INCH-POUND

MIL-DTL-0053022C (MR)
25 January 2007
USED IN LIEU OF
MIL-P-53022B
1 June 1988

DETAIL SPECIFICATION

PRIMER, EPOXY COATING, CORROSION INHIBITING 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-53022B.

1. SCOPE

1.1 Scope. This specification covers a flash drying, corrosion inhibiting epoxy primer for ferrous and nonferrous metals. The primer is lead and chromate free and meets the air pollution requirements for solvent emissions (see 1.2 and 6.1).

1.2 Types. The coating will be furnished in the following types as specified (see 6.2):

Type I - Lead and chromate-free formulation to meet Rule 102, South Coast Air Quality Management District (see 3.3.2.3.1).

Type II - High solids, lead and chromate free formulation to meet a maximum volatile organic compound content of 420 grams/liter (3.5 pounds/gallon) as packaged (see 4.12).

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/.

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).

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	-	Colors Used in Government Procurement 26622.

DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-DTL-5541	-	Chemical Conversion Coatings on Aluminum and Aluminum Alloys.
MIL-DTL-12468	-	Decontaminating Agent, STB.
MIL-DTL-64159	-	Coating, Water Dispersible, Aliphatic Polyurethane, Chemical Agent Resistant.
MIL-T-81772	-	Thinner, Aircraft Coating.

(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.)

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

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY (EPA)

EPA Method 311	-	Analysis of Hazardous Air Pollutant Compounds in Paints and Coatings by Direct Injection into a Gas Chromatograph.

(Copies of these documents are available online at www.epa.gov/ttn/emc/ or from the Environmental Protection Agency, Ariel Rios Building, 1200 Pennsylvania Avenue, N.W., Washington, DC 20460.)

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 D185	-	Standard Test Methods for Coarse Particles in Pigments, Pastes, and Paints. (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 D562	-	Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer. (DoD adopted)
ASTM D610	-	Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces. (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 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 D1652	-	Standard Test Method for Epoxy Content of Epoxy Resins. (DoD adopted)
ASTM D2369	-	Standard Test Method for Volatile Content of Coatings. (DoD adopted)
ASTM D2371	-	Standard Test Method for Pigment Content of Solvent-Reducible Paints. (DoD adopted)
ASTM D2698	-	Standard Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging. (DoD adopted)
ASTM D3272	-	Standard Practice for Vacuum Distillation of Solvents From Solvent-Reducible Paints for Analysis. (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)
ASTM D3960	-	Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings. (DoD adopted)
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.)

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

Rule 102 – Definition of Terms: Photochemically Reactive Solvent.

(Copies of this document are available from www.aqmd.gov/aqmd/Interfaces/onsiteservices.html or South Coast Air Quality Management District, 21865 Copley drive, Diamond Bar, CA 91765.

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 6.3). 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 Color characteristics. The color of the primer shall be characteristic of titanium dioxide pigments or a light grey not darker than color chip number 26622 of FED-STD-595.

3.3 Composition. The primer shall be furnished in 2 parts: part A (a pigmented epoxy resin component) in 1-quart, 1-gallon, 4-gallon or 5-gallon primary containers (see 6.2), and part B (catalyst components) in 1/2-pint, 1-quart or 1-gallon primary containers (see 6.2). When mixed four parts by volume of part A to one part by volume of part B, a product meeting the applicable requirements of this specification shall result.

3.3.1 Pigment. The pigment portion of part A shall conform to the percent by weight requirements specified in table I. When the part A of the primer is tested in accordance with section 4, the analysis shall conform to the requirements of table II. Zinc chromate or neutral lead chromate shall not be employed alone or as a component part of any pigment. Small amounts of tinting pigments are permissible to achieve color as in 3.2. Extender pigments must be siliceous in nature; however the use of silica-alumina ceramic spheres is permitted.

TABLE I. Pigment composition.

Characteristics	Type I		Type II	
	Percent by weight		Percent by weight	
	Min	Max	Min	Max
Titanium dioxide	50.0	-	32.0	-
Zinc phosphate	9.0	11.0	5.0	-
Corrosion inhibiting pigment 1/	0.9	1.1	0.5	-
Extenders	-	40.0	-	45.00
Hexavalent chromium	negative		negative	

1/ Sicorin RZ, Basf Wyandatte Corp. or equivalent.

