

MIL-C-46487 (Ord)  
6 February 1961  
 Superseding  
 FA-PD-MI-1808 (Rev. 1)  
 dated 5 December 1956

MILITARY SPECIFICATION  
 FOR  
 CLEANING, PREPARATION AND ORGANIC COATING OF STEEL CARTRIDGE CASES

1. SCOPE

1.1 This specification covers the requirements for cleaning, surface preparation and organic coating for steel artillery ammunition cartridge cases, 37MM and larger and steel small arms ammunition cartridge cases, ammunition, through 30 millimeter.

1.2 The cartridge case finish system shall be one of the following types, as specified (see 6.1).

Type I Epoxide Modified Phenol Formaldehyde Varnish  
 Type II - Phenol Formaldehyde Varnish

2. APPLICABLE DOCUMENTS

2.1 The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

SPECIFICATIONS

Federal

O-A-115 - Acid - Sulphuric, Technical Grade  
 O-C-303 - Chromic Acid, Technical Grade  
 O-P-313 - Phosphoric Acid, Technical Grade  
 O-A-51 - Acetone, Technical  
 UU-T-101 - Tape, Gummed, Mending Reinforcing and Securing

Military

MIL-G-2550 - General Specification for Ammunition Except Small Arms Ammunition  
 MIL-V-12276 - Varnish, Phenolic Baking

THIS DOCUMENT CONTAINS 13 PAGES.

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## STANDARDS

### Federal

Federal Test Method Standard No. 151 Metals; Test Methods

(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.)

## 3. REQUIREMENTS

3.1 General. - Unless otherwise specified by the procuring activity or unless previous approval has been granted on continuing or supplemental contracts, details of the proposed procedure, equipment and chemicals to be used shall be forwarded in writing to Frankford Arsenal, Philadelphia 37, Pennsylvania, ATTN: ORDBA-6111 for review, comment and approval. The contractor shall not commence operations until such time as he has received written approval of the proposed methods, procedures and chemicals to be used. The exact designation of each material proposed for use shall be stated. The proposed procedure shall include a detailed method of control, including limits for time, temperature, pH values, method of application of the organic coating, method of reworking, repair and all other pertinent details which will insure compliance with the requirements of this specification.

3.2 Preproduction Sample. - Unless otherwise directed by the contracting officer, a preproduction sample shall be required.

3.3 Finish Systems. - Type I and Type II systems shall consist of a surface treatment followed by the application of an organic coating.

### 3.3.1 Surface Treatment.

3.3.1.1 Foreign Matter. - All surfaces of the cartridge case shall be thoroughly cleaned to remove all traces of grit, oil, wax, grease, rust or other foreign matter prior to the start of each process or treatment.

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3.3.1.2 Finish System Type I. - Unless otherwise specified by the contracting officer, the surface treatment for the Type I finish system shall consist of a chemical conversion treatment which will produce a continuous amorphous iron phosphate coating on the metal surfaces.

3.3.1.3 Finish System Type II. - Unless otherwise specified by the contracting officer, the surface treatment for the Type II finish system shall consist of either an acid etch treatment or a chemical conversion treatment which will produce a continuous amorphous iron phosphate coating on the metal surfaces.

3.3.1.4 Iron Phosphate Coating. - The iron phosphate treatment shall deposit a hard iridescent coating on the previously cleaned metal surfaces. The processing for the iron phosphate treatment shall consist of not less than 5 stages, as follows:

- a. Cleaning
- b. Water rinse
- c. Phosphating
- d. Water rinse
- e. Conditioning rinse

When pickling to remove rust and scale is necessary, the process for this treatment shall consist of not less than 7 stages as follows:

- a. Cleaning
- b. Water rinse
- c. Pickling
- d. Water rinse
- e. Phosphating
- f. Water rinse
- g. Conditioning rinse

3.3.1.4.1 Weight of Phosphate Coating. - The iron phosphate coating weight on the finished surface shall be 2.5 milligrams per square decimeter (mg/sq. dec) minimum and 9 mg/sq. dec maximum (25 mg per square foot minimum and 80 mg/sq. ft maximum).



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3.3.1.5 Acid Etch Treatment. - When an acid etch treatment is used for the Type II finish system, it shall leave the metal surface bare. The surface treatment shall consist of not less than five (5) stages as follows:

- a. Cleaning
- b. Water rinse
- c. Acid treatment
- d. Water rinse
- e. Conditioning rinse

3.3.1.5.1 When sulphuric acid which shall comply with the requirements of Federal Specification O-A-115 is used for rust removal, such treatment may be considered sufficient for the acid etch treatment, required in paragraph 3.3.1.5, stage C.

