

TT-P-664D

INCH-POUND

TT-P-664D  
 September 1, 1988  
 SUPERSEDING  
 TT-P-664C  
 January 13, 1969

## FEDERAL SPECIFICATION

PRIMER COATING, ALKYD, CORROSION-INHIBITING,  
 LEAD AND CHROMATE FREE, VOC-COMPLIANT

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

## 1. SCOPE

1.1 Scope. This specification covers the requirements for a quick drying, corrosion-inhibiting, high solids alkyd primer for pretreated ferrous and non-ferrous metals. The primer is lead and chromate free and contains no more than 420 grams per liter (3.5 pounds per gallon) of volatile organic compounds (VOC) as applied.

## 2. APPLICABLE DOCUMENTS

2.1 Government documents. The following specifications and standards form a part of this specification to the extent specified herein.

## Federal Specifications

QQ-A-250	- Aluminum and Aluminum Alloy Plate and Sheet, General Specification for.
QQ-A-250/5	- Aluminum Alloy Alclad 2024, Plate and Sheet.
TT-C-490	- Cleaning Methods and Pretreatment of Ferrous Surfaces.
TT-E-489	- Enamel, Alkyd, Gloss, Low VOC Content.
TT-S-735	- Standard Test Fluids; Hydrocarbon.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: USA Belvoir Research, Development, and Engineering Center, ATTN: STRBE-TSE, Fort Belvoir, VA 22060-5606 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

FSC 8010

DISTRIBUTION STATEMENT A. Approved for public release, distribution is unlimited.

## TT-P-664D

- |            |  |
|------------|--|
| TT-T-266   | - Thinner, Dope and Lacquer (Cellulose-Nitrate).                                       |
| TT-T-306   | - Thinner, Synthetic Resin Enamel.   |
| PPP-P-1892 | - Paint, Varnish, Lacquer and Related Materials;<br>Packaging, Packing and Marking of. |
| PPP-T-60   | - Tape, Packaging, Waterproof.   |

## Federal Standards

- |             |  |
|-------------|--|
| FED-STD-141 | - Paint, Varnish, Lacquer, and Related<br>Materials; Methods of Inspection, Sampling and<br>Testing. |
| FED-STD-313 | - Preparation and Submission of Material Safety<br>Data Sheets.                                      |

(Activities outside the Federal Government may obtain copies of Federal specifications, standards, and commercial item descriptions, as outlined under General Information in the Index of Federal Specifications and Standards, and Commercial Item Descriptions. The Index, which includes cumulative bimonthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

(Single copies of this specification, other Federal specifications, and commercial item descriptions required by activities outside the Federal Government for bidding purposes are available without charge from General Services Administration Business Service Centers in Boston, MA; New York, NY; Philadelphia, PA; Washington, DC; Atlanta, GA; Chicago, IL; Kansas City, MO; Fort Worth, TX; Houston, TX; Denver, CO; San Francisco, CA; Los Angeles, CA; and Auburn, WA.

(Federal Government activities may obtain copies of Federal standardization documents and the Index of Federal Specifications, Standards, and Commercial Item Descriptions from established distribution points in their agencies.)

## Military Specifications

- |             |   |
|-------------|---|
| MIL-C-5541  | - Chemical Films and Chemical Film Materials for<br>Aluminum and Aluminum Alloys. |
| MIL-L-11195 | - Lacquer, Lustreless, Hot Spray.   |
| MIL-C-81706 | - Chemical Conversion Materials for Coating Aluminum<br>and Aluminum Alloy.       |

(Unless otherwise indicated, copies of federal and military specifications, standards, and handbooks are available from the Naval Publications and Forms Center, (ATTN: NPODS), 5801 Tabor Avenue, Philadelphia, PA 19120-5099.)

2.2 Non-Government publications. The following document(s) form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted shall be those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issue of documents not listed in the DODISS are the issues cited in the solicitation (see 6.2).

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American Society for Testing and Materials Publications (ASTM):

- B 117 - Salt Spray (Fog) Testing.
- D 50 - Chemical Analysis of Yellow, Orange, Red and Brown Pigments Containing Iron and Manganese.
- D 523 - Specular Gloss.
- D 562 - Consistency of Paints Using the Stormer Viscometer.
- D 610 - Evaluating Degree of Rusting on Painted Steel Surfaces.
- D 1210 - Fineness of Dispersion of Pigment-Vehicle Systems.
- D 1308 - Effect of Household Chemicals on Clear and Pigmented Organic Finishes.
- D 1364 - Water in Volatile Solvents (Fisher Reagent Titration Method.)
- D 1542 - Qualitative Tests for Rosin in Varnishes.
- D 1544 - Color of Transparent Liquids (Gardner Color Scale).
- D 1639 - Acid Value of Organic Coating Materials.
- D 3335 - Low Concentrations of Lead, Cadmium and Cobalt in Paint by Atomic Absorption Spectroscopy.
- D 3960 - Determining Volatile Organic Content (VOC) of Paints and Related Coatings.