3.3.2 Vehicle.

3.3.2.1 Part A (epoxy resin component). Part A shall consist of a bisphenol type epoxy resin and pigment combined with necessary amounts of flow control agents and volatile solvents to meet the requirements of this specification.

3.3.2.2 Part B (catalyst component). Part B shall consist of an aliphatic polyamine-epoxy resin adduct combined with the necessary amounts of volatile solvents to meet the requirements of the specification.

3.3.2.3 Volatile content. The volatile content of the mixed primer shall conform to the following requirements of volume when tested as in Table V.

3.3.2.3.1 For type I. The volatile portion of the mixed primer shall conform to Rule 102 of South Coast Air Quality Management District as described below:

a. A combination of hydrocarbons, alcohols, aldehydes, ester, ethers, and ketones having an olefinic or cyclo-olefinic type of unsaturation: 5 percent maximum.
b. A combination of aromatic compounds with eight or more carbon atoms to the molecule except ethyl benzene: 8 percent maximum.
c. A combination of ethyl benzene, ketones having branched hydrocarbon structures, or toluene: 20 percent maximum.
d. Total a + b + c: 20 percent maximum.

3.3.2.3.2 For type II. The volatile organic compound content shall not exceed 420 grams/liter (3.5 pounds/gallon) as packaged (see 4.12).

3.4 Quantitative requirements.

3.4.1 Part A (epoxy resin component). Part A shall conform to the quantitative requirements of table II when tested as in section 4.

TABLE II. Part A requirements.

Characteristics	Type I		Type II	
	Min	Max	Min	Max
Total solids, percent by weight of part A	60	-	70	-
Pigment, percent by weight of part A	38	-	41	-
Vehicle solids, percent by weight of part A	22	-	19	-
Epoxy resin, percent by weight of vehicle solids	90	-	80	-
Fineness of grind	5	-	5	-
Viscosity, Kerbs Stormer, Shearing rate-200 rpm Equivalent K.U.	63	73	65	80
Coarse particles and skins (retained on a 325 standard sieve), percent by weight of pigment.	-	1.0	-	1.0

3.4.2 Part B (catalyst component). Part B shall conform to the quantitative requirements of table III when tested as in section 4.

TABLE III. Part B requirements.

Characteristics	Type I		Type II	
	Min	Max	Min	Max
Amine nitrogen content, percent by weight of part B	2.0	3.0	3.0	4.0
Epoxy resin	Positive		Positive	
Weight per gallon, pounds	7.6	8.0	7.5	8.5

3.4.3 Mixed primer. The mixed primer shall conform to the quantitative requirements of table IV when tested as in section 4.

3.5 Qualitative requirements.

3.5.1 Condition in container.

3.5.1.1 Part A. When tested as specified in 4.10.1, part 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.

3.5.1.2 Part B. When tested as specified in 4.10.2, part 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.

TABLE IV. Mixed primer requirements.

Characteristics	Type I		Type II	
	Min	Max	Min	Max
Specular gloss, 60 degrees	10	30	10	30
Drying time				
Set to touch	-	15	-	30
Dry hard, minutes	-	90	-	240
Dry through, hours	-	4	-	6
Lead metal, percent by weight of total solids	-	0.06	-	0.06

3.5.2 Storage stability.

3.5.2.1 Part A. A full quart can of part A shall show no skinning, livering, curdling, hard dry caking or tough gummy sediment when tested as specified in 4.11.1. It shall remix readily to a smooth homogeneous state and shall meet all other requirements of the specification.

3.5.2.2 Part B. When tested as specified in 4.11.2, a full 8 ounce can of part 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 container and shall meet all other requirements of the specification.

3.5.3 Mixing properties. When tested as specified in 4.12, smooth homogeneous mixture shall result. The primer shall be free from grit, seeds, skins, or lumps. After aging as specified in 4.12, the primer shall show no signs of gelation.

