NOT MEASUREMENT SENSITIVE

MIL-DTL-11414F 24 July 2013 SUPERSEDING MIL-P-11414E 22 July 1992

#### **DETAIL SPECIFICATION**

# PRIMER ALKYD, FAST DRY, CORROSION INHIBITING, LEAD AND CHROMATE FREE

This specification is approved for use by all Departments and Agencies of the Department of Defense.

## 1. SCOPE

1.1 <u>Scope</u>. This specification covers the requirements of a fast drying corrosion inhibiting, low VOC content alkyd primer for coating interior and exterior surfaces of ammunition. This enamel primer is formulated free of lead and chromate (hexavalent chromium), with a maximum VOC content of 420 grams/liter (g/l) (3.5 pounds/gallon (lbs/gal)) as applied. The primer is also formulated free of hazardous air pollutants (HAP-free). This primer is not to be used on tactical or combat equipment. Equipment primed with this material is not to be coated with chemical agent resistant coating (CARC) primers or topcoats.

Beneficial comments, recommendations, additions, deletions, clarifications, etc. and any data that may improve this document should be sent to: Director, U.S. Army Research Laboratory, Weapons and Materials Research Directorate, Materials Manufacturing Technology Branch, Specification and Standards Office, ATTN: RDRL-WMM-D, Aberdeen Proving Ground, MD 21005-5069 or emailed to <a href="mailto:richard.j.squillacioti.civ@mail.mil">richard.j.squillacioti.civ@mail.mil</a>. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <a href="https://assist.dla.mil">https://assist.dla.mil</a>.

AMSC N/A FSC 8010

Distribution Statement A. Approved for public release; distribution is unlimited.

### 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.

## 2.2 Government documents.

2.2.1 <u>Specifications</u>, 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.

## INTERNATIONAL STANDARDIZATION AGREEMENTS

# **MILITARY STANDARDS**

STANAG 4147 - Chemica

- Chemical Compatibility of Ammunition Components with Explosives (Non-Nuclear Applications)

# FEDERAL STANDARDS

FED-STD-313

- Material Safety Data, Transportation Data, and Disposal Data For Hazardous Materials Furnished To Government Activities.

(Copies of these documents are available online at <a href="http://quicksearch.dla.mil">http://quicksearch.dla.mil</a> 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 this document are available online at <a href="http://www.epa.gov/ttn/emc/">http://www.epa.gov/ttn/emc/</a> or from the Environmental Protection Agency, Ariel Rios Building, 1200 Pennsylvania Avenue, N.W., Washington, DC 20460.)

# CODE OF FEDERAL REGULATION 29 CFR 1910.1200 - Hazardous Communication Standard

(Copies of this document are available online at www.gpoaccess.gov/cfr/index.html

or U.S.Government Printing Office, P.O. Box 979050, St. Louis, MO 63197-9000).

2.3 <u>Non-Government publications</u>. 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.
ASTM D185	-	Standard Test Methods for Coarse Particles in Pigments.
ASTM D522	-	Standard Test Methods for Mandrel Bend Test of Attached Organic
		Coatings.
ASTM D523	-	Standard Test Method for Specular Gloss.
ASTM D562	-	Standard Test Method for Consistency of Paints Measuring Krebs Unit
		(KU) Viscosity Using a Stormer Type Viscometer.
ASTM D610	-	Standard Practice for Evaluating Degree of Rusting on Painted Steel
		Surfaces.
<b>ASTM D1210</b>	-	Standard Test Method for Fineness of Dispersion of Pigment Vehicle
		Systems by Hegman Type Gage.
ASTM D1364	-	Standard Test Method for Water in Volatile Solvents (Karl Fischer Reagent
		Titration Method).
ASTM D1640	-	Standard Test Methods for Drying, Curing, of Film Formation of Organic
		Coatings at Room Temperature.
ASTM D1654	-	ı
		Subjected to Corrosive Environments.
ASTM D1729	-	± ±
		Diffusely Illuminated Opaque Materials.
ASTM D2369		$\epsilon$
ASTM D2371		$\epsilon$
ASTM D2805		
ASTM D3271	-	$\mathbf{J}$
		Gas Chromatograph for Solvent Analysis.
ASTM D3335	-	,
		Cobalt in Paint by Atomic Absorption Spectroscopy.
ASTM D3359		
ASTM D3363		<b>3</b>
ASTM D3891	-	
4 GFD 4 D 2024		Varnish, Lacquer, and Related Products.
ASTM D3924	-	
A CITINA D 20.60		and Testing Paint, Varnish, Lacquer, and Related Materials.
ASTM D3960	-	
A COTA DE COO		Content of Paints and Related Coatings.
ASTM D5380	-	· · · · · · · · · · · · · · · · · · ·
A CTM DAGG		Extenders in Paint by X Ray Diffraction Analysis.
AS IM D00//	-	Standard Test Method for Evaluating Adhesion by Knife.