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

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

2.3 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, (except for related associated detail specifications, specification sheets or MS standards), 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. Primer furnished under this specification shall be a product which is qualified for listing on the applicable Qualified Products List (QPL) at the time set for opening of bids (see 4.2 and 6.3). Any changes in the formulation of a qualified product will necessitate its requalification. The material supplied under the contract shall be identical, within manufacturing tolerances, to the product receiving qualification.

3.2 Color. The color of the primer shall be characteristic of red or brown iron oxide pigments (see 6.6).

#### 3.3 Compositions.

3.3.1 Pigment. The pigment portion of the primer shall conform to the percent by weight requirements of table I when tested as specified in 4.3.2. Only synthetic iron oxides and only siliceous extenders may be used, except that barium sulfate may replace up to 20 percent of the extender content.

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TABLE I. Quantitative requirements of pigment.

Pigment	Percent By Weight	
	Minimum	Maximum
Iron oxide (Fe <sub>2</sub> O <sub>3</sub> by analysis)	45.0	-
Zinc phosphate	9.0	11.0
Corrosion inhibiting pigment [1]	0.9	1.1
Siliceous extenders	-	45.0
Barium sulfate	-	9.0
Hexavalent chromium	Negative	

[1] Sicorin RZ, BASF Wyandotte Corporation, or equivalent.

3.3.2 Vehicle. The vehicle shall be a resin modified, drying oil phthalic alkyd resin conforming to the requirements of table II, together with shall amounts of driers, antioxidants, wetting agents and stabilizers as required.

TABLE II. Characteristics of alkyd resin.

Property	Percent By Weight	
	Minimum	Maximum
Alkyd resin solution		
Total solids percent by weight	75	-
Viscosity (Gardner)	-	Z3
Color (Gardner color standards of 1953)	-	13
Alkyd resin solids		
Phthalic anhydride, percent by weight	25	-
Unsaponifiable matter, percent by weight	-	5
Acid number	-	22
Rosin/phenol	Positive	

3.4 Quantitative requirements. The primer shall conform to the quantitative requirements of table III when tested as specified in 4.3.

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TABLE III - Quantitative requirements of primer.

Characteristics	Requirements	
	Minimum	Maximum
VOC, grams volatile per liter of primer reduced for spray	-	420
Total solids, percent by weight of primer	78.0	-
Pigment, percent by weight of primer	50.0	-
Vehicle solids, percent by weight of primer	28.0	-
Lead metal, percent by weight of total solids	-	0.06
Phthalic anhydride, percent by weight of vehicle solids	25	-
Unsaponifiable matter, percent by weight of vehicle solids	-	5
Water, percent by weight of primer	-	1.0
Coarse particles and skins (retained on No. 325 mesh seive), percent by weight of pigment	-	0.5
60 deg. specular gloss	5	15
Viscosity, (reduced for spray) Krebs-Stormer shearing rate 200 rpm:		
Grams	-	125
Equivalent KU	-	67
Fineness of grind	5	-
Drying time, air dry minutes		
Set to touch, minutes	-	10
Dry hard, minutes	-	45
Dry through, hours	-	4

### 3.5 Qualitative requirements.

3.5.1 Condition in container. A freshly opened full container of the primer, tested as specified in 4.3.10, shall be free from grit, seeds, lumps, abnormal thickening, or livering, and shall show no more pigment settling or caking that can be readily reincorporated to a smooth, homogeneous state.

#### 3.5.2 Storage stability.

3.5.2.1 Partially full container. The primer shall show no skinning when tested as specified in 4.3.11.1.

3.5.2.2 Full container. A full quart container of the primer shall show no skinning, livering, curdling, hard or dry caking, or tough gummy sediment when tested as specified in 4.3.11.2. The primer shall remix readily to a smooth, homogeneous state and shall meet all other requirements of this specification.

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3.5.3 Accelerated stability. When tested as specified in 4.3.12, the package material shall show no livering, curdling, hard caking, or tough gummy sediment and shall mix readily to a smooth, homogeneous state.