3.5.4 Spraying properties. When tested as specified in 4.13, 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.

3.5.5 Adhesion. A film of primer, tested as specified in 4.14, shall show no removal of the primer by the adhesive tape beyond one-sixteenth inch on either side of the scored lines.

3.5.6 Knife test. A film of primer, tested as specified in 4.15, 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.

3.5.7 Flexibility. A film of primer, tested as specified in 4.16, shall withstand bending without cracking or flaking.

3.5.8 Water resistance. A film of primer, tested as specified in 4.17, shall show no wrinkling or blistering immediately after removal of the panel from the water. The primer shall be no more than slightly affected when examined 2 hours after removal; and after 24 hours air drying, the portion of the panel which was immersed shall be almost

indistinguishable with regard to hardness and adhesion from the portion which was not immersed.

3.5.9 Hydrocarbon fluid resistance. A film of primer, tested as specified in 4.18, shall show no blistering or wrinkling and no more than a slight yellow to beige color change on submerged area of panel. Upon removal from the fluid slight softening is acceptable. After 2 hours air drying, the panel that was immersed shall be almost indistinguishable with regard to hardness and gloss from a panel prepared at the same time but not immersed.

3.5.10 Salt spray resistance. A film of primer, tested as specified in 4.19 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. On removal of the primer, there shall be no more than a trace of rusting, pitting or corrosion of the substrate.

3.5.11 Topcoating. A film of primer, tested as specified in 4.20, shall show no blistering, wrinkling or other evidence of lifting. The topcoat shall be difficult to remove from the primer and the primer from the panel when cut with the knife blade.

3.5.12 Weather resistance. A film of the primer, exposed as specified in 4.21, shall show no rusting, cracking, checking, flaking, or loss of adhesion. On 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).

3.5.13 Super tropical bleach (STB) resistance. When tested as specified in 4.22, 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.

3.5.14 Toxic ingredients. The primer shall contain no benzene (benzol), chlorinated solvents or ethylene based glycol ethers and their acetates.

3.6 User instruction markings. All primary containers shall be legibly marked or labeled "Part A (epoxy resin component) or "Part B (catalyst component)" as applicable, with the manufacturer’s mixing 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: Mix part A well; then add 1 part by volume of part B to 4 parts by volume of part A and mix well. For spray application, type I material shall be thinned by reducing 4 parts of the mixed primer by volume with one part by volume of MII-T-81772. For spray application of type II material, follow the manufacturer's instructions in order to allow a 20 minute induction time before use. During spray application, avoid inhalation and eye or skin contact.

TYPE I MATERIAL SHALL BE USED WITHIN 8 HOURS AFTER MIXING.
TYPE II MATERIAL SHALL BE USED WITHIN 4-8 HOURS AFTER MIXING.

3.7 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 6.8). The MSDS shall be included with each shipment of the material covered by this specification and submitted to pertinent Government agencies as stated in a FED-STD-313.

3.8 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 6.9).

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. The qualification inspection shall consist of tests for all requirements specified in section 3 and table V (see 6.3). 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.

4.3.1 Lot and batch formation. For purposes of conformance inspection, 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 6.3.2). 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 6.3.2). The addition of any substance to a batch shall constitute a new lot.

4.3.2 Conformance tests. Conformance tests for acceptance of individual lots shall consist of total solids, viscosity, fineness of grind, 60° specular gloss, drying time,

condition in container, mixing properties, and spraying properties. At a minimum, the contractor shall select representative samples from the first and last containers from each lot of each component, and subject the samples to all conformance tests. Results shall meet the applicable requirements in section 3. There shall be no failures (see 6.6).

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

4.5 Material safety data sheet (MSDS). The MSDS shall address components A and B and be in compliance with FED-STD-313 (see 3.7). Noncompliance shall be cause for rejection.

4.6 Test methods.

4.6.1 Test conditions. The testing conditions shall be in accordance with section 9 of FED-STD-141 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 3.5, as applicable, shall constitute failure of the applicable test. For all tests requiring the use of the mixed primer, parts A and B shall be mixed in the proportions specified in paragraph 4.12.

4.6.2 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.

4.6.3 Test procedures. The following tests (see table V), shall be conducted in accordance with FED-STD-141, the EPA test method, the ASTM test method, 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.

