MIL-V-13750B(AR)
18 February 1972
SUPERSEDING
MIL-V-13750A(MR)
9 February 1966

MILITARY SPECIFICATION

VARNISH, PHENOL-FORMALDEHYDE, CLEAR AND ALUMINUM PIGMENTED

1. SCOPE

1.1 Scope. This specification covers two grades and four types of clear and aluminum pigmented para-phenyl phenol-formaldehyde resin air drying varnishes for use on ammunition (see 6.1). It also provides for two compositions one of which is suitable for use under AIR POLLUTION REGULATIONS (see 6.4).

1.2 Classification.

1.2.1 Grades. The varnishes (clear and aluminized) covered by this specification shall be furnished in the following grades as specified.

Grade A - Tung oil para-phenyl phenol-formaldehyde resin varnish.

Grade B - Linseed oil para-phenyl phenol-formaldehyde resin varnish.

1.2.2 Types. Grade A and B varnishes shall be furnished in the four types listed in Table I.

1.2.3 Composition. The varnishes covered by this specification shall be of the following compositions as specified.

Composition G - General use.

Composition L - Limited use (see 6.4).

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2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

Federal

TT-L-1155 - Linseed Oil, Alkali Refined

TT-P-143 - Paint, Varnish, Lacquer and Related Materials; Packaging Packing, and Marking of

TT-P-320 - Pigment, Aluminum, Powder and Paste, For Paint

TT-R-271 - Resin Phenol-Formaldehyde, Para-Phenyl

TT-T-548 - Toluene, Technical

TT-T-775 - Tung Oil, Raw (China Wood) (For Use in Organic Coating)

TT-X-916 - Xylene, (For Use in Organic Coatings)

STANDARDS

Federal

Fed. Test Method Std. No. 141 - Paint, Varnish, Lacquer and Related Materials; Methods of Inspection, Sampling and Testing

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

3. REQUIREMENTS

3.1 Materials. The materials used in the manufacture of the varnishes purchased under this specification shall be of high quality, entirely suitable for the purpose, and shall conform to the applicable specifications.

3.2 Composition. The composition of the varnishes shall be as specified in Table I. For grade A varnishes the oil portion shall be raw tung oil (TT-T-775). For grade B varnishes the oil portion shall be alkali refined linseed oil (TT-L-1155). Driers and anti-oxidants may be used as required.

3.2.1 Composition L. The vehicle shall be the same as 3.2 and Table I except the volatile solvents used shall conform to the following requirements by volume when tested as in 4.4.3.

(a) Aromatic compounds with eight or more carbon atoms except ethyl benzene: 8 percent maximum.

(b) Ethyl benzene and toluene: 20 percent maximum.

(c) Solvents with an olefinic or cyclo-olefinic type of unsaturation: negative test (see 6.5).

(d) Ketones: negative.

(e) Total of a + b: 20 percent maximum.

Type	Ingredients	Percent by Weight
I (Clear)	Para-phenyl phenol-formaldehyde resin, TT-R-271	25.0 ± 1.0
	Oil (see 1.2.1 and 3.2)	25.0 ± 1.0
	Xylene (TT-X-916, Grade B)	50.0 ± 2.0
II (Clear)	Varnish conforming to Type I	50.0
	Toluene (TT-T-548)	50.0
III (Pigmented)	Varnish conforming to Type I	84.0 ± 0.5
	Aluminum Powder (TT-P-320, Type I, Class B)	16.0 ± 0.5
IV (Pigmented)	Varnish conforming to Type I	42.0 ± 2.0
	Aluminum Powder (TT-P-320, Type I, Class B)	16.0 ± 1.0
	Toluene (TT-T-548)	42.0 ± 2.0

3.3 Quantitative requirements. The varnishes shall conform to the quantitative requirements specified in Table II when tested as specified in 4.4.