3.5.4 Suspension properties. The primer shall completely redisperse to a smooth, homogeneous state when tested as specified in 4.3.13.

3.5.5 Spraying properties. The primer, tested as specified in 4.3.14, 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, uniform finish free from seeds.

3.5.6 Flexibility. A film of the primer tested as specified in 4.3.15 shall withstand bending without cracking or flaking.

3.5.7 Adhesion. Films of the primer tested as specified in 4.3.16 shall show no removal of the primer by the adhesive tape beyond one-sixteenth inch on either side of the score line.

3.5.8 Knife test. Films of the primer tested as specified in 4.3.17 shall be hard and tough and shall adhere tightly to the metal 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.

3.5.9 Water resistance. Films of the primer tested as specified in 4.3.18 shall show no wrinkling or blistering immediately after removal of the panel from 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 from the portion which was not immersed, with regard to hardness, and shall show a color change equivalent to a lightness index difference not exceeding 2.5 units.

3.5.10 Hydrocarbon fluid resistance. Films of the primer tested as specified in 4.3.19 shall show no wrinkling or blistering immediately upon removal of the panel. After 24 hours air drying, the portion of the panel which was immersed shall be almost indistinguishable with regard to hardness, color and gloss, from a panel prepared at the same time but not immersed.

3.5.11 Lacquer resistance. A film of primer tested as specified in 4.3.20 shall show no bleeding, blistering, wrinkling, film irregularities, or other evidence of lifting. The system shall have a gloss comparable to the gloss of the MIL-L-11195 lacquer applied over glass and shall show satisfactory adhesion between lacquer and primer, and between primer and metal.

3.5.12 Enamel resistance. A film of primer tested as specified in 4.3.21 shall show no blistering, wrinkling, or other evidence of lifting. The system shall have a gloss of not less than 90 percent of the gloss of the olive drab enamel applied over glass and shall show satisfactory adhesion between enamel and primer, and between primer and metal.

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3.5.13 Salt spray resistance. Films of the primer tested as specified in 4.3.22 and examined immediately after removal from the salt spray test shall show no more than a trace of rusting (No. 9, ASTM D 610) 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 on the panels.

3.5.14 Weather resistance. Films of the primer prepared and exposed as specified in 4.3.23 shall show no cracking, checking, flaking, or loss of adhesion, and no more than a trace of rusting (No. 9, ASTM D 610). On removal of the coating system, the surface of the metal shall show no more than a trace of rusting, pitting, or corrosion on the panels.

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

3.5.16 Material Safety Data Sheet. A Material Safety Data Sheet (MSDS) shall be prepared in accordance with FED-STD-313 for the primer coating. The contractor shall overpack a copy of the MSDS with each shipment of material (see 6.5).

3.5.17 User instruction marking. In addition to the markings specified in 5.2, all containers shall include the VOC content in grams per liter of coating (less water) when reduced as specified with TT-T-306, type II, and shall be legibly marked or labeled with the following:

CAUTION: 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 adequate ventilation.  
For other safety recommendations refer to the Material Safety Data Sheet.  
Keep containers closed.

#### 4. QUALITY ASSURANCE PROVISIONS.

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

4.1.1 Responsibility for compliance. All items must meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The

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absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling in quality conformance does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to acceptance of defective material.

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

4.1.3 Material Safety Data Sheet. Material Safety Data Sheets not prepared in accordance with FED-STD-313 shall be cause for rejection.

4.2 Classification of test. Testing under this specification shall be for the following:

- a. Qualification.
- b. Acceptance of individual lots.
- c. Acceptance for use as component on end item (conformance).

4.2.1 Qualification testing. The qualification tests shall consist of tests for all requirements specified in section 3, and as specified in table IV (see 6.3).

4.2.2 Acceptance of lots testing. Acceptance testing for individual lots shall be as follows: VOC, condition in container, total solids, viscosity, fineness of grind, 60 degrees specular gloss, drying time, spraying properties, and lacquer resistance as specified in sections 3 and 4.

4.2.3 Conformance testing. When approved by the cognizant activity, acceptance of lots for use as a component on an end item shall be based on conformance with requirements specified in section 3 and 4 for the following characteristics: Fineness of grind, 60 degrees specular gloss, drying time, flexibility, knife test, water resistance, hydrocarbon resistance, and enamel resistance.

#### 4.3 Test methods.

4.3.1 Test conditions. The routine 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, 3.3, 3.4, and 3.5, as applicable, shall constitute failure of the applicable test.