4.7 Analysis of primer.

4.7.1 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 dish 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 ^{o}F (105 ^{o}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 solid) as required. Nonconformance to the requirements in table II shall constitute failure of this test.

TABLE V. Index.

Item	Applicable Tests in FED-STD-141 or EPA Test Method	ASTM Test Method	Test Paragraph	Requirement Paragraph or Table
Pigment analysis	-	D2371	4.7.6	3.3.1
Titanium dioxide	-	D1394	4.7.6.1	Table I
Zinc phosphate	-	-	4.7.6.2	Table I
Hexavalent chromium	-	-	4.7.6.3	Table I
Isolation of vehicle (Supercentrifuge)	-	D2698	-	3.3.2
Solvent distillation	-	D3272	4.7.3	3.3.2.3.1
Aromatic compounds	EPA Method 311	-	-	3.3.2.3.1
Olefinic and Cyclo-olefinic	EPA Method 311	-	-	3.3.2.3.1
Ketones	EPA Method 311	-	-	3.3.2.3.1
Volatile organic compounds	-	D3960	4.7.3.1	3.3.2.3.2
Total solids	-	-	4.7.1	Table II
Pigment solids	-	-	4.7.2	Table II
Vehicle solids	-	-	4.7.2	Table II
Coarse particles and skins	-	D185	-	Table II
Viscosity	-	D562	-	Table II
Fineness of grind	-	D1210	-	Table II
Amine nitrogen content	-	-	4.7.5.3	Table III
Epoxy resin				
Part A		D1652	4.7.5.1	Table III
Part B		-	4.7.5.2	Table III
Weight per gallon	-	D1475	-	Table III
60° specular gloss	-	D523	4.8	Table IV
Drying time	4061.3	-	4.9	Table IV
Lead metal	-	D3335	4.7.4	Table IV
Condition in container				
Part A	3011.3	-	4.10.1	3.5.1.1
Part B	4261.1	-	4.10.2	3.5.1.2
Storage stability				
Full container				
Part A	-	-	4.11.1	3.5.2.1
Part B	4261.1	-	4.11.2	3.5.2.2

TABLE V. Index – Continued.

Item	Applicable Tests in FED-STD-141 or EPA Test Method	ASTM Test Method	Test Paragraph	Requirement Paragraph or Table
Mixing properties	-	-	4.12	3.5.3
Spraying properties	4331.2	-	4.13	3.5.4
Adhesion	-	D5486/D5486M, type IV	4.14	3.5.5
Knife test	6304.2	-	4.15	3.5.6
Flexibility	-	D522 Method B	4.16	3.5.7
Water resistance	-	D1308	4.17	3.5.8
Hydrocarbon resistance	-	-	4.18	3.5.9
Salt spray resistance	-	B117	4.19	3.5.10
Topcoating	-	-	4.20	3.5.11
Weather resistance	-	G90	4.21	3.5.12
STB resistance	-	-	4.22	3.5.13
Toxic ingredients	-	-	4.23	3.5.14

4.7.2 Pigment and vehicle solids. Place approximately 75 ml of well mixed part A in the bowl of a super centrifuge capable of developing at least 40,000 rpm. Rotate at 40,000 to 50,000 rpm for a period of 30 minutes, or until clear. Pour off the clear liquid into a small flask. Immediately stopper to prevent evaporation of the volatile portion. Run the vehicle solids on the recovered portion as in 4.7.1 but do not thin. Save rest of recovered vehicle for other tests.

Calculations:

Symbols: P = % Pigment in primer
T.S. = % Total solids in primer (4.4.1)
V.S. = % Vehicle solids in primer
N.V.V. = % Nonvolatile in vehicle (above)

a. Pigment = P = 100 (T.S. - N.V.V.)
b. Vehicle solids = (100 - N.V.V.) = V.S. = T.S. - P

Nonconformance to the requirements in table II shall constitute failure of this test.

4.7.3 Solvent analysis. For type I, vacuum distill the solvents from parts A and B using ASTM D3272. Analyze a mixture of 8 ml A and 1 ml B using EPA method 311. Determine compliance with 3.3.2.3.1.

