

INCH-POUND

MIL-DTL-0053030B (MR)
6 September 2006
USED IN LIEU OF
MIL-P-53030A
9 March 1992
w/AMENDMENT 2
20 August 1992

DETAIL SPECIFICATION

PRIMER COATING, EPOXY, WATER REDUCIBLE, LEAD AND CHROMATE FREE

This specification is approved for interim use by the U.S. Army Research Laboratory. Other activities in the Department of Defense may use this interim revision or may continue using MIL-P-53030A with AMENDMENT 2.

1. SCOPE

1.1 Scope. This specification covers the requirements for a water-reducible, air-drying, corrosion-inhibiting, epoxy-type primer for pretreated ferrous and non-ferrous metals. The primer is lead and chromate-free and is compatible with chemical agent resistant aliphatic polyurethane topcoats. The primer contains no more than 340 grams per liter (2.8 pounds per gallon) of volatile organic compounds (VOC), as applied.

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3, 4, or 5 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3, 4, or 5 of this specification, whether or not they are listed.

Comments, suggestions, or questions on this document should be addressed to: Director, U.S. Army Research Laboratory, Weapons and Materials Research Directorate, Materials Applications Branch, Specifications and Standards Office, Attn: AMSRD-ARL-WM-MC, Aberdeen Proving Ground, MD 21005-5069 or emailed to rsquilla@arl.army.mil . Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at http://assist.daps.dla.mil/ .
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2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

FEDERAL SPECIFICATIONS

TT-C-490	-	Chemical Conversion Coatings and Pretreatments for Ferrous Surfaces (Base for Organic Coatings).
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FEDERAL STANDARDS

FED-STD-141	-	Paint, Varnish, Lacquer and Related Materials: Methods Of Inspection, Sampling and Testing.
FED-STD-313	-	Material Safety Data, Transportation Data, and Disposal Data for Hazardous Materials Furnished to Government Activities.
FED-STD-595 Color Chip Number	-	Colors Used in Government Procurement 27722.

DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-DTL-5541	-	Chemical Conversion Coatings on Aluminum and Aluminum Alloys.
MIL-DTL-12468	-	Decontaminating Agent, STB.
MIL-PRF-23699	-	Lubricating Oil, Aircraft Turbine Engine, Synthetic Base, NATO Code Number O-156.
MIL-DTL-64159	-	Coating, Water Dispersible Aliphatic Polyurethane, Chemical Agent Resistant.
MIL-T-81772	-	Thinner, Aircraft Coating.
MIL-PRF-83282	-	Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Metric, NATO Code Number H-537.
MIL-PRF-87257	-	Hydraulic Fluid, Fire Resistant; Low Temperature, Synthetic Hydrocarbon Base, Aircraft and Missile.

(Copies of these documents are available online at <http://assist.daps.dla.mil/quicksearch/> or <http://assist.daps.dla.mil/> or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

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2.3 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

ASTM INTERNATIONAL

ASTM B117	-	Standard Practice for Operating Salt Spray (Fog) Apparatus. (DoD adopted)
ASTM D522	-	Standard Test Methods for Mandrel Bend Test of Attached Organic Coatings. (DoD adopted)
ASTM D523	-	Standard Test Method for Specular Gloss. (DoD adopted)
ASTM D610	-	Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces. (DoD adopted)
ASTM D1193	-	Standard Specification for Reagent Water. (DoD adopted)
ASTM D1210	-	Standard Test Method for Fineness of Dispersion of Pigment-Vehicle Systems by Hegman-Type Gage. (DoD adopted)
ASTM D1308	-	Standard Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes. (DoD adopted)
ASTM D1364	-	Standard Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method). (DoD adopted)
ASTM D1394	-	Standard Test Methods for Chemical Analysis of White Titanium Pigments. (DoD adopted)
ASTM D1475	-	Standard Test Method for Density of Liquid Coatings, Inks, and Related Products. (DoD adopted)
ASTM D2371	-	Standard Test Method for Pigment Content of Solvent-Reducible Paints. (DoD adopted)
ASTM D3335	-	Standard Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy. (DoD adopted)
ASTM D3363	-	Standard Test Method for Film Hardness by Pencil Test. (DoD adopted)

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ASTM D4214	-	Standard Test Methods for Evaluating the Degree of Chalking of Exterior Paint Films. (DoD adopted)
ASTM D5486/D5486M-		Standard Specification for Pressure-Sensitive Tape for Packaging, Box Closure, and Sealing. (DoD adopted)
ASTM G90	-	Standard Practice for Performing Accelerated Outdoor Weathering of Nonmetallic Materials Using Concentrated Natural Sunlight.

(Copies of these documents are available from www.astm.org or ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.)

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

3. REQUIREMENTS

3.1 Qualification. The primer furnished under this specification shall be a product which is qualified for listing on the applicable qualified products list (QPL) before contract award (see 4.2 and 6.4). 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 Color. The color of the primer shall be characteristic of titanium dioxide pigments, or no darker than light gray No. 27722 of FED-STD-595.

3.3 Toxic ingredients. The manufacturer shall certify that the primer contains no benzene (benzol), chlorinated solvents, or ethylene based glycol ethers and their acetates.