4.3.1.1 Test panels. Except as otherwise specified, steel test panels shall be pretreated with a phosphate coating conforming to TT-C-490, type I and aluminum test panels shall be aluminum clad aluminum alloy conforming to QQ-A-250 and QQ-A-250/5 and given the film treatment with materials conforming to form I and II, method C (immersion), class 1A of MIL-C-81706 to produce coatings meeting the requirements of MIL-C-5541.

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4.3.1.2 Primer application. Unless specified otherwise, the primer shall be applied to the test panels by spraying one cross-coat to a dry film thickness of 0.0009 to 0.0011 inches after reduction to spray viscosity with the appropriate solvent (see 4.3.6).

4.3.1.3 Test procedures. The following tests (see table IV), shall be conducted in accordance with FED-STD-141 or ASTM 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.

TABLE IV. Index.

Item	Application method in FED-STD-141	Applicable ASTM test Method	Test paragraph	Requirement paragraph
Pigment Analysis	4021	-----	4.3.2	3.3.1
Iron oxide - Fe <sub>2</sub> O <sub>3</sub>	----	D 50	4.3.2.1	Table I
Zinc phosphate	----	-----	4.3.2.2	Table I
Hexavalent chromium	----	-----	4.3.2.3	Table I
Isolation of vehicle (super centrifuge)	4032	-----	-----	3.3.2
Color of transparent liquids	----	D 1544	-----	Table II
Acid number	----	D 1639	-----	Table II
Rosin	----	D 1542, Sec. 4a	-----	Table II
Phenol	5141	-----	-----	Table II
Total solids	----	-----	4.3.3	Table III
Pigment	----	-----	4.3.4	Table III
Vehicle solids	----	-----	4.3.4	Table III
Lead metal	----	D 3335	4.3.5	Table III
Phthalic anhydride	7014	-----	-----	Table III
Unsaponifiable	7014	-----	-----	Table III
Water	----	D 1364	-----	Table III
Coarse particles and skins	4092	-----	-----	Table III
Volatile organic compounds (VOC)	----	D 3960	4.3.6	Table III
Viscosity	----	D 562	4.3.7	Table III
60 deg. specular gloss	----	D 523	4.3.8	Table III
Fineness of grind	----	D 1210	-----	Table III

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TABLE IV. Index. (Cont'd)

Item	Application method in FED-STD-141	Applicable ASTM test Method	Test paragraph	Requirement paragraph
Drying time	4061	-----	4.3.9	Table III
Condition in container	3011	-----	4.4.10	3.5.1
Storage stability				
Partially full container	3021	-----	4.3.11.1	3.5.2.1
Full container	----	-----	4.3.11.2	3.5.2.2
Accelerated stability	----	-----	4.3.12	3.5.3
Suspension properties	----	-----	4.3.13	3.5.4
Spraying properties	4331	-----	4.3.14	3.5.5
Flexibility	6221	-----	4.3.15	3.5.6
Adhesion	----	-----	4.3.16	3.5.7
Knife Test	6304	-----	4.3.17	3.5.8
Water resistance	----	D 1308, Sec. 5D	4.3.18	3.5.9
Hydrocarbon resistance	----	-----	4.3.19	3.5.10
Lacquer resistance	----	-----	4.3.20	3.5.11
Enamel resistance	----	-----	4.3.21	3.5.12
Salt spray resistance	----	B 117	4.3.22	3.5.13
Weather resistance	----	-----	4.3.23	3.5.14
Toxic ingredients	----	-----	4.3.24	3.5.15

4.3.2 Pigment analysis. Extract the pigment as in method 4021 of FED-STD-141 using extraction mixture C. Make appropriate qualitative and quantitative tests on the extracted pigment to determine if only permissible pigments were used. Nonconformance to the requirements in 3.3.1 shall constitute failure of this test.

4.3.2.1 Quantitative  $\text{Fe}_{27}\text{O}_{37}$ . Determine  $\text{Fe}_{27}\text{O}_{37}$  content by ASTM D 50, section 10 and 11. Nonconformance to the requirement in table I shall constitute failure of this test.

4.3.2.2 Zinc phosphate content. Determine the zinc phosphate content in accordance with 4.3.2.2.1 and 4.3.2.2.2.

4.3.2.2.1 Determination of zinc.

4.3.2.2.1.1 Reagents.

- a. Buffer solution (pH 10): 350 mL conc.  $\text{NH}_{47}\text{OH}$  +  $\text{NH}_{47}\text{Cl}$  +  $\text{H}_{27}\text{O}$  to give 1000 mL.

