2/} See: MIL-PRF-32550

TERMS: 1 mil = 25.4 microns.

4.7.7.1 Ferrous surfaces. The specimens/coupons, prepared above (see 4.7.2), shall then be exposed to the 5 percent salt spray specified in ASTM B117. The selected primer shall be tested as specified in table II. When specified, the painted specimens/coupons shall be scribed with two intersecting lines using the scribe tool as described in ASTM D1654 (paragraph 5.1.2). Upon removal, rinse the specimens/coupons gently in warm running or deionized water (DI) until free from any visible salt deposits and examine for compliance as specified in 3.5.7 for CARC systems. Nonconformance shall constitute failure of test.

4.7.7.2 Zinc or zinc alloy coated surfaces. All pretreatments and primers used on zinc or zinc alloy coated surfaces shall be qualified by the GMW 14872 accelerated cyclic corrosion test. The test protocol and calibration tests for mass loss shall be conducted as noted in the test specification. All pretreatments and primers shall be tested as specified in table II, CARC Approved Primers. The test specimens/coupons shall be subjected to the required number of

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cycles of testing specified in table II. The specimens/coupons shall meet the acceptance criteria as specified in 3.5.7 for CARC systems.

4.7.7.3 Aluminum surfaces. Pretreatments shall be applied in accordance with manufacturer's instructions to clean aluminum surfaces. Unless specifically called for in the contract, a chromate containing primer shall not be used for qualification of a pretreatment. All pretreatments and primers shall be tested as specified in table II. Upon removal, rinse the specimens/coupons gently in warm running or DI water until free from any visible salt deposits and examine for compliance as specified in 3.5.7 for CARC systems. When visual examination is indeterminate, the specimens/coupons shall undergo coating removal along the scribe as per ASTM D1654 and inspected for pitting or corrosion not evident with paint present. Evaluate for compliance as specified in 3.5.7 for CARC systems.

4.7.8 Hydrogen embrittlement relief test. Unless otherwise specified, the test for the effectiveness of the procedures used to control hydrogen embrittlement shall be performed using procedures outlined in ASTM F519 using type 1a.1, which is a standard size notched specimen/coupon at a frequency of every 120 days.

4.8 Qualification testing of pretreatments. Tests specified in 4.8.1-4.8.11 pertain to the chemical and coating suppliers submitting products for qualification to types III, IV and VIII.

TABLE III. Qualification test methods.^{1/}

Item	ASTM Method	Test Paragraph	Requirement Paragraph or Table
Storage stability	D1849	4.8.5	3.6.1
Adhesion	D3359	4.8.6	3.6.2
Flexibility	D522	4.8.7	3.6.3
Water resistance	-	4.8.8	3.6.4
Hydrocarbon fluid resistance	-	4.8.9	3.6.5
Salt spray resistance	B117	4.8.10.1	3.6.6.1
Cyclic corrosion resistance ^{2/}	-	4.8.10.2	3.6.6.2
Atmospheric corrosion	G50	4.8.10.3	3.6.6.3
Hydrogen embrittlement	F519	4.8.11	3.6.7

^{1/} These test methods pertain to qualification submissions of types III, IV and VIII pretreatments only.

Tests in this table do not apply to the end user and applicator of pretreatments.

^{2/} General Motors Standard: GMW 14872.

4.8.1 Test conditions. Except as otherwise specified herein, the routine testing and referee testing shall be conducted in accordance with ASTM D3924. The dry film thickness of the epoxy primer being used in this testing shall be as specified in 4.8.4. Failure of any test result to fall within the specified ranges in section 3 shall constitute failure of the applicable test.

4.8.2 Test specimens/coupons. Steel test specimens/coupons shall be cold rolled SAE 1010 steel conforming to ASTM A109/A109M or ASTM A1008/A1008M or as agreed upon by the

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manufacturer and ARL. Hydrogen embrittlement specimens shall be type 1a.1 as described in ASTM F519. The specimens/coupons shall have Rockwell C hardness (HRC) 55 to 75 and a surface roughness of 30 to 45 micro inches (arithmetic average) as rolled. Aluminum test specimens/coupons shall be aluminum alloy 2024-T3 or as agreed upon by the manufacturer and ARL. Standard steel and aluminum test specimens/coupons shall be 4 x 6 x 0.0312 inches in size, except as specified herein.