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

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

# 3. REQUIREMENTS

- 3.1 Qualification. The coatings furnished under this specification shall be products which are authorized by the qualifying activity for listing on the applicable Qualified Products Database (QPD) at the time set for opening of bids (see 4.2 and 6.3). Any change in the formulation or processing of a qualified product shall necessitate its requalification. The material supplied under the contract shall be identical, within manufacturing tolerance, to the product that received qualification.
- 3.2 <u>Materials</u>. The materials used in the coating shall be as specified herein. Materials not specified shall be selected by the contractor and shall be subject to all provisions of this specification.
- 3.2.1 <u>Toxic products and formulations</u>. The material shall have no adverse effect on the health of the personnel when used for its intended purpose. Questions pertinent to this effect shall be referred by the contracting activity to the appropriate departmental medical service, which will act as an advisor to the contracting agency.
- 3.2.2 <u>Hazardous material</u>. The contractor shall comply with the hazardous material requirements of FED-STD-313 (see 6.4).
- 3.3 <u>Color</u>. When tested as specified in 4.5.4, the color of the primer shall be characteristic of the iron oxide red pigment.

## 3.4 Composition.

3.4.1 <u>Pigment</u>. The pigment portion of the primer shall conform to the requirements of table I when tested as specified in 4.5.5, 4.5.6, and 4.5.7. Lead or chromate (hexavalent) pigments shall not be used. Compounds of antimony, arsenic, beryllium, cadmium, cobalt, chromium, cyanide, lead, manganese, mercury, nickel and selenium shall be absent. Extender pigments shall be siliceous matter and barium sulfate, in any ratio, and shall not exceed the amount specified in table I. The use of the specified pigments does not guarantee that a product will meet all the requirements of this specification, as the choice of vendors, amounts, methods of dispersion and incorporation can significantly affect the quality of the end product. Other corrosion inhibiting pigments, in addition to the specified zinc phosphate in table I can be used.

TABLE I. Quantitative requirements of pigment.

	PERCENT BY WEIGHT		
PIGMENT	MINIMUM	MAXIMUM	
Iron oxide, red, color index- PR101	30.0	-	
Zinc phosphate <u>1</u> /	9.0	11.0	
Corrosion inhibiting pigment <u>2</u> /	0.9	1.1	
Siliceous extenders	-	60.0	
Barium sulfate	-	10.0	
Hexavalent chromium	Absent	Absent	

<sup>1/</sup> Zinc phosphate or metal phosphate complex.

- 3.4.2 <u>Lead and chromate (hexavalent) content</u>. When tested as specified, the lead content shall not exceed 0.06 percent by weight of the total nonvolatile content (see 4.5.8) and the test for hexavalent chromium shall be negative (see 4.5.7).
- 3.4.3 <u>Vehicle</u>. The vehicle shall be a drying oil alkyd, modified or unmodified.
- 3.4.4 <u>Solvent analysis</u>. When tested as specified in 4.5.9, solvents used shall be in accordance with the guidelines established by local, state and federal regulations and shall be volatile organic hazardous air pollutants-free (VOHAP-free).
- 3.5 <u>Quantitative characteristics</u>. When tested as specified in 4.5.2, the enamel shall conform to the quantitative requirements of table II.

TABLE II. Quantitative requirements of primer.

	Requirements	
Characteristics	Minimum	Maximum
VOC, grams volatile / liter of enamel reduced for	-	420
spray (see 4.5.18)		
Pigment: Hexavalent chromium, cadmium, cadmium	Absent	
compounds		
Inorganic HAP compounds	Absent	
VOHAP compounds	Absent	
Total solids, % by weight of primer	70	-

<sup>2/</sup> Heucorin RZ, Heubach GmbH Company or equivalent.

TABLE II. Quantitative requirements of primer - Continued

	Requirements	
Characteristics	Minimum	Maximum
Total pigment, % by weight of nonvolatile	-	70
Water, % by weight of enamel	-	0.5
Coarse particles and skins, % by weight of pigment	-	0.5
Specular gloss, 60 degree	2	6
Viscosity, reduced as specified in 4.5.11, KU	-	70
Fineness of grind	5	-
Drying time, air dry:		
Set to touch, minutes	-	6
Dry hard, minutes	-	12
Contrast ratio	0.98	-

# 3.6 Qualitative requirements.

3.6.1 <u>Condition in container</u>. When tested as specified in 4.5.14, a freshly opened full container of the enamel shall be free from grit, coarse particles, skins, lumps, seeds, livering or abnormal thickening. The enamel shall show no pigment settling or caking that cannot be readily reincorporated to a smooth homogeneous state.

#### 3.6.2 Storage stability.

- 3.6.2.1 <u>Full container</u>. When tested as specified in 4.5.15.1, a full quart of the enamel shall be free from coarse particles, grit, skins, lumps, seeds, livering, hard caking and tough, gummy sediment. The enamel shall remix readily to a smooth homogeneous state, shall show a maximum viscosity increase of 15 KU and shall meet all the requirements of this specification.
- 3.6.2.2 <u>Partially filled container</u>. When tested as specified in 4.5.15.2, the enamel shall show no skinning. After being aged as specified in 4.5.15.2, the enamel shall show no livering, curdling, seeding, hard caking, or tough gummy sediment. The enamel shall show no pigment settling or caking that cannot be readily re-incorporated to a smooth homogeneous state.
- 3.6.3 <u>Accelerated stability</u>. When tested as specified in 4.5.16, the enamel shall show no livering, curdling, hard caking, or tough gummy sediment and shall mix readily to a smooth homogenous state.
- 3.6.4 <u>Suspension properties</u>. When tested as specified in 4.5.17, the enamel shall completely redisperse to a smooth, homogeneous state.
- 3.6.5 <u>Spraying properties</u>. When tested as specified in 4.5.18, the enamel shall show no running, sagging or streaking. The dried film shall show no dusting, mottling, color separation, flooding or floating, and shall present a smooth, uniform finish free from defects.

