

MIL-P-53032
7 December 1983

MILITARY SPECIFICATION

PRIMER COATING, WATER REDUCIBLE, EPOXY ESTER-LATEX TYPE,

LEAD AND CHROMATE FREE

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 water reducible air drying blended epoxy ester-latex resin based primer that is lead and chromate free, and also meets air pollution requirements for solvent emissions. It is intended for use on treated ferrous metals, treated aluminum, and wood (see 6.1).

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. Unless otherwise specified (see 6.2), the following specifications and standards of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

| | |
|------------|--|
| TT-C-490 | - Cleaning Methods and Pretreatment of Ferrous Surfaces. |
| TT-E-489 | - Enamel; Alkyd Gloss (For Exterior and Interior Surfaces). |
| TT-S-735 | - Standard Test Fluids, Hydrocarbon. |
| PPP-P-1892 | - Paint, Varnish, Lacquer, and Related Materials; Packaging. |

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 and Development Center, ATTN: STRBE-DS, Fort Belvoir, VA 22060 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 8010

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STANDARDS

FEDERAL

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

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- MIL-STD-1188 - Commercial Packaging of Supplies and Equipment.

(Copies of specifications and standards required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Other publications. The following document(s) form a part of this specification to the extent specified herein. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

- B117 - Salt Spray Testing.
- D50 - Chemical Analysis of Yellow, Orange, Red, Brown Pigments Containing Iron and Manganese.
- D93 - Flash Point of Pigmented Materials (Pensky Martin).
- D562 - Tests of Viscosity of Paints using Stormer Viscosimeter.
- D1006 - Recommended Practices for Conducting Exterior Exposures Tests of Paint on Wood.
- D1301 - Chemical Analysis of White Lead Pigments.
- D1308 - Effect on Household Chemicals on Clear and Pigmented Coatings.
- D1475 - Density of Paint, Varnish, Lacquer, and Related Products.
- D3335 - Tests for Low Concentrations of Lead, Cadmium and Cobalt in Paint by Atomic Absorption Spectroscopy.
- D3792 - Water Content.
- D3960 - Volatile Organic Content (VOG).

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

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT (SCAQMD)

- Rule 1107 - Manufactured Metal Parts and Product Coatings.

(Application for copies should be addressed to the South Coast Air Quality Management District, 9150 East Flair Dr., El Monte, CA 91731).

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(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

2.3 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.

3. REQUIREMENTS

3.1 Qualification. Primer furnished under this specification shall be a product which is qualified for listing on the applicable Qualification Products List at the time set for opening of bids (see 4.2 and 6.3). Any change 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 iron oxide pigments.

3.3 Composition.

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 high purity iron oxide and calcium carbonate should be used. Small amounts of antissettlers may be added, but are not recommended. The non-reactive extenders must be water insoluble; nonalkali-sensitive low to medium oil adsorption talcs are recommended. The percentage of non-reactive extender is determined by difference.

TABLE I. Quantitative requirements of pigment.

| Characteristics | Requirements (% by weight) | |
|---|----------------------------|---------|
| | minimum | maximum |
| Iron Oxide (Fe ₂ O ₃) (99 + %) | 45 | 50 |
| Calcium Carbonate | 25 | 30 |
| Other Non-reactive extenders | 20 | 30 |

3.3.2 Vehicle. The vehicle shall be a water reducible epoxy ester blended with a 100 percent acrylic latex conforming to requirements in table II and the spectra in figures 1 and 2. The volatile portion of the vehicle shall conform to Rule 1107 of South Coast Air Quality Management District and the requirements stated in 3.3.2.1. Small amounts of antifoamers and driers should be added to insure foam control and good drying. The use of any significant amounts of surfactants is not recommended.

3.3.2.1 Composition of the volatile vehicle (VOC). When tested as in 4.3.3 and table III the total volatile content (VOC) of the primer as defined by

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SCAQMD Rule 1107 shall be equal to or less than 2.8 lbs. per gallon after subtracting water.

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

TABLE II. Quantitative requirements of primer.

| Characteristics | Minimum | Maximum |
|---|---------|---------------|
| Total solids, percent by weight of primer | 51.0 | - |
| Pigment, percent by weight of total solids | 53.0 | 58.0 |
| Epoxy ester, percent by weight of total solids | 36.0 | 42.0 |
| 100% acrylic latex resin, percent by weight of total solids | 5.0 | 10.0 |
| Water, percent by weight of primer | 30.0 | 35.0 |
| Flash Point | 120° F | - |
| Viscosity (package): | | |
| Krebs-Stormer, Shearing rate 200 rpm: | | |
| Grams | 150 | 200 |
| Equivalent K.U. | 72 | 82 |
| pH of Primer | 8.5 | - |
| Drying Time: | | |
| Air-drying: | | |
| Set to touch, minutes | 10 | 60 |
| Dry through, hours | - | 18 |
| Full hardness, hours | - | 72 |
| 60-degree specular gloss | 10 | 50 |
| Fineness of grind | 4 | - |
| Lead Metal (percent of total solids) | - | 0.06 |
| Hexavalent Chromium (in pigment) | - | Negative Test |

3.5 Qualitative requirements.

3.5.1 Condition in container. A freshly opened full container of primer, when tested as specified in 4.3.13 shall be free from grit, seeds, skins, lumps, abnormal thickening, or livering and shall show no more pigment settling or caking than can be readily reincorporated to smooth homogeneous state. The primer shall not have an offensive smell.