3.4 Compositions. The primer shall consist of two components, one of which shall contain a bisphenol-A type epoxy resin. Component A shall be a resin solution containing all of the corrosion inhibitors and pigments and shall be furnished in primary containers of 1-quart, 1-gallon, and 5-gallon capacities, as specified (see 6.2). Component B shall be a clear resin solution and shall be furnished in primary containers of 1/2-pint, 1-quart or 1-gallon capacities, as specified (see 6.2). The primer shall be furnished as a kit and when the components are mixed and reduced as specified by the manufacturer, a product meeting the applicable requirements of this specification shall result. The primer kit sizes shall be as specified in 3.4.1

3.4.1 Primer kit sizes. Kit sizes, as specified (see 6.2), shall be designated as 1-quart, 1-gallon, and 4-gallon, and they shall be categorized to correspond with the rated capacity of the primary containers for component A, except for the 4-gallon size. The primary container combinations for the kit sizes shall be as follows:

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1-quart primer kit	=	one, 1-quart can for component A one, 1/2-pint can for component B
1-gallon primer kit	=	one, 1-gallon can for component A one, 1-quart can for component B
4-gallon primer kit	=	one, 5-gallon can for component A one, 1-gallon can for component B

3.4.2 Primer kit contents. Each primer kit shall consist of prescribed amounts of both components, each in a separate primary container. The primary containers for the components shall be filled to such a level that when the components are mixed together according to the manufacturer's specified volumetric proportions (see 3.4), the total volume of the mixture equals the specified kit size. The filling levels for volumetric proportions of 3:1, expressed as a percent of each primary container's rated capacity, are as follows:

Kit size	Primary container size	Component	Proportion 3:1 level
1-quart	1-quart	A	75%
	1/2-pint	B	100%
1-gallon	1-gallon	A	75%
	1-quart	B	100%
4-gallon	5-gallon	A	60%
	1-gallon	B	100%

3.4.3 Pigment. The pigment portion of the primer shall conform to the percent by weight requirements of table I when tested as specified in 4.5.4.2.

TABLE I. Quantitative requirements of pigment.

Pigment	Percent by weight	
	Minimum	Maximum
Titanium dioxide	50.0	--
Zinc phosphate	10.0	20.0
Corrosion inhibiting pigment <u>1/</u>	0.9	1.1
Siliceous extenders	--	30.0
Hexavalent chromium	Negative	

1/ Sicorin RZ, BASF Wyandotte Corp. or equivalent.

3.5 Quantitative requirements. The primer shall conform to the quantitative requirements of table II when tested as specified in 4.5.

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TABLE II. Quantitative requirements.

Characteristics	Minimum	Maximum
VOC, grams volatile per liter admixed primer	--	340
Total solids:		
Percent by weight of component A	70	--
Percent by weight of component B	70	--
Lead metal, percent by weight of total solids	--	0.06
Fineness of grind	5	--
Coarse particles and skins (retained on No. 325 sieve), percent by weight of pigment	--	1.0
60° specular gloss	--	25
Pot life (thinned to spray viscosity), hours	6	--
Drying time - Set to touch, minutes	--	45
Dry hard, hours	--	2
Full hardness, hours	--	24
Full cure, days	--	7

3.6 Qualitative requirements – liquid.3.6.1 Condition in container.

3.6.1.1 Component A. When tested as specified in 4.5.7.1, component A shall be free from grit, seeds, skins, abnormal thickening or livering in a freshly opened container and shall show no more pigment settling or caking than can be easily and completely reincorporated to a smooth homogeneous state.

3.6.1.2 Component B. When tested as specified in 4.5.7.2, component B shall be clear and free from sediment and suspended matter when examined by transmitted light. It shall show no livering, curdling, gelling or skinning in a freshly opened full container.

3.6.2 Storage stability. After being tested as specified in 4.5.8, the primer shall meet all of the requirements of this specification.

3.6.2.1 Component A. A full container of component A shall show no skinning, livering, curdling, hard dry caking nor tough gummy sediment when tested as specified in 4.5.8. It shall mix readily to a smooth homogeneous state and meet all other requirements of this specification.

3.6.2.2 Component B. A full container of component B shall be clear and free from sediment and suspended matter when examined by transmitted light as specified in 4.5.8. It shall show no livering, curdling, gelling or skinning and shall meet all other requirements of this specification.

3.6.3 Accelerated storage stability. After being tested as specified in 4.5.9, the primer shall meet all of the requirements of this specification. Each component shall be free of the defects listed in

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3.6.2.1 and 3.6.2.2, and when prepared as specified in 4.5.11.1, the admixed primer shall be a smooth homogeneous mixture free from grit, seeds, lumps, and skins.

3.6.4 Freeze-thaw stability. After being tested as specified in 4.5.10, the primer shall meet all of the requirements of this specification. Each component shall be free of the defects listed in 3.6.2.1 and 3.6.2.2, and when prepared as specified in 4.5.11.1, the admixed primer shall be a smooth homogeneous mixture free from grit, seeds, lumps, and skins.

3.6.5 Mixing properties.

3.6.5.1 Mixing. When tested as specified in 4.5.11.1, a smooth homogeneous mixture shall result.

3.6.5.2 Dilution. When the admixed primer is reduced with water as specified in 4.5.11.2, there shall be no evidence of incompatibility other than that of a transient nature during the first half of water addition. The primer shall not separate into visually distinct layers in the first hour after reduction. The pot life is acceptable if the viscosity does not exceed 25 seconds in a number 2 Zahn cup after aging as specified in 4.5.11.2.

3.6.6 Spraying properties. When tested as specified in 4.5.12, the primer shall spray satisfactorily in all respects and shall show no running, sagging, or streaking. The dried film shall show no dusting, mottling, or color separation and shall present a smooth finish free from seeds.