3.3.1.6 Conditioning Rinse. - The conditioning rinse shall be maintained at a pH value of from 2.0 to 5.0 by the addition of a sufficient amount of chromic acid which shall comply with the requirements of Federal Specification O-C-303 (approximately .08 ounces per gallon) or a mixture of phosphoric acid which shall comply with the requirements of Federal Specification O-P-313 and chromic acids (approximately .04 ounces of each per gallon).

3.3.1.7 Rinsing. - All water rinses shall employ clean running water in a spray unit or in a continuous overflowing tank. After rinsing, the cartridge case shall be free of harmful chemicals.

3.3.1.8 Drying. - The drying procedure shall be such that at the end of the drying period or cycle the temperature of the cartridge case shall be not less than 70 degrees fahrenheit (°F).

### 3.3.2 Organic Coating

3.3.2.1 Type I. - The organic coating for Type I finish system shall consist of an epoxide modified phenol formaldehyde varnish which shall comply with the requirements of Specification MIL-V-12276, Type III when cured at the baking schedule required by the contractor's process.

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3.3.2.2. Type II. - The organic coating for Type II finish system shall consist of a phenol formaldehyde varnish which shall comply with the requirements of Specification MIL-V-12276, Type I when cured at the baking schedule required by the contractor's process.

3.3.2.3 Coating Application. - The organic coating shall be applied to the cartridge case within 4 hours after surface treatment has been completed, preferably while cartridge case is still warm. The organic coating applied to the cartridge case shall produce an adherent film with no visible pinholes, craters, blisters or unevenness. Particular care shall be taken to obtain a smooth coating on roughened or machined surfaces.

3.3.2.4 Cured Coating. - After curing, the organic coating shall be smooth and continuous, completely covering the surface of the cartridge cases.

3.3.2.4.1 Thickness of Coating. - Unless otherwise specified, on the cartridge case drawing, the thickness of the organic protective coating shall be not less than 0.0003 inches and not greater than 0.001 inches.

3.3.2.4.2 Salt Spray. - For lot acceptance and approval of the preproduction sample, the cartridge cases with approved treatment and finish shall withstand a twenty four (24) hour salt spray exposure in accordance with Federal Test Method Standard No. 151 Metals; Test Methods 811 and shall be without any evidence of rust. However, rust on stamped letters on the head, within one thirty second ( $1/32$ ) inches of all other edges or on any surface not requiring coating shall be permitted.

3.3.2.4.3 Curing. - Type I and Type II finish systems after curing, shall not soften or loosen when subjected to the acetone curing test. The acetone shall comply with the requirements of Federal Specification O-A-51.

3.3.2.4.4 Flexibility. - Type I finish system, after curing, shall not crack or flake when subjected to the flexibility test.



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3.4 Rework or Repair. - When a lot of cartridge cases is rejected for noncompliance with the requirements of the specification, the coating shall be completely removed and the cases reworked with the type finish specified for this application. The recoated cases shall comply with 3.3.2. When repair to damaged or defective areas is made, the varnish shall be removed from the area to be repaired. The area shall be solvent cleaned prior to recoating. Type I or Type II finishes shall be repaired with the varnish or other materials specified for this application.

#### 4. QUALITY ASSURANCE PROVISIONS

4.1 General. - The quality assurance provisions in this section and other documents referenced in this specification are applicable to inspection performed by or for the Government. Inspection shall be in accordance with Specification MIL-G-2550, except as specified herein.

4.1.1 Contractor Inspection. - 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 requirement.

4.1.2 Contractor Quality Assurance System. - The contractor shall provide and maintain an adequate quality assurance system, acceptable to the Government covering the supplies under the contract. A current written description of the system shall be submitted to the contracting officer prior to the start of production. The written description will be considered acceptable when, as a minimum, it provides the quality assurance required by this specification and other applicable documents referenced in this specification. The contractor shall notify the Government of and obtain approval for any change to the written procedure that might affect the degree of assurance required by this specification or other applicable documents referenced herein.

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4.1.3 Government Verification. - All quality assurance operations performed by the contractor will be subject to Government verification at unscheduled intervals. Verification will consist of (a) surveillance of the operations to determine that practices, methods, and procedures of the written inspection plan are being properly applied, and (b) Government product inspection to measure quality of product offered for acceptance. Deviations from the prescribed or agreed upon procedures or instances of poor practices which might have a detrimental effect upon the quality of the product will be immediately called to the attention of the contractor. Failure of the contractor to promptly correct deficiencies discovered will be cause for suspension of acceptance until correction has been made or until conformance of product to prescribed criteria has been demonstrated. To avoid interference with operations, the contractor shall designate a responsible official or officials to whom the Government inspector will report such instances.