3.5.2 Storage stability.

3.5.2.1 Heat-freeze resistance. An 80 to 90 percent filled, tightly closed, pint can of primer shall show no skinning when tested as specified in 4.3.14.1. After heat aging as specified in 4.3.14, the primer shall show no livering, curdling, seeding, hard caking, or gummy sediment. It shall mix readily to smooth homogeneous state and any skin formed shall be continuous and easily removed. The viscosity change shall not exceed +10 percent of the initial value. The condition of the heated-frozen primer shall be judged as

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above as to hard settling and redispersion; and rejected if difficult to restore back to a usable condition. The viscosity change of the heated-frozen primer shall not exceed +10 percent of the initial value. The heated-frozen primer shall meet all other requirements of this specification.

3.5.2.2 Full container. A full quart container of primer shall show only no skinning, livering, curdling, seeding, hard dry caking; or tough, gummy sediment when tested as specified in 4.3.14.2. Soft settling is permitted provided the primer shall remix readily to a smooth homogeneous state, shall have a maximum viscosity change of +10 percent of the initial value and shall meet all other requirements of the specification.

3.5.3 Dilution stability. When thinned as specified in 4.3.15, the primer shall remain stable and uniform, showing no precipitation curdling, or separation. Slight pigment settling shall be permitted. Slight surface floating of additives will also be permitted; provided they are easily stirred back in.

3.5.4 Brushing properties. The primer, tested as specified in 4.3.16, shall be capable of being brushed out and laid off without dragging the brush. When dry, the brushed surface shall be free from sags and runs, and shall show a minimum of brush marks.

3.5.5 Spraying properties. The primer, tested as specified in 4.3.17, shall spray satisfactorily in all respects, and show no running, sagging, or streaking. The dried film shall show no dusting, mottling, or color separation, and shall present a smooth finish with no seediness and minimal pinholes.

3.5.6 Flexibility. A film of primer tested as specified in 4.3.18 shall withstand bending without cracking or flaking.

3.5.7 Knife test. A film of primer tested as specified in 4.3.19 shall be hard and tough and shall adhere tightly to the metal. It shall be difficult to furrow off with the knife and shall not flake, chip, or powder. The knife cut shall show beveled edges.

3.5.8 Recoating. A film of primer tested as specified in 4.3.20 shall show no blistering, wrinkling, or other evidence of lifting. The test 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 glass. The drying time of the enamel top coat over the primer shall not differ significantly from the drying time of the enamel top coat applied to bare glass.

3.5.9 Water resistance. A film of primer tested as specified in 4.3.21 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 immediately after removal. After 2 hours air drying, the portion of 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.

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3.5.10 Hydrocarbon resistance. A film of primer tested as specified in 4.3.22 shall show no blistering or wrinkling immediately after 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, adhesion, and general appearance from a panel prepared at the same time but not immersed.

3.5.11 Salt spray resistance. A film of primer tested as specified in 4.3.23 on cold rolled steel and examined immediately after removal from the salt spray test shall show no more than a trace of rusting and no more than 10 scattered blisters no larger than 1 mm in diameter. On removal of the primer there shall be no more than a trace of rusting, pitting, or corrosion of the substrate. On the phosphated steel panels, after removal from salt fog (after 336 hours) the condition of the surface shall be as good as the cold rolled steel panels; except for score rusting. Stripping shall also be the same; except score corrosion shall not exceed 1/8-inch in width on either side of the score.

3.5.12 Weather resistance. Films exposed as specified in 4.3.24 shall show only trace rusting, cracking, checking, flaking, blistering or loss of adhesion. Some fading and chalking are permitted as weathering proceeds. On removal of the coating the surface of the metal shall show no more than a trace of rusting, pitting, or corrosion.

3.5.13 Toxicity. The product shall contain no benzene (benzol), chlorinated compounds, or hydrolyzable chlorine derivatives.

3.6 Material safety data sheet. A Material Safety Data Sheet shall be prepared for the primer by the manufacturer in accordance with FED-STD-313 and submitted to the contracting officer (see 6.2).

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 desired, for conformance to prescribed requirements.

4.1.1 Sampling and inspection. Sampling and inspection shall be performed in accordance with the following procedures:

- a. For lot size up to 1000 gallons, the sample size shall be one quart.
- b. For lot size greater than 1000 gallons, the sample size shall be one gallon.

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

- a. Qualification.

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- b. Acceptance of individual lots.
- c. Acceptance for use as component on end item.

4.2.1 Qualification tests. The qualification tests shall consist of tests for all requirements specified in section 3 (see 6.3).

4.2.2 Acceptance tests. Acceptance tests for acceptance of individual lots shall consist of tests specified in section 4 with the exception of storage stability (see 3.5.2.2 and 4.3.14.2) and weather resistance (see 3.5.12 and 4.3.24).

4.2.3 Acceptance of lots. When approved by the cognizant activity, acceptance of lots for use as a component on an end item shall be based on conformance with specified requirements for the following characteristics:

- Epoxy ester
- Acrylic latex
- Calcium carbonate
- Iron oxide
- Lead and chromate levels
- Flash point
- Flexibility
- Knife test
- Water resistance
- Hydrocarbon fluid resistance
- Total solids
- Pigment, percent by weight of total solids

4.3 Tests.

4.3.1 Test conditions. The routine and referee testing conditions shall be in accordance with the pertinent test methods of FED-STD-141 except as otherwise specified herein.

4.3.2 Test methods. The following tests shall be conducted in accordance with methods in table III, and as hereinafter specified.