4.8.3 Test procedures. Tests shall be conducted in accordance with ASTM, GM, or as specified herein (see 4.3). The right is reserved to make any additional tests deemed necessary to determine that the pretreatment meets the requirements of this specification.

4.8.4 Epoxy primer. Test specimens/coupons shall be prepared with the appropriate pretreatment that is being tested. These test specimens/coupons shall be solvent wiped and cleaned prior to applying epoxy primer. Apply epoxy primer conforming to MIL-DTL-53022, type IV or MIL-DTL-53030, type II to a dry film thickness of 1.5 ± 0.2 mils (37.5 ± 5 microns). For aviation system application evaluation, MIL-PRF-23377 or MIL-PRF-85582 epoxy primers shall be used and applied to a dry film thickness of 0.6 - 0.9 mils (15 - 22.5 microns).

4.8.5 Storage stability. Along with the submission of the samples for testing, the manufacturer shall provide a statement describing the storage life of the material, the conditions for storage, and the containers for storage. Check for compliance with 3.6.1.

4.8.6 Adhesion. Prepare three (3) steel and three (3) aluminum test specimens/coupons as specified in 4.8.2 and 4.8.4. Air dry the specimens/coupons for seven (7) days. Perform adhesion testing as specified in ASTM D3359, method B and examine for compliance with 3.6.2.

4.8.7 Flexibility. Prepare a steel and aluminum test specimen/coupon as specified in 4.8.2 and 4.8.4. Air dry the specimens/coupons for seven (7) days. Bend the coated specimens/coupons over a 1/4 inch mandrel as specified in ASTM D522. Examine the coating for cracks over the area of the bend for compliance with 3.6.3.

4.8.8 Water resistance. Prepare a steel and aluminum test specimen/coupon as specified in 4.8.2 and 4.8.4. Air dry the specimens/coupons for seven (7) days. Coat all exposed unpainted metal surfaces with wax or suitable protective coating and immerse in DI water at 73 ± 2 °F (23 ± 1 °C) for 168 hours. Specimens/coupons shall be immersed at a minimum depth of 50%. At the end of the test period, remove and examine for compliance with 3.6.4.

4.8.9 Hydrocarbon fluid resistance. Prepare a steel and aluminum test specimen/coupon as specified in 4.8.2 and 4.8.4. Air dry the specimen/coupon for 168 hours and then immerse for 168 hours in a hydrocarbon fluid conforming to JP8 at 70 ± 5 °F (21 ± 3 °C). Specimens/coupons shall be immersed at a minimum depth of 50%. At the end of the test period, remove and examine for compliance with 3.6.5.

4.8.10 Corrosion testing. Prepare fifteen (15) pretreated and primed 4 x 6 inch specimens/coupons as specified in 4.8.2 and 4.8.4 for each substrate class being tested (see 4.8.2). Five (5) each prepared specimens/coupons shall be exposed to the salt spray, cyclic corrosion, and atmospheric corrosion resistance testing. Allow to air dry for seven (7) days. Coat the edges and uncoated metal surfaces with suitable coating for exposure. Tape, wax, and

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chromated coatings are unacceptable for edge and back coatings. The five (5) specimens/coupons to undergo atmospheric corrosion resistance testing shall also be CARC top-coated. For pretreatments that qualified to only class A or B, use only steel or aluminum specimens/coupons as appropriate.

4.8.10.1 Salt spray resistance. Draw five (5) specimens/coupons from 4.8.10 of each substrate class being tested and make an “X” scribe through the primer (see 4.7.7). Expose these for the number of hours as specified in table II in accordance with ASTM B117. Upon removal, wash the specimens/coupons gently in warm running water or DI water until free from any visible salt deposits and examine immediately for compliance with 3.6.6.1. When visual examination is indeterminate, the specimens/coupons shall undergo coating removal along the scribe as per ASTM D1654 and inspection for rust, pitting or corrosion not evident with paint present. Nonconformance shall constitute failure of this test.

4.8.10.2 Cyclic corrosion resistance. Draw five (5) specimens/coupons from 4.8.10 of each substrate class being and make an “X” scribe through the primer (see 4.7.7). Expose the specimens/coupons to accelerated cyclic corrosion for the number of cycles as specified in table II in accordance with GMW 14872, Ext, Exposure C. Upon removal, wash the specimens/coupons gently in warm running water or DI water until free from any visible salt deposits and examine immediately for compliance with 3.6.6.2. When visual examination is indeterminate, the specimens/coupons shall undergo coating removal along the scribe as per ASTM D1654 and inspection for rust, pitting or corrosion not evident with paint present. Nonconformance shall constitute failure of this test.

