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FEDERAL SPECIFICATION

PINE OIL

This specification was approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers the product generally known in the naval stores industry as pine oil, the terpene oil obtained from the wood of the longleaf yellow pine tree (see 6.1).

1.2 Classification.

1.2.1 *Types and grade.* The pine oil covered by this specification shall be of the grade generally known in the trade as "standard", and shall be of the following types, as specified (see 6.2):

Type I.—Steam distilled pine oil, sulphate pine oil, or synthetic pine oil.
Type II.—Destructively distilled pine oil.
Type III.—"Commercial" pine oil.

2. APPLICABLE SPECIFICATIONS, STANDARDS, AND OTHER PUBLICATIONS

2.1 Specifications and Standards. The following specifications and standards, of the issues in effect on date of invitation for bids, form a part of this specification:

Federal Specifications:

TT-P-143 — Paint, Varnish, Lacquer, and Related Materials; General Specifications for Packaging, Packing and Marking.
PPP-B-585—Boxes; Wood, Wirebound.
PPP-B-621—Boxes, Wood, Nailed and Lock-Corner.
PPP-B-636—Box, Fiberboard.
PPP-P-704—Pails: Shipping, Steel (1 through 12 Gallon).

Federal Standards:

Fed. Std. No. 102—Preservation, Packaging, and Packing Levels.
Fed. Std. No. 123—Marking for Domestic Shipment (Civilian Agencies).

Federal Test Method Standards:

No. 141—Paint, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling, and Testing.
No. 791—Lubricants, Liquid Fuels, and Related Products; Methods of Testing.

(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards, and Handbooks as outlined under General Information in the Index of Federal Specifications, Standards, and Handbooks and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.

(Single copies of this specification and other product specifications required by activities outside the Federal Government for bidding purposes are available without charge at the General Services Administration Regional Offices in Boston, New York, Atlanta, Chicago, Kansas City, Mo., Dallas, Denver, San Francisco, Los Angeles, Seattle, and Washington, D. C.

(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications, Standards, and Handbooks from established distribution points in their agencies.)

Military Standards:

MIL–STD–105 — Sampling Procedures and Tables for Inspection by Attributes.

MIL–STD–129—Marking for Shipment and Storage.

(Copies of Military Specifications and Standards 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 documents form a part of this specification. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

Governmental:

49–CFR(1950)–71.1 — Interstate Commerce Commission Rules and Regulations for Transportation of Explosives and Other Dangerous Articles.

(The above Governmental regulations is part of the *Code of* Federal Regulations and is available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Orders for this publication should cite the title and code as listed above. Prices may be obtained from the Superintendent of Documents.)

Nongovernmental:

ASTM Standards—Part 4

ASTM Designation: D 156—Standard Method of Test for Saybolt Color of Refined Petroleum Products.

ASTM Designation: D 802–57—Standard Methods of Sampling and Testing Pine Oils.

ASTM Designation: D 890—Standard Method of Test for Water in Liquid Naval Stores.

ASTM Designation: E 1—Specifications for ASTM Thermometers.

(Applications for copies should be addressed to the American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pennsylvania.)

Uniform Freight Classification:

Ratings, Rules and Regulations.

(Applications for copies should be addressed to the Official Classification Committee, 1 Park Avenue at 33rd Street, New York 16, New York.)

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

2.2.1 The requirements of this specification and the methods of test applicable to pine oil are essentially similar to the following:

American Society for Testing Materials:

ASTM Designation: D 804—Standard Definitions and Terms Relating to Naval Stores and Related Products.

ASTM Designation: D 1364–55T—Tentative Method of Test for Water in Lacquer Solvents and Diluents (Fischer Reagent Titration Method).

3. REQUIREMENTS

3.1 Quality. The pine oil supplied under this specification shall be of the type and kind specified (see 6.5), without admixture with another kind of pine oil or with other terpenic or nonterpenic material.