4.2 Preproduction Inspection. - Unless otherwise specified in the detail specification, 4 cartridge cases from the preproduction sample required by the detail specification shall be subjected to the test prescribed herein. The sample shall be processed in the same manner, using the same material and procedures as will be used in regular production and shall be obtained from the same source of supply. After inspection and provisional acceptance at source the preproduction sample shall be inspected for all requirements of this specification. The Government reserves the right to require new preproduction samples until such time as an acceptable sample is submitted.

4.3 Inspection Provisions. - Alternative inspection procedures and inspection equipment may be used by the contractor when such procedures and equipment provide, as a minimum, quality assurance required in the contractual documents. The inspection procedures or equipment specified in this specification will prevail in case of dispute.

4.3.1 Material. - Authenticated reports of objective tests performed on the material by the contractor shall be required to insure compliance with the chemical requirements. Reports shall include positive identification of the material, the applicable requirements, the specific tests performed and the results obtained.

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Numerical results of each specimen or sample inspected shall be included except that where numerical results are impossible an adjective description shall be used. Initial test reports shall accompany the preproduction sample. Prior to inspection the inspector shall determine from the information stated on test reports whether the material submitted complies with the requirements. Material which does not comply shall be rejected.

**4.3.2 Submittal of Product.** - The contractor shall submit the product in accordance with the requirements of the detail specification.

**4.3.3 Inspection Testing.** - The following tests shall be in accordance with the provisions of 4.4 except as provided in 4.3.

**4.3.3.1 pH Factor.** - The pH factor of the conditioning rinse shall be determined every 2 hours by either testing the surfaces of 2 cartridge cases as they come from the rinse or by checking the rinse itself. Failure of the rinse to comply with 3.3.1.6 shall be reported to the contractor. Failure on the part of the contractor to take necessary corrective action will be cause for suspension of inspection for acceptance until the corrective action has been taken.

**4.3.3.2 Weight of Phosphate Coating.** -

**4.3.3.2.1 Preproduction Sample.** - Two test panels shall be processed with the preproduction sample up to the completion of the phosphate coating operation. Failure of any of the test panels to comply with 3.3.1.4.1 shall be cause for rejection of the preproduction sample.

**4.3.3.2.2 Production Sample.** - Two test panels shall be processed with each 4 hours of production up to the completion of the phosphate coating operation. Failure of any test panels to comply with 3.3.1.4.1 shall be cause for rejection of all cartridge case processed since the last acceptable test panels for this test.

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#### 4.3.3.3 Thickness of Organic Coating.

4.3.3.3.1 Preproduction Sample. - Unless otherwise specified in the detailed specification, 4 cartridge cases from the preproduction sample shall be subjected to this test. Failure of any specimen to comply with 3.3.2.4.1 shall be cause for rejection of the preproduction sample.

4.3.3.3.2 Production Lot Sample. - Unless otherwise specified in the detailed specification, one cartridge case from each 1000 cartridge cases or fraction thereof shall be subjected to this test, except that in no instance shall there be less than 2 cartridge cases selected and tested for each 8 hours production. Failure of any specimen to comply with 3.3.2.4.1 shall be cause for rejection of the production represented by the failing sample.

#### 4.3.3.4 Salt Spray.

4.3.3.4.1 Preproduction Sample. - Unless otherwise specified by the detail specification, 2 cartridge cases from the preproduction sample shall be subjected to this test. Failure of any specimens to comply with 3.3.2.4.2 shall be cause for rejection of the preproduction sample.

4.3.3.4.2 Production Lot Sample. - One cartridge case from each 5000 cartridge cases produced, during the first 5 days of production, shall be subjected to this test, except that not less than 2 samples from each 8 hours daily production shall be selected and tested. Failure of any specimens to comply with 3.3.2.4.2 shall be cause for rejection of the lot. When the samples selected from the first 5 days production complies with 3.3.2.4.2, the sample size may be reduced to one cartridge case from each 10,000 cartridge cases or fraction thereof, but in no instance shall there be less than 2 cartridge cases per lot subjected to this test. Failure of any specimens to comply with 3.3.2.4.2 shall be cause for rejection of the lot. When on reduced sampling, rejection of a lot for non-compliance with 3.3.2.4.2 shall be cause for returning to the original method of sampling until 5 successive days production has been accepted, after which reduced sampling may again be instituted, until such time as another lot failure occurs.