4.8.10.3 Atmospheric corrosion resistance. Draw five (5) specimens/coupons from 4.8.10 of each substrate class being tested and make an “X” scribe through the primer and top-coat (see 4.7.7). Expose these for two (2) years at a suitable test site in accordance with ASTM G50, with equivalent or greater mass loss than 1.5mpy such as the ARL site at Cape Canaveral. Specimen/coupons may be given a conditional approval after 1 year exposure for use determinate upon successful completion of the full 2 year exposure. Upon completion of the 2 year exposure, specimen/coupons shall be field evaluated, then returned to ARL for a final evaluation. Upon return, wash the specimens/coupons gently in warm running water or DI until free from any visible salt deposits and examine immediately for compliance with 3.6.6.3. When visual examination is indeterminate, the specimens/coupons shall undergo coating removal along the scribe as per ASTM D1654 and inspection for rust, pitting or corrosion not evident with paint present. Nonconformance shall constitute failure of this test.

4.8.11 Hydrogen Embrittlement. Apply pretreatment to three (3) notched round bars, type 1a.1 as described in ASTM F519 according to manufacturer’s recommended cleaning and application procedures. The notch should be masked for any mechanical cleaning procedures. Coupons shall be tested in accordance with ASTM F519 with particular reference to Annex A1. If product is found to contribute to hydrogen embrittlement, a statement to that effect will be required on packaging and relief procedures in accordance with MIL-STD-171 shall be provided with application procedure.

4.8.12 Toxic ingredients. Other than PCBTF, the manufacturer of the chemical or coating being supplied for qualification to ARL shall certify that the pretreatments contain no benzene (benzol), chlorinated solvents or ethylene based glycol ethers and their acetates. All products

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being submitted for qualification, with the exception of type VI, shall be absent of pigments containing compounds of antimony, arsenic, beryllium, cadmium, chromium, cobalt, cyanide, lead, manganese, mercury, nickel and selenium. Pigments contained in these materials shall be HAP-free. Nonconformance to 3.7 constitutes failure of this requirement.

5. PACKAGING

This section is not applicable to this specification.

6. NOTES

INFORMATION FOR GUIDANCE ONLY. (This section contains information of a general or explanatory nature that is helpful, but is not mandatory.)

6.1 Intended use. This specification is intended to provide the acceptable methods for cleaning, surface preparation, conditioning and pretreatment of metals prior to the application of the CARC and non CARC coating systems. The pretreatment methods for the metal preparation are by the use of chemical conversion coatings and corrosion inhibiting and/or adhesion promoting base coatings for subsequent military coatings. The intent for the use of the cleaning and pretreatment coatings is described in 6.1.1, 6.1.2 and 6.1.3. The cleaning methods and chemical conversion coatings covered in this specification are suitable for use as adhesion promoters and corrosion preventatives/removers for metallic substrates prior to application of paints. Organic finishing of prepared surfaces should not be delayed as contamination from ambient sources may occur causing a reduction in adhesion, compromising the corrosion resistance of the organic coating.

6.1.1 Metal surfaces. Cleaning methods are intended primarily for metal cleaning, rust removing, descaling, or surface etching purposes in conjunction with chemical pretreatment processes or a pre-primer coating prior to the application of paint. Method I (abrasive blasting) is generally recommended to remove heavy rust and mill scale on metals with thicknesses greater than 1/8 inch and to produce an even surface profile. Abrasive blasted high strength steels and corrosion resistant metals may not be coated without being given a conversion coating unless authorized by the procuring activity. 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. The cleaning processes in this specification may also be used on nonferrous surfaces. Care should be exercised to ensure that cleaning materials do not have detrimental effects on the items being cleaned. Mixed metal assemblies or fabrications will need additional attention to prevent damage or unwanted results.

6.1.3 Nonferrous substrate testing variations. Coated substrates are tested in accordance with the GMW 14872 accelerated cyclic corrosion test as specified in table II (see 4.8.10.2), then scraped after exposure and examined using the same acceptance criteria noted for the accelerated corrosion test in 3.6.6.2 of this specification, unless an accelerated corrosion test such as the

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sulfur dioxide salt spray test specified in ASTM G85 is specified in the contract. In that case, the test protocol as indicated in the contract should be used to qualify the pretreatment/primer. MIL-DTL-5541 requires some testing other than that covered in this document for aluminum and aluminum alloys.