3.2 Appearance. The pine oil shall be clear and transparent, and free from sediment, suspended matter or visible evidence of separated water when tested as specified in 4.4.1.

3.3 Color. The pine oil shall be not darker than a pale straw yellow, which color corresponds to that of the No. 6.0 yellow Lovibond colorimeter glass, when viewed through a one-inch thick layer of the oil as specified in 4.4.2.1. This color is also similar to No. 19 on the Saybolt Color Scale (see 4.4.2.2).

3.4 Odor. When tested as specified in 4.4.3, the pine oil shall have the pleasantly mild and aromatic ("piney") odor characteristic of the kind or type of oil indicated by the

name of the product. It shall be free from strongly empyreumatic, unpleasantly pungent or offensive odor. The vapors above the oil in an open vessel at room temperature shall not be irritating to the nasal passages. Upon request of the purchaser, the seller shall submit a sample by which the acceptability of the proposed delivery of pine oil as to odor may be established prior to shipment.

3.5 Chemical requirements. The pine oil shall conform to the requirements specified in table I.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examination and tests shall be kept complete and available to the Government as specified in the contract or order. 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.

4.1.1 *Sampling for inspection of filled containers at contractor's plant.* A random sample of filled containers shall be taken by the inspector in accordance with Military Standard MIL–STD–105 at inspection level I and acceptable quality level = 2.5 percent defective to verify compliance with this specification in regard to fill, closure, marking, and other requirements not involving tests.

4.1.2 *Sampling for tests at laboratory.*

4.1.2.1 From each inspection lot, (see 4.1.1), the inspector shall take two containers at random. From each of the two containers, 1-quart samples shall be taken and placed in separate, clean, dry, metal or glass containers, and then sealed, marked and forwarded to the test laboratory designated by the activity concerned.

4.1.2.2 When requested by the contractor, the inspector shall select samples in accord-

TABLE I.—*Quantitative requirements*

Requirement	Type I		Type II		Type III		Test Paragraph
	Min.	Max.	Min.	Max.	Min.	Max.	
Specific gravity at 15.5/15.5°C.	0.930	0.945	0.900		0.925		4.4.4
Refractive index at 20°C.	1.479	1.492	1.479	1.498	1.475	1.490	4.4.5
Dissolved water, percent by weight		1.0		1.5		1.5	4.4.6
Acid number		2.0		5.0		2.0	4.4.7
Distillation range, percent by volume:							4.4.8
Distilled below 185°C.		5.0		5.0		5.0	
Distilled below 200° C.		25.0		20.0		25.0	
Distilled below 230° C.	95.0		85.0		90.0		
Total terpene alcohols, percent by weight	70		40		60		4.4.9
Residue after polymerization, percent by volume		2.0		3.0		3.0	4.4.10

ance with 4.1.2.1 with the following exception: Three-quart samples shall be taken instead of 1-quart samples. Each 3-quart sample shall be divided into three equal parts, one part to be delivered to the contractor, one part to the testing laboratory, and one part to be held by the inspector in case of dispute.

4.1.2.3 *Testing activity.* Unless otherwise specified, the samples taken by the inspector as required for test purposes shall be forwarded to the Industrial Laboratory, Mare Island Naval Shipyard, Vallejo, California; to the Chemical Laboratory, Norfolk Naval Shipyard, Portsmouth, Virginia; or to the Materiel Laboratory, New York Naval Shipyard, Brooklyn 1, New York.

4.2 Inspection. Inspection shall be performed in accordance with method 1011 of Federal Test Method Standard No. 141.

4.2.1 *Inspection of filled containers at the contractor's plant.* Each sample filled container, selected as specified in 4.1.2 shall be examined for defects of construction of the container and closure, for evidence of leakage, and for unsatisfactory markings. Each filled container shall also be weighed to determine the amount of contents. Any container in the sample having one or more defects, or under required fill, shall be rejected, and if the number of defective containers in any sample exceeds the acceptance number for the appropriate sampling plan of Military Standard MIL-STD-105, the lot represented by the sample shall be rejected. Rejected lots may be resubmitted for acceptance tests provided the contractor has removed or reworked all nonconforming containers.