3.7 Qualitative requirements - dried film.

3.7.1 Knife test. A film of primer, tested as specified in 4.5.13, shall adhere tightly to the test panel. It shall be difficult to furrow off with the knife and shall not flake, chip or powder. The knife cut shall show beveled edges.

3.7.2 Flexibility. A film of primer tested as specified in 4.5.14 shall withstand bending without cracking or flaking.

3.7.3 Wet adhesion. A film of primer tested as specified in 4.5.15 shall show no removal of the primer by the tape beyond one-sixteenth inch on either side of the scored lines.

3.7.4 Water resistance. A film of primer, tested as specified in 4.5.16, shall show no wrinkling or blistering immediately after removal of the panel from the water. The primer shall be no more than slightly softened when examined 2 hours after removal. After 24 hours air drying, the portion of the panel which was immersed shall be practically the same with regard to hardness and adhesion compared to the portion which was not immersed.

3.7.5 Hydrocarbon fluid resistance. A film of primer, tested as specified in 4.5.17, shall show no blistering or wrinkling and no more than a slight yellowing or softening upon removal from the fluid. After 2 hours air drying, the portion of the panel that was immersed shall be practically the same with regard to hardness, color and gloss from a panel prepared at the same time but not immersed.

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3.7.6 Salt spray resistance. A film of primer tested as specified in 4.5.18 and examined immediately after removal from the salt spray test shall show no more than a trace of rusting (ASTM D610, table I, rust grade 9) or corrosion, and no more than five scattered blisters, none larger than 1 mm in diameter. Upon removal of the primer, there shall be no more than a trace of rusting, pitting, or corrosion on the panels.

3.7.7 Fluid resistance. A film of primer tested as specified in 4.5.19 shall show no blistering, wrinkling or loss of adhesion immediately after removal. Slight initial softening is acceptable. After a four hour recovery period, the hardness of the primer shall be fully recovered. Discoloration of the primer is acceptable and shall not be cause for rejection.

3.7.8 Recoating. A film of primer, tested as specified in 4.5.20, shall show no blistering, wrinkling or other evidence of lifting. The topcoat shall adhere tightly to the primer, and the primer shall adhere tightly to the panel when cut with the knife blade.

3.7.9 Weather resistance. Films of the primer prepared and exposed as specified in 4.5.21 shall show no rusting, cracking, checking, flaking, or loss of adhesion. Primer that has a topcoat coating with Green 383, 34094 as specified in MIL-DTL-64159 type II shall show no more than light chalking (see ASTM D4214). Upon removal of the coating system, the surface of the metal shall show no more than a trace of rusting, pitting, or corrosion (ASTM D610, table I, rust grade 9).

3.7.10 Super tropical bleach (STB) resistance. When tested as specified in 4.5.22, a film of the coating shall show no blistering, wrinkling, or film softening when examined immediately after washing with water. Film softening shall not exceed a 2 pencil hardness difference (see ASTM D3363) from an unexposed film with identical cure history prior to STB exposure. After drying, there shall be a maximum color change of 2.5 NBS units when comparing a portion of the untested panel to that of the tested area. The STB composition shall be in accordance with MIL-DTL-12468.

3.8 User instruction marking and precaution sheet. All primary containers shall be legibly labeled "Component A (Pigmented Base Component)" or "Component B (Curing Component)" as applicable, with the manufacturer's mixing and thinning instructions, the VOC content (in grams per liter) and the following:

PRECAUTION: The Surgeon General requires airline respirators to be used unless air sampling shows exposure to be below standards. Then, either chemical cartridge respirators or airline respirators are required. Avoid contact with skin and eyes. Use with adequate ventilation. For other safety recommendations, refer to the material safety data sheet (MSDS). Keep containers closed.

INSTRUCTIONS FOR USE:

- a. The surface to be primed shall be clean and free of oil and dust.
- b. Apply over pretreated metal.
- c. Equipment shall be adequately grounded. Clean spray equipment immediately after use.
- d. The primer from one vendor, or component thereof, shall never be mixed with that of another vendor.

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MIX ONLY THAT AMOUNT TO BE USED IN 6 HOURS.

3.9 Toxicity clearance. All new chemicals and materials being added to the Army supply system shall have a toxicity clearance. A toxicity clearance involves a toxicological evaluation of materials prior to introduction into the Army supply system. The Army 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).

3.10 Material safety data sheet (MSDS). A MSDS shall be prepared for the primer in accordance with FED-STD-313 and forwarded to the qualifying activity (see 6.4.2). The MSDS 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.

4. VERIFICATION

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

- a. Qualification inspection (see 4.2).
- b. Conformance inspection (see 4.3)

4.2 Qualification inspection. Qualification shall be conducted by the Qualifying activity (see 6.4). The qualification test sample shall consist of four quarts of the paint. The samples shall be legibly identified (see 6.4.3). Qualification inspection shall consist of tests for all requirements specified in section 3 in accordance with table III, and examination for user instruction marking (see 3.8). The results of each test shall be compared with the applicable requirement in section 3. Failure to conform to any requirement shall be counted as a defect, and paint represented by the sample test shall not be approved for inclusion on the qualified products list (QPL) under this specification.