- 3.6.6 <u>Flexibility</u>. When tested as specified in 4.5.19, a film of the enamel shall withstand bending without cracking or flaking.
- 3.6.7 <u>Knife test</u>. When tested as specified in 4.5.20, the enamel shall adhere tightly to and not flake or crack from the metal surface. The film shall ribbon or curl from the metal on cutting and the cut shall show beveled edges.
- 3.6.8 Water resistance. When tested as specified in 4.5.21, a film of the coating shall show no blistering or wrinkling and no more than a slight whitening or softening immediately upon removal from the water. Film softening shall not exceed a 2 pencil hardness difference (see ASTM D3363) from an unexposed film with identical cure history prior to water exposure. When examined two hours after removal, there shall be only a slight softening, whitening or dulling. After 24 hours air drying, the panel which was immersed shall be almost indistinguishable with regard to hardness, adhesion and general appearance from a panel prepared at the same time but not immersed.
- 3.6.9 <u>Hydrocarbon resistance</u>. When tested as specified in 4.5.22, a film of the coating shall show no blistering or wrinkling when examined immediately after removal from the hydrocarbon test fluid. When examined 2 hours after removal, there shall be no excessive softening, whitening, or dulling. Film softening shall not exceed a 2 pencil hardness difference (see ASTM D3363) from an unexposed film with identical cure history prior to hydrocarbon exposure. After 24 hours drying the portion of the panel which was immersed shall be the same with regard to hardness, adhesion, and general appearance from a panel prepared at the same time but not immersed.
- 3.6.10 <u>Adhesion</u>. The assessment of the adhesion of the coating film shall be determined by its ability to not peel from the substrate when tested in accordance with ASTM D3359, as specified in 4.5.23. The resultant test rating shall be classified as scale 4B or better.
- 3.6.11 <u>Salt spray resistance</u>. When tested as specified in 4.5.24 and examined immediately after removal from the salt spray test, films of the enamel primer coating shall have a minimum rating of No. 7, based on test method ASTM D1654, and the failure evaluation procedure for unscribed specimens, procedure B. Upon completion of the salt spray fog test, remove the primer coating film and inspect the substrate. There shall be no more than a trace of rusting, pitting, or corrosion on the panels.
- 3.6.12 <u>Weather resistance</u>. When prepared and exposed as specified in 4.5.25, films of the enamel primer coating shall show no 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 (see ASTM D610, table I, rust grade 9).
- 3.6.13 <u>Reactivity</u>. When subjected to the vacuum stability test as in 4.5.26, the reactivity of the primer with the explosives listed in table III shall meet the requirements of Test 1, Procedure A of STANAG 4147. Energetics that meet the reactivity requirements of Test 1, Procedure A of

STANAG 4147, shall have their compatibility verified with the "Onset of Decomposition" test specified in 4.5.28.

TABLE III. High Explosives.

a) TNT	h) HBU-88
b) C-4	i) PAX-2A
c) IMX-101	j) Comp-B5
d) DEMN-3J	k) PAX-3
e) HMX	1) PAX-21
f) Black powder	m) Comp-A3
g) Comp-A5	n) Comp-B

- 3.6.14 <u>Corrosion</u>. Panels tested as in 4.5.27 shall show no evidence of corrosion within one-eighth inch of the edge and shall show no breaks or cracks in the film.
- 3.6.15 Onset of Decomposition. When tested as in 4.5.28, mixtures of the dried paint film and the explosives listed in table III shall meet the temperature requirements as noted by Test 4 of STANAG 4147. Energetics that meet the onset of decomposition requirements of Test 4 of STANAG 4147 shall have their compatibility verified with the reactivity test specified in 4.5.26.
- 3.6.16 <u>User instruction marking</u>. All containers shall include the VOC content and VOHAP content in g/l or lbs/gal of coating, both un-thinned and when reduced as specified with the manufacturer's recommended HAP-free thinner. All containers shall be legibly marked or labeled with precautionary information as follows:

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 (MSDS). Keep containers closed.

- 3.6.17 <u>Material Safety Data Sheet</u>. The manufacturer shall comply with the requirements set forth by the Hazardous Communication Standard, 29 CFR 1910.1200. A MSDS shall be prepared for the coating in accordance with FED-STD-313 and forwarded to the qualifying activity (see 6.4). The MSDS for the material covered by this specification shall be included with each shipment, and submitted to pertinent Government agencies as stated in FED-STD-313.
- 3.6.17.1 <u>Safety Data Sheets (SDS)</u>. Safety Data Sheets (SDSs) shall be phased into the system by the following deadline of June 1, 2015 and after June 1, 2016 the Material Safety Data Sheets (MSDSs) becomes extinct and you must ensure that each hazardous chemical in your workplace has an SDS and only an SDS as specified in the OSHA Brief

http://www.osha.gov/dsg/hazcom/osha-brief.html and as specified in Appendix D of 29 CFR 1910.1200 (see:

http://www.osha.gov/pls/oshaweb/owadisp.show\_document?p\_table=standards&p\_id=10103\_).