4.3 Lot acceptance tests. The samples selected in accordance with 4.1.2 shall be subjected to the tests specified in 4.4 except that the laboratory may perform complete tests on only one sample from each lot provided sufficient tests are made on the second sample to verify that both are alike. Lots shall be accepted or rejected by the inspector on the basis of the laboratory test report on the transmitted samples. Rejected lots may be resubmitted for acceptance tests provided the contractor has removed or reworked all nonconforming products.

4.4 Test procedures.

4.4.1 *Appearance.* The appearance of the pine oil shall be examined in accordance with method 4261 of Federal Test Method Standard No. 141.

4.4.2 *Color.*

4.4.2.1 *Lovibond scale.* Fill a glass colorimeter cell, having an internal length of one inch, with the oil or pour the oil to a depth of one inch into a cylindrical colorimeter tube having a polished flat bottom. Fill a second cell or tube to the same depth with distilled water. Place a No. 6.0 yellow Lovibond colorimeter glass in front of the cell containing the water, and compare the two cells against a neutral white light (clouded sky); or place the colorimeter glass on the tube containing the water and hold the two tubes side by side over a white reflecting surface such as a sheet of white paper or block of magnesia. The color of the one-inch layer of pine oil shall be not darker than the color of the glass plus water in the cell or tube.

4.4.2.2 *Alternate color test (Saybolt color scale).* Determine the color of the pine oil according to the Standard Method of Test for Color of Refined Petroleum Oils by Saybolt Chromometer (method 101 of Federal Test Method Standard No. 791, or ASTM Designation: D 156). The color shall be not darker than Saybolt No. 19.

4.4.3 *Odor.* The odor of the pine oil shall be determined in accordance with method 4401 of Federal Test Method Standard No. 141. When specified, the odor of the sample shall be compared with that of an agreed-upon reference sample preserved in a tightly stoppered bottle.

4.4.4 *Specific gravity.* Determine the specific gravity of the pine oil with a hydrometer or specific gravity balance standardized at 15.56°C. (60°F.). If the temperature of the oil when tested is above or below 15.56° C., correct the specific gravity reading to that base by adding or subtracting from the observed reading a correction factor of 0.00080 for each degree Centigrade or 0.00045 for each degree Fahrenheit that the temperature is above or below the specified level, respectively.

4.4.5 *Refractive index.* Determine the refractive index of the pine oil with an Abbe refractometer in accordance with method 4371 of Federal Test Method Standard No. 141, at any convenient room temperature. If the temperature of test is above or below 20°C. (68°F.), the reading shall be corrected to 20°C. by adding or subtracting from the observed reading the factor 0.00040 for each degree Centigrade, of 0.000222 for each degree Fahrenheit that the test temperature of the oil is above or below 20°C., respectively.

4.4.6 *Dissolved water.* Fill a test tube with the pine oil, stopper, and immerse in an ice-bath for 10 minutes. If the pine oil remains clear, the dissolved water is within the specified limit, and no further tests need be made. If turbidity develops, determine the water content quantitatively in accordance with method 4081 or method 4082 of Federal Test Method Standard No. 141. In the distillation method, the flask must be free from etched surfaces, as these can bring about some slight decomposition of the terpene alcohols, resulting in too high a value for the water content.

4.4.6.1 *Alternate titration method for water.* The water in pine oil may be determined by titration with Karl Fischer reagent, a complex solution of iodine, pyridine, methanol, and sulfur dioxide, according to the procedure described in method 4082 of Federal Test Method Standard No. 141, or the modification thereof entitled, "Standard Method of Test for Water in Liquid Naval Stores", ASTM Designation: D 890. In the latter method, the reagent is prepared and stored as two separate solutions, which are brought together in the reactive stage during the course of the determination.