TABLE III. Index.

| Item | Test Methods | | | Requirement Paragraph |
|---|--------------|------|----------------|-----------------------|
| | FTMS 141 | ASTM | Test Paragraph | |
| Analysis of Pigment: | 4021 | - | 4.3.6 | - |
| Qualitative Chromate | - | - | 4.3.6.1 | table II |
| CaCO ₃ - Calcium Carbonate | - | - | 4.3.6.2 | table I |
| Fe ₂ O ₃ - Iron Oxide | - | D50 | 4.3.6.3 | table I |
| Brushing properties | 4321 | - | 4.3.16 | 3.5.4 |
| Condition in container | 3011 | - | 4.3.13 | 3.5.1 |
| Dilution Stability | 4203 | - | 4.3.15 | 3.5.3 |
| Drying Time | 4061 | - | 4.3.12 | table II |
| Fineness of grind | - | - | 4.3.11 | table II |

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TABLE III. Index. (con't)

| Item | Test Methods | | | |
|--------------------------------|--------------|-------|-------------------|--------------------------|
| | FTMS 141 | ASTM | Test Paragraph | Requirement Paragraph |
| Flash Point | - | D93 | - | table II |
| Flexibility | 6221 | - | 4.3.18 | 3.5.6 |
| Hydrocarbon resistance | - | D1308 | 4.3.22 | 3.5.10 |
| Knife test | 6304 | - | 4.3.19 | 3.5.7 |
| Epoxy ester | - | - | 4.3.7 | table II, 3.3.2 |
| Acrylic latex | - | - | 4.3.8 | table II, 3.3.2 |
| Pigment content | - | - | 4.3.5 | table II |
| pH of primer | - | - | 4.3.9 | table II |
| Recoating | - | - | 4.3.20 | 3.5.8 |
| Salt Spray resistance | - | B117 | 4.3.23 | 3.5.11 |
| Specular Gloss | - | - | 4.3.10 | table II |
| Spraying properties | 4331 | - | 4.3.17 | 3.5.5 |
| Storage stability | - | - | 4.3.14 | - |
| Partially full container | 3021 | - | 4.3.14.1 | 3.5.2.1 |
| Full container | - | - | 4.3.14.2 | 3.5.2.2 |
| Total solids | - | - | 4.3.4 | table II |
| Toxicity, Solvents | - | - | 4.3.25 | 3.5.13 |
| Toxicity, Lead Metal | - | D3335 | 4.3.26 | table II |
| Viscosity: | - | - | - | - |
| Package | - | D562 | - | table II |
| Volatile Organic Content (VOC) | - | D3960 | 4.3.3 | 3.3.2.1 |
| Water Content | - | D3792 | - | table II |
| Water Resistance | - | D1308 | 4.3.21 | 3.5.9 |
| Weather Resistance | - | D1006 | 4.3.24 | 3.5.12 |

4.3.3 Volatile organic compounds (VOC) determination. Calculate the VOC using the following equation:

$$\text{VOC (pounds per gallon)} = \frac{D_c \times (100 - X_c - X_w)}{100 - (X_w \times \frac{D_c}{8.32})}$$

Where D_c = coating density in lbs per gallon as determined in accordance with ASTM D1475.

X_c = Weight percent solids as determined in 4.3.4.

X_w = Weight percent water as determined in ASTM D3792.

Nonconformance to 3.3.2.1 shall constitute failure of this test.

4.3.4 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 with fourth-decimal accuracy. Transfer a small sample that does not exceed 0.3g to the dish, determine its exact weight by loss in weight of the bottle. Dilute the sample with 2 mls of distilled water and dry in a gravity convection oven at 220 \pm 4° F for 2 hours. Upon cooling,

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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).

4.3.5 Total pigment. Extract the pigment as specified in method 4021 of FED-STD-141 using an extraction mixture of equal volumes of benzene and acetone. Save the pigment and thinned vehicle for other tests. Calculate pigment as percent of total solids.

$$\text{Percent pigment} = \frac{\text{Weight of pigment} \times 10,000}{(\text{Weight of primer used}) \times (\% \text{ total solids})}$$

4.3.6 Pigment analysis. With the pigment extracted in 4.3.5 perform the following tests:

4.3.6.1 Qualitative chromate.

a. Reagents:

25 percent aq. KOH

b. Procedure:

- (1) Add 5 ml of 25 percent aq. KOH to 1/2g of the extracted pigment contained in 15 ml centrifuge tube.
- (2) Agitate by shaking the tube for a few minutes then centrifuge.
- (3) The supernatant liquid should be colorless. A yellow color (chromate) constitutes a failure.

4.3.6.2 Quantitative calcium carbonate.

4.3.6.2.1 Carbonate as calcium carbonate. Determine carbon dioxide on a 2g sample of the extracted pigment (see 4.3.5) as in ASTM method D1301 (using the method for determination of CO₂ in lead carbonate). The factor to convert CO₂ to CaCO₃ is 2.2742.

4.3.6.2.2 Calcium as calcium carbonate. Weigh 0.3 to 0.4g to the fourth decimal of the extracted pigment (see 4.3.5) into a 250 ml beaker. Add 25 ml of concentrated HCl. Cover the beaker with a watch glass and boil on a hot plate for 1 hour. Add 25 ml of water and filter through fine filter paper into a 400 ml beaker. Wash the residue 2 times with 10 ml of 1N HCl. To the filtrate add 100 ml of 18N H₂SO₄. While stirring add 150 ml of ethyl alcohol. Let stand 12 hours and filter through fine filter paper. Wash filter and residue 3 times with 10 ml of 75 percent ethyl alcohol. Ignite in a tared crucible to 500° C. Cool in a desiccator and weigh. Calculate calcium carbonate as percent of pigment:

$$\text{CaCO}_3 = \frac{\text{Weight of residue}}{\text{Weight of sample}} \times 73.52$$

4.3.6.3 Quantitative Fe₂O₃. Use ASTM method D50 or method 7141 of FED-STD-141.

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4.3.7 Determination of epoxy ester resin.