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- 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. N/5 Disodium ethylenediaminetetraacetate dihydrate (EDTA): 37.2g EDTA per liter aqueous solution.

## 4.3.2.2.1.2 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 (L<sub>V</sub>J<sub>S</sub>).
- 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 (L<sub>V</sub>J<sub>B</sub>).

## 4.3.2.2.1.3 Calculations.

$$\text{Percent Zn} = \frac{(\text{L}_{\text{V}}\text{J}_{\text{B}} - \text{L}_{\text{V}}\text{J}_{\text{S}}) \times 0.2 \times 3.269}{\text{Sample Wt.}}$$

$$\text{Percent zinc phosphate} = \frac{(\text{L}_{\text{V}}\text{J}_{\text{B}} - \text{L}_{\text{V}}\text{J}_{\text{S}}) \times 0.2 \times 7.035}{\text{Sample Wt.}}$$

Where: L<sub>V</sub>J<sub>B</sub> - Milliliters of ZnO for blank, and  
L<sub>V</sub>J<sub>S</sub> - Milliliters of ZnO for sample

## 4.3.2.2.2 Determination of phosphate.

## 4.3.2.2.2.1 Reagents.

- a. Conc NH<sub>4</sub>OH
- b. Conc HNO<sub>3</sub>
- c. NH<sub>4</sub>NO<sub>3</sub>
- d. Ammonium molybdate - Johnson's Formula: Mix 55g of (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub> · 4H<sub>2</sub>O and 50 g of NH<sub>4</sub>NO<sub>3</sub> with 18 mL of conc NH<sub>4</sub>OH and 20 mL H<sub>2</sub>O. Stir. Dilute to about 700 mL with H<sub>2</sub>O, 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.

## 4.3.2.2.2.2 Procedures.

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- a. Accurately weigh approximately 2g of pigment into a 250 mL glass-stoppered Erlenmeyer flask.
- b. Add 25 mL of conc  $\text{NH}_4\text{OH}$ , stopper, and shake vigorously every few minutes over a period of 60 minutes.
- c. Add 25 mL of  $\text{H}_2\text{O}$  and filter through fine paper into a 400 mL beaker, washing well with water.
- d. Neutralize the filtrate with 7.5N  $\text{HNO}_3$  (requires about 35 mL).
- e. Add 15 mL conc  $\text{HNO}_3$  and 6g of  $\text{NH}_4\text{NO}_3$ . Stir.
- f. Heat the clear solution to 80 deg. C (no higher) 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  $\text{HNO}_3$  (5 mL conc.  $\text{HNO}_3$  per 500 mL solution). The washing should be thorough.
- i. Give the collected precipitate a final wash with a small amount of water.
- j. Dry the crucible for 2 hours in a 105 deg. C oven.
- k. Cool crucible in a desiccator and determine the weight of the precipitate to the nearest one-tenth mg. (It should not exceed 3g; if it does, repeat the determination with a smaller sample).

## 4.3.2.2.2.3 Calculations.

$$\text{Percent PO}_4 = \frac{\text{Wt. PPT.} \times 5.029}{\text{Sample Wt.}}$$

$$\text{Percent zinc phosphate} = \frac{\text{Wt. PPT.} \times 11.18}{\text{Sample Wt.}}$$

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

4.3.2.3 Hexavalent chromium ( $\text{Cr}^{6+}$ ) must be absent.

- (1) Reagents:  
25 percent aqueous KOH
- (2) Procedure:
  - a. Add 5 mL of 25% aq. KOH to 1/2g of the extracted pigment contained in a 15 mL centrifuge tube.
  - b. Agitate by shaking the tube for a few minutes, then centrifuge.
  - c. The supernatant liquid should be colorless. A yellow color indicates presence of chromate. Nonconformance to the requirement in table I shall constitute failure of this test.

4.3.3 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 of the 60 mm aluminum dishes to the nearest one-tenth mg. Transfer a small sample that does not exceed 0.3 g 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 toluene and dry in a gravity convection oven at 105

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deg. C for 30 minutes. 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 III shall constitute failure of this test.

4.3.4 Pigment solids and vehicle solids by supercentrifuging. Place approximately 75 mL of well mixed paint in the bowl of a supercentrifuge capable of developing at least 40,000 rpm. Rotate at 40,000 to 50,000 rpm for period of 30 minutes, or until clear. Pour off the clear liquid into small flask which is immediately stoppered to prevent evaporation of the volatile portion. Run the percent nonvolatile vehicle on the recovered portion as specified in 4.3.3. but do not thin at all. Save rest of recovered vehicle for other tests.