TABLE II - Quantitative requirements

Characteristics	Type I	Type II	Type III	Type IV
Total solids, percent by weight of product, minimum	48.0	24.0	56.0	35.0
Pigment, percent by weight of product, minimum	--	--	15.5	15.5
Pigments other than aluminum	None	None	None	None
Rosin and rosin derivatives	None	None	None	None
Phenols other than para-phenyl phenol	None	None	None	None
Isoctane insoluble, percent by volume, minimum	50	25	50	20
Drying oils:				
Grade A	Tung	Tung	Tung	Tung
Grade B	Linseed	Linseed	Linseed	Linseed
Viscosity, No. 4 Ford Cup, seconds	35-65	10-25	85-200	10-25
Drying time:				
Air drying, minutes, maximum				
Set to touch	30	10	30	10
Dry through	90	60	90	20
Directional reflectance, percent	--	--	15-30	15-30

3.4 Qualitative requirements.

3.4.1 Condition in container. The clear varnishes from freshly opened full containers shall be transparent liquids free from moisture, skins or lumps. The aluminum pigmented varnishes shall not show hard settling in freshly opened full containers, shall be free from skins or lumps and shall readily mix to a smooth homogeneous state. There shall be no evidence of livering, curdling or gassing in either the clear or aluminum pigmented varnishes when tested as in 4.4.9.

3.4.2 Accelerated storage stability (pigmented type only). When Type III and Type IV are tested as in 4.4.10 there shall be no pressure build up. The viscosity of Type III shall not increase more than 10 seconds over the viscosity of the material as received. The viscosity of Type IV shall not increase more than 5 seconds over the viscosity of the original material. A spray coat of the varnishes shall show no loss of leafing as compared to a spray coat of the materials as received (visual comparison).

3.4.3 Flexibility. When tested as in 4.4.11 all grades and types except Grade B, Type I, II and III shall withstand the bend test without evidence of cracking or flaking. Grade B, Types I, II and III may crack but shall not lift or flake.

3.4.4 Resistance to boiling water. When tested as in 4.4.12 Types I and II and the isolated vehicles from Types III and IV shall show no wrinkling or blistering and no more than slight dulling or softening immediately after removal from boiling water. When inspected half hour after removal the portion of the panel which was immersed shall be almost indistinguishable with regard to hardness and adhesion from the duplicate unexposed panel. The tendency for a slight whitening to remain shall be disregarded.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier 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 that supplies and services conform to the prescribed requirements.

4.2 Sampling, inspection and testing. Unless otherwise specified, sampling, inspection and testing shall be in accordance with method 1031 of Fed. Test Method Std. No. 141.

4.3 Classification of tests. Testing under this specification shall be for the purpose of acceptance of individual lots.

4.4 Test methods.

4.4.1 Test conditions. The routine and referee testing conditions shall be in accordance with section 7 of Fed. Test Method Std. No. 141 except as otherwise specified herein.

4.4.2 The following tests shall be conducted in accordance with applicable methods of Fed. Test Method Std. No. 141 or as hereinafter specified. The right is reserved to make any additional tests deemed necessary to determine that the varnish meets the requirements of this specification.

TABLE III - Index

Tests	Test Method		Paragraph of this specification giving requirements
	Applicable method in Fed. Test Method Std. No. 141	Paragraph of this specification giving further references	
Aromatic hydrocarbons	--	4.4.3.2	3.2.1
Olefinic and cyclo-olefinic compounds	--	4.4.3.3	3.2.1
Ketones	--	4.4.3.4	3.2.1
Total solids	4041	--	Table II
Pigment	--	4.4.4	Table II
Pigments other than aluminum	--	4.4.5	Table II
Vehicle isolation	4032	--	--
Rosin	5031	--	Table II
Phenolic resins	5141	--	Table II
Isooctane insoluble	--	4.4.6	Table II
Identification of drying oils	7501	4.4.7	Table II
Viscosity	4282	--	Table II
Drying time	4061	4.4.8	Table II
Directional reflectance	6121	--	Table II
Condition in container	3011	4.4.9	3.4.1
Accelerated storage stability	--	4.4.10	3.4.2
Flexibility	6221	4.4.11	3.4.3
Resistance to boiling water	6011	4.4.12	3.4.4