4.3.7.1 Quantitative epoxy ester resin. Weigh, by difference, 5-7g of well-mixed sample accurately (+1) mg using a weighing bottle and add to 30g of C.P. sodium chloride which has been moistened with 2.5 ml of distilled water, contained in a 200-ml glazed porcelain casserole. Mix well and place in a vacuum desiccator over indicator type Drierite. Dry overnight or 16 hours with pump running.^{1/} Grind the salt paint mixture in a mortar and transfer to a 33 x 80 mm Soxhlet extraction cup and cover with a plug of glass wool. Extract with 130 ml of technical grade benzene under vigorous reflux for 4 hours. Use a Soxhlet extraction apparatus equipped with a calcium chloride tube and a few Berl saddles as boiling stones in the 250 ml erlenmeyer flask. At the conclusion of the extraction period, remove the extraction thimble and rinse with benzene. Save the extraction thimble and contents for use in infrared identification of the acrylic resin. Rinse the Soxhlet with benzene and transfer extract and rinsings to a tared 250 ml beaker containing Berl saddles. Rinse out any residue from the flask or saddles with benzene and add to the extract. (If pigment has escaped through the thimble into the extract, then the extract must be filtered.) Evaporate the extract on a cooler portion of the steam bath under a light stream of nitrogen. When all of the solvent has evaporated, dry in a vacuum oven at 140°-160° F overnight. Weigh the beaker and determine the benzene extract.

4.3.7.2 Calculation. Calculate the percent epoxy ester resin as follows:

$$\text{Epoxy ester resin (percent)} \frac{2/}{Z} = \frac{Y \times 100}{Z}$$

Where Y = Net Weight of benzene extract.

Z = (weight of paint sample) x $\frac{(\% \text{ total solids})}{100}$.

4.3.7.3 Qualitative epoxy ester resin. Vacuum dry a film of the epoxy ester resin from 4.3.7.2 taken up in benzene on a sodium chloride plate and scan the infrared spectrum from 2 to 15 micro meters. The spectrum shall closely resemble that in figure 1 (see 3.3.2).

4.3.8 Acrylic latex resin.4.3.8.1 Qualitative acrylic latex.

4.3.8.1.1 Sample preparation. Allow the benzene to evaporate from the extraction thimble (see 4.3.7.1). Drain the Soxhlet to remove any residual benzene. Extract the residue in the thimble with 130 ml of acetone under

^{1/} Moistening the salt with water before addition of the paint facilitates dispersion of the paint and subsequent grinding of the dried material.

^{2/} It should be noted that the benzene extract contains not only the epoxy ester resin, but also nonvolatile additives that are benzene soluble. This fact should be considered when calculating the percent acrylic latex resin although no adjustment is necessary.

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vigorous reflux for two hours, using the same apparatus as for the benzene extraction. Remove the extraction thimble and evaporate the acetone extract down to a concentrated solution on the steam bath.

4.3.8.1.2 Infrared spectrum. Vacuum dry a film of the extracted latex resin (see 4.3.8.1.1) on a sodium chloride plate and obtain a spectrum from 2.5 to 15 micrometers. The spectrum shall closely resemble that in figure 2 (see 3.3.2).

4.3.8.2 Quantitative acrylic latex resin. Calculate the percent acrylic latex resin by subtracting the percent epoxy ester (see 4.3.7) plus percent pigment (see 4.3.5) from 100 percent, and check obtained value against value for acrylic latex in table II.

4.3.9 pH of primer. Stir a freshly opened quart can of primer. Obtain a 2 to 2-1/2 inch piece of short range (6 to 9) pH paper. Add one or two drops of distilled water to vertical tip of paper to wet about 1/2 inch to 1-1/2 inch. Dip wet end in primer about half the wetted distance. The color in the unsubmerged wetted portion should develop in less than thirty seconds to the minimum pH acceptable (see table II).

4.3.10 Specular gloss (60 degrees). Run specular gloss in accordance with the following:

4.3.10.1 Apparatus.

- a. Instrumental components. The apparatus shall consist of an incandescent light source, means for locating the specimen surface, and a receptor located to receive the required pyramid of rays reflected by the specimen.
- b. Geometric conditions. The axis of the incident beam shall be 60 degrees \pm 0.1 degrees from the perpendicular to the test surface. The axis of receptor shall be at the mirror reflection of the axis of the incident beam. The angular dimensions (apertures) and tolerances of the source and receptor shall be as indicated.
- c. Measurement mechanism. The receptor-measurement mechanism shall give a numerical indication proportional to the light flux passing the receptor field stop within \pm 1 percent of full scale.

4.3.10.2 Procedure.

- a. Apparatus adjustment. Test aperture ratio and image centering by adjusting the instrument to read correctly the gloss of a polished glass standard and then reading the gloss of one or more standards having poor image-forming characteristics. If the instrument readings of the latter standards do not agree closely with the calibration values for these standards, readjust the instrument according to instructions of the manufacturer, or return the instrument for readjustment.
- b. Preparation of test specimens. Prepare films of the finish to be tested by drawing down with a doctor blade of 0.007-inch gap clearance to yield a wet film thickness of approximately 0.0035 inch on plane opaque white

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glass panels as prescribed in 1.5 of method 2021, FED-STD-141. Dry panels for 48 hours at a temperature of $75^{\circ} \pm 2^{\circ}$ F, a relative humidity of 90 ± 4 percent, and under dust-free conditions.

- c. Calibration. If the apparatus is properly adjusted, calibrate the glossmeter at the start and completion of every period of operation, and during operation at sufficiently frequent intervals to assure constancy of instrument response. To calibrate, adjust the instrument to read correctly the gloss of a standard whose assigned value is approximately that of the glossiest specimens to be measured.
- d. Measurement. Measure at least three portions of the specimen surface to obtain an indication of uniformity.