4.3 Conformance inspection. The contracting officer shall require that an appropriate sample from each production lot (see 4.3.1) be forwarded to the U.S. Army Research Laboratory, ATTN: AMSRD-ARL-WM-MC (Coatings Team), Building 4600, Deer Creek Loop, Aberdeen Proving Ground, MD 21005-5069. Conformance inspection for individual lots shall be VOC, condition in container, total solids, fineness of grind, mixing properties, spraying properties, drying time, and 60 degree specular gloss. There shall be no failures (see 6.6).

4.3.1 Lot and batch formation. Unless otherwise specified in the contract or purchase description (see 6.2), a lot shall consist of all coatings of the same type, composition and color, from a single uniform batch, produced and offered for delivery at one time (see 6.4.4). Unless otherwise specified in the contract or purchase description (see 6.2), a batch shall consist of all coating material (in U.S. gallons) manufactured during one continuous operation and forming part of one contract or order for delivery (see 6.4.4). The addition of any substance to a batch shall constitute a new lot.

4.4 Inspection conditions. Unless otherwise specified, sampling, inspection and testing shall be in accordance with section 1000 of FED-STD-141.

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4.4.1 Material safety data sheet (MSDS). The MSDS shall address all components of the primer and be in compliance with the requirements of FED-STD-313. Nonconformance to 3.10 shall constitute failure of this requirement.

4.5 Test methods.

4.5.1 Test conditions. The testing conditions shall be in accordance with FED-STD-141, section 9 or in accordance with the appropriate ASTM method except as otherwise specified herein. Failure of any test result to fall within the ranges specified in 3.2 through 3.7, as applicable, shall constitute failure of the applicable test.

4.5.2 Test panels. Steel test panels shall be pretreated with a zinc phosphate coating in accordance with TT-C-490 type I. Aluminum test panels shall be aluminum alloy 3003H14 treated with alodine 1200S to produce a coating meeting the requirements of MIL-DTL-5541.

4.5.2.1 Primer preparation and application. For all tests requiring the use of admixed primer, components A and B shall be thoroughly mixed separately, combined as specified in 4.5.11.1, and thinned with water according to the manufacturer's instructions to a spraying viscosity of approximately 20 seconds in a number 2 Zahn cup. The primer shall be sprayed to a dry film thickness of 0.0009 to 0.0011 inches (0.02286 to 0.02794 mm).

4.5.3 Test procedures. Tests (see table III), shall be in accordance with FED-STD-141, ASTM, or as specified herein. The right is reserved to make any additional tests deemed necessary to determine that the primer meets the requirements of this specification.

4.5.4 Analysis of component A.

4.5.4.1 Nonvolatile (total solids) content. Place a portion of the thoroughly mixed sample in a dropping bottle and weigh to the nearest one-tenth mg. Weigh one 60 mm aluminum dishes to the nearest one-tenth mg. Transfer a small sample that does not exceed 0.3g to the dish, determine its exact weight to the nearest one-tenth mg by loss in weight of the bottle. Dissolve the sample in 2 mL of A.C.S. reagent grade ethanol and dry in a gravity convection oven at 221 °F (105 °C) for 1 hour. Upon cooling, reweigh the dish to the nearest one-tenth mg. From the weight of the residue in the dish and the weight of the sample taken, calculate the percent nonvolatile (total solids) as required. Nonconformance to the requirements in table II shall constitute failure of this test.

4.5.4.2 Pigment analysis. Extract the pigment as in ASTM D2371, but use ethanol for extraction. Make appropriate qualitative and quantitative tests on the extracted pigment to determine if only permissible pigments were used. Nonconformance to 3.4.3 shall constitute failure of this test.

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TABLE III. Index.

ITEM	FED-STD-141 Method	ASTM Method	Test Paragraph	Requirement Paragraph
Pigment analysis	--	--	4.5.4.2	3.4.3
Extraction of pigment	--	D2371	4.5.4.2	Table I
Titanium dioxide	--	D1394	4.5.4.2.1	Table I
Zinc phosphate	--	--	4.5.4.2.2	Table I
Acid insoluble	--	--	4.5.4.2.3	Table I
Hexavalent chromium	--	--	4.5.4.2.4	Table I
Fineness of grind	--	D1210	--	Table II
Coarse particles and skins	--	--	--	Table II
Total solids	--	--	4.5.4.1	Table II
Lead metal	--	D3335	4.5.4.3	Table II
Drying time	4061.3	--	4.5.6.1	Table II
60° specular gloss	--	D523	4.5.6.2	Table II
Volatile organic compounds (VOC)	--	--	4.5.6.3	Table II
Condition in container	--	--	4.5.7	3.6.1
Component A	3011.3	--	4.5.7.1	3.6.1.1
Component B	4261.1	--	4.5.7.2	3.6.1.2
Storage stability	--	--	4.5.8	3.6.2
Component A	3011.3	--	4.5.8	3.6.2.1
Component B	4261.1	--	4.5.8	3.6.2.2
Accelerated storage stability	--	--	4.5.9	3.6.3
Freeze-thaw stability	--	--	4.5.10	3.6.4
Mixing properties	--	--	4.5.11	3.6.5
Spraying properties	4331.2	--	4.5.12	3.6.6
Knife test	6304.2	--	4.5.13	3.7.1
Flexibility	--	D522 Method B	4.5.14	3.7.2
Wet adhesion	6301.3	--	4.5.15	3.7.3
Water resistance	--	D1308	4.5.16	3.7.4
Hydrocarbon fluid resistance	--	--	4.5.17	3.7.5
Salt spray resistance	--	B117	4.5.18	3.7.6
Fluid resistance	--	--	4.5.19	3.7.7
Recoating	--	--	4.5.20	3.7.8
Weather resistance	--	--	4.5.21	3.7.9
STB resistance	--	--	4.5.22	3.7.10

4.5.4.2.1 Quantitative titanium dioxide. Determine titanium dioxide content by ASTM D1394. Nonconformance to the requirements in table I shall constitute failure of this test.