Calculations:

Symbols:                    P = % pigment in primer  
                               T.S. = % Total solids in primer (see 4.3.3)  
                               V.S. = % Vehicle solids in primer  
                               N.V.V. = % Nonvolatile in vehicle (above)

$$(1) \text{ Pigment solids} = P = \frac{100 (T.S. - N.V.V.)}{(100 - N.V.V.)}$$

$$(2) \text{ Vehicle solids} = (V.S.) = (T.S. - P)$$

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

4.3.5 Lead content.

4.3.5.1 Determination of lead by atomic absorption spectroscopy. Determine percent of lead in accordance with ASTM D 3335. Nonconformance to table III shall constitute failure of this test.

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

4.3.5.2.1 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 naphthenate of a known lead content. Thoroughly mix the aliquots to incorporate the lead and drawdown 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 23 +/-1.1 deg. C (73.4 +/-2 deg. F), 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.

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4.3.5.2.2 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:

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

Where I = Gross intensity and the background it takes 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 III shall constitute failure of this test.

4.3.6 Volatile organic compound (VOC) determination. Reduce four parts by volume of the primer with one part by volume of thinner conforming to TT-T-306, type II as if for spray application. Determine the VOC of the mixture in accordance with ASTM D 3960. Nonconformance to table III shall constitute failure of this test.

4.3.7 Viscosity. Reduce the primer as specified in 4.3.6 and determine reduced viscosity according to ASTM D 562. Nonconformance to table III shall constitute failure of this test.

4.3.8 Specular gloss (60 degrees). Drawdown the primer to a dry film thickness of 0.001 +/-0.0001 inches on a plane glass panel of one of the types described in method 2021 of FED-STD-141. Measure the 60 degrees gloss as specified in ASTM D 523. Nonconformance to table III shall constitute failure of this test.

4.3.9 Drying time. Drawdown the primer as specified in 4.3.8 and determine the drying time under the test conditions described in 4.3.1 and in accordance with method 4061 of FED-STD-141. Nonconformance to table III shall constitute failure of this test.

4.3.10 Condition in container. Determine package conditions for acceptance testing in accordance with method 3011 of FED-STD-141 and observe for compliance with 3.5.1. For qualification testing, evaluate pigment settling or caking by proceeding as in method 3011 but do not stir. Reseal the can and then agitate

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for 3 minutes on paint shaker.[1] On re-examination of the contents, the presence of any gel particles or undispersed pigment indicates unsatisfactory settling properties. Nonconformance to 3.5.1 shall constitute failure of this test.

#### 4.3.11 Storage stability.

4.3.11.1 Partially full container. Determine skinning after 48 hours in accordance with method 3021 of FED-STD-141. Reseal and age for 7 days at 60 deg. C. Nonconformance to 3.5.2.1 shall constitute failure of this test.

4.3.11.2 Full container. Allow a full standard quart can of the primer to stand undisturbed for one year at 72 to 80 deg. F and then examine the contents. Evaluate pigment settling or caking as in 4.3.10 except agitate the can for 5 minutes on the paint shaker prior to reexamination. Determine viscosity and make other applicable tests. Nonconformance to 3.5.2.2 shall constitute failure of this test.

4.3.12 Accelerated stability. Fill an 8 ounce wide-mouth glass jar, approximately 4-1/2 inches high and 2 inches in diameter, with the packaged primer. Secure the cover tightly and invert the jar momentarily to check for leaks. Place the sample in a 60 +/-2 deg. C (140 +/-4 deg. F) oven in an upright position for 7 days. After this period, allow to cool to room temperature and examine the contents. Nonconformance to 3.5.3 shall constitute failure of this test.

4.3.13 Suspension properties. Reduce the primer as specified in 4.3.6. Place six ounces of the reduced material in an 8 ounce glass jar. Allow the stoppered jar to remain undisturbed for 24 hours and then place the unopened jar on a paint shaker as specified in 4.3.10 and agitate the contents for 20 seconds. Reexamine the material for any evidence of non-homogeneity or undispersed pigment. Nonconformance to 3.5.4 shall constitute failure of this test.

4.3.14 Spraying properties. Reduce the primer as specified in 4.3.6. Spray on a steel panel to a dry film thickness between 0.0009 to 0.0011 inch and observe for spraying properties as in method 4331 of FED-STD-141. Nonconformance to 3.5.5 shall constitute failure of this test.

