

METRIC

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 SUPERSEDING
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 (see 6.4)

MILITARY SPECIFICATION

RESIN, CHLORINATED ALKYD, SOLUTION (METRIC)

This specification is approved for use by the Naval Sea Systems Command and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers chlorinated, alkyd resin solution for use as an ingredient in fire-retardant interior paints.

2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATION

FEDERAL

TT-T-291 - Thinner-Paint, Volatile Spirits, Petroleum Spirits.

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.

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

2.2 Other publications. The following document forms a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

- D 600 - Liquid Paint Driers, Spec. for.
- D 1296 - Odor of Volatile Solvents and Diluents, Test for.
- D 1398 - Fatty Acid Content of Alkyd Resins and Resin Solutions, Test for.
- D 1475 - Density of Paint, Varnish, Lacquer, and Related Products, Test for.
- D 1545 - Viscosity of Transparent Liquids by Bubble Time Method, Test for.
- D 1549 - Zinc in Lubricating Oils and Additives (Polarographic Method), Test for.
- D 1639 - Acid Value of Organic Coating Materials, Test for.
- D 1959 - Iodine Value of Drying Oils and Fatty Acids, Test for.
- D 2245 - Oils and Oil Acids in Solvent-Type Paints, Identification of.
- D 2369 - Volatile Content of Paints, Test for.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Ship Engineering Center, SEC 6124, Department of the Navy, Washington, DC 20362 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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

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

3. REQUIREMENTS

3.1 Toxicity. The material shall have no adverse effect on the health of personnel when used for its intended purpose (see 4.3). Questions pertinent to this effect shall be referred by the procuring activity to the appropriate service medical department which will act as advisor to the procuring activity.

3.2 Ingredient requirements. The nonvolatile, chlorinated alkyd resin shall be manufactured from safflower oil fatty acids, hexachloroendomethylenetetrahydrophthalic (chloroendic acid (or anhydride), and glycerol. (Other dibasic acids or anhydrides may be used in small amounts, if necessary, but not more than one percent based on the above chlorinated dibasic acid.) The nonvolatile resin shall be in solution in paint thinner conforming to type II, grade A of TT-T-291.

3.3 Quantitative requirements. The resin solution shall conform to table I.

TABLE I. Quantitative requirements.

Characteristics	Minimum	Maximum
Nonvolatile resin, percent by weight of solution	59	61
Volatiles, percent by weight of solution	39	41
Chlorinated dibasic acid, percent by weight of resin solids	47	---
Chlorine, percent by weight of dibasic acid	52	---
Oil acids, percent by weight of resin solids	45	---
Iodine number of oil acids	142	---
Acid number, based on nonvolatile resin	---	6
Viscosity at 25°C (77°F), Gardner bubble viscometer tube	U	W
Color, Gardner color standards	---	10
Compatibility, zinc oxide, K.U. increase	---	15
Linoleic acid, percent by weight of oil acids	70	---
Linolenic acid, percent by weight of oil acids	---	1.0
Viscosity at 25°C (77°F) after reduction to 50 percent nonvolatile content, Gardner bubble viscometer tube	C	F
Weight per gallon, Kg (pounds)	3.71(8.30)	3.88(8.70)

3.3.1 Volatile portion. The volatile portion of the chlorinated alkyd resin solution shall conform to the following requirements by volume:

- (a) A combination of hydrocarbons, alcohols, aldehydes, ethers, esters, or ketones having an olefinic or cycloolefinic type of unsaturation except perchloroethylene: 5 percent maximum.
- (b) A combination of aromatic compounds with eight or more carbon atoms to the molecule except ethylbenzene, methyl benzoate, and phenyl acetate: 8 percent maximum.
- (c) A combination of ethylbenzene, ketones having branched hydrocarbon structures, trichloroethylene, or toluene: 20 percent maximum.

3.4 Qualitative requirements.

3.4.1 Appearance. The resin solution shall be clear, transparent, and homogeneous when examined by transmitted light.

3.4.2 Odor. The odor of the resin solution shall be characteristic of the petroleum spirits permitted.

3.4.3 Setting and drying time. A film of the resin solution, prepared and tested as specified (see 4.2.6) shall set to touch in not more than 1 hour and shall dry hard in not more than 5 hours.

3.4.4 Water resistance. When tested by the method specified (see 4.2.7), the resin film shall show no more than very slight evidence of whitening, dulling, or other defects.

3.4.5 Other resins. Rosin, rosin derivatives, and other natural or synthetic resins (excepting the specified chlorinated alkyd resin) shall not be present.

3.4.6 Paint thinner compatibility. There shall be no clouding or other evidence of incompatibility when the resin solution is tested as specified (see 4.2.8).

3.4.7 Zinc oxide compatibility. When tested as specified (see 4.2.4), the viscosity after five days storage shall be ± 5 Krebs units when compared to the adjusted viscosity of 80 Krebs units prior to storage.