4.4.7 *Acid number.* The acid number of the pine oils shall be determined in accordance with method 5071 of Federal Test Method Standard No. 141.

4.4.8 *Distillation range.* The distillation range of the pine oil shall be determined in accordance with method 4301 of Federal Test Method Standard No. 141. Since the distilling temperature of pine oil is affected by changes in atmospheric pressure, the temperatures specified in table I must be adjusted whenever the barometer reading (after correcting to 0°C. for a mercurial barometer) is above or below 760 mm. The adjusted temperature is determined by adding or subtracting from the figures in the table the factor 0.059°C. for each millimeter that the corrected barometer reading differs from 760 mm. (The corrections to be applied, over a range of barometer readings, for both aneroid and mercurial barometers, and for various room temperatures are given in table II.

4.4.8.1 The thermometer used for pine oil distillation shall be either the ASTM Partial Immersion Thermometer No. 2C, or the ASTM Solvents Distillation Thermometer No. 42C (see Standard Specifications for ASTM Thermometers, ASTM Designation: E 1–52, or method 9501 of Federal Test Method Standard No. 791). A Liebig glass condenser having 400 mm. of its length in contact with the cooling water may be used for the distillation test.

4.4.9 *Total terpene alcohols.*

4.4.9.1 *Apparatus.*

4.4.9.1.1 *Flask.* A round bottom, short neck flask of 500-ml. capacity.

TABLE II.—*Correction for Barometric pressure*[1]

Observed barometic pressure (mm.)	Aneroid barometer	Mercurial barometer			
		Temperature of barometer			
		20°C.	25°C.	30°C.	35°C.
780	−1.18	−1.03	−0.99	−0.96	−0.92
770	−0.59	−0.44	−0.41	−0.37	−0.33
760	0.00	+0.15	+0.18	+0.22	+0.25
750	+0.59	+0.73	+0.77	+0.81	+0.84
740	+1.18	+1.32	+1.36	+1.39	+1.43
730	+1.77	+1.91	+1.94	+1.98	+2.01
720	+2.36	+2.50	+2.53	+2.57	+2.60
710	+2.95	+3.09	+3.12	+3.15	+3.19
700	+3.54	+3.67	+3.71	+3.74	+3.77

Note 1—See ASTM Designation: D 802–57; Standard Methods of Sampling and Testing Pine Oil.

4.4.9.1.2 *Water trap.* A trap and adjustable mercury reservoir, constructed in accordance with the dimensions shown in figure 1; or a Dean & Stark (Barrett type) moisture trap of 20-ml. capacity with a ground glass stopcock and graduation markings at each 0.1 ml.

4.4.9.1.3 *Condenser.* A 12-inch straight water-cooled Liebig glass condenser.

4.4.9.2 *Procedure.* Weigh 0.1 gram of powdered silica gel or Kontak clay and transfer it to the flask along with a few glass beads. Tare the flask to the nearest 0.1 gram. Using a pipette, weigh into the flask 100 ± 0.25 gram of the sample, taking care to avoid getting oil on the neck or outside of the flask (remove with dipentene or other solvent if this occurs). From a 1-ml. graduated pipette, add 0.3 ml. of an 85 percent reagent grade syrupy phosphoric acid. Apply a thin film of stopcock lubricant to all ground glass joints and connect the apparatus. Bring the mercury up into the trap and fasten the ring support of the reservoir at a height to hold the top of the mercury column about one inch below the side tube. Insert a snug fitting, long-handled burette brush into the top of the condenser, keeping the brush near the top. The handle must be long enough to permit occasional lowering of the brush down to the area where condensation of the distillate takes place. Apply heat to the flask, gently at first, until the reaction gets underway. As the distillation proceeds, the decomposition products (water and dipentene) are collected in the trap. Gradually lower the mercury level in the graduated leg of the trap and increase the rate of boiling. Bring down any droplets of water that might adhere to the sides of the condenser by adding a few drops of xylene, dry dipentene or mineral spirits at the top of the condenser, in order to wet the brush. By moving the brush up and down at the lower end of the condenser where the droplets of water are hung up, all water can be brought into the trap. At 15 to 20 minute intervals during the test, raise the mercury reservoir slowly so as to return the supernatant dipentene back into the flask, but retaining the water in the trap. Again lower the mercury level to permit additional distillate to collect. Continue the distillation for 4 hours. Free the condenser tube from any hung-up water droplets and measure the vol-