4.3.11 Fineness of grind. Run fineness of grind according to the following procedures.

4.3.11.1 Apparatus:

- a. The apparatus shall consist of a device having two parts made in accordance with the following requirements.
- b. Gage. A hardened steel block approximately 7 inches in length, 2-1/2 inches in width, and 1/2 inch in thickness. The top surface of the block shall be ground smooth and flat and shall have the dimensional requirements corresponding to the Hegman Standard graduations.
- c. Scraper. A double-wedge steel blade 3-1/2 inches long, 1-1/2 inches wide and 1/4 inch thick. The two edges on the long sides shall be rounded to a radius of approximately 0.01 inch.

4.3.11.2 Procedure.

- a. Preparation of sample. Thoroughly mix by stirring, and strain an appropriate amount of the material to be tested. Allow to stand for at least one hour in a partially filled closed container to permit all entrapped air to escape.
- b. Performance of test. Place the thoroughly cleaned and dried gage on a flat, non-slippery surface. Pour a sufficient amount of the prepared sample into the deep end (well) of the groove so that it overflows the groove slightly. Grasp the scraper between the thumbs and fingers of both hands and place it edge-wise in contact with the surface of the gage at the extreme deep ends of the grooves with the long dimension of the scraper across the width and parallel to the short dimension of the gage. While holding the scraper perpendicular to the surface of the gage and at right angles to the length of the grooves, draw it at a uniform moderate rate over the surface of the gage to a point beyond the zero ends of the grooves (graduation 8). Sufficient downward pressure should be exerted on the scraper to just fill the grooves and clean the level surface of the gage. Immediately determine the fineness value of the material by viewing the gage from the side in such a manner that the line of vision is at right angles to the long dimension of the grooves and not more than 30 degrees nor less than 20 degrees to the face of the

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gage while it is held in a light that will make the pattern of the material in the grooves readily visible. Observe the point along the groove where the material first shows a predominately speckled appearance and note the number between 0 and 8 graduations that most nearly corresponds to that point. Report this number as the fineness of grind. Disregard any scattered specks which appear prior to the point where the predominantly speckled appearance begins.

4.3.12 Drying time. Determine drying time as in method 4061 of FED-STD-141 under referee conditions, except that the primer shall be drawn down with a 0.0035 inch (0.007 inch gap) clearance film applicator.

4.3.12.1 Full hardness. The film shall be considered to have reached full hardness when it is very difficult to remove with a knife blade.

4.3.13 Condition in container. Determine package condition on acceptance testing in accordance with method 3011 of FED-STD-141 and observe for compliance with 3.5.1. On qualification testing evaluate pigment settling of caking by proceeding as in method 3011 but do not stir. Stir with a spatula for three minutes. Use a moderate rate of stirring. On re-examination of the contents, the disclosure of any gel bodies, or undispersed pigment indicates unsatisfactory settling properties.

4.3.14 Storage stability.

4.3.14.1 Heat-freeze resistance. Determine skinning after 48 hours in accordance with method 3021 of FED-STD-141. Reseal and age for seven days at $125^{\circ} \pm 3^{\circ}$ F and observe for compliance with 3.5.2.1 after bringing sealed can to $77^{\circ} \pm 1^{\circ}$ F. If acceptable, reseal and hold at 5° F $\pm 5^{\circ}$ F for 24 hours. Bring sealed can to 77° F $\pm 1^{\circ}$ F and test for compliance to 3.5.2.1 (freeze cycle).

4.3.14.2 Full container. Store a full standard pint can of the primer at 72° F to 80° F undisturbed for six months and then examine the contents. Evaluate pigment settling or caking as specified in 4.3.13 but stir with a spatula for 3 minutes as specified in 4.3.13 prior to the re-examination. Let stand at least one hour at 77° F $\pm 1^{\circ}$ F. Determine viscosity and make other applicable tests to determine compliance with 3.5.2.2.

4.3.15 Dilution stability. Reduce five parts by volume of primer to one part room temperature tap water. Stir water gradually in. Let stand two hours, and observe for compliance to 3.5.3.

4.3.16 Brushing properties. Apply the primer as packaged using a 2-1/2-inch brush in accordance with method 4321 of FED-STD-141 and observe for compliance with 3.5.4.

4.3.17 Spraying properties. Reduce five parts by volume of primer with up to one part by volume of tap water as specified in 4.3.5. Stir well. Filter. Spray on a steel panel and observe for spraying properties in accordance with method 4331 of FED-STD-141 for compliance with 3.5.5. For referee test use automatic applicaton per method 2131 of FED-STD-141.

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4.3.18 Flexibility. Determine flexibility in accordance with method 6221 of FED-STD-141. Apply a 2-inch wide film of primer with a film applicator that will give a dry film thickness of 0.0014 to 0.0016 inch on a smooth finish steel panel prepared in accordance with method 2011 of FED-STD-141, using the aliphatic naphtha ethylene glycol ether mixture. The panel shall be prepared from rust-free cold rolled carbon steel 0.010 \pm 0.001 inch thick with a Rockwell 15-T maximum hardness of 82 and finished with a surface roughness of 8 to 12 microinches. Air-dry the primer in a horizontal position for 18 hours and then bake for 24 hours at 220° F \pm 4° F. Condition the panel for 1/2 hour under reference conditions. Bend over a 1/2-inch mandrel. Examine the coating for cracks over the area of the bend in a strong light at a 7-diameter magnification for compliance with 3.5.6.

4.3.19 Knife test. Perform the knife test on the flat portion of the flexibility test panel (see 4.3.18) in accordance with method 6304 of FED-STD-141, and observe the results for compliance with 3.5.7.