- 3.6.18 <u>Toxic ingredients</u>. Other than parachlorobenzotrifluoride (PCBTF), the coating shall contain no benzene, chlorinated solvents or ethylene based glycol ethers and their acetates (see 4.5.29). The coating shall have no adverse effects on the health of personnel when used for its intended purpose.
- 3.7 <u>Toxicity clearance</u>. 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 6.3.4).

# 4. VERIFICATION

- 4.1 <u>Classification of inspections</u>. The inspection requirements specified herein are classified as follows:
  - a. Qualification inspection (see 4.2 and 6.3).
  - b. Conformance inspection (see 4.3).
- 4.2 <u>Qualification inspection</u>. Qualification shall be conducted by the qualifying activity (see 6.3). The qualification test sample shall consist of four quarts of the paint. The samples shall be legibly identified (see 6.3.1). Qualification inspection shall consist of tests for all requirements in section 3 and table IV and examination for user instruction marking (see 3.6.16). 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 the paint represented by the sample test shall not be approved for inclusion in the QPD under this specification.
- 4.3 <u>Conformance inspection</u>. The manufacturer shall forward from each production lot (see 4.3.1) a batch validation letter detailing the batch number, manufacturer's code, specification and type number, QPL number and batch volume to U.S. Army Research Laboratory (ARL), ATTN: RDRL-WMM-C, Organic Coatings Team, Building 4600, Deer Creek Loop, Aberdeen Proving Ground (APG), MD 21005-5066. The manufacturer shall perform conformance inspection testing on each production lot and have these test results on file when requested by the contracting officer. Conformance inspection shall consist of tests for VOC content, condition in container, total solids, viscosity, color, contrast ratio, fineness of grind, gloss, and dry time as specified in sections 3 and 4. There shall be no failures (see 6.5).
- 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). When required, the manufacturer shall furnish with each lot and/or batch a certified test report showing that the material has passed the conformance inspection, and that there has been no formulation or process change from that which resulted in the production of the qualification inspection sample. The addition of any substance to a batch shall constitute a new lot.

- 4.3.2 <u>Inspection conditions</u>. Unless otherwise specified, all inspections shall be performed in accordance with ASTM D3924. Also, unless otherwise stated in the test method or paragraph, room temperature shall be 73  $\pm$  4 °F (23  $\pm$  2 °C) and a 40 70 percent relative humidity.
- 4.4 <u>Hazardous material</u>. Failure to comply with the hazardous material requirements of FED-STD-313 shall be cause for rejection.

# 4.5 Test methods.

4.5.1 <u>Test conditions</u>. Except as otherwise specified herein, the routine testing and referee testing shall be conducted in accordance with ASTM D3924. A dry film thickness of  $1.5 \pm 0.2$  mils (37.5  $\pm$  5 microns) shall be used whenever film thickness is requested in any test, unless otherwise required by the test. Failure of any test result to fall within the specified ranges in section 3 shall constitute failure of the applicable test.

TABLE IV. Test method index.

	Test	Requirement	ASTM
Test	Paragraph	Paragraph	Method
Color	4.5.4	3.3	D1729
Pigment analysis	4.5.5	3.4.1	D2371
Extender pigment	4.5.6	Table I	D5380
Chromium content	4.5.7	3.4.2	-
VOC content	-	Table II	D3960
Nonvolatile matter	-	Table II	D2369
Pigment content	-	Table II	D2371
Lead content	4.5.8	3.4.2	D3335
Water	-	Table II	D1364
Coarse particles	-	Table II	D185
Solvent analysis <sup>1/</sup>	4.5.9	3.4.4	D3960, D3271
Specular gloss	4.5.10	Table II	D3891, D523
Viscosity, reduced	4.5.11	Table II	D562
Fineness of grind	-	Table II	D1210
Drying time	4.5.12	Table II	D1640
Contrast ratio	4.5.13	Table II	D2805
Condition in container	4.5.14	3.6.1	-
Storage stability	4.5.15	-	-
Full container	4.5.15.1	3.6.2.1	-
Partially full container	4.5.15.2	3.6.2.2	-
Accelerated stability	4.5.16	3.6.3	-
Suspension properties	4.5.17	3.6.4	-
Spraying properties	4.5.18	3.6.5	-
Flexibility	4.5.19	3.6.6	D522
Knife test	4.5.20	3.6.7	D6677

1/ EPA Method 311.

TABLE IV. Test meth	noa maex – Conunu
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	Test	Requirement	ASTM
Test	Paragraph	Paragraph	Method
Hydrocarbon resistance	4.5.22	3.6.9	-
Adhesion	4.5.23	3.6.10	D3359
Salt spray resistance	4.5.24	3.6.11	B117
Weather resistance	4.5.25	3.6.12	-
Reactivity	4.5.26	3.6.13	-
Corrosion	4.5.27	3.6.14	-
Onset of	4.5.28	3.6.15	-
decomposition			