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4.5.4.2.2 Zinc phosphate content. Determine the zinc phosphate content in accordance with 4.5.4.2.2.1 and 4.5.4.2.2.2.

4.5.4.2.2.1 Determination of zinc.

4.5.4.2.2.1.1 Reagents.

- a. Buffer solution (pH 10): 350 mL conc. NH_4OH + 54g NH_4Cl + H_2O to give 1000 mL.
- b. Eriochrome black T (0.5%) 0.25g eriochrome black T + 2.2g hydroxylamine hydrochloride per 50 mL methanol solution.
- c. Primary standard zinc oxide (0.200N): Accurately weigh 4.069g of oven-dried ZnO . Dissolve it in 250 mL of the buffer solution and dilute to 500.0 mL.
- d. 0.5N Disodium ethylenediaminetetra-acetate dihydrate (EDTA): 37.2g EDTA per liter aqueous solution.

4.5.4.2.2.1.2 Procedure.

- a. Accurately weigh approximately 1.0 gram of pigment into a 250 mL glass-stoppered Erlenmeyer flask.
- b. Add 25 mL of buffer, stopper, and shake vigorously every few minutes over a period of 30 minutes.
- c. Filter through fine paper into a 400 mL beaker, washing well with water until 200 mL of filtrate are collected.
- d. Add 20.0 mL of the EDTA (an excess) to the filtrate.
- e. Add 10 drops of eriochrome black T.
- f. Titrate with standard ZnO to a wine-red end point (V_s).
- g. Run a blank by titrating 20.0 mL of the EDTA in 200 mL of an aqueous solution containing 25 mL of the buffer (V_b).

4.5.4.2.2.1.3 Calculations.

$$\text{percent Zn} = ((V_b - V_s) \times 0.2 \times 3.269) / (\text{Sample wt.})$$

$$\text{percent zinc phosphate} = ((V_b - V_s) \times 0.2 \times 7.035) / (\text{Sample wt.})$$

Where: V_b = Milliliters of ZnO for blank and
 V_s = Milliliters of ZnO for sample.

4.5.4.2.2.2 Determination of phosphate.

4.5.4.2.2.2.1 Reagents.

- a. Conc NH_4OH
- b. Conc HNO_3
- c. NH_4NO_3

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- d. Ammonium molybdate - Johnson's formula: Mix 55g of $(\text{NH}_4)_2\text{MoO}_7 \cdot 4\text{H}_2\text{O}$ and 50g of NH_4NO_3 with 18 mL of concentrated NH_4OH and 20 mL H_2O . Stir. Dilute to about 700 mL with H_2O , heat with occasional stirring until all salts have dissolved. Dilute to 1000 mL. Let stand overnight. Filter through fine paper but do not wash the residue.

4.5.4.2.2.2.2 Procedure.

- a. Accurately weigh approximately 2g of pigment into a 250 mL glass stoppered Erlenmeyer flask.
- b. Add 25 mL of conc NH_2OH , stopper and shake vigorously every few minutes over a period of 60 minutes.
- c. Add 25 mL of H_2O and filter through fine paper into a 400 mL beaker, washing well with water.
- d. Neutralize the filtrate with 7.5 HNO_3 (requires about 35 mL).
- e. Add 15 mL conc HNO_3 and 6g of NH_4NO_3 . Stir.
- f. Heat the clear solution to 176 °F (80 °C) maximum and add 75 mL of ammonium molybdate with constant stirring.
- g. Stir for several minutes and let the precipitate settle for 2 hours.
- h. Filter through a tared crucible (gooch or medium glass), transfer the precipitate, and wash with 1 percent HNO_3 (5 mL conc. HNO_3 per 500 mL solution). The washing shall be thorough.
- i. Give the collection precipitate a final wash with a small amount of water.
- j. Dry the crucible for 2 hours in a 221 °F (105 °C) oven.
- k. Cool crucible in a desiccator and determine the weight of the precipitate to the nearest one-tenth mg (it shall not exceed 3g; if it does, repeat the determination with a smaller sample).

4.5.4.2.2.2.3 Calculations.

$$\text{percent PO}_4 = (\text{wt. ppt.} \times 5.029) / (\text{Sample wt.})$$

$$\text{percent zinc phosphate} = (\text{wt. ppt.} \times 11.18) / (\text{Sample wt.})$$

4.5.4.2.2.2.4 Failure criteria. Nonconformance to table I shall constitute failure of this test.

4.5.4.2.3 Acid insoluble. Determine matter insoluble in acid in the extracted pigment in accordance with the appropriate test method. Nonconformance to the extender requirement of table I shall constitute failure of this test.

4.5.4.2.4 Hexavalent chromium (Cr6+ shall be absent).

- a. Reagents: 25 percent aqueous KOH
- b. Procedure:
 - (1) Add 5 mL of 25 percent aq. KOH to 1/2g of the extracted pigment contained in a 15 mL centrifuge tube.
 - (2) Agitate by shaking the tube for a few minutes, then centrifuge.

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- (3) The supernatant liquid shall be colorless. A yellow color indicates presence of chromate. Nonconformance to the requirement in table I shall constitute failure of this test.