4.7.3.1 Volatile organic compound (VOC) determination. For type II, determine the VOC of part A and part B in accordance with ASTM D3960. The density is determined by using the weight-per-gallon cup described in ASTM D1475. The nonvolatile content is determined by using ASTM D2369.

Calculate the VOC for the coating as mixed with the equation:

$$\frac{4(\text{VOC of part A}) + (\text{VOC of part B})}{5} = \text{VOC pounds per gallon of mixed coating}$$

Determine compliance with 3.3.2.3.2.

4.7.4 Lead content.

4.7.4.1 Determination of lead by atomic absorption spectroscopy. Determine percent of lead in accordance with ASTM D3335. Nonconformance to table IV shall constitute failure of this test.

4.7.4.2 Determination of lead by X-ray emission spectrometric analysis (alternate method).

4.7.4.2.1 Test panel preparation. Using 100 grams of a known lead free 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 napthenate of a known lead content. Thorouqhly mix the aliquots to incorporate the lead and draw down the standards and mixed 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 23 ± 1.1 °C (73.4 ± 2 °F), a relative humidity of 50 ± 4 percent, and under dust free conditions. Cut the draw downs into a suitable size and shape to fit the sample holder of the x-ray fluorescence spectrometer.

4.7.4.2.2 X-ray analytical procedure. Lead content shall be determined using an x-ray fluorescence spectrometer capable 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:

$$R = \frac{I_{pb} - \left(\frac{I_{pb}\ \text{Background I} + I_{pb}\ \text{Background II}}{2}\right)}{I_{\text{Compton Line}}}$$

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 IV shall constitute failure of this test.

4.7.5 Analysis of vehicle.

4.7.5.1 Epoxy resin - part A. Determine the epoxy resin content on the extracted vehicle in accordance with ASTM D1652 for compliance with table II. Nonconformance to table II constitutes failure of this test.

4.7.5.2 Epoxy resin - part B. Place 4 drops of part B in a test tube. Add about 10 drops of toluene and 10 drops of ethyl alcohol, mix and dry completely in an oven at 105 °C (221 °F). After cooling, add 1 mL concentrated sulfuric acid and warm to about 60 °C (140 °F) in a water bath for 10 minutes. Cool and add 2 drops of 40 percent formaldehyde solution. Allow the sample to stand a few minutes. Dilute with 10 mL of water added all at one time. A blue or green color shall form almost immediately if epoxy resins of the bisphenol type are present. Nonconformance to table III constitutes failure of this test.

4.7.5.3 Amine nitrogen content – part B. Determine the amine nitrogen content in accordance with 4.7.5.3.1. Nonconformance to table III constitutes failure of this test.

4.7.5.3.1 Determination of amine nitrogen content – part B.

4.7.5.3.1.1 Reagents.

a. Perchloric acid in acetic acid, 0.1 N. : Prepare by adding 24 ml of acetic anhydride and 8.9 ml of 70 percent perchloric acid to 1,000 ml of glacial acetic acid. Mix, cool, and standardize against weighed quantities of potassium acid phthalate dissolved in glacial acetic acid using methyl violet as indicator. Correct the titration, if necessary, by running a blank on the same volume of acetic acid used to dissolve the standard. Calculate the normality of the reagent.

$$\text{Normality} = \frac{\text{grams of potassium biphthalate} \times 1{,}000}{(\text{ml of reagent for standard - ml for blank}) \times 204.22}$$

4.7.5.3.1.2 Procedure.

a. Accurately weigh, approximately 0.5 g of the activator into a 250 ml Erlenmeyer flask.
b. Add 25 ml glacial acetic acid followed by 50 ml of methyl isobutyl ketone. Protect the sample, solvents and reagents from exposure to moisture of the atmosphere.
c. Add a few crystals of methyl violet as indicator, sufficient to give a strong purple color. Insert a dry teflon-covered magnetic stirring bar.

d. Attach a vented rubber stopper to the tip of a dry, 25 ml buret in such a manner that the sample shall be protected from moisture during titration. When the buret is filled with reagent, attach a drying tube to the upper end.
e. Attach the sample flask to the stopper on the buret, stir slowly and titrate with a perchloric acid reagent to a blue-green end point.
f. Run a blank titration on a mixture of 25 ml of the acetic acid and 50 ml of methyl isobutyl ketone.