- 4.5.2 <u>Test procedures</u>. Tests shall be conducted in accordance with table IV. The right is reserved to make any additional tests deemed necessary to determine that the coating meets the requirements of this specification.
- 4.5.3 <u>Test panels</u>. Unless otherwise specified, metal panels used for test purposes shall be two types:
  - a. Steel, cold rolled, pretreated with zinc phosphate coating B-952 with P60 chrome rinse, 0.032 inches (0.8128 mm) thick, for all tests except flexibility.
  - b. Steel, tinplated, 0.010 inches (0.254 mm) thick, for flexibility only.
- 4.5.4 <u>Color</u>. Determine the color in accordance with ASTM D1729 by applying films with a 0.002 inch (0.004 inch gap clearance) film applicator on a black and white hiding chart until complete hiding is obtained. Evaluate for compliance with 3.3.
- 4.5.5 <u>Pigment analysis</u>. Extract the pigment as in ASTM D2371, except use ethanol for the extraction. Make appropriate qualitative and quantitative tests on the extracted pigment to determine if only permissible pigments were used. Nonconformance to 3.4.1 shall constitute failure of this test.
- 4.5.5.1 <u>Antimony</u>, arsenic, beryllium, cadmium, cyanide, manganese, mercury, nickel and <u>selenium</u>. Manufacturers need to review applicable environmental and safety regulations and comply with material shipment requirements (see 6.2). The MSDS needs to include a notarized statement that verifies that the composition of the material (coating formulation) is free of the antimony, arsenic, beryllium, cadmium, cyanide, manganese, mercury, nickel, selenium and their compounds.
- 4.5.6 Extender pigment. Determine siliceous matter and barium sulfate by the applicable portions of ASTM D5380. Evaluate for compliance with table I.

- 4.5.7 <u>Chromium (hexavalent) content</u>. Determine the presence or absence of hexavalent chromium by either of the following chemical reagent screening techniques. Nonconformance to the 3.4.2 requirements shall constitute failure of this test.
  - a. Weigh approximately 250 mg of pigment into a small glass test tube. Add 5 ml of 25 percent aqueous KOH and shake vigorously for 1-2 minutes. Decant a one ml aliquot into a plastic micro-centrifuge tube and centrifuge for five (5) minutes at 17,000 rpm, balancing the centrifuge with a tube containing one ml of the KOH solution. The resulting supernatant liquid shall be nearly colorless. Use the tube containing the KOH solution as a reference. A distinct yellow color indicates the presence of hexavalent chromium and therefore shall constitute failure of the test requirement.
  - b. Weigh approximately 50 mg of pigment into a small glass test tube. Add 5ml of 10 percent aqueous H<sub>2</sub>SO<sub>4</sub> and shake vigorously for 1-2 minutes. Decant a one ml aliquot into a plastic micro-centrifuge tube and centrifuge for five (5) minutes at 17,000 rpm, balancing the centrifuge with a second tube containing one ml of the H<sub>2</sub>SO<sub>4</sub> solution. The solution in the second tube shall act as the test "blank". Immerse a chromate ion (CrO<sub>4</sub><sup>2-</sup>) test strip into the supernatant from each tube. Shake off the excess liquid and after approximately one minute compare the color change in the reaction zone of the strips to the color scale (concentration levels) provided by the manufacturer. The appearance of a purple/violet color from the "sample" strip is indicative of hexavalent chrome and therefore shall constitute failure of the test requirement. If a color change is apparent from the "blank" strip rerun the test using fresh, chromium-free reagents. The appearance of a pronounced color change is indicative of hexavalent chrome and therefore shall constitute failure of the test requirement.
- 4.5.8 <u>Lead content</u>. Determine the presence or absence of lead by weighing 50 mg of pigment into a small glass test tube. Add 5 ml of dilute HNO<sub>3</sub> and shake vigorously for 1-2 minutes. Decant a one ml aliquot into a plastic micro centrifuge tube and centrifuge for five (5) minutes at 17,000 rpm, balancing the centrifuge with a second tube containing one ml of the dilute HNO<sub>3</sub> solution. The solution in the second tube shall act as the test "blank". Immerse a lead ion test strip into the supernatant from each tube. Shake off the excess liquid and after approximately one minute compare the color change in the reaction zone of the strips to the color scale (concentration levels) provided by the manufacturer. If a color change is apparent from the "blank" strip rerun the test using fresh, lead-free reagents. Confirmation of the exact lead concentration in the coating's solids shall be quantified using ASTM D3335. Nonconformance to the 3.4.2 requirements shall constitute failure of this test.

## 4.5.9 Solvent analysis.

- 4.5.9.1 <u>Solvent analysis for VOC content determination</u>. Determine the VOC content in accordance with ASTM D3960. Check for compliance with table II.
- 4.5.9.2 <u>VOHAP content</u>. Hazardous solvent content of the reduced enamel shall be determined in accordance with ASTM D3271 or EPA Method 311, as applicable. Check for compliance with table II.