3.4.8 Material Safety Data Sheet. The procuring activity shall be provided a material safety data sheet (MSDS) at the time of contract award. The MSDS is DD Form 1813 and found in and part of FED-STD-313. The MSDS shall be included with each shipment of the material covered by this specification.

4. QUALITY ASSURANCE PROVISIONS

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

4.2 Quality conformance inspection. Quality conformance inspection shall be performed in accordance with method 1031 of FED-STD-141.

4.2.1 Test procedures.

4.2.1.1 Test conditions. The routine and referee testing conditions shall be in accordance with section 7 of FED-STD-141 except as otherwise specified herein.

4.2.1.2 Tests shall be conducted in accordance with table II and as hereinafter specified.

TABLE II. Test procedures.

Test	Applicable method of FED-STD-141	Applicable ASTM method
Nonvolatile resin	----	D 2369
Volatiles	----	D 2369
Oil acids	----	D 1398
Iodine number	----	D 1959
Linoleic and linolenic acid contents of oil acids	----	D 2245
Acid number of resin	----	D 1639
Viscosity	----	D 1545
Weight per gallon	----	D 1475
Color	4242	----
Reduced viscosity	----	D 1545
Appearance	4261	----
Odor	----	D 1296
Setting and drying time	4061	----
Other resins	----	D 1549
Paint thinner compatibility	4203	----

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4.2.2 Chlorinated dibasic acid. Weigh accurately about 1.5 grams (g) of the resin solution into a 500-milliliter (-mL) Erlenmeyer flask with a ground-glass joint. Dissolve the resin solution in 100 mL of benzene and add 50 mL of 1N potassium hydroxide in isopropyl alcohol. Add a magnetic stirring bar and fit the flask with an air condenser. Reflux while stirring for 2 hours. Stopper the flask, cool, and allow to stand overnight. Filter through a Gooch crucible having a glass filter pad on the bottom covered with diatomaceous earth. Wash the flask and precipitate with a solution of one volume of isopropyl alcohol to two volumes of benzene. After final washing with 25 mL of ethyl ether, draw air through the crucible for 1 minute. Save the filtrate for the analysis of the oil acids. Wash the precipitate from the flask and through the Gooch crucible with about 100 mL of water. Transfer the washing to a separatory funnel. Acidify with 1:4 sulfuric acid. Extract the chlorinated dibasic acid with consecutive volumes of 75 mL, 50 mL, and 50 mL of ethyl ether. Collect the ether extracts and wash with water until the washings are acid-free. Transfer the ether to a 250-mL beaker. Add 5 mL of m-cresol purple indicator (0.025 g in 100 mL of absolute ethyl alcohol) and titrate to a purple end point with 0.2N potassium hydroxide in methyl alcohol.

$$\text{Percent chlorinated dibasic acid} = \frac{\text{mL of alkali} \times \text{normality} \times 19.45}{\text{g of nonvolatile vehicle}}$$

4.2.3 Chlorine. Chlorine shall be determined using the Schoniger Low Pressure Combustion Apparatus. Weigh 30 milligrams (mg) of nonvolatile vehicle from the nonvolatile determination of the resin solution on a Schoniger paper sample holder. Fold the paper over the sample and place in the platinum sample holder on the flask head. Add 15 mL of N/10 sodium hydroxide to the flask. Flush the flask with oxygen and insert the flask head. Place the flask in the safety ignition unit and fire the sample. Allow the flask to cool, remove from unit, and allow to stand until the mist in the flask condenses. Shake the flask vigorously to absorb vapors and then transfer the solution to a 150-mL beaker. Add 5 drops of chloride indicator composed of 5.0 g of chemically pure diphenyl carbazone and 1.0 g of bromphenol blue indicator dissolved in 1 liter (L) of 95 percent ethanol or propanol. Add 0.2N nitric acid until the color changes from purple to yellow and then add 4 drops excess. Titrate with 0.025N mercuric nitrate solution until the color of the solution changes from yellow to lavender.

Percent chlorine in chlorinated dibasic acid =

$$\frac{\text{mL} \times \text{titration} \times \text{normality of mercuric nitrate} \times 355.0}{\text{g sample} \times \text{percent chlorinated dibasic acid in nonvolatile vehicle}}$$

4.2.4 Zinc oxide compatibility. The zinc oxide compatibility test shall be made by pebble mill grinding, using a one-size porcelain-lined jar. Assuming that the nonvolatile content of the resin solution is 60 percent, the following quantities of ingredients shall be placed in the jar:

<u>Ingredient</u>	<u>Grams</u>
Zinc oxide (American Zinc Sales Company AZO-22)	240
Resin solution	200
Paint thinner	65

After the ingredients are weighed into the jar, they shall be mixed with a spatula until fluid. Add 1000 g of 2.06-centimeters (-cm) outside diameter (OD) x 2.06-cm long "Burundum Tabular" stones (U.S. Stoneware Company or equal) to the contents of the jar. Close the jar and place on rolls operating at 86 revolutions per minute for 5 hours (25,800 revolutions). Remove from rolls, open jar, and add 200 g of resin solution. Close jar and again rotate on mill for 15 minutes. Remove from rolls and weigh 530 g of the milled batch into a 1-pint friction top can. Adjust viscosity to 80 Krebs units by adding petroleum spirits. Store the reduced batch in the tightly closed can at room temperature for 5 days and determine the viscosity at this time.