4.7.5.3.1.3 Calculation. Calculate the amine nitrogen content:

$$\text{Percent nitrogen} = \frac{(\text{ml of reagent for sample} - \text{ml for blank}) \times \text{normality} \times 14.0}{10 \times \text{weight of sample}}$$

4.7.5.4 Weight per gallon – part B. Determine the weight per gallon of part B in accordance with ASTM D1475 and check for compliance with table III. Nonconformance to table III constitutes failure of this test.

4.7.6 Analysis of pigment. 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 3.3.1 shall constitute failure of this test.

4.7.6.1 Titanium dioxide ($Ti0_2$) content. Determine the titanium dioxide ($Ti0_2$) content on the extracted pigment in accordance with ASTM D1394 for compliance with table I. Nonconformance to table I shall constitute failure of this test.

4.7.6.2 Zinc phosphate content. Determine the zinc phosphate content in accordance with 4.7.6.2.1 and 4.7.6.2.2.

4.7.6.2.1 Determination of zinc.

4.7.6.2.1.1 Reagents.

a. Buffer solution (pH 10): 350 ml concentrated NH_4OH + 54g NH_4Cl + H_20 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 ml.
d. 0.5N Disodium ethylenediaminetetraacetate dehydrate (EDTA): 37.2g EDTA per liter aqueous solution.

4.7.6.2.1.2 Procedure.

a. Accurately weigh approximately 1.0 gram of pigment into a 250 ml glass stoppered Erlenmeyer flask.
b. Add 25 ml 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 (Vs).
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).

4.7.6.2.1.3 Calculations.

$$\text{Percent Zn} = \frac{(V_b - V_S) \times 0.02 \times 3.269}{\text{Sample weight}}$$

$$\text{Percent zinc phosphate} = \frac{(V_b - V_S) \times 0.2 \times 7.035}{\text{Sample weight}}$$

4.7.6.2.2 Determination of phosphate.

4.7.6.2.2.1 Reagents.

a. Concentrated NH_4OH.
b. Concentrated HNO_3.
c. NH_4NO_3.
d. Ammonium molybdate – Johnson's formula: Mix 55g of $(NH_4)_6MO_7O_{24} \cdot 4H_2O$ 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 1000ml. Let stand overnight. Filter through fine paper but do not wash the residue.

4.7.6.2.2.2 Procedure.

a. Accurately weigh approximately 2g of pigment into a 250 ml glass-stoppered Erlenmeyer flask.
b. Add 30 ml 7.5N HNO_3 and agitate the sample every few minutes over a period of 30 minutes.
c. Filter through Whatman 50 paper into a 400 ml beaker washing well with water.
d. Add 6 grams of NH_4NO_3, stir.
e. Heat the clear solution to 80 °C (no higher) and add 75 ml of ammonium molybdate with constant stirring.
f. Stir for several minutes and let the precipitate settle for 2 hours.
g. Filter through a tared crucible (gooch or medium glass), transfer the precipitate, and wash with 1 percent HNO_3 5 ml concentrated HNO_3 per 500 mL solution. The washing shall be thorough.
h. Give the collected precipitate a final wash with a small amount of water.
i. Dry the crucible for 2 hours in a 105 °C oven.

j. Cool crucible in a desiccator and determine the weight of the precipitate (it shall not exceed 3g; if it does, repeat the determination with a smaller sample).

4.7.6.2.2.3 Calculations.

$$\text{Percent } PO_4 = \frac{\text{Weight of precipitate x } 5.029}{\text{Sample weight}}$$

$$\text{Percent zinc phosphate } [Zn_3(PO_4)_2 \cdot 2H_2O] = \frac{\text{Weight of precipitate x } 11.18}{\text{Sample weight}}$$

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

4.7.6.3 Hexavalent chromium (Cr^{6+} shall be absent).

a. Reagents:

1. 25 percent aqueous KOH

b. Procedure:

1. Add 5 ml of 25 percent aqueous KOH to 0.5g of the extracted pigment contained in a 15 ml centrifuge tube.
2. Agitate by shaking the tube for a few minutes then centrifuge.
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.