6.1.4 Method of application. New design documents, engineering drawings and ordering data should indicate the type of coating required, cleaning requirements and methods of application when practicable.

6.1.5 Application conditions. The contractor should be cognizant of the environmental controls necessary so as not to apply any coating outside the coating manufacturer's recommendations. Organic coatings should not be applied when the surface temperature, air temperature, or relative humidity would cause a negative effect on the application and subsequent function of the applied coating. When surface temperature or air temperature are < 5 °F above dew point, the coating is adversely affected. At relative humidity in excess of 50%, a dew point calculation may be necessary to avoid the formation of moisture on the substrate to be coated. Limits on humidity during application of a coating should be determined in the guidelines set forth by the manufacturer of the coating (see table IV).

TABLE IV. Dew point.

		AMBIENT AIR TEMPERATURE (°F)										
		20	30	40	50	60	70	80	90	100	110	120
% RELATIVE HUMIDITY	90	18	28	37	47	57	67	77	87	97	107	117
	85	17	26	36	45	55	65	75	84	95	104	113
	80	16	25	34	44	54	63	73	82	93	102	110
	75	15	24	33	42	52	62	71	80	91	100	108
	70	13	22	31	40	50	60	68	78	88	96	105
	65	12	20	29	38	47	57	66	76	85	93	103
	60	11	19	27	36	45	55	64	73	83	92	101
	55	9	17	25	34	43	53	61	70	80	89	98
	50	6	15	23	31	40	50	59	67	77	86	94
	45	4	13	21	29	37	47	56	64	73	82	91
	40	1	11	18	26	35	43	52	61	69	78	87
	35	-2	8	16	23	31	40	48	57	65	74	83
30	-6	4	13	20	28	36	44	52	61	69	77	

Note 1: Dew point is the temperature at which moisture will condense on the surface.
No organic coatings should be applied unless the surface temperature of the substrate is a minimum of 5 °F above the dew point at the point of application.

Note 2: In the example shown above, if the air temperature is 70 °F and relative humidity is 65%, the dew point is 57 °F. In this example, no organic coating should be applied

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unless the surface temperature is 62 °F minimum.

6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number, revision letter, amendment, and date of this specification.
- b. Additional requirements for specific Non-CARC applications (see 1.1).
- c. Surface cleaning method, surface preparation process and metal class required (see 1.2).
- d. Whether a SDS is required with each shipment (see 3.9/3.9.1 and 4.6).
- e. Whether toxicity clearance is required (see 3.10).
- f. If/which qualification samples are required and where to send them (see 4.3 and 6.3).
- g. Sampling and inspection requirements.
- h. When certification is acceptable (see 6.3.3)

6.3 Qualification. With respect to products requiring qualification, awards will be made only for products which are, at the time of award of contract, qualified for inclusion in the applicable qualified products list whether or not such products have actually been so listed by that date. The attention of the supplier is called to this requirement, and manufacturers are urged to arrange to have the products they propose to offer to the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or orders for the products covered by this specification. The activity responsible for the qualified products list is Combat Capabilities Development Command Army Research Laboratory, ATTN: FCDD-RLW-MC, Camouflage, Coatings and Corrosion Team, 6300 Rodman Road, Building 4600, Aberdeen Proving Ground, MD 21005-5069, email: thomas.braswell2.civ@mail.mil. An online listing of products qualified to this specification may be found in the Qualified Products Database (QPD) at <https://qpd.dla.mil/>.

6.3.1 Retention of qualification. In order to retain qualification of a product approved for listing on the qualified products list (QPD), the manufacturer will verify by certification to the qualifying activity that the manufacturer's product complies with the requirements of this specification. Unless otherwise specified, the time of periodic verification by certification will be in two year intervals from the date of the original qualification, and will be initiated by the qualifying activity. No change will be made in formulation, raw materials or supplier(s) of raw materials, methods of manufacture, equipment, or geographic location without prior written Government approval. The Government reserves the right to re-examine the qualified product whenever deemed necessary to determine that the product continues to meet any or all of the specification requirements.