4.2.5 Reduced viscosity at 25°C (77°F). Reduced viscosity shall be determined as follows. Reduce the nonvolatile content of the resin solution to 50 ± 0.5 percent by weight with paint thinner conforming to type II, grade A of TT-T-291. Determine viscosity by procedure in ASTM test method D 1545.

4.2.6 Drying time. Drying time shall be determined on the resin solution, which has been reduced to 50 percent nonvolatile content using paint thinner conforming to type II, grade A of TT-T-291. Based on the resin solids present, add the equivalent of 0.5 percent lead (metal) and 0.05 percent cobalt (metal) using naphenate driers conforming to class B of ASTM D 600. Let stand 24 hours after addition of driers and then determine time required to set to touch and to dry hard in accordance with method 4061 of FED-STD-141.

4.2.7 Water resistance. Water resistance shall be determined on the 50 percent non-volatile content resin solution to which driers have been added as specified in 4.2.6. Pour the reduced resin on a tin panel of the type described in method 2012 of FED-STD-141. After pouring on the coating, allow it to spread over the entire face of the panel, except the upper 1.27 cm, and then allow to drain in a nearly vertical position and dry for 48 hours. Cut off a 1.27-cm strip of the panel from the edge which was at the bottom during the draining and drying periods. (This is done to remove the accumulation resulting in a thick film at this point.) Cut the panel in half lengthwise and save one-half for comparison. Entirely immerse the other half vertically in a beaker of distilled water at room temperature for a period of 24 hours. Two hours after removal from the water, examine the panel by comparison with the unexposed half for whitening, dulling, blooming, blistering, softening, and loss of adhesion.

4.2.8 Paint thinner compatibility. Paint thinner compatibility shall be determined as follows: Place 50-ml of the resin solution in a 200-ml beaker. Add 50-ml paint thinner conforming to type II, grade A of TT-T-291. Stir until thoroughly mixed. Allow to stand 30 minutes and examine. Re-examine at the end of 24 hours.

4.2.9 Appearance. When tested in accordance with 4.2.1.2, the resin solution shall conform to paragraph 3.4.1.

4.2.10 Odor. When tested accordance with 4.2.1.2, the resin solution shall conform to paragraph 3.4.2.

4.2.11 Other resins. The presence of rosin and other resins shall be determined by the methods of 4.2.1.2 and conform to paragraph 3.4.5.

4.3 Toxicity. A manufacturer of material shall disclose the formulation of his product to the Navy Bureau of Medicine and Surgery, Navy Department, Washington, DC 20372. The disclosure of proprietary information, which shall be held in confidence by the Bureau of Medicine and Surgery, shall include: the name, formula, and approximate percentage by weight and volume of each ingredient in the product; the results of any toxicological testing of the product; identification of its pyrolysis products; and any such other information as may be needed to permit an accurate appraisal of any toxicity problem associated with the handling, storage, application, use, or disposal of the material.

5. PREPARATION FOR DELIVERY

5.1 Special marking. Each container shall be marked with the following statement:

"The volatile content of this container is not photochemically reactive as defined by Rule 102 of the South Coast Air Quality Management District." (see 6.3.)

6. NOTES

6.1 Intended use. This specification covers an ingredient for use in specifications for paints and related materials and, as such, is not intended for use for direct procurement by the Government. In the event of small purchases for experimental purposes and for purchases by industry, suitable preparation for delivery requirements should be made a part of the contract. The quality assurance provisions are provided for use in connection with inspection of this resin when used as an ingredient in paints purchased for the Government.

6.2 Ordering data. Procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Special marking required (see 5.1).

6.3 Volatile content. Although the container marking specifically refers to the South Coast Air Quality Management District, the paint may be used anywhere else paint complying with 3.3 is allowed. This includes all other Air Pollution Control Districts or similar areas controlling this emission of solvents into the atmosphere. Information regarding Los Angeles County Air Pollution Rules 102, 442, and 443 may be obtained from: South Coast Air Quality Management District, Metropolitan Zone, 434 South San Pedro Street, Los Angeles, California 90013.

6.4 Changes from previous issue. The symbol "\$" is not used in this revision to identify changes with respect to the previous issue, due to the extensiveness of the changes.

Preparing activity:
Navy - SH
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