4.5.4.3 Lead content.

4.5.4.3.1 Atomic absorption spectroscopy. Determine the percent of lead in accordance with ASTM D3335. Nonconformance to table II shall constitute failure of this test.

4.5.4.3.2 X-ray emission spectrometric analysis (alternate method).

4.5.4.3.2.1 Test panel preparation. Using 100 grams of a known lead free coating which meets all of the requirements for this primer, prepare standard aliquots containing 0.00, 0.03, 0.06, and 0.09 percent lead metal, based on total nonvolatile paint, by adding calculated amounts of lead naphthenate of a known lead content. Thoroughly mix the aliquots to incorporate the lead and draw down the standards and primer to be tested on duplicate black and white Morest cards using a 0.0020 inch (0.004 inch gap clearance) film applicator. Dry for 48 hours at a temperature of 73.4 ± 2 °F (23 ± 1.1 °C), a relative humidity of 50 ± 4 percent, and under dust free conditions. Cut the drawdowns into a suitable size and shape to fit the sample holder of the X-ray fluorescence spectrometer.

4.5.4.3.2.2 X-ray analytical procedure. Lead content shall be determined using an X-ray fluorescence spectrometer came of determining lead content at a minimum level of 0.03 percent by weight of the total nonvolatile paint. The parameters of angle, crystal, pulse height selection, counting time, collimator, X-ray tube, voltage and amperage, shall be established for a wave length dispersive fluorescence spectrometer according to conventional X-ray analytical procedures. The analytical line Pb L-alpha or Pb L-beta shall be used. To calibrate, place the known standards in the X-ray unit and measure the count rates of lead, lead background and the Compton scattered background from the X-ray tube. The ratio R, of net lead intensity and Compton scattered background is calculated as follows:

$$R = ((I_{Pb} (I_{Pb} \text{ Background I} + I_{Pb} \text{ Background II})) / 2) / I_{\text{Compton Line}}$$

Where I = Gross Intensity and the background is taken on each side of the Pb line.

Establish a lead calibration curve using these results. Determine the lead content of the test paint using the above procedure and calibration curve. When using an energy dispersive fluorescence spectrometer, it shall be set up in accordance with the manufacturer's manual. Nonconformance to table II shall constitute failure of this test.

4.5.5 Analysis of component B.

4.5.5.1 Nonvolatile (total solids) content. Determine the percent nonvolatile (total solids) as specified in 4.5.4.1, except that toluene shall be used to disperse the material. Nonconformance to table II shall constitute failure of this test.

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4.5.6 Analysis of admixed primer.

4.5.6.1 Drying time. Prepare a film of the primer on steel as specified in 4.5.2.1 and determine drying time in accordance with FED-STD-141, method 4061.3 under referee conditions. The film has reached full hardness when it is very difficult to remove with a knife blade. Nonconformance to table II shall constitute failure of this test.

4.5.6.2 Specular gloss (60 degrees). Prepare a film of the primer on steel as specified in 4.5.2.1 and allow to dry 48 hours. Determine the 60 degree specular gloss in accordance with ASTM D523. Nonconformance to table II shall constitute failure of this test.

4.5.6.3 Volatile organic compounds (VOC). Separately mix components A and B, and then combine them as specified in 4.5.11.1, but do not thin the mixture. Keep the container tightly covered.

4.5.6.3.1 Nonvolatile (total solids) content. Determine the weight percent total solids, X_m , of the admixed primer in accordance with the procedure specified in 4.5.4.1.

4.5.6.3.2 Density. Determine the density, D_m , of the admixed primer in grams per liter in accordance with ASTM D1475.

4.5.6.3.3 Water content. Determine the weight percent water, X_w , of the admixed primer in accordance with ASTM D1364.

4.5.6.3.4 Calculation.

$$\text{VOC (grams per liter)} = (D_m(100 - X_m - X_w)) / (100 - X_w \times D_m/997).$$

4.5.6.3.5 Failure criterion. Nonconformance to table II shall constitute failure of this test.

4.5.7 Condition in container.

4.5.7.1 Component A. Determine package condition of component A for acceptance testing in accordance with FED-STD-141, method 3011.3. For qualification testing, evaluate pigment settling or caking in accordance with FED-STD-141, method 3011.3, and then stir by hand for five minutes. The presence of any gel particles or undispersed pigment after stirring indicates unsatisfactory settling properties. Nonconformance to 3.6.1.1 shall constitute failure of this test.

4.5.7.2 Component B. Determine package condition of component B in accordance with FED-STD-141, method 4261.1. Nonconformance to 3.6.1.2 shall constitute failure of this test.

4.5.8 Storage stability. Allow unopened containers of component A and component B to stand undisturbed for one year at 72 to 80 °F (22 to 27 °C) and then examine the contents. Evaluate the condition of each component as specified in 4.5.7, then mix as specified in 4.5.11.1 and examine the admixed primer. Nonconformance to 3.6.2 shall constitute failure of this test.

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4.5.9 Accelerated storage stability. Allow tightly sealed glass jars filled with each component to stand undisturbed for 7 days at 140 °F (60 °C). Allow to cool to room temperature and examine the contents. Mix the components as specified in 4.5.11.1. Nonconformance to 3.6.3 shall constitute failure of this test.

4.5.10 Freeze-thaw stability. Subject tightly sealed containers of each component to a 4 day cycle of 16 hours at 10 °F (-12 °C) and 8 hours at room temperature. After completion, examine the contents separately, and then mix as specified in 4.5.11.1. Nonconformance to 3.6.4 shall constitute failure of this test.