6.3.2 Sample identification for qualification inspection. Samples for QPD testing are to be identified in a cover letter with the following information:

- a. Manufacturer's name and product number.
- b. Submitted by (name and date).
- c. Specify the number of samples.
- d. Specify the reason for submitting the samples.
- e. Specification TT-C-490F; Method _____, Type _____, Class _____, Form _____, "Chemical Conversion Coatings and Pretreatments for Metallic Substrates (Base for Organic Coatings)".

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- f. Provide a copy of the SDS.
- g. Provide a copy of the notarized statement of composition.
- h. Provide a copy of the technical data sheet.
- i. Provide a copy of the test report.

6.3.3 Process Approval. When specified in the contract (see 4.2 and 6.2), for types I, II, and V; in lieu of submitting written procedures identifying cleaning, pretreatment and painting processes, applicators certified as specified herein are acceptable. Be prepared to offer evidence of the certification if requested.

- a. National Aerospace and Defense Contractors Accreditation program (NADCAP) Performance Review Institute (PRI) accreditation to SAE AC7108, Chemical Processing. See <https://www.eauditnet.com>
- b. The Society for Protective Coating (SSPC) Contractor Certification for SSPC QP3/AISC 420, Qualification of Paint Shops. See <https://www.sspc.org/>
- c. NACE International Institute for Contractor Accreditation Program (NIICAP) AS-1S Shop Accreditation. See <http://www.niicap.net/>
- d. ISO 9001/2015 or ISO/IEC 17025 Accreditation. See: <https://www.iso.org/home.html>.

In reviewing and validating certification by the Engineering Support Activity (ESA), applicators certified should submit their accreditation from the external certification body or registrar.

6.4 SDS. Contracting officers will identify those activities requiring copies of a completed SDS prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

6.5 Toxicity request. Department of the Army Regulation (AR) 40-5, Preventive Medicine, (AR) 70-1, Acquisition Policy, and Department of the Army Pamphlet 70-3, Acquisition Procedures, require a toxicity clearance. Army toxicity questions and/or a toxicity clearance request should be addressed to: US Army Public Health Command (USAPHC), ATTN: MCHB-IP-TTE, 5158 Blackhawk Road, APG-EA, MD 21010-5403 or emailed to usaphctepinfo@amedd.army.mil.

6.6 Cyclic Corrosion Laboratory Test. A properly calibrated cyclic chamber performing GM9540P will, after 40 cycles, cause mass loss in the range of 3712 - 4312 mg on a standardized 3.18 mm thick mass loss coupon. The target range for calibration with respect to GMW14872, Exterior, 4 sprays/cycle, Exposure C is 3660 - 4220 mg at 26 ±3 cycles on a standardized 3.18mm mass loss coupon. The congruency of the mass loss ranges given in GM9450P and GMW14872 had led ARL to decide to change the required cycles for accelerated corrosion testing from 40 to 30 cycles for GMW14872. To achieve an equal corrosive environment between the two test methods, the cycles for GMW 14872 would be 75% of the requirement that was established for GM 9540. This new requirement would be reflected into each CARC pretreatment, primer and application specification as they are amended or revised. Where the present requirement was 60 cycles, it would become 45 cycles. Where 120 cycles was referenced, it would become 90 cycles.

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6.7 Measuring paint thickness. Eddy current, magnetic, ultrasonic or other paint thickness measurement gages recognized by SSPC or NACE International are appropriate provided regular calibration is maintained.

6.8 CARC approved primers. Listed in table II are the primers that are approved for use in a CARC coatings system. Contact CCDC Army Research Laboratory for any updates to this table.

6.9 Appearance. After application of chemical conversion coating or use of one of the cleaning methods, changes in appearance are to be expected. Conversion coatings leave a visible chemical deposit. Some variation in uniformity is to be expected. Appearance should be in accordance with good commercial practices.

6.9.1 Type II coatings. The color ranges, when using an aqueous iron phosphate pretreatment, from light brown to violet to blue to gold to gray.

6.10 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.

6.11 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.

6.12 Water break testing on low surface energy substrates. Checking for cleanliness using the water break test is not appropriate for some pretreatments and previously applied primers and topcoats. Freshly applied MIL-DTL-5541, for example, will pass the water break test as described herein. However, as the film cures, the change in surface energy will present a water break test failure even though the substrate is clean. Primers and topcoats too may present a water break failure even though they are meticulously cleaned. Clean these substrates in accordance with this specification using the materials recommended by the coating supplier and use visual verification for cleanliness.