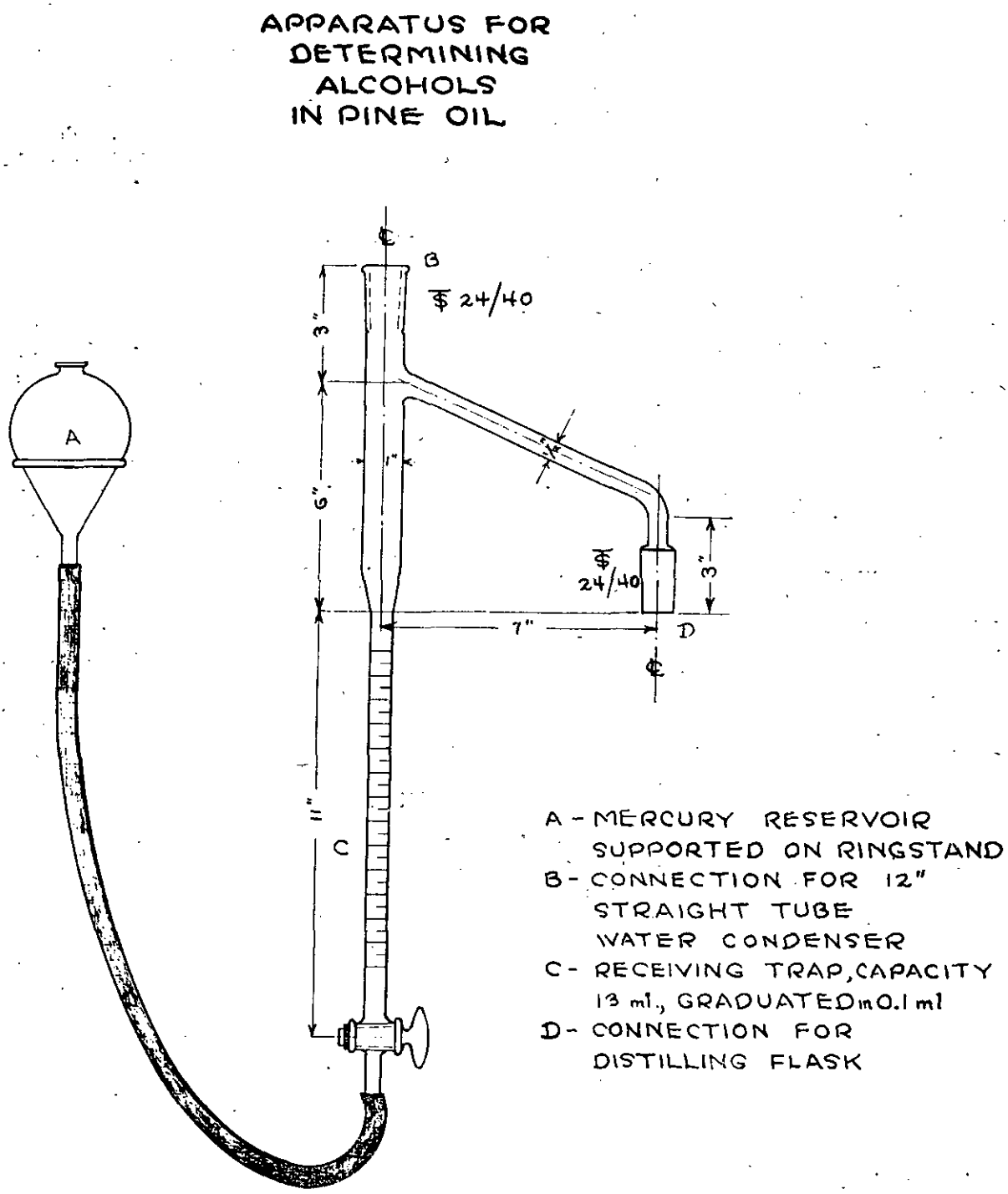

FIGURE 1.—Terpene alcohol determination apparatus.

ume of water collected in the trap. From this volume deduct the amount of dissolved water in the sample, as determined in 4.4.6. Compute the percent of total terpene alcohols as follows:

Total alcohols, percent = ml. water collected × 8.56

(Factor: 1.0 ml. water is equivalent to 8.56 grams terpineol.)

4.4.10 *Residue after polymerization.* The residue after polymerization shall be determined in accordance with method 5121 of Federal Test Method Standard No. 141.

5. PREPARATION FOR DELIVERY

For civil agency procurement, the definitions and applications of the levels of packaging and packing shall be in accordance with Federal Standard No. 102.

5.1 Packaging.

5.1.1 *Level A.* Pine oil shall be furnished in 1-gallon cans, or 5- or 55-gallon drums conforming to the requirements specified in Federal Specification TT–P–143. Five-gallon pails shall conform to type I of Federal Specification PPP–P–704; exterior coating shall be olive drab and containers shall be furnished with pour spouts. Bulk shipments shall be made in accordance with existing Interstate Commerce Commission Regulations. When specified, pine oil may be furnished in 5-gallon square cans in accordance with Federal Specification TT–P–143. One-gallon and 5-gallon square cans shall be made from a minimum commercial tinplate having a 0.50 pound of tin per base box of metal.

5.1.2 *Level C.* Pine oil shall be packaged in accordance with the manufacturer's commercial practice.

5.2 Packing.

5.2.1 Levels A and C shall be packed in accordance with the requirements of Federal Specification TT–P–143.