4.8 Specular gloss (60 degree). Prepare a drawdown of the catalyzed primer on a black and white Morest card to a dry film thickness of 0.001 ± 0.0001 inch. Dry for 48 hours and determine the 60 degree gloss in accordance with ASTM D523. Nonconformance to table II shall constitute failure of this test.

4.9 Drying time. Determine drying time as in method 4061.3 of FED-STD-141, except that the primer shall be sprayed to a dry film thickness of 0.0009 to 0.0011 inch for type I and to a dry film thickness of 0.0010 to 0.0015 inch for type II. Nonconformance to table IV shall constitute failure of this test.

4.10 Condition in container.

4.10.1 Part A. Determine package condition of part A on acceptance testing in accordance with method 3011.3 of FED-STD-141 and observe for compliance with 3.5.1.1. On qualification testing, evaluate pigment settling or caking by proceeding as in method 3011.3 of FED-STD-141, but do not stir. Reseal and then agitate the can for 3 minutes on a paint shaker. On reexamination of the contents, the disclosure of any gel bodies or undispersed pigment indicates unsatisfactory settling properties. Nonconformance to 3.5.1.1 constitutes failure of this test.

4.10.2 Part B. Determine package condition of part B in accordance with method 4261.1 of FED-STD-141 and observe for compliance with 3.5.1.2. Nonconformance to 3.5.1.2 constitutes failure of this test.

4.11 Storage stability.

4.11.1 Part A. Allow a full can of part A to stand undisturbed for 12 months at 72 to 80 °F and then examine the contents. Evaluate the pigment settling as in 4.10.1 except agitate the can for 5 minutes on the paint shaker prior to reexamination. Determine viscosity and other applicable tests for compliance with 3.5.2.1. Nonconformance to 3.5.2.1 shall constitute failure of this test.

4.11.2 Part B. Allow a full 8 ounce can of part B to stand undisturbed for 12 months. At the end of this period examine the contents in accordance with method 4261.1 of FED-STD-141 for compliance with 3.5.2.2. Nonconformance to 3.5.2.2 shall constitute failure of this test.

4.12 Mixing properties. Thoroughly mix 4 parts by volume of part A with one part by volume of part B and examine for compliance with 3.5.3. Place 5 ounces of the material in an eight ounce glass jar and do not agitate or disturb for 4 hours. At the end of this period examine for compliance with 3.5.3.

4.13 Spraying properties. If necessary for application, reduce four parts of the mixed primer by volume with one part of MIL-T-81772. For type II, the solvent MIL-T-81772 can be used or follow manufacturer's recommendations not to exceed the VOC level (see1.2). Spray on a steel panel to a dry film thickness between 0.0009 and 0.0011 inch and observe for spraying properties in accordance with method 4331.2 of FED-STD-141 for compliance with 3.5.4.

4.14 Adhesion. Spray the primer as in 4.13 on a steel and aluminum panel pretreated as in 4.6.2. Air dry type I specimens for four hours and type II specimens for six hours and then score a line through to the metal across the width of the film using a sharp pointed knife. The film shall then be taped perpendicular to and across the score line. The tape used shall meet the requirements of ASTM D5486/D5486M, type IV. Nonconformance to 3.5.5 shall constitute failure of this test.

4.15 Knife test. Prepare films of primer as in 4.14 and air dry 3 days for type I and 7 days for type II. Perform the knife test as in method 6304.2 of FED-STD-141. Nonconformance to 3.5.6 shall constitute failure of this test.

4.16 Flexibility. Prepare films of the primer as in 4.14 except substitute a thin gauged steel panel prepared as specified in 4.13, using the petroleum naphtha propylene glycol monomethyl ether mixture. Allow the panels to air dry 3 days for type I and 7 days for type II. Bend the coated panels according to ASTM D522, method B. Examine the coating for cracks over the area of the bend for compliance with 3.5.7. Nonconformance to 3.5.7 constitutes failure of this test.