- 4.5.10 <u>Specular gloss</u>. Prepare a 0.002 inch dry film thickness drawdown of the enamel using a film applicator with the appropriate gap clearance on a glass panel according to ASTM D3891, and air dry for 24 hours. Measure the 60 degree specular gloss in accordance with ASTM D523 and evaluate for compliance with table II.
- 4.5.11 <u>Viscosity (reduced)</u>. Reduce eight parts, by volume, of the enamel with one part, by volume, of HAP-free thinner recommended by the manufacturer. Measure the viscosity in accordance with ASTM D562 and evaluate for compliance with table II.
- 4.5.12 <u>Drying time</u>. Prepare a 0.002 inch dry film thickness drawdown of enamel using a film applicator with the appropriate gap clearance on a glass panel and air dry for the specified time. Check the drying time in accordance with ASTM D1640 and evaluate for compliance with table II.
- 4.5.13 <u>Contrast ratio</u>. Prepare a 0.002 inch dry film thickness drawdown of the enamel using a film applicator with the appropriate gap clearance and allow to air dry for 24 hours. Determine the contrast ratio in accordance with ASTM D2805 and evaluate for compliance with table II.
- 4.5.14 <u>Condition in container</u>. Upon opening a full previously unopened container the condition of the contents shall be examined for compliance with 3.6.1. Reseal, then agitate the container for 3 minutes on a paint shaker and examine for compliance with 3.6.1. On re-examination of the contents, the disclosure of any gel bodies or undispersed pigment indicates unsatisfactory settling properties.

# 4.5.15 Storage stability.

- 4.5.15.1 <u>Full container</u>. Allow a full standard quart can of the enamel to stand undisturbed for one year at standard conditions (see 4.3.2) and then re-examine for pigment settling or caking as specified in 4.5.14. Determine viscosity and perform other applicable tests. Nonconformance to 3.6.2.1 shall constitute failure of this test.
- 4.5.15.2 <u>Partially filled container</u>. Fill a 1 pint friction top can three-fourths full with enamel. Secure the top tightly and invert the can momentarily. Store the can in an upright position for 48 hours and check for skinning. Reseal the can and age for 72 hours at 60 °C (140 °F). After the aging period, evaluate for compliance with 3.6.2.2.
- 4.5.16 <u>Accelerated stability</u>. Fill an 8 ounce wide-mouth glass jar, approximately 4 1/2 inches high and 2 inches in diameter, with the packaged enamel. Secure the cover tightly and invert the jar momentarily to check for leaks. Place the sample in a  $60 \pm 2$  °C ( $140 \pm 4$  °F) oven in an upright position for 7 days. After this period, allow to cool to room temperature and examine the contents. Non-conformance to 3.6.3 shall constitute failure of this test.
- 4.5.17 <u>Suspension properties</u>. Reduce the enamel as specified in 4.5.11. 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 4.5.14 and agitate the

contents for 20 seconds. Reexamine the material for any evidence of non-homogeneity or undispersed pigment. Nonconformance to 3.6.4 shall constitute failure of this test.

- 4.5.18 Spraying properties. If reduction is necessary for spray application, reduce the enamel as specified in 4.5.11. Spray the coating on a solvent cleaned test panel (see 4.5.3) to a dry film thickness of  $1.5 \pm 0.2$  mils (37.5  $\pm 5$  microns). Observe the spraying properties for compliance with 3.6.5.
- 4.5.19 <u>Flexibility</u>. Determine flexibility in accordance with ASTM D522, method B. Spray the coating on a steel panel, tinplated 0.010 inches (0.254 mm) thick to a dry film thickness of  $1.5 \pm 0.2$  mils (37.5  $\pm 5$  microns). Air dry for 168 hours. Bend the coated panels over a  $\frac{1}{4}$  inch mandrel. Examine the coating for cracks over the area of the bend for compliance with 3.6.6.
- 4.5.20 <u>Knife test</u>. Using the flat portion of the panel used for the flexibility test (see 4.5.19), perform this test in accordance with ASTM D6677 and check for compliance with 3.6.7.
- 4.5.21 Water resistance. Spray the coating on a test panel (see 4.5.3) to a dry film thickness of  $1.5 \pm 0.2$  mils (37.5  $\pm$  5 microns) and air dry for 168 hours. Coat all exposed unpainted metal surfaces with wax or suitable protective coating and immerse in deionized water at  $25 \pm 1$  °C (77  $\pm 2$  °F) for 18 hours. Panels shall be immersed at a minimum depth of 50%. At the end of the test period, remove and examine for compliance with 3.6.8.
- 4.5.22 <u>Hydrocarbon resistance</u>. Spray the coating on a test panel (see 4.5.3) to a dry film thickness of  $1.5 \pm 0.2$  mils ( $37.5 \pm 5$  microns) and air dry for 168 hours. Immerse halfway in a hydrocarbon fluid conforming to JP8 at  $70 \pm 5$  °F ( $21 \pm 3$  °C) for 4 hours. Evaluate for compliance with 3.6.9.
- 4.5.23 <u>Adhesion</u>. Spray the coating as in 4.5.18 on a steel panel pretreated as in 4.5.3. Air dry the specimen for seven (7) days. Perform adhesion testing as specified in ASTM D3359, method B and examine for compliance with 3.6.10.
- 4.5.24 Salt spray resistance. Prepare three 4 x 12 inch steel panels as specified in 4.5.3. Reduce the enamel as specified in 4.5.11 and spray the panels to a dry film thickness of  $1.5 \pm 0.2$  mils  $(37.5 \pm 5 \text{ microns})$  and air dry for 7 days. Coat edges and uncoated metal surfaces with wax or other suitable coating, but do not score. The enamel-coated panels shall be exposed to a 5 percent salt spray fog test in accordance with ASTM B117. Panels shall be exposed for 48 hours. Upon completion of the test, panels shall be removed and washed gently in running water, no warmer than  $100 \,^{\circ}\text{F}$  (38  $^{\circ}\text{C}$ ), until surface is free from any visible salt deposits and then immediately examined for compliance with 3.6.11. Strip the enamel from the panels and inspect the panels for rust, pitting or corrosion. Nonconformance to 3.6.11 shall constitute failure of this test.
- 4.5.25 <u>Weather resistance</u>. Prepare two panels as specified in 4.5.24. Allow to air dry for a minimum of 7 days. Panels shall be placed outdoors for 24 months. The exposure racks shall be weathered at latitude 33° 23' north and 112° 35' west. At the end of the exposure, strip the enamel film from the surface of the metal and examine it for conformance with 3.6.12. Nonconformance to 3.6.12 shall constitute failure of this test.