4.4.3 Solvent analysis for composition L.

4.4.3.1 Separation of volatile portion. Pour about 15 grams of the varnish into a 50ml. distilling flask. Add 10 ml. of tricresyl phosphate and several anti-bumping stones or Berl saddles. Fit a release valve into the mouth of the flask and attach a delivery tube to the side arm, extending into a receiver consisting of a test tube (20 x 150 mm) with side arm for attaching to a vacuum pump. The glass delivery tube should reach 1-1/2 inches from the bottom of the tube. Immerse the receiver in a dry ice-acetone bath. Preheat a silicone oil bath to 180°C. Raise the oil bath until the oil reaches the sample level. Reduce the pressure slowly to 10 mm. of mercury. After all solvent has distilled, carefully release the vacuum using the valve that is connected to the distilling flask. Reserve the collected distillate for the aromatic solvent determination and the test for ketone, olefinic and cyclo-olefinic compounds.

4.4.3.2 Determination of aromatic hydrocarbons.

Apparatus: A gas chromatograph equipped with a thermal conductivity detector.

Column Preparation: Two lengths of 1/4-inch copper tubing, 6-ft. and 18-ft long, are packed with 35% N, N-Bis (2-cyanoethyl) formamide on 60- to 80- mesh Chromosorb P.

Operating Conditions:	6-ft.	18-ft.
Detector cell temperature, °C.	300	300
Detector cell current, ma.	150	150
Injection port temperature, °C.	300	300
Helium flow at exit, cc/minute	175	110
Column temperature, °C.	125	100

4.4.3.2.1 Total aromatic content - procedure A. Transfer precisely 3 ml. of distillate or thinner to a 25-ml. glass-stoppered volumetric flask and add exactly 0.3 ml. of high purity phenylcyclohexane. While cooling the graduate under tap water, add 15 ml. of 85% sulfuric acid slowly. After all the acid has been added, shake vigorously for 2 minutes and allow the layers to separate. Add sufficient 85% acid to force the top layer into the neck of the flask and then transfer most of the top layer to a micro-separatory funnel. Wash the distillate with 5 ml. portions of distilled water until all acid has been removed and reserve the washed solvent for chromatographic analysis. Install the 6-ft. column and follow the operating conditions described above. Inject about 5 microliters of the acid-treated sample and allow the chromatogram to develop until the internal standard, phenylcyclohexane, emerges.

$$\% \text{ total aromatic solvents, v/v} = \frac{A \times 10^{*} \times 1.07^{**}}{B}$$

where, A = area of aromatic solvent peaks
B = area of internal standard peak
* = percent of internal standard
** = detector response correction factor

NOTE: If the above determination exceeds 8 percent, continue with the following procedure:

4.4.3.2.2 Toluene and ethylbenzene - procedure B. Treat 3 ml. of solvent in the same manner as described in procedure A except substitute benzene for phenylcyclohexane. Install the 18-ft. column and follow the operating conditions described for that column. Inject about 3 microliters of sample and allow the chromatograph to develop until all of the xylene isomers appear. Purge the column by raising the column temperature to 120°C. After the high boiling materials emerge, reset the column temperature to 100°C. Calculate the percent of toluene and ethylbenzene as follows:

$$\% \text{ toluene, v/v} = \frac{(\text{area of toluene peak})\ (1.017)^{*}\ (10)^{**}}{(\text{area of benzene peak})}$$

$$\% \text{ ethylbenzene, v/v} = \frac{(\text{area of ethylbenzene peak})\ (1.054)^{*}\ (10)^{**}}{(\text{area of benzene peak})}$$

where, * is the correction factor for the detector response
** is the percentage of internal standard added

NOTE: Sensitivity of the instrument should be adjusted to keep peaks from running off the scale. Appropriate corrections must be made for changes in sensitivity when computing the peak areas.