4.3.15 Flexibility. Determine flexibility as in method 6221 of FED-STD-141. Using a film applicator that will deposit a dry film thickness of 0.0009 to 0.0011 inch, drawdown a 2-inch wide film of primer on a flat tinfoil panel cleaned with the petroleum naphtha-propylene glycol monomethyl ether mixture as in method 2012 of FED-STD-141. Air dry 1/2 hour then bake for 24 hours at 105 +/-2 deg. C (221 +/-4 deg. F). Allow the panel to return to room temperature, then bend over a 1/4 inch mandrel. Nonconformance to 3.5.6 shall constitute failure of this test.

[1] An apparatus of this type, powered by a 1/4 hp motor, operates at a rate of 1350 shakes per minute and is manufactured by Red Devil Tools, Irvington, New Jersey.

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4.3.16 Adhesion. Prepare drawdowns of the primer as specified in 4.3.15 on a steel and aluminum panel pretreated as specified in 4.3.1.1. Air dry the specimens for one hour under referee conditions 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 with water resistant, pressure sensitive adhesive tape (3/4-inch width) conforming to PPP-T-60, type IV. The tape shall be pressed down with two passes of a 4-1/2 pound rubber covered roller, approximately 3-1/2 inches in diameter by 1-3/4 inches in width. The surface of the roller shall have a durometer hardness value within the range of 70 to 80[2]. Allow 10 seconds for the test area to return to room temperature. Grasp a free end of the tape and at a rapid speed, strip it from the specimen by pulling the tape back upon itself at 180 degrees and check for film removal. Nonconformance to 3.5.7 shall constitute failure of this test.

4.3.17 Knife test. Prepare films of the primer as specified in 4.3.1.2 on a steel and aluminum panel pretreated as specified in 4.3.1.1. Air dry for 168 hours. Perform the knife test as in method 6304 of FED-STD-141. Nonconformance to 3.5.8 constitutes failure of this test.

4.3.18 Water resistance. Prepare films of primer as specified in 4.3.17 and air dry for 168 hours. Measure the directional reflectance of the coating and coat all exposed, uncoated metal surfaces with wax or other suitable coating. Immerse the panels for 18 hours in distilled water at 23 +/-1 deg. C as in method 6011 of FED-STD-141. On removal, observe the panel for compliance with 3.5.9. Determine the amount of color change expressed as lightness index difference ( $[W-DELTA]L$ ), using method 6122 of FED-STD-141. Nonconformance to 3.5.9 shall constitute failure of this test.

4.3.19 Hydrocarbon fluid resistance. Prepare films of primer as specified in 4.3.17 and air dry 168 hours. Do not wax or coat the exposed metal surfaces. Immerse the panels for 4 hours in a hydrocarbon fluid conforming to TT-S-735, type III. Upon removal, examine for compliance with 3.5.10. Nonconformance to 3.5.10 constitutes failure of this test.

4.3.20 Lacquer resistance. Prepare three test panels of the primer on steel as specified in 4.3.17. Allow to air dry 1 hour, 24 hours, and 96 hours, respectively, and then spray a wet coat of olive drab lacquer conforming to MIL-L-11195 over the specimens and over a glass panel. The dry film thickness of the lacquer shall be 0.0009 to 0.0011 inch. The dry film thickness of the primer-lacquer system shall be 0.0017 to 0.0019 inch. The lacquer shall be prepared for spraying at room temperature by reducing two parts by volume of lacquer with one part by volume of thinner conforming to TT-T-266. After the lacquer topcoat has air dried 48 hours, examine for lifting and check for compliance with 3.5.11. Allow the specimens to air dry 1 week after recoating, and determine adhesion between lacquer and primer and between primer and steel using the knife test as in method 6304 of FED-STD-141. Nonconformance to 3.5.11 shall constitute failure of this test.

[2] A roller of this type is available from the Pressure Sensitive Tape Council, 1201 Waukegan Road, Glenview, Illinois 60025.