4.3.3.5 Curing Test. - Unless otherwise specified in the detail specification, one cartridge case from the preproduction sample and one cartridge case from each 4 hours production or



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fraction thereof shall be subjected to this test. Failure of the specimen to comply with 3.3.2.4.3 shall be cause for rejection of the preproduction sample or the 4 hours production represented by the failing specimen.

#### 4.3.3.6 Flexibility Test.

4.3.3.6.1 Small Arms Ammunition Cartridge Cases, Preproduction Sample. - Unless otherwise specified in the detail specification, 2 cartridge cases or sections of 2 cartridge cases from the preproduction sample, shall be subjected to this test. Failure of any specimens to comply with 3.3.2.4.4 shall be cause for rejection of the lot.

4.3.3.6.1.1 Small Arms Ammunition Cartridge Cases, Production Sample. - Unless otherwise specified in the detail specification, 2 cartridge cases or sections of 2 cartridge cases, from each 8 hours production shall be subjected to this test. Failure of any specimens to comply with 3.3.2.4.4 shall be cause for rejection of the lot.

4.3.3.6.2 Artillery Ammunition Cartridge Cases, Production Sample. - Unless otherwise specified in the detail specification, 2 test panels 4 inches by 6 inches by 0.0125 inches, made from the same material as the cartridge case, or plain carbon cold rolled steel which have been processed with the lot of cartridge cases, shall be subjected to this test. Failure of any specimens to comply with 3.3.2.4.4 shall be cause for rejection of the lot.

4.3.3.6.2.1 Artillery Ammunition Cartridge Cases, Production Lot Sample. - Unless otherwise specified in the detail specification 2 test panels 4 inches by 6 inches by 0.0125 inches, made from the same material as the cartridge case, or plain carbon cold rolled steel, which have been processed with the lot of cartridge cases, shall be subjected to this test. Failure of any specimens to comply with 3.3.2.4.4 shall be cause for rejection of the lot.

4.3.4 Retest. - In the event of a lot failure in any test phase, at the request of the contractor, a retest will be permitted, using 6 cartridge cases from the failing lot. The retest will be confined to the phase or phases in which the original specimen failed. Failure of more than one specimen from the retest sample to comply with the applicable requirements shall be cause for rejection of the lot. No retest will be permitted on preproduction sample.

4.3.5 Inspection Equipment. - The examination and tests shall be made using equipment approved by the Government.

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#### 4.4 Test Methods and Procedures.

4.4.1 pH Factor. - The pH factor of the conditioning rinse shall be determined by the use of Universal pH papers or equivalent, by applying the paper to the side of the cartridge case immediately after removal from the rinse or to the rinse bath itself.

4.4.2 Weight of Phosphate Coating. - Two (2) test panels, 4 inches wide by 6 inches long, made from the same material as the cartridge cases shall be processed through all stages with the cartridge cases. When the panels have been processed, they shall be dried and accurately weighed, after which the coating shall be completely removed, by immersion in 5 percent chromic acid solution at 165 degrees fahrenheit plus or minus 2 degrees fahrenheit (°F) for 15 minutes. The test panels shall again be rinsed, dried and weighed and the surface of both sides calculated. Coating weight shall be determined from the following formulas.

$$\text{Coating Weight (mg/sq. dec.)} = \frac{\text{Initial weight in grams} - \text{final weight in grams}}{\text{Total Area in Sq. Cms.}} \times 100,000$$

$$\text{Coating Weight (mg/sq. ft.)} = \frac{\text{Initial weight in grams} - \text{final weight in grams}}{\text{Total Area in Sq. Inches}} \times 144,000$$

The chromic acid used in this test shall conform to the requirements of Specification O-C-303. The solution shall be used one time only.

4.4.3 Thickness of Organic Coating. - The thickness of the protective organic coating shall be tested using gaging equipment designed for this purpose, using the method prescribed by the manufacturer of the equipment. (See 6.5)

4.4.4 Salt Spray. - The salt spray test shall be performed using a 20 percent sodium chloride solution in accordance with Method 811. In performing this test the equipment and criteria prescribed by Federal Test Method Standard No. 151 shall be utilized.

4.4.5 Gurink Test. - An area of at least one (1) inch square on the sample cartridge case shall be kept wet with acetone, complying

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with Federal Specification O-A-51, for two (2) minutes. The area being tested shall be vigorously rubbed with the thumb or the hand and visually inspected for evidence of lifting, blistering or softening.