- 4.5.26 <u>Reactivity.</u> Determine the reactivity of the paint in contact with the explosives listed in table III using the vacuum stability test as specified by Test 1, Procedure A of STANAG 4147. The reactivity shall be determined before and after storage for 60 days at 71° C and ambient relative humidity and at 71° C and 100 percent relative humidity. The 100 percent relative humidity test shall not be made on the black powder which is deteriorated by moisture. Prepare the samples as specified in 4.5.26.1.
- 4.5.26.1 Preparation of samples. A sufficient amount of the paint to provide approximately 140 grams of dried film shall be poured on glass or stainless steel plates. The films shall be air dried under ambient conditions for 48 hours, or until no longer tacky, and then peeled off with a sharp edged tool in strips. These strips are then suspended on glass rods in an oven or cabinet with circulating air at a temperature not to exceed 30 °C for 48 hours. If an air circulating oven is not available, it will be satisfactory to place the suspended strips before a fan in a warm room for 48 hours. The paint strips are then removed, cut into smaller pieces, and then ground in a mortar fine enough to pass through a 16-mesh sieve. The explosives shall be reduced by grinding or rasping to a fineness of 20 mesh or less. The black powder shall not be ground, but is to be used in the granulation furnished for the test. Ten samples shall then be prepared consisting of 10 grams of each of the explosives listed in table III (with the exception of black powder) with 10 grams of the paint. With the black powder, 7.0 grams of each shall be mixed with 7.0 grams of paint. The mixing shall be thorough using a wood spatula with the mixture on a piece of glazed paper. After mixing, the mixtures shall be spread out on large watch glasses, and dried in a desiccator at room temperature for 24 hours. They are then placed in dry glass bottles with rubber or cork stoppers and reserved for tests. At the same time, 10-gram samples of each of the explosives and a 20gram sample of the paint are prepared and bottled in the same way. Samples are taken directly from these bottles to make the initial vacuum stability and onset of decomposition tests as required in sections 4.5.26.3 and 4.5.28. For the storage tests, the remainder of the mixed samples and controls, after the initial tests, are divided into equal portions and placed in separate bottles. One series of the samples is arranged, unstoppered, in individual Mason jars, sealed under ambient conditions, and placed in a surveillance oven at 71° C. The other series of mixtures and controls, except the black powder, is placed, also unstoppered, in individual Mason jars, each containing a test tube with 10 cc of water. The bottles containing the samples are protected in such a way that condensation from the top of the Mason jar will not fall into the sample bottle. The jars are fitted with rubber gaskets, closed, and placed in a surveillance oven at 71° C. After 60 days, the samples of both series are removed, dried in a circulating oven or cabinet at approximately 35° to 40° C for 48 hours, and tested according to sections 4.5.26.3 and 4.5.28.
- 4.5.26.2 <u>Calibration</u>. Calibration of the glass tube shall be as specified by Test 1, Procedure A of STANAG 4147.
- 4.5.26.3 <u>Testing procedure</u>. The procedure shall be as specified by Test 1, Procedure A of STANAG 4147. Two and one-half gram portions of the dried paint, as controls, shall be added to each of two tubes~ and 2-1/2 grams of each of the explosives, also as controls, shall be added to individual tubes. Five-gram samples of each of the mixtures of any one of the series prepared under 4.5.26.1, after drying for 24 hours in a desiccator at room temperature, are placed in individual tubes. After assembly, the test is run as specified by Test 1, Procedure A of STANAG 4147 for 40 hours at 100°C. All readings shall be made with the sample removed from the bath

and at room temperatures. The readings of both the controls and the test samples shall then be corrected to standard conditions of temperature and pressure and checked for compliance with Test 1, Procedure A of STANAG 4147.

- 4.5.26.4 <u>Calculation of reactivity</u>. The reactivity of each of the explosive materials with the coating compound shall be calculated as specified by Test 1, Procedure A of STANAG 4147.
- 4.5.27 Corrosion. Coat on both sides 26 smooth SAE 1020 steel panels, approximately 4 x 2 x 1/20 inches, with  $0.001 \pm 0.0002$  inch of the primer and thoroughly dry. Using two panels for each test, make into 12 sandwiches with slabs or compressed pellets of each explosive shown in Table III. The slabs or pellets shall be at least one-eight inch in thickness, and shall be held in close contact with the coated surfaces of the panel by means of tying the sandwich together with a cotton string or cord. Each sandwich is then placed in a separate glass jar in an inclined position under ambient conditions, then closed and placed in a controlled temperature oven at 71° C for 60 days. Two of the coated panels shall be stored under the same conditions, to serve as controls for all of the explosives under tests. After 60 days, the samples are removed, the panels separated from the sandwiches and the surfaces, which have been in contact with the explosives examined visually for cracks, pits, or other signs of corrosion as specified under 3.6.14. The control panels will serve as a comparison in this examination.
- 4.5.28 Onset of Decomposition. Determine the onset of decomposition in duplicate on mixtures of the dried paint film and the explosives listed in table III both prior to and after storage in closed glass containers for 60 days under ambient relative humidity at 71° C and under 100 percent relative humidity at 71° C. The amount of sample prepared and testing shall be conducted in accordance with Test 4 Differential Scanning Calorimetry, of STANAG 4147. The onset of decomposition shall be in compliance with the requirements of Test 4 of STANAG 4147.
- 4.5.29 <u>Toxic ingredients</u>. Other than PCBTF, the manufacturer shall certify that the coating contains no benzene, chlorinated solvents or ethylene based glycol ethers and their acetates. Check for compliance with 3.6.18. Nonconformance to 3.6.18 shall constitute failure of this test.