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4.3.21 Enamel resistance. Prepare two test panels of the primer as specified in 4.3.20. Allow to air dry 1 hour, and 24 hours, and then spray a wet coat of olive drab gloss enamel conforming to TT-E-489, over the test specimens and over a glass panel. The dry film thickness of the enamel shall be 0.0014 to 0.0016 inch. The dry film thickness of the primer-enamel system shall be 0.0017 to 0.0019 inch. The enamel shall be prepared for spraying by reducing eight parts by volume of enamel to one part by volume of thinner conforming to TT-T-306, type II. After the topcoat has air dried 48 hours, examine for evidence of lifting; and after 72 hours, compare the 60 degree specular gloss with that of the enamel on glass and check for compliance with 3.5.12. Allow the specimens to air dry 1 week after recoating, and determine the adhesion between enamel and primer and between primer steel using the knife tests as in method 6304 of FED-STD-141. Nonconformance to 3.5.12 shall constitute failure of this test.

4.3.22 Salt spray resistance. Prepare three, 4 x 12 inch panels each of pretreated steel and aluminum as specified in 4.3.17 and 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 B 117. Remove the panels, wash gently in running water no warmer than 100 deg. F (38 deg. C) until free from any visible salt deposits. Examine immediately for compliance with 3.5.13. Strip the primer from the panels and inspect the panels for rust, pitting or corrosion. Nonconformance to 3.5.13 shall constitute failure of this test.

4.3.23 Weather resistance. Prepare two unscored 4- by 12-inch test specimens of the primer as specified in 4.3.17. Allow to air dry 24 hours and spray a coat of olive drab enamel conforming to TT-E-489, to a dry film thickness of 0.0014 to 0.0016 inch. Allow to air dry for 168 hours and place on outdoor exposure for 24 months at an angle of 45 degrees facing south in the latitude of Washington, D.C. After exposure, examine for compliance with 3.5.14. Then strip the primer film from the metal and inspect the surface. Nonconformance to 3.5.14 shall constitute failure of this test.

4.3.24 Toxic ingredients. The manufacturer shall certify that the primer contains no benzene (benzol), chlorinated solvents, or ethylene based glycol ethers and their derivatives. Nonconformance to 3.5.15 shall constitute failure of this test.

4.4 Inspection of preparation for delivery. The packaging, packing and marking specified in 5.1 and 5.2 shall be examined for quality conformance in accordance with the applicable requirements of PPP-P-1892.

## 5. PREPARATION FOR DELIVERY

5.1 Packaging and packing. The primer shall be packaged and packed in accordance with PPP-P-1892. The level of packaging and packing shall be level A, B, or C as specified (see 6.2). The primer shall be furnished in 1-quart or 1-gallon friction plug cans, 5-gallon lug cover steel pails, or 55-gallon steel drums as specified (see 6.2).

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5.2 Marking. Marking of each container, intermediate containers, shipping containers, and palletized loads as applicable, shall be in accordance with PPP-P-1892. Special markings shall be as specified in the contract or purchase order (see 6.2) and as specified in 3.5.17.

#### 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 contains no more than 420 grams per liter (3.5 pounds per gallon) of VOC as applied, and is suitable for use under synthetic enamel or lacquer topcoats. It is not intended for use on the inside of potable water tanks or for marine use.

6.2 Ordering data. Procurement documents should specify the following:

- a. Title, number and date of this specification.
- b. Level of packaging and packing required (see 5.1).
- c. Size of container required (see 5.1).
- d. Any special marking requirement (see 5.2).

6.3 Qualification. With respect to products requiring qualification, awards will be made only for products which are at the time set for opening of bids, qualified for inclusion in the applicable Qualified Products List whether or not such products have actually been so listed by that date. The attention of 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 orders for the products covered by this specification. The activity responsible for the Qualified Products List is the USA Belvoir Research, Development and Engineering Center, ATTN: STRBE-VO, Fort Belvoir, VA 22060-5606, and information pertaining to qualification of products may be obtained from this activity.

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

6.5 Material Safety Data Sheets. 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 appendix B of FED-STD-313.

6.6 Color. It is important to note that the color of the primer will vary within limits according to the type and amounts of color pigment used.

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## 6.7 Subject term (key work) listing.

Alkyd  
 Coating  
 Lead and chromate free  
 Primer  
 VOC-complaint

6.8 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

6.9 Changes from previous issue. Asterisks (or vertical lines) are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

## MILITARY INTERESTS:

Custodians

Army - ME  
 Navy - SH  
 Air Force - 99

Review activities

Army - AR, CR, ER, MR  
 Navy - YD  
 Air Force - 84

User activities

Navy - AS, OS

## CIVIL AGENCY COORDINATING

FSA- FSS

## PREPARING ACTIVITY:

Army - ME

Project: 8010-1209

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Orders for this publication are to be placed with General Services Administration, acting as an agent for the Superintendent of Documents. See section 2 of this specification to obtain extra copies and other documents referenced herein.