4.5.11 Mixing properties.

4.5.11.1 Mixing. Thoroughly stir component A by hand until uniform. Unless otherwise specified, mix one volume of component B with three volumes of component A. Nonconformance to 3.6.5.1 shall constitute failure of this test.

4.5.11.2 Dilution. Reduce the admixed primer with water meeting the type IV requirements of ASTM D1193 to a spraying viscosity of 20 seconds in a number 2 Zahn cup. Stir or shake well. Allow to stand undisturbed for at least 6 hours to check the pot life requirement. Nonconformance to 3.6.5.2 shall constitute failure of this test.

4.5.12 Spraying properties. Prepare a film of the primer on steel as specified in 4.5.2.1. Observe for spraying properties as in FED-STD-141, method 4331.2. For referee test, use the automatic application per FED-STD-141, method 2131.2. Nonconformance to 3.6.6 shall constitute failure of this test.

4.5.13 Knife test. Spray the primer as specified in 4.5.2.1 on one steel and one aluminum panel. Allow to dry for 168 hours. Perform the knife test in accordance with FED-STD-141, method 6304.2. Nonconformance to 3.7.1 shall constitute failure of this test.

4.5.14 Flexibility. Spray a film of primer to a dry film thickness between 0.0009 and 0.0011 inch (0.02286 mm to 0.02794 mm) on smooth finish steel panel prepared as specified in 4.5.12, using the aliphatic naphtha-propylene glycol monomethyl ether mixture. The panel shall be prepared from new cold rolled carbon steel, rust-free, 0.009 to 0.011 inch (32 gauge) thick with a Rockwell 15-T maximum hardness of 82 and finished with a surface roughness of 8 to 12 microinches. Allow the panel to air dry 168 hours. Bend the coated panels according to ASTM D522, method B. Examine the coating for cracks over the area of the bend for compliance with 3.7.2. Nonconformance to 3.7.2 shall constitute failure of this test.

4.5.15 Wet adhesion. Prepare two panels as specified in 4.5.13. Allow to dry for 168 hours. Immerse in distilled water at room temperature for 24 hours. Test for wet adhesion in accordance with FED-STD-141, method 6301.3, except that the tape used shall meet the requirements of ASTM D5486/D5486M, type III. Nonconformance to 3.7.3 shall constitute failure of this test.

4.5.16 Water resistance. Prepare two panels as specified in 4.5.13. Allow to dry for 168 hours. Coat all exposed, uncoated metal surfaces with wax or other suitable coating. Immerse the panels

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in distilled water at room temperature for 168 hours in accordance with ASTM D1308. Nonconformance to 3.7.4 shall constitute failure of this test.

4.5.17 Hydrocarbon fluid resistance. Prepare films of primer as specified in 4.5.13. Air dry 168 hours. Do not wax or coat the exposed metal surfaces. Immerse the panels for 168 hours in a hydrocarbon fluid conforming to JP8 at 77 ± 2 °F (25 ± 1 °C). Panels shall be immersed at a minimum depth of 50 %. At the end of the test period, remove and examine for compliance with 3.7.5. Nonconformance to 3.7.5 shall constitute failure of this test.

4.5.18 Salt spray resistance. Prepare four, 4 x 12 inch panels each of pretreated steel and aluminum as specified in 4.5.2.1. Air dry for 168 hours. Coat edges and uncoated metal surfaces with wax or other suitable coating, but do not score. Expose the panels to 5 percent salt spray for 336 hours as specified in ASTM B117. Remove the panels, wash gently in running water no warmer than 100 °F (38 °C) until free from any visible salt deposits. Examine immediately for compliance with 3.7.6. Strip the primer from the panels with a suitable paint remover. Inspect the panels for rust, pitting or corrosion. Nonconformance to 3.7.6 shall constitute failure of this test.

4.5.19 Fluid resistance. Prepare two panels as specified in 4.5.13. Allow to dry for 168 hours. Immerse in lubricating oil conforming to MIL-PRF-23699 at 250 °F (121 °C) for 24 hours. Also test sets of panels using hydraulic fluids conforming to MIL-PRF-87257 and MIL-PRF-83282 at 150 °F (66 °C). Nonconformance to 3.7.7 shall constitute failure of this test.

4.5.20 Recoating. Prepare three steel panels as specified in 4.5.12. Allow the primer to dry 2, 24 and 168 hours respectively, then spray each panel with a topcoat of camouflage green 383 polyurethane conforming to MIL-DTL-64159 type II. If necessary, the polyurethane shall be thinned according to the specification with thinner conforming to MIL-T-81772 and sprayed to a dry film thickness of 0.0018 to 0.0022 inches (0.04572 to 0.05588 mm). After the topcoat has dried for 24 hours, examine the panels for evidence of lifting. After the topcoat has dried for 168 hours, determine the intercoat adhesion using the knife test as in FED-STD-141, method 6304.2. Nonconformance to 3.7.8 shall constitute failure of this test.