6.13 OQE. A documented statement of fact, or supporting record, either quantitative or qualitative, pertaining to the quality of an item or process, based on observations, measurements, or tests that can be verified as required within the document. Evidence will be expressed in terms of specific quality requirements or characteristics in a certified lab report. These characteristics are identified in drawings, specifications and other documents which describe the item, process or procedure.

6.14 Requirements by responsible parties. The following table is a resource that defines which party is responsible for which testing and includes the applicable paragraph numbers. Requirements for Qualification Testing are covered in table III and are the responsibility of the manufacturer and the qualifying activity.

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TABLE V. Requirements by responsible parties.

Test Name	Responsible Party	Paragraph
Process approval	Applicator	3.2.1
Preproduction approval	Applicator	3.2.2
Stress relief	Applicator	3.2.3
Qualification	Manufacturer	3.2.4
Hydrogen embrittlement	Manufacturer / Applicator	3.2.7
Visual inspection	Applicator	3.5.1
Lighting	Applicator	3.5.2
Coating weight	Applicator	3.5.3
Coating thickness	Applicator	3.5.5
Paint adhesion	Applicator	3.5.6
Accelerated corrosion	Applicator	3.5.7

6.15 Subject term (key word) listing.

Aluminum	Cleaning	Process	Type
Class	Method	Steel	

6.16 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

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APPENDIX A

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirements shall apply for Non-CARC applications unless otherwise specified by the contract.

A.1 SCOPE

A.1.1 This supplementary requirements appendix section of this document provides additional requirements for Non-CARC material applications, and is a mandatory part of the specification and are intended as additional critical requirements of the process.

A.2 APPLICABLE DOCUMENTS

ASTM INTERNATIONAL

ASTM B117	-	Standard Practice for Operating Salt Spray (Fog) Apparatus
ASTM D610	-	Standard Practice for Evaluating Degree of Rusting on Painted Steel Surfaces
ASTM D1654	-	Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments
ASTM D5486/D5486M	-	Standard Specification for Pressure-Sensitive Tape for Packaging, Box Closure, and Sealing
ASTM F519	-	Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments.

(Copies of these documents are available from <https://astm.org/>.)

A.3 REQUIREMENTS

A.3.1 Preproduction validation panels. For a single coat system, dry film thickness shall be no greater than 1.5 mils unless otherwise specified in the contract. Non-primed panels shall be checked for coating weight (see A.4.5). For zinc coated surfaces, the panels shall be made of the same material as that specified in the contract. When coated steels are used other than cold rolled SAE 1010 steel, the standard test panels shall be made of the same material as that specified in the contract.

A.3.2 Quantitative requirements. When tested as specified, the pre-treatment and/or subsequently applied coating system shall conform to the quantitative requirements in table A-1.

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TABLE A-1. Quantitative requirements of pre-treatment and/or subsequently applied coating system.

Item	Requirement Paragraph	Supplementary Requirement Paragraph	Test Paragraph
Preproduction Validation Panels	3.2.2	A.3.1	4.7.2
Hydrogen Embrittlement	3.2.7	A.3.11	A.4.14
Cleaning	3.3	3.3, A.3.5	4.7.3.1
Coating Weight	3.4.1, 3.4.5	A.3.6	A.4.5
Application of Organic Coating	3.5.4	A.3.7	N/A
Dry Film Thickness	3.5.5	A.3.1	4.7.6.1
Salt Spray Resistance	3.5.7	A.3.10	A.4.12
Total Alkali Contamination	None	A.3.5	A.4.2
Total Acid Contamination	None	A.3.5	A.4.3
Adhesion	4.7.6.2	A.3.9	A.4.11

A.3.3 Cleaning. Acid pickling shall not be used unless specifically approved or authorized by the drawing or specification for the item being processed.

A.3.4 Contamination Test. Inorganic contamination test (SSPC-Guide 15) is not required for confined, continuous systems.

A.3.5 Rinsing. Water rinses after the cleaning and phosphate operation shall be tested for contamination after every four hours of production. 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. 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.

A.3.6 Phosphate coating weight for Non-CARC.

- Type I - Zinc phosphate spray application (150 - 500 mg/ft²)
Zinc phosphate immersion or dip application (300 - 500 mg/ft²)
- Type II - Aqueous iron phosphate (35 mg/ft²).
- Type III - Organic pretreatment (unless otherwise specified, DoD-P-15328 and MIL-C-8514)
- Type V - Zinc phosphate (500 - 1100mg/ft²).