#### 4.4.6 Flexibility Test.

4.4.6.1 Small Arms Ammunition Cartridge Cases. - The cartridge cases or sections thereof shall be held in a vice and bent 180 degrees, care shall be taken so as not to damage the organic coating at the bend with the creasing tool. A strip of transparent pressure sensitive tape conforming to the requirements of Federal Specification UU-T-101 shall be pressed firmly over the bend area, and then stripped off. Upon removal, the tape shall be visually examined for presence of organic coating adhering thereto.

4.4.6.2 Artillery Ammunition Cartridge Cases. - The sections removed from the cartridge cases of the preproduction sample and the coated test panels representing each production lot shall be tested by bending the sample 180 degrees rapidly around a mandrel 0.125 inches plus or minus 0.01 inches in diameter, after which the coating of the bend area shall be visually examined for cracks. A strip of transparent pressure sensitive tape conforming to Federal Specification UU-T-101 shall be pressed firmly over the bend area, and then stripped off. Upon removal, the tape shall be visually examined for presence of organic coating adhering thereto.

### 5. PREPARATION FOR DELIVERY

5.1 Preparation for delivery not applicable.

### 6. NOTES

6.1 Intended Use. - Type I coating should be used on all cartridge cases 20MM and smaller and on all other sizes when the steel may be subjected to deformation, after the application of the coating. Phenol formaldehyde varnish which has been modified by the addition of an epoxide resin has increased flexibility permitting deformation of a coated steel item without damage to the coating. Type II coating should be used where there is no further deformation of the metal after the application of the coating.

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6.2 Color. - Slight variation in color of the coated surface due to chromic acid rinse shall not be cause for rejection.

6.3 Rinsing. - Adequate rinsing is very important in the cleaning procedure. When the water film remaining on the case after the conditioning rinse maintains its continuity (is free from water break) the surface may be considered free from oil and grease.

6.4 Optimum results are obtained with varnish thickness between 0.0004 inches and 0.0006 inches.

6.5 For testing thickness of the organic coating the following instruments are recommended.

Amico - Brenner Magne Gages - American Instrument Co.,  
Silver Springs, Md.  
Elcometer - Distributed by Henry H. Gardner Laboratory, Inc.  
4723 Elm St. Bethesda 14, Maryland.  
G.E. Gage - General Electric Co., Schenectady, N. Y.  
Lea Gage - Lea Manufacturing Co., Waterbury, Conn.  
Krouse, Portable Magnifying Micrometer - McNary Engineering  
Service Springfield, Ohio

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**CUSTODIAN:**

Army - Ordnance Corps

**PREPARING ACTIVITY:**

Army - Ordnance Corps

13.



MIL-V-12276D(MR)  
AMENDMENT-1  
1 August 1973

MILITARY SPECIFICATION

VARNISH, PHENOLIC, BAKING

This amendment forms a part of Military Specification  
MIL-V-12276D(MR) dated 4 January 1971.

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Paragraph 4.4.7, corrosion resistance, line 7: Delete fourth sentence and substitute "Expose the panels for 250 hours to 20 percent salt spray in accordance with method 6061 of Fed. Test Method Std. No. 141."

Custodian:  
Army - MR

Preparing activity:  
Army - MR

Review activities:  
Army - MR, MU, MD

Project No. 8010-A087

FSC 8010

THIS DOCUMENT CONTAINS 1 PAGES.

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MIL-V-12276D(MR)  
4 January 1971  
SUPERSEDING  
MIL-C-12276C(MR)  
8 September 1965

MILITARY SPECIFICATION  
VARNISH, PHENOLIC, BAKING

1. SCOPE

1.1 Scope. This specification covers three types of heat hardening phenolic varnishes suitable for use as a coating for cartridge cases and as a lining for munitions and other containers. It provides for an additional composition suitable for use under Air Pollution Regulations.

1.2 Classification. The varnish covered by this specification shall be of the following compositions, types, and classes as specified (see 6.2 and 6.6).

Compositions

Composition G - General use  
Composition L - Limited use (see 6.6).

Type I - For use on cartridge cases

Class A - For spray applications.  
Class B - For dip applications.

Type II - For use in lining munitions and chemical containers.

Type III - For use on cartridge cases under special conditions (see 6.1).

Class A - For spray applications  
Class B - For dip applications  
Class C - For roller, curtain or similar coating applications

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## MIL-V-12276D(MR)

## 2. APPLICABLE DOCUMENTS

2.1 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:

## SPECIFICATIONS

## FEDERAL

- L-T-90