4.3.20 Recoating. Using a 0.0035 inch (0.007 inch gap clearance) film applicator, draw down a 2 inch wide film of the primer on a clear plate glass panel. Air dry for 24 hours, then apply a 3-inch wide film of white gloss enamel conforming to TT-E-489, class A, across the width of the dried primer film with a 0.0030-inch (0.0060-inch gap clearance) film applicator. After the enamel has dried 24 hours compare the 60 degree specular gloss of the enamel over the primer with that of the enamel on glass and check for compliance with 3.5.8. Allow the specimens to air dry for 72 hours after recoating and determine the adhesion between enamel and primer and between primer and glass using the knife test as in method 6304 of FED-STD-141; observe for compliance with 3.5.8.

4.3.21 Water resistance. Draw down a film of the primer with a 0.0035-inch (0.007-inch gap clearance) film applicator on a thin Alodine aluminum panel in accordance with method 2101 of FED-STD-141 using the aliphatic naphtha-ethylene glycol monoethyl ether mixture. Air dry the primer for 168 hours. Immerse these panels in distilled water at 75° F \pm 2° F for 18 hours. Test in accordance with ASTM D1308 for compliance with 3.5.9.

4.3.22 Hydrocarbon resistance. Prepare a film of the primer as specified in 4.3.21 and air dry 168 hours. Do not wax or coat the exposed metal surfaces. Immerse the panel for 2 hours in a hydrocarbon fluid conforming to TT-S-735, type III, in accordance with ASTM D1308 for compliance with 3.5.10.

4.3.23 Salt spray resistance. Clean three 4-inch x 10-inch phosphated steel panels (prepared in accordance with TT-C-490, light phosphate treatment) and three cold rolled steel panels in accordance with method 2011 of FED-STD-141 using the petroleum naphtha-ethylene glycol monethyl ether mixture. Reduce the primer for spray application as specified in 4.3.17 and spray the test panels to a dry film thickness between .0014 and .0016 inches in a one coat application. Air dry for 168 hours. Score one panel of phosphate steel only. Expose the panels to five percent salt spray for 168 hours as described in ASTM B117. Remove the cold rolled set, and let phosphate set run for total 336 hours. Wash

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the panels gently in running water not warmer than 100° F until free from any visible salt deposits and examine immediately for compliance with the requirements of 3.5.11. Strip the primer film from the panels by means of lacquer thinner and inspect steel for rusting, pitting or corrosion. Upon completion of the 336 hours for the phosphate set, remove as above, clean and examine, then strip and reexamine for compliance to 3.5.11.

4.3.24 Weather resistance. Prepare four phosphated steel panels as specified in 4.3.23. Spray one coat of the primer at 0.0014 to 0.0016 inch dry film thickness. Air dry for 18 hours in a horizontal position before applying one coat of yellow enamel conforming to TT-E-489 (dry film .0012 to .0015 inches). Permit the test panels to air dry 168 hours and then expose three panels, saving one for control in accordance with method ASTM D1006 for 18 months in the latitude of Washington, D.C. Examine the panels for compliance with 3.5.12 every six months.

4.3.25 Toxicity: solvents. The manufacturer shall certify that the primer contains no benzene (benzol), chlorinated compounds, or hydrolyzable chlorine derivatives.

4.3.26 Toxicity: lead content of nonvolatile (ASTM method). Determine lead content according to ASTM D3335 and check for compliance with table II. The following alternate method may be used.

4.3.26.1 Toxicity: lead content by X-ray emission spectrometric analysis (alternate method).

4.3.26.1.1 Test panel preparation. Using 100 grams of known lead free specification primer prepare standard aliquots containing 0.00, 0.03, 0.06, and 0.09 percent lead metal, based on total nonvolatile content by adding calculated amounts of lead naphthenate (water reducible type) of a known lead content. Thoroughly mix the aliquots to incorporate the lead and draw down the standards, and the primer to be tested on duplicate black and white Moresst cards using a 0.0035 inch (0.007 inch gap clearance) film applicator. Dry for 48 hours at a temperature of 75° F \pm 2° F, a relative humidity of 50 \pm 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 spectrometer.

4.3.26.2 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 content. 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:

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$$R = I_{Pb} - \frac{(I_{Pb} \text{ background I} + I_{Pb} \text{ background II})}{2}$$

I Compton Line

Where I = gross intensity
and the background is taken on each side of the Pb line.

Establish a lead calibration curve using these results. Determine the lead content on the test primer using the above procedure and calibration curve. When using an energy dispersive fluorescence spectrometer, it shall be setup in accordance with the manufacturer's manual.

4.3.27. Inspection of packaging. The preservation, packing, and marking shall be examined for compliance with the quality assurance requirements of PPP-P-1892 for level A and B. Commercial preservation, packing and marking shall be examined for compliance to MIL-STD-1188.

5. PACKAGING

5.1 Preservation, packing and marking. Preservation, packing and marking of the primer shall be level A, level B or commercial as specified (see 6.2). Levels A and B preservation, packing and marking shall be in accordance with PPP-P-1892. Commercial preservation, packing and marking shall be in accordance with MIL-STD-1188 along with the special marking requirements of PPP-P-1892. The primer, for all degrees of packaging, shall be furnished in 1-quart or 1-gallon multiple friction top containers, in 5-gallon lug cover steel pails, or in 55-gallon steel drums as specified (see 6.2). The container shall be coated on the inside with a coating which shall neither affect nor be affected by the primer when the filled and sealed container is stored at room temperature for one year.

6. NOTES

6.1 Intended use. The primer covered by this specification is intended mainly for priming clean phosphate-treated ferrous metal and treated aluminum where exposure to lead or chromate pigments is not permitted and organic solvents are controlled. It may be used on wooden parts of components in the same context as above. It is not intended for use on the inside of potable water tanks, for marine use, for steel exposed to long term weathering. It is not intended for use as a lacquer resistant primer; TT-P-664 is recommended for this application.