4.4.3.3 Test for olefinic or cyclo-olefinic compounds. Take 2 test tubes and place 2 drops of the distillate in each. Dissolve the first sample in 1 ml. of carbon tetrachloride and add 1 drop of 1 percent bromine in carbon tetrachloride. Shake and allow to set for 5 minutes. A positive test is indicated by the complete absence of yellow color when observed against a white background. Dissolve the second sample in 1 ml. of acetone and add 1 drop of 1 percent permanganate solution (1 gram of potassium permanganate crystals in 95 mls. of acetone and 5 mls. of water). Shake and allow to set for 2 minutes. A positive test is indicated by the decolorization of the purple solution. The solvent is considered to fail the test for olefinic and cyclo-olefinic compounds if either of the above tests is positive (see 3.2.1 and 6.5).

4.4.3.4 Test for ketones.

4.4.3.4.1 Reagent. Two grams of 2,4-dinitrophenylhydrazine + 4 mls. of concentrated sulfuric acid + 30 mls. methanol (add slowly) + 10 mls. water.

4.4.3.4.2 Procedure. Pipette 1 ml. of reagent into a 20 x 170 mm. test tube. Add 10 drops of distillate (see 4.4.3.1) and shake for 30 seconds. A yellow precipitate or cloud in the reagent layer indicates the presence of ketones. Run a blank using one ml. of reagent and 10 drops of mineral spirits.

4.4.4 Pigment determination. Using a 10 cc. syringe transfer an 8 to 10 gram sample of the thoroughly mixed varnish to a 250-ml. beaker. Determine the weight of the sample to the 0.001 gram by difference using the weight of the syringe and varnish and the weight of the syringe after discharging the varnish. Add 150-ml. of the benzol to the beaker and stir thoroughly. Filter the mixture through a tared 30-ml. fritted glass filtering crucible having a medium porosity. Wash any residue in the beaker into the crucible with a stream of benzol. Thoroughly wash the residue in the crucible first with benzol and then with acetone. Dry at 100° to 105°C. for one hour, cool in a desiccator and weigh. Calculate the percent pigment in the sample from the increase in weight of the crucible. Save the extracted aluminum pigment for subsequent sodium hydroxide solubility test.

4.4.5 Pigments other than aluminum. Weigh and approximate 0.5 gm. sample of the isolated pigment (from 4.4.4) and transfer to a 600-ml. Erlynmeyer flask. Add 300-ml. of 0.1 normal sodium hyrdoxide solution. Stopper and shake the flask frequently during a one half hour period. Release the stopper while the material is at rest to permit escape of hydrogen gas. Note whether any insoluble material is present. A slight cloud which does not settle out on standing may be observed. Filter immediately through a tared Gooch crucible. Wash thoroughly with hot water and dry to constant weight. From the increase in the weight of the crucible and the weight of the original sample calculate the percent insoluble in 0.1 Normal NaOH as follows:

$$\frac{\text{Wt. of 0.1 Normal NaOH insoluble} \times 100}{\text{Weight of sample}}$$

Any amount of 0.1 Normal NaOH insoluble in excess of 2.0% shall be considered pigment other than aluminum.

4.4.6 Isooctane insoluble. Into a 500 ml. graduated centrifuge tube pour exactly 10 ml. of isooctane. Add exactly 10 ml. of the clear varnish or isolated vehicle. Fill the tube with isooctane, stopper and agitate thoroughly, centrifuge for approximately one hour and read the insoluble (bottom layer) in ml. Multiply ml. by ten and report as percent isooctane insoluble.

4.4.7 Identification of drying oils. Extract the fatty acids as in methods 7021 and 7031 of Fed. Test Method Std. No. 141 and identify the drying oil by its characteristic fatty acids following the procedure in method 7501 of Fed. Test Method Std. No. 141. Check for compliance with Table II.

4.4.8 Drying time. Draw down a 2 inch wide film of the varnish on a clear plate glass panel with a 0.0015 inch film applicator (0.0030 inch gap clearance). Determine drying time under referee conditions as in method 4061 of Fed. Test Method Std. No. 141 and observe for compliance with Table II.

4.4.9 Condition in container. Determine package condition as in method 3011 of Fed. Test Method Std. No. 141 and observe for compliance with 3.4.1.