5.2.2 *Level C.* Pine oil shall be packed to afford protection against damage during direct shipment from the supply source to the first receiving activity for immediate use. Containers shall be of the size commonly used for the purpose, and shall comply with the Uniform Freight Classification Regulations or other common carrier regulations applicable to the mode of transportation.

5.3 Marking.

5.3.1 Individual containers and the outer cases or carton holding small containers shall show the correct legal designation and type of pine oil contained therein, as set forth in the specifications.

5.3.2 *Civil agencies.* In addition to the marking specified in 5.3.1 and any special marking required by the contract or order, marking shall be in accordance with Federal Standard No. 123.

5.3.3 *Military agencies.* In addition to the marking specified in 5.3.1 and any special marking required by the contract or order, marking shall be in accordance with Federal Specification TT–P–143.

6. NOTES

6.1 Kinds of pine oil. The article known in the trade as "pine oil" is a high boiling fraction of the terpene oils obtained from pine wood by any of the processes described below. The principal constituents of pine oils are the secondary and tertiary cyclic terpene alcohols, of which alpha-terpineol, borneol, and fenchyl alcohol are present. In addition, there are also present in lesser quantity, variable proportions of several terpene hydrocarbons as well as other related terpene compounds, the proportion and character of which, depend on the source of the oil and the method of manufacture. The four kinds or classification of pine oil recognized in the trade, depending on the source, kind, and condition of the wood from which the oil is

derived, or the method of production, are designed or described as follows:

(a) Steam distilled (or "natural") pine oil, a high boiling fraction of the distillate obtained from pine stumpwood by steam distillation, or by extraction followed by such distillation.