6.2 Ordering data. Purchasers should exercise any desired options offered herein and procurement documents should specify the following:

- a. Title, number, and date of this specification.
- b. Date of issue of DoDISS applicable and exceptions thereto (see 2.1.1).
- c. A material Safety Data Sheet shall be prepared (see 3.6).

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- d. Degree of preservation and degree of packing required (see 5.1).
- e. Size of container required (see 5.1).

6.3 Qualification. With respect to products requiring qualification, awards will be made only for such products as have, prior to the time set for opening of bids, been tested and approved for inclusion in the applicable qualified products list whether or not such products have actually been so listed by that date. The attention of the 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 products covered by this specification. The activity responsible for the qualified products list is: Commander, US Army Belvoir Research and Development Center, Fort Belvoir, VA 22060, ATTN: STRBE-VO and information pertaining to qualification of products may be obtained from that activity.

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

6.5 Sample formulation. The following formula was developed by the Preparing Activity and was found to meet the requirement of this specification. However, the Government disclaims any responsibility for any supplier's submission based on this formula. It should serve as a useful guide.

Formula 154 - 190:

| <u>Ingredient</u> | <u>Percent by weight</u> |
|--|--------------------------|
| Water | 31.88 |
| Triethyl Amine | 2.15 |
| Ethyl Alcohol | 4.40 |
| Butyl Cellosolve | 8.74 |
| Activ 8 (Vanderbilt) | 0.08 |
| Epoxy Ester (100% basis) R.C.I.38690 | 19.54 |
| Acrylic Latex (100% basis) U.C. 4358 | 2.98 |
| Defoamer 675 (Colloid) | 0.55 |
| Driers: CoN (100% basis) | 0.11 |
| ZrN (100% basis) | 0.26 |
| Fe ₂ O ₃ (Pfizer 8098) | 13.95 |
| CaCO ₃ (Pfizer 25-11) | 7.68 |
| Talc Nytal 300 | 7.68 |

6.6 Samples. It is believed that this specification adequately describes the characteristics necessary to secure the desired material and that normally no samples will be necessary prior to award to determine compliance with this specification. If, for any particular purpose, samples with bids are necessary, they should be specifically asked for in the invitation for bids, and the particular purpose to be served by the bid sample should be definitely stated; and the specification to apply in all other respects.

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Custodians:

Army - ME

Navy - YD

Preparing activity

Army - ME

Review activity:

Army - MR

Project: 8010-A253

User activity:

Army - AT

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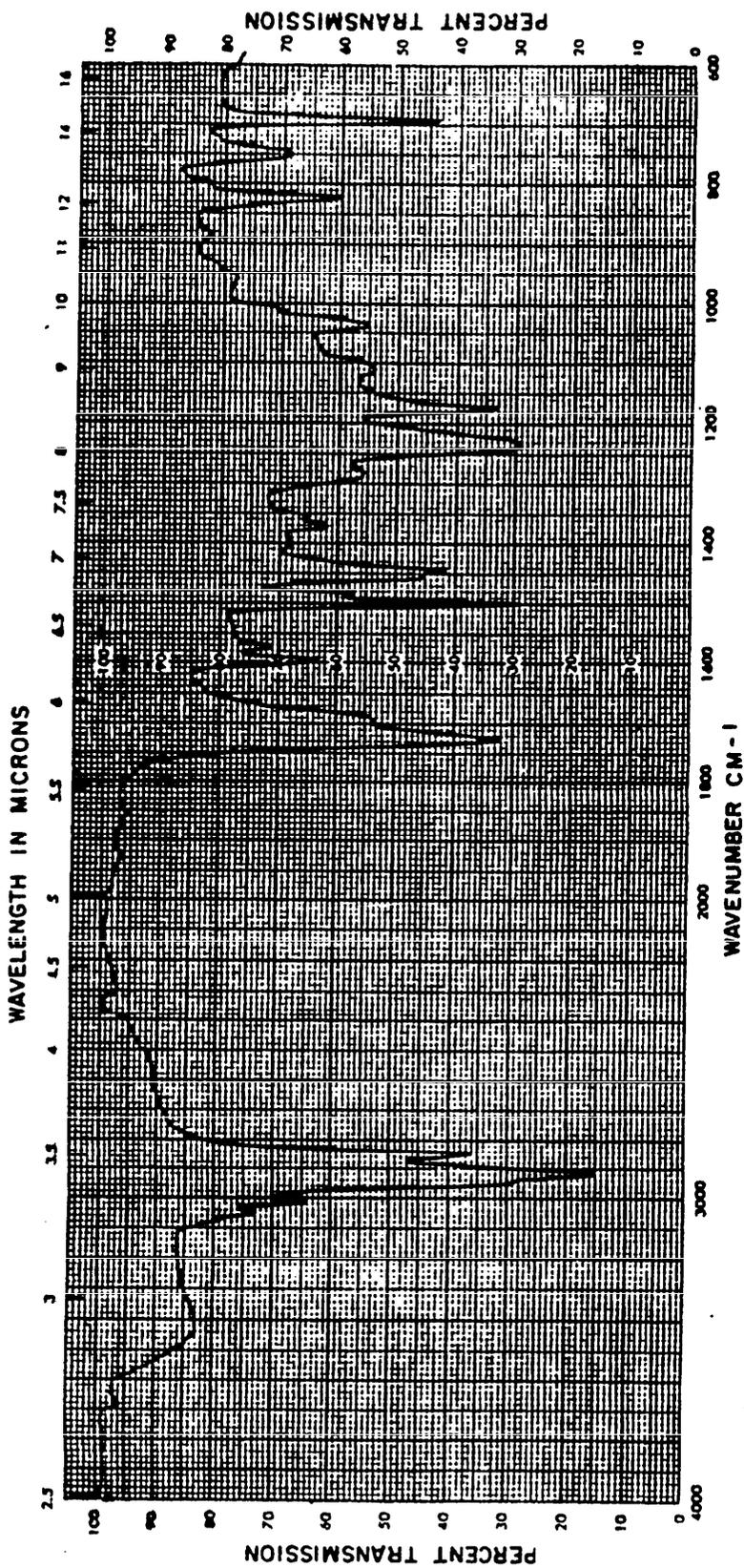


FIGURE 1. Infrared spectrum of epoxy ester resin.