A.3.7 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

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or soiling prior to painting. The temperature of the metal surface shall be controlled to eliminate blistering, poor adhesion or unsightly film results.

A.3.8 Application of organic pretreatment coating. For organic pretreatment 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. The coating 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 after the gage has been standardized using the same surface as that over which the organic finish has been applied.

A.3.9 Paint adhesion. 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 average diameter.

A.3.10 Salt spray resistance. After pretreatment and primer (or paint for single coat systems) application, the specimens subjected to the salt fog test 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 x 6 inch test panel or equivalent area of a 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.

A.3.11 Hydrogen embrittlement relief. Adequate verification tests shall be performed for all parts having a hardness of Rockwell C39 or greater. Treated parts shall not reveal any developing cracks upon subsequent inspection (see A.4.14).

A.4 QUALITY ASSURANCE PROVISIONS

A.4.1 Total alkali and total acid contamination. Water rinses shall be tested for contamination by using total alkali and acid contamination tests after cleaning and phosphate operations every four hours during production.

A.4.2 Total alkali contamination test. Prepare a 10 mL sample of the rinse solution, dilute to 50 mL with distilled water rinse 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.

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A.4.3 Total acid contamination test. Prepare a 10 mL sample of the rinse solution, dilute to 50 mL with distilled water rinse add 5 drops of phenolphthalein indicator. Slowly add alkaline testing solution (0.1N NaOH) from a 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.

A.4.4 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.

A.4.5 Phosphate coating weight (types I, II, and V). Three test specimens, 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 A.4.9. Acceptance criteria specified in A.3.6 are determined as outlined in A.4.6, A.4.7, and A.4.8. Unless otherwise approved by the procuring agency, the following stripping procedures shall be used.

A.4.6 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 70°F (21°C). The solution can be formulated by adding 125 grams of sodium hydroxide and 125 grams of tetrasodium EDTA to 750 mL deionized water. Upon removal from 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.

A.4.7 Iron phosphate stripping (type II) for coating weight determination on steel. The phosphate shall be completely removed by immersion in a sodium hydroxide/sodium tartrate solution for 15 minutes at 70°F (21°C). 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.

A.4.8 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 70°F (21°C). 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.

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A.4.9 Coating weight formula. The coating weight is determined by the formula below:

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

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

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

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 A.3.6) 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 requirements. 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.

A.4.10 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 supplier's product technical bulletin, these additional tests shall also be performed by the contractor. Adequate rinsing shall be performed following any chemical process to remove residual material remaining from the cleaning operation and rinses shall be tested for contamination. The final rinse shall be tested, replenished, and discarded in accordance with procedures designated by the chemical suppliers. All process control tests shall be conducted every four hours during production or as specified in the contract.

A.4.11 Paint adhesion test. Unless otherwise specified by contract, a minimum of two test specimens (see 4.7.2) 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 time 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. Non-conformance to A.3.9 shall constitute failure of this test, and all items processed since last acceptance shall be rejected and corrective action shall be taken.

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A.4.12 Salt spray resistance test. Prior to initiation of production, or whenever a change in production occurs, or when required by the procuring activity, a minimum of three test specimens (see 4.7.2), shall be run through all steps of the regular production process including primer application. Specimens having a hardness of Rockwell C39 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. 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 from each day's production shall be subjected to the accelerated corrosion test specified below (see A.4.13), 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 60° on either side of the 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.

A.4.13 Ferrous surfaces. The specimens, prepared above (see 4.7.2), shall then be exposed to the 5 percent salt spray specified in ASTM B117 for a duration as specified by the contract. Within 24 to 48 hours after removal from the salt fog cabinet, the test specimen 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 A.4.12.

A.4.14 Hydrogen embrittlement relief test. Unless otherwise specified, the test shall be performed as outlined in ASTM F519 using a specimen of type 1a every 90 to 120 days.

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CONCLUDING MATERIAL

Custodians:

Army - MR
Navy - SH
Air Force - 20

Preparing Activity:

Army - MR
(Project MFFP-2019-004)

Review Activities

Army - AV, CR, MI, PT
Navy - AS, MC
Air Force - 13, 19
DLA – CQ, DH, IS, GS4
DISA - DC5

Civil agency:

GSA - FAS

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at <https://assist.dla.mil/>.