## 5. PACKAGING

5.1 <u>Packaging and markings</u>. 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. Containers of paint shall be marked with a label that identifies the specification number, QPL number and material designation so that traceability to the QPD can be verified.

#### 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 covers an alkyd primer that when reduced as specified shall have a maximum volatile organic compound (VOC) content of not more than 420 grams per liter (g/l) (3.5 pounds/gal (lbs/gal)). This primer is also formulated free of hazardous air pollutants (HAP-free). The intended use is on properly solvent cleaned and pretreated metal surfaces where exposure to lead or chromate pigments is not permitted. This primer is intended for painting the interior of ammunition items; such as bombs, shells, rockets and mines prior to being filled with explosives. It may also be used for the exterior surfaces of these items as a primer. It is not intended for use on the inside of potable water tanks or for marine use. This primer is not to be used with chemical agent resistant coating (CARC) primers or topcoats.
- 6.2 <u>Acquisition requirements</u>. Acquisition documents should specify the following:
  - a. Title, number, and date of the specification.
  - b. Required markings (see 3.6.16).
  - c. Whether a MSDS is required with each shipment (see 3.6.17 and 4.5.5.1).
  - d. If a toxicity clearance is required (see 3.7).
  - e. If qualification samples are required and where to send them (see 4.2 and 6.3).
  - f. If conformance samples are required and where to send them (see 4.3).
  - g. Lot and batch formation (see 4.3.1).
  - h. Packaging requirements (see 5.1).
- 6.2.1 <u>Basis of purchase</u>. The enamel covered by this specification should be purchased by volume, the unit being one U.S. liquid gallon of 231 cubic inches at 68 °F (20 °C).
- 6.3 Qualification. With respect to products requiring qualification, awards will be made only for products which are, at the time of award of contract, qualified for inclusion in the QPD, whether or not such products have actually been so listed by that date. The attention of contractors is called to this requirement and manufacturers are urged to arrange to have the products that they propose to offer to the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or purchase orders for the products covered by this specification. Samples for QPD testing (see 4.2) and for the extension testing program (see 4.3) should be submitted to ARL, ATTN: RDRL-WMM-C, Organic Coatings Team, Building 4600, Deer Creek Loop, APG, MD 21005-5066. Listings of qualified products can be obtained online through ASSIST.
- 6.3.1 <u>Sample identification for qualification inspection</u>. Samples for QPD testing and for the extension testing program are to be identified in a cover letter with the following information:

Manufacturer's name and product number.

Submitted by (name and date).

Specify the number of samples.

Specify the reason for submitting the samples.
Specification MIL-DTL-11195H; Type II, Color, Color Number
"Enamel, Lusterless, Fast Dry, VOC Compliant, (For Use On Ammunition And Other
Metals)".
Provide a copy of the MSDS.
Provide a copy of the notarized statement of composition.
Provide a copy of the technical data sheet.
Provide a copy of the test report.

- 6.3.2 <u>Conformity to qualified sample</u>. 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.
- 6.3.3 <u>Retention of qualification</u>. To retain qualification of products approved for listing in the QPD, the manufacturer will be requested to verify by certification to the qualifying activity that its product(s) comply with the requirements of this specification. Unless otherwise specified by the qualifying activity, the time of periodic verification by certification will be in two year intervals from the date of original qualification and will be initiated by the qualifying activity.
- 6.3.4 <u>Toxicity clearance request</u>. 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, ATTN: MCHB-TS-TTE, 5158 Blackhawk Road, APG, MD 21010-5403.
- 6.4 <u>MSDS</u>. 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.
- 6.5 <u>Conformance rejection and retest</u>. Failure in any conformance inspection will result in the rejection of the batch from which it was obtained. Rejected material cannot be resubmitted for acceptance without written approval from the qualification activity (see 4.4). The application for resubmission will contain all details concerning previous rejections and measures taken to correct these deficiencies.
- 6.6 Subject term (key word) listing.

Ammunition HAP-free Lead Pigment Reactivity Surfaces

6.7 <u>Changes from previous issue</u>. 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**

Custodians:

Army - MR Navy - SH

Air Force - 99

Review activities:

Army - MD1, MI Navy - AS, CG, MC

Air Force - 11, 84

Civil agency: GSA/FAS

Preparing activity: Army – MR

Project 8010-2013-014

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 <a href="https://assist.dla.mil">https://assist.dla.mil</a>/.