4.17 Water resistance. Prepare sprayed films of primer as in 4.14. Allow the panels to air dry 3 days for type I and 7 days for type II. Coat all exposed, uncoated metal surfaces with wax or other suitable coating. Immerse the panels for 168 hours in distilled water at 23 ± 1 °C as specified in ASTM D1308. On removal, observe panels for compliance with 3.5.8. Nonconformance to 3.5.8 constitutes failure of this test.

4.18 Hydrocarbon fluid resistance. Prepare films of primer as in 4.14 and air dry 3 days for type I and 7 days for type II. 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 3.5.9. Nonconformance to 3.5.9 shall constitute failure of this test.

4.19 Salt spray resistance. Prepare three 4 by 12 inch test panels each as in 4.14 and air dry type I for 3 days and type II for 7 days. Expose the panels to 5 percent salt spray for 336 hours in accordance with ASTM B117. Upon removal, wash the panels gently in warm running water until free from any visible salt deposits and examine immediately for compliance with 3.5.10. Nonconformance to 3.5.10 shall constitute failure of this test.

4.20 Topcoating. Prepare 2 test panels each as in 4.14. Allow to air dry 15 minutes and 24 hours respectively and spray a coat of camouflage green 383, 34094 polyurethane conforming to MIL-DTL-64159, type II over the specimens. 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 minimum of 0.0018. Examine the panels for evidence of lifting after the topcoat has air dried 2 hours. Allow the specimens to air dry 168 hours after recoating. Cut film with a knife blade and check for compliance with 3.5.11. Nonconformance to 3.5.11 constitutes failure of this test.

4.21 Weather resistance. Prepare four 4 by 12 inch panels (2 of each substrate) of the primer as in 4.14. Allow the primer to air dry for 24 hours and then apply a coat of camouflage 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/m^2 of total UV irradiance, in an accelerated outdoor exposure according to ASTM G90. At 70 MJ/m^2 intervals examine the panels for compliance with 3.5.12. 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 3.5.12 shall constitute failure of this test.

4.22 Super tropical bleach (STB) resistance. Prepare a steel panel as specified in 4.13. 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 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 3.5.13.

4.23 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 3.5.14 constitutes 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. This specification is intended for use on properly cleaned and pretreated ferrous and nonferrous substrates where exposure to lead or chromate pigments is not permitted. Type II formulations will meet a 420 gram/liter maximum (3.5 pound/gallon) volatile organic compound (VOC) content requirement.

6.2 Acquisition requirements. Acquisition documents should specify the following:

a. Title, number and date of this specification.
b. Type of coating required (see 1.2).
c. Size of primary container required for part A and size of primary container required for part B (see 3.3).
d. If a toxicity clearance is required (see 3.8).
e. Packaging requirements (see 5.1).
f. Preparation of material safety data sheets (MSDS) in accordance with FED-STD-313 for the epoxy primer coating and inclusion of MSDS with shipment of material.

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 Qualified Products List QPL-53022, 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. Information pertaining to qualification of products may be obtained from the U.S. Army Research Laboratory, ATTN: AMSRD-ARL-WM-MC (Coatings Team), Building 4600, Deer Creek Loop, Aberdeen Proving Ground, MD 21005-5069.

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

6.4 Basis of purchase. The primer covered by this specification should be purchased by volume, the unit being one US gallon of 231 cubic inches at 68 °F (20 °C).

6.5 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.

6.6 Conformance rejection and retest. Failure in any conformance inspection will result in the rejection of the batch from which it was obtained and constitutes justification for removal from the qualified products list. Rejected material cannot be resubmitted for acceptance without written approval from the qualification activity (see 6.3). The application for resubmission will contain all details concerning previous rejections and measures taken to correct these deficiencies.

6.7 Limitations of olefinic test. The test for olefinic and cycle-olefinic compounds will not be positive for solvents containing less than 1 percent of these compounds.

6.8 Material safety data sheet (MSDS). Contracting officers will identify those activities requiring copies of a completed MSDS prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

6.9 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.

6.10 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.

6.11 Subject term (key word) listing.

Ferrous substrates
Flash drying
Nonferrous substrates
VOC content

6.12 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: 8010-2006-012

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

Civil agency:
GSA/FSS – 6FEE

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/.