4.4.10 Accelerated storage stability (pigmented type only). Provide the lid for a standard triple tight half pint paint can with a 1/4 inch air tight copper outlet tube approximately 1-1/2 inches long. Insert a rubber tube approximately 3 inches long well down over the end of the outlet tube and stopper with two screw clamps spaced about 1/2 inch apart. Pour 7-1/2 fluid ounces of the varnish as received into a half pint can and seal with the lid described above. Submerge in a water bath at 130° ± 2°F. for 72 hours. Examine periodically for any indication of gas escape. Cool to room temperature and attach the free end of the rubber tube to a gauge sensitive to pressures from 0 to 300 mm. of mercury. Release screw clamps and check pressure build-up, viscosity and leafing properties for compliance with 3.4.2.

4.4.11 Flexibility. Solvent clean a tinplate panel as in method 2012 of Fed. Test Method Std. No. 141 using the aliphatic naphtha-ethylene glycol monoethyl ether solvent mixture. Flow a coat of the varnish on the panel and let stand in a nearly vertical position at 70° to 90°F (21° to 32°C.) for 1 hour. Then place the panel in a horizontal position in a properly ventilated oven and bake for 5 hours at 203° to 212°F. (95° to 100°C.). Remove from the oven, condition the panel at 73.4° ± 2°F. (23° ± 1.1°C.) for one-half hour and bend over a 1/4 inch mandrel as in method 6221 of Fed. Test Method Std. No. 141 and observe for compliance with 3.4.3.

4.4.12 Resistance to boiling water. Using a 0.002 inch (0.004 inch gap clearance) film applicator draw down a 2 inch wide film of the clear varnish or isolated vehicle on two flat tinplate panels prepared as in method 2012 of Fed. Test Method Std. No. 141 using the aliphatic naptha-ethylene glycol monoethyl ether mixture. Allow to air dry for 72 hours under referee conditions and immerse one panel in boiling water for 15 minutes as in method 6011 of Fed. Test Method Std. No. 141. At the end of the test period remove the panel from the water and inspect for compliance with 3.4.4.

5. PREPARATION FOR DELIVERY

5.1 Packaging and packing. The varnish shall be delivered 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 varnish shall be packaged level A or C; packed level A, B, or C as specified (see 6.2) in accordance with TT-P-143.

5.2 Marking. The containers shall be marked in accordance with TT-P-143.

6. NOTES

6.1 Intended use. The Types I and II clear varnishes and Types III and IV ready mixed aluminum paints covered by this specification are intended for use as a sealer for detonators.

6.2 Ordering data. Procurement document should specify the following:

(a) Title, number and date of this specification.
(b) Grade, type and composition varnish required (see 1.2).
(c) Level of packaging and level of packing (see section 5).
(d) Size of containers (see section 5).

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

6.4 Composition L varnishes should be specified for use in areas with regulations controlling the emission of solvents into the atmosphere.

6.5 The test for olefinic and cyclo-olefinic compounds will not be positive for solvents containing less than 1 percent of these compounds.

Custodian:

Army - AR

Review activities:

Army - MR

Preparing activity:

Army - AR

(Project No. 8010-A006)

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revisio letter should be given.
2. The submitter of this form must complete blocks 4, 5, 6, and 7.
3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization t waive any portion of the referenced document(s) or to amend contractual requirements.

I RECOMMEND A CHANGE:	1. DOCUMENT NUMBER	2. DOCUMENT DATE (YYMMDD)
	MIL-V-13750B(AR)	720218

3. DOCUMENT TITLE
VARNISH, PHENOL-FORMALDEHYDE, CLEAR AND ALUMINUM PIGMENTED

4. NATURE OF CHANGE *(Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets if needed.)*

5. REASON FOR RECOMMENDATION

8. PREPARING ACTIVITY

a. NAME	b. TELEPHONE (Include Area Code)
US ARMY ARDEC STANDARDIZATION OFFICE	(1) Commercial: (201) 724-6671 (2) AUTOVON: 880-6671
c. ADDRESS (Include Zip Code) ATTN: SMCAR-BAC-S PICATINNY ARSENAL, NJ 07806-5000	IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT: Defense Quality and Standardization Office 5203 Leesburg Pike, Suite 1403, Falls Church, VA 22041-3466 Telephone (703) 756-2340 AUTOVON 289-2340