

NOT MEASUREMENT  
SENSITIVE

MIL-DTL-3688D  
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SUPERSEDING  
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DETAIL SPECIFICATION  
WAX EMULSION (RUST INHIBITING)

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

1. SCOPE

1.1 Scope. This specification covers a type of wax emulsion suitable for use as a rust inhibiting coating for general weatherproofing when applied to articles (see 6.1).

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

FEDERAL SPECIFICATIONS

QQ-S-698 - Steel, Sheet and Strip, Low Carbon .

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

MIL-DTL-3688D

FEDERAL STANDARDS

- FED-STD-141 - Paint, Varnish, Lacquer and Related Materials: Methods Of Inspection, Sampling and Testing.

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

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

American Society for Quality (ASQ)

- ASQC-Z1.4 - Sampling Procedures and Tables for Inspection by Attributes. (DoD adopted)

(Copies of this document are available from [www.asq.org](http://www.asq.org) or American Society for Quality, 600 Plankinton Avenue, Milwaukee, WI 53203.)

ASTM INTERNATIONAL

- ASTM B117 - Standard Practice for Operating Salt Spray (Fog) Apparatus. (DoD adopted)
- ASTM D823 - Standard Practice for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels. (DoD adopted)
- ASTM D3891 - Standard Practice for Preparation of Glass Panels for Testing Paint, Varnish, Lacquer, and Related Products
- ASTM D5347 - Standard Test Method for Determination of the Ash Content of Fats and Oils.
- ASTM D5486/D5486M - Standard Specification for Pressure-Sensitive Tape for Packaging, Box Closure, and Sealing. (DoD adopted)

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

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

## MIL-DTL-3688D

## 3. REQUIREMENTS

3.1 Material. The material furnished under this specification shall be a uniform, stable aqueous emulsion of waxes, resins, and emulsifying agents together with suitable rust inhibitors capable of meeting all the requirements of this specification. The emulsion shall be supplied ready for application at room temperature  $77^{\circ} \pm 5^{\circ}\text{F}$  ( $25^{\circ} \pm 2^{\circ}\text{C}$ ) by spray, dip or brush methods.

3.2 Recycled, recovered, or environmentally preferable materials. Recycled, recovered, or environmentally preferable materials shall be used to the maximum extent possible provided that the material meets or exceeds the operational and maintenance requirements and promotes economically advantageous life cycle costs.

3.3 pH value. The pH of the wax emulsion shall be between 8.0 and 9.0 when tested as specified in 4.4.1.

3.4 Volatile matter. The emulsion shall be free from volatile organic solvents when tested as specified in 4.4.2. The volatile shall be water with not more than 1.0 percent by weight perfume or perfume solvents.

3.5 Nonvolatile matter (total solids). The nonvolatile content of the wax emulsion shall be not less than 12 percent by weight when tested as specified in 4.4.3.

3.6 Wax and resin content. The wax and resin content of the emulsion shall be not less than 8 percent by weight when tested as specified in 4.4.4.

3.7 Ash content. The ash content of the wax emulsion shall not exceed 0.5 percent when tested as specified in 4.4.5.

3.8 Free alkali. There shall be no free alkali present when tested as specified in 4.4.6.

3.9 Softening point (dropping point). The softening point of the wax shall be not less than  $165^{\circ}\text{F}$  ( $74^{\circ}\text{C}$ ) when tested as specified in 4.4.7.

3.10 Stability to centrifuging. The emulsion shall be free from particles, granules and foreign agents as shown by centrifuging as in 4.4.8. The material shall remain homogeneous and no more than 3 percent by weight shall settle out.

3.11 Drying time. The wax emulsion shall dry to a hard flexible surface within 20 minutes and shall form a non-tacking film when tested as specified in 4.4.9.

3.12 Appearance of dried film. A film of the wax, prepared and tested as specified in 4.4.10, shall show no signs of whiteness and shall have a smooth, lustrous finish. The film shall be clear, free from particles and practically colorless.

3.13 Dipping properties. The unreduced wax emulsion shall dip and flow out in a continuous film and shall deposit 0.30 gm maximum per  $\text{ft}^2$  of coating when prepared and tested as in 4.4.11.

## MIL-DTL-3688D

3.14 Spraying properties. The unreduced wax emulsion shall spray and flow out in a continuous film and shall deposit 0.30 gm maximum per ft<sup>2</sup> of coating when prepared and tested as in 4.4.12.