(b) Sulphate pine oil, a refined oil obtained by fractional distillation from certain high boiling portions of the condensed vapors sulphate wood turpentine) recovered during the alkaline digestion of freshly cut pine wood in the sulphate process of making paper.

(c) Synthetic pine oil, the product made by chemical hydration of pinene or by dehydration of terpin hydrate to form terpene alcohols.

(d) Destructively distilled pine oil, an oil obtained by fractional redistillation of the lighter portion of the condensate recovered in the dry digestive distillation (carbonization) of pine stumpwood.

6.2 Types of pine oil. Steam distilled or natural pine oil from pine stumpwood is the best known kind of pine oil, and is usually available in the greatest quantity and highest quality. Sulphate and synthetic pine oils are refined to simulate quite closely the composition and properties of steam distilled pine oil. For this reason, the three kinds are often used interchangeably for many purposes, and may therefore be considered to be of the same type of material, described herein as type I. Destructively distilled pine oil has a noticeably different odor and composition, including lower terpene alcohol content, so that it cannot conform with the test requirements specified for type I. Such oil is classified under a separate designation as type II. The quality and properties of pine oils can be varied at will by the manufacturer. For certain uses, an oil similar to but of lower quality and price than envisioned by type I may be adequate for the purpose. For such cheaper, lower quality oil, a separate classification and test requirements are provided, with the designation type III. This comprises oils having a lower density, distillation range, and terpene alcohol content than are required for oils of type I.

6.3 Intended use. The pine oil covered by this specification is intended for use in the vehicle of anti-fouling ship-bottom paints; as an ingredient of insecticide, germicidal and deodorizing compositions; as a component of textile soaps and other cleaning, detergent and degreasing compounds; as a frothing agent; and for other uses. For some uses, the odor of the pine oil may have a bearing on its suitability for the proposed use.

6.4 The article described in the pharmaceutical trade as "oil of pine", derived from pine needles, and the dark, empyreumatic oil generally known as "oil of pine tar", obtained by redistilling pine tar oil, do not come within the meaning of the term "pine oil" as covered by this specification.

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

(*a*) Number, title, and date of this specification.

(*b*) The type and kind of pine oil required, if limitation is desired (see 1.2.1).

(*c*) The size of the unit containers (see 5.1).

(*d*) The selection of the applicable levels of packaging and packing (see section 5).

(*e*) Whether a pre-shipment sample of the proposed delivery is required (see 3.4).

6.6 Basis of purchase. Pine oil shall be purchased by volume.

6.7 Identification of pine oil. The purchasing agency may state in the invitation for bids, or in the contract, whether delivery will be limited to one or more kinds of pine oil, depending on the intended use thereof.

The bidder shall in every case state the kind of pine oil he proposes to furnish under the bid submitted.

6.8 Inspection. The United States Department of Agriculture maintains a Federal naval stores inspection and grading service in the areas where pine oil is produced. An agency purchasing pine oil may, by consulting the Naval Stores Branch, Agricultural Marketing Service, U. S. Department of Agriculture, Washington 25, D. C., determine whether it appears practicable and possible, on shipments originating in such producing areas, to effect a saving by delegating authority to that Branch to have its inspectors make the initial sampling and inspection, under reimbursement from the purchasing agency.

6.9 Scope of this specification. This specification describes properties and characteristics of the grade of pine oil suitable for the needs of the Federal Government. It is not intended to include all types or grades of pine oil which may be commercially available. Except when stated in the invitation for bids, and the proposed use of the material indicates the desirability of a preliminary test of the odor, samples will not be required prior to the award of the contract.

6.10 Transportation description. Transportation description and minimum weight application to this commodity are:

Rail:
Pine Oil
Carload minimum weight 30,000 pounds.

Motor:
Pine oil
Motor volume minimum weight, 30,000 pounds.

MILITARY CUSTODIANS:
Army—Q
Navy—Sh.