4.5.21 Weather resistance. Prepare two steel panels as specified in 4.5.12. After drying for 24 hours, topcoat all primed panels with Green 383, 34094 as specified in MIL-DTL-64159 type II to a dry film thickness of 0.0020 ± 0.0002 inch (0.0508 ± 0.00508 mm). Allow to air dry for a minimum of 7 days and record color and 60° gloss readings for each panel. Panels shall be placed outdoors, for the equivalent of 560 MJ/m² of total UV irradiance, in an accelerated outdoor exposure according to ASTM G90. At 70 MJ/m² intervals examine the panels for compliance with 3.7.9. Determine chalking according to ASTM D4214. Wash the panels with a warm soap solution using a soft sponge or cloth, rinse, dry and examine for color change at each interval. The exposure racks shall be angled at a latitude of 33° 23' North and 112° 35' West. Nonconformance to 3.7.9 shall constitute failure of this test.

4.5.22 Super tropical bleach (STB) resistance. Prepare a film of primer on steel as specified in 4.5.12. Scribe a 1 inch diameter wax ring using a china marker on the painted surface of the panel. Place approximately 1 ml of STB agent on the panel surface. Do not cover. Allow to

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stand 30 minutes then thoroughly wash with water. An STB slurry mix of 40 parts STB and 60 parts water by weight shall be used. Examine for compliance with 3.7.10.

4.5.23 Toxic ingredients. The manufacturer shall certify that the primer contains no benzene (benzol), chlorinated solvents, or ethylene based glycol ethers and their acetates. Nonconformance to 3.3 shall constitute failure of this requirement.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Service or Defense Agency, or within the military service's system command. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

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

6.1 Intended use. The primer described in this specification is intended for use on clean, chemically pretreated metal surfaces where exposure to lead or chromate pigments is not permitted. It is a water reducible epoxy primer with maximum VOC content of 340 grams per liter (2.8 pounds per gallon) and is compatible with chemical agent resistant aliphatic polyurethane topcoats.

6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number, and date of this specification.
- b. Size of containers for Component A and Component B (see 3.4).
- c. Kit size (see 3.4.1)
- d. If a toxicity clearance is required (see 3.9).
- e. If qualification samples are required and where to send them (see 4.2 and 6.4).
- f. If conformance samples are required and where to send them (see 4.3).
- g. Lot and batch formation (see 4.3.1).
- h. Packaging requirements (see 5.1).
- i. Whether material safety data sheets (MSDS) are required with each shipment (see 6.4.2).

6.3 Basis of purchase. The primers covered by this specification should be purchased by volume, the unit being a kit comprised of 1 quart of 57.75 cubic inches or 1 gallon of 231 cubic inches. The kit components need not be the same size.

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6.4 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 Qualified Products List QPL-53030, whether or not such products have actually been so listed by that date. The attention of contractors is called to this requirement and manufacturers are urged to arrange to have the products that they propose to offer to the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or purchase orders for the products covered by this specification. Samples for qualified products list (QPL) testing (see 4.2) should be submitted to the U.S. Army Research Laboratory, ATTN: AMSRD-ARL-WM-MC (Coatings Team), Building 4600, Deer Creek Loop, Aberdeen Proving Ground, MD 21005-5069.

6.4.1 Retention of qualification. In order to retain qualification of a product approved for listing on the qualified products list (QPL), 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.

6.4.2 Material safety data sheet (MSDS). The contracting activity should be provided a material safety data sheet at the time of contract award. The MSDS should be provided in accordance with OSHA section 1910.1200, 29 CFR Chapter XVII and found as part of FED-STD-313. OSHA section 1910.1200 requires reporting threshold criteria for known or suspected human carcinogens on MSDS 0.1 percent or greater, and 1 percent or greater for other health hazards. The MSDS should be included with each unit of issue of material covered by the specification, when specified (see 6.2). Contracting officers will identify those activities requiring copies of completed material safety data sheets prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

6.4.3 Sample identification for qualification inspection. Samples for qualified products list (QPL) testing are to be identified in a cover letter with the following information:

- Manufacturer's name and product number.
- Submitted by (name and date).
- Specify the number of samples.
- Specify the reason for submitting the samples.
- Specify the specification number MIL-DTL-0053030B.
- Provide a copy of the material safety data sheet (MSDS).
- Provide a copy of the statement of composition.
- Provide a copy of the technical data sheet.
- Provide a copy of the test report.

6.4.4 Conformity to qualified sample. All lots of coatings supplied under this specification must be manufactured using the same formulation, raw materials and supplier(s) of raw materials,

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methods of manufacture, equipment, and geographic location as the qualification sample, unless changes have been approved by the qualifying activity.

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: Commander, US Army Center For Health Promotion And Preventive Medicine (MCHB-TS-T), 5158 Blackhawk Road, Aberdeen Proving Ground, MD 21010-5403.

6.6 Conformance rejection and retest. Failure in any conformance inspection will result in the rejection of the batch from which it was obtained. Rejected material cannot be resubmitted for acceptance without written approval from the qualification activity (see 4.3). The application for resubmission will contain all details concerning previous rejections and measures taken to correct these deficiencies.

6.7 Detail specification. MIL-DTL-53072, Chemical Agent Resistant Coating (CARC) System Application Procedures and Quality Control Inspection, is available for application procedures and quality control inspection of this coating.

6.8 Subject term (key word) listing.

Camouflage
CARC
Corrosion inhibiting
Lead and chromate free
Primer
VOC compliant
Water reducible

6.9 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.

CONCLUDING MATERIAL

Custodian:
Army - MR

Preparing activity:
Army - MR

Project 8010-2006-011

Review activities:
Army - AR, AT, CR, EA, MD1, MI

Civil agency:
GSA/FSS – 6FEE

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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 <http://assist.daps.dla.mil/>.