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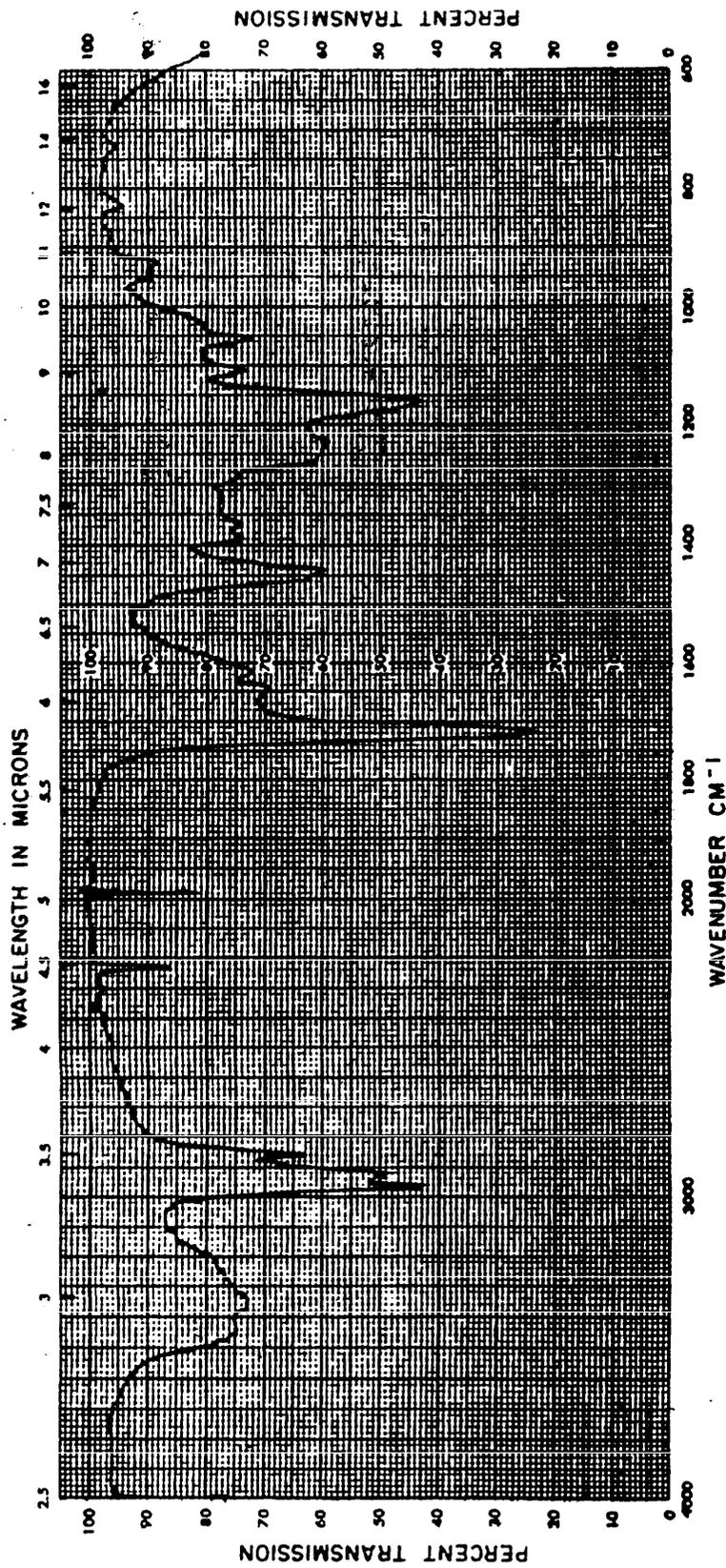


FIGURE 2. Infrared spectrum of acrylic latex resin.

X-4137

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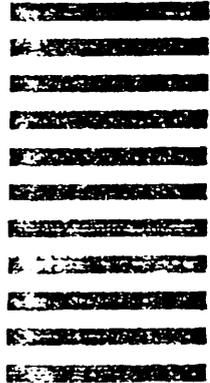


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| | | | |
|---|--|--|--|
| 1. DOCUMENT NUMBER MIL-P-53032 | | 2. DOCUMENT TITLE Primer Coating, Water Reducible, Epoxy Ester- Latex Type, Lead and Chromate Free | |
| 3a. NAME OF SUBMITTING ORGANIZATION | | 4. TYPE OF ORGANIZATION (Mark one): | |
| b. ADDRESS (Street, City, State, ZIP Code) | | <input type="checkbox"/> VENDOR <input type="checkbox"/> USER <input type="checkbox"/> MANUFACTURER <input type="checkbox"/> OTHER (Specify): _____ | |
| 5. PROBLEM AREAS | | | |
| a. Paragraph Number and Wording: | | | |
| b. Recommended Wording: | | | |
| c. Reason/Rationale for Recommendation: | | | |
| 6. REMARKS | | | |
| 7a. NAME OF SUBMITTER (Last, First, MI) - Optional | | b. WORK TELEPHONE NUMBER (Include Area Code) - Optional | |
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