3.15 Brushing properties. The unreduced wax emulsion shall brush and flow out in a continuous film and shall deposit 0.30 gm maximum per ft<sup>2</sup> of coating when prepared and tested as in 4.4.13.

3.16 Adhesion of tape to wax film. When tested, both before and after aging, as specified in 4.4.14, the adhesion of the tape shall not be less than 12 oz per inch width.

3.17 Flexibility. The wax coating shall not crack or chip from the panel when tested as specified in 4.4.15.

3.18 Resistance to salt spray corrosion. When examined visually there shall be no corrosion of the wax coated steel panels after exposure to salt spray as specified in 4.4.16.

3.19 Accelerated storage stability. The wax emulsion shall remain stable and shall show no caking, thickening or any other significant change in properties after storage as specified in 4.4.17.

#### 4. VERIFICATION

4.1 Classification of inspections. The inspection requirements specified herein are classified as conformance inspection (see 4.2).

#### 4.2 Conformance inspection.

4.2.1 Conformance inspection of filled containers. Each sample filled container, as specified in 4.3.2, shall be examined for defects of the closure and for evidence of leakage. Each filled container shall also be weighed to determine the amount of contents. Any container having one or more defects or not filled as required shall be rejected and the lot (see 4.3.1) represented by the container shall be rejected.

4.2.2 Testing for lot conformance tests. The samples selected in accordance with 4.3.3 shall be subjected to all the tests specified in 4.4. Acceptance of the wax emulsion shall be based upon compliance of the material with the requirements of this specification.

#### 4.3 Sampling.

4.3.1 Lot. For purposes of sampling and testing, a lot shall consist of one batch of wax emulsion, produced by one manufacturer, in accordance with the same specification revision. Each batch shall consist of that quantity of wax emulsion that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogeneous.

4.3.2 Sampling of filled containers. Samples shall be selected at random from each lot in accordance with ASQC-Z1.4 to verify compliance with this specification in regard to fill, closure and other requirements.

## MIL-DTL-3688D

4.3.3 Sampling for conformance tests. From each inspection lot (see 4.3.1), containers shall be selected at random, in accordance with ASQC-Z1.4. From each of the containers, 1 quart samples shall be used in the following tests.

4.4 Test methods.

4.4.1 pH value. Add 10 ml of distilled water to 10 ml of the wax emulsion and determine the pH with a meter using state of the art electrodes. Check for compliance with 3.3.

4.4.2 Volatile matter. About 100 ml of the wax emulsion shall be distilled and the distillate tested for specific gravity. Check for compliance with 3.4.

4.4.3 Nonvolatile matter (total solids). Accurately weigh 2 to 3 gm of the sample in a tared aluminum weighing dish about 8 cm in diameter. Spread the material over the bottom. Heat the material in an oven at 221° to 230°F (105° to 110°C) for about 4 hours and then cool. Weigh the dish and contents. Repeat the heating until the last two weightings do not differ by more than 0.5 mg. Usually a second heating of 2 hours will be sufficient. Calculate the percentage of nonvolatile matter as follows and check for compliance with 3.5.

$$\text{Percent nonvolatile} = \frac{B}{A} \times 100$$

where A = weight (gm) before drying  
B = weight (gm) after drying

4.4.4 Wax and resin content. Weigh from 2 to 5 gm of the emulsion into a 250 ml Erlenmeyer or round-bottom flask. Add several anti-bumping stones or Berl saddles and 75 ml of methyl amyl ketone (MAK). Attach a Dean-Stark trap and condenser. Reflux on a hot plate or mantle until the trap is filled with the mixed water and MAK, then continue for an additional hour. Cool and filter the contents of the flask through paper of medium porosity into a weighed 250 ml beaker. Wash the filter contents with additional MAK and evaporate the MAK in a hot water bath with the aid of a current of air, then dry in a 140°F (60°C) oven for several hours. Cool in a desiccator and weigh. Calculate the percent of wax and resin combined and check for compliance with 3.6. Retain for softening point test (see 4.4.7).

4.4.5 Ash content. Determine the ash content of the emulsion using a sample of about 10 gm according to ASTM D5347. Check for compliance with 3.7.

4.4.6 Free alkali. Twenty ml of distilled water shall be added to a 10 ml sample of the wax emulsion and the mixture boiled, keeping the volume constant by the addition of water, until any ammonia vapor has been driven off. After all ammonia has been removed as determined by testing periodically by wet indicator paper held over the sample, the sample shall be cooled, 100 ml of neutral ethyl alcohol (94% or higher) added and the mixture agitated. It shall then be allowed to settle and the liquid decanted through filter paper. A few drops of phenolphthalein indicator shall be added. If any pink color appears, the emulsion contains free alkali and fails to meet the requirements for free alkali in compliance with 3.8.

4.4.7 Softening point. A small amount of the material isolated in paragraph 4.4.4 shall be tested for softening point by sandwiching between two micro cover glasses and placing on the stage of

## MIL-DTL-3688D

a Fisher-Johns melting point apparatus. The temperature increase shall not exceed 35.6°F (2°C) per minute. Record the temperature at which the sample is completely liquefied. Check for compliance with 3.9.

4.4.8 Stability to centrifuging. A sample of the wax emulsion shall be centrifuged for 15 minutes at a temperature of 100° ± 2°F (38° ± 1°C) and a speed of 750 revolutions per minute. Check for compliance with 3.10.

4.4.9 Drying time. Draw down a film of wax emulsion with a 3 ml drawdown knife on a clear glass plate prepared as in ASTM D3891. The thickness of the dried emulsion shall be 1.5 ml ± 0.2 ml. Perform the "free from after-tack" procedure described in Method 4061.3 of FED-STD-141 for compliance with 3.11.

4.4.10 Appearance of dried film. Glass plates with wax emulsion, prepared and dried as in 4.4.9, shall be viewed through the under side with bright daylight transmitted through it. The film shall comply with the requirements of 3.12.

4.4.11 Dipping properties. Obtain two phosphate coated, low carbon steel (SAE 1010) panels, 4 x 12 inch x 0.026 inch thick (10 x 30 cm x 0.065 cm) conforming to QQ-S-698 (see 6.3). Record weight of phosphate coated panel and begin dip application under conditions according to ASTM D823 method B. One minute after immersion, withdraw panel instantly and keep it in a vertical position for approximately 30 minutes. Air dry the panel to a constant weight. Subtract weight of phosphate coated panel from weight of wax coated panel and record difference as weight of wax coating. Compute a gm/ft<sup>2</sup> of coating, based on a test area coverage of 2/3 ft<sup>2</sup>. Observe dipping properties for compliance with 3.13.

4.4.12 Spraying properties. Obtain two phosphate coated, low carbon steel (SAE 1010) panels, 4 x 12 inch x 0.026 inch thick (10 x 30 cm x 0.065 cm) as in 4.4.11. Record weight of phosphate coated panel and spray the unreduced wax emulsion on both sides of the panel to produce a uniform coating surface. Dry the panel in a vertical position and compute weight per ft<sup>2</sup> of coatings as in 4.4.11. Observe spraying properties for compliance with 3.14

4.4.13 Brushing properties. Obtain two phosphate coated, low carbon steel (SAE 1010) panels, 4 x 12 inch x 0.026 inch thick (10 x 30 cm x 0.065 cm) as in 4.4.11. Record weight of phosphate coated panel, and brush the unreduced wax emulsion on both sides of the panel and examine as it dries. Dry the panel in a vertical position and compute weight per ft<sup>2</sup> of coating as in 4.4.11. Observe brushing properties for compliance with 3.15.

4.4.14 Adhesion of tape to wax film.

4.4.14.1 Apparatus.

- (a) Eight phosphate coated, low carbon steel (SAE 1010) panels, 4 x 12 inch x 0.026 inch thick (10 x 30 cm x 0.065 cm) as in 4.4.11. Cut these panels to 1 ½ inch x 4 inch (3.8 x 11.4 cm).
- (b) Eight strips of tape, each 1 x 8 inch (2.5 x 20 cm), conforming to ASTM D5486/D5486M.

## MIL-DTL-3688D

- (c) Use a commercially available rubber covered inking roller, 2 inch (5.0 cm) diameter x 3 inch (7.5 cm) width.

4.4.14.2 Preparation of test specimens. The panels, prepared as in 4.4.14.1 (a), shall be dipped in wax emulsion and allowed to dry for 30 minutes at room temperature  $77^{\circ} \pm 5^{\circ}\text{F}$  ( $25^{\circ} \pm 2^{\circ}\text{C}$ ). Apply a tape strip (see 4.4.14.1 (b)) to each coated panel with the adhesive face down and in a manner such that 4 1/2 inch (11.4 cm) of the tape extends beyond one end of the panel. Pass the roller twice over the surface of the tape, once in each direction, using only the weight of the roller in applying the tape to the panel.

4.4.14.3 Unaged. Using four of the test specimens prepared as in 4.4.14.2, double the free end of the tape and pull the adhering tape from the panel at a rate of 20 inches (50 cm) per minute. The test shall be performed at a temperature of  $70^{\circ}$  to  $80^{\circ}\text{F}$  ( $21^{\circ}$  to  $27^{\circ}\text{C}$ ) and a relative humidity of 50 to 60 percent. The test shall be conducted within 2 minutes after the tape has been applied to the panel.

4.4.14.4 Aged. Four of the test specimens, prepared as in 4.4.14.2, shall be aged in a drying oven maintained at  $145^{\circ} \pm 5^{\circ}\text{F}$  ( $63^{\circ} \pm 3^{\circ}\text{C}$ ) and not less than 50 percent humidity for 24 hours. At the end of 24 hours, remove the panels from the oven and allow them to stand for two hours at  $70^{\circ}$  to  $80^{\circ}\text{F}$  ( $21^{\circ}$  to  $27^{\circ}\text{C}$ ). They shall then be tested for adhesion as specified in 4.4.14.3.

4.4.15 Flexibility. Obtain two phosphate coated, low carbon steel (SAE 1010) panels, 4 x 12 inch x 0.026 inch thick (10 x 30 cm x 0.065 cm) as in 4.4.11. The panels shall be prepared as outlined in 4.4.14.2 except that they shall be allowed to dry for 48 hours at room temperature ( $77^{\circ} \pm 2^{\circ}\text{F}$ ) ( $25^{\circ} \pm 2^{\circ}\text{C}$ ). At the end of 48 hours, subject the panels to a  $180^{\circ}$  bend over a stationary 1/8 inch (0.32 cm) diameter mandrel. Examine for compliance with 3.17.

4.4.16 Resistance to salt spray corrosion. Obtain six phosphate coated, low carbon steel (SAE 1010) panels, 4 x 12 inch x 0.026 inch thick (10 x 30 cm x 0.065 cm) as in 4.4.11 and air dry for 72 hours. After air drying, expose the unscored panels to 5 percent salt spray for 6 hours according to ASTM B117. Upon removal, examine for corrosion. Strip the wax coating from the panels by solvent cleaning. Re-examine for compliance with 3.18.

4.4.17 Accelerated storage stability. A one quart container of the wax emulsion (see 4.3.3) shall be stored at  $145^{\circ} \pm 5^{\circ}\text{F}$  ( $63^{\circ} \pm 2^{\circ}\text{C}$ ) for a period of 15 days. At the end of this time, the sample shall be examined for compliance with 3.19.

## 5. PACKAGING

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

## MIL-DTL-3688D

### 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 rust inhibiting wax emulsion covered by this specification is military unique. It is used as a combination preservative and rust inhibitor on cartridge cases for major caliber artillery.

6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number, and date of this specification.
- b. Size of panels desired.
- c. Quantity of panels desired.
- d. Capacity of containers and type of container to be furnished.
- e. Packaging requirements (see 5.1).

6.3 Panel suppliers. Phosphate coated low carbon steel panels (SAE 1010), that conform to the requirements of this specification, may be obtained from the following suppliers:

ACT Laboratories  
273 Hillside Drive  
Hillsdale, MI 49242

Q Panel Company/Lab Products  
800 Canterbury Road  
Westlake, OH 44145

6.4 Subject term (key word) listing.

Cartridge cases  
Coating  
Preservative  
Weatherproofing

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



MIL-DTL-3688D

CONCLUDING MATERIAL

Custodians:

Army - MR  
Navy - YD  
Air Force - 99

Preparing activity:

Army - MR

Project 8030-2005-003

Review activities:

Army - MI  
Navy - AS, CG, OS, SH  
Air Force - 03, 84

Civil agency:

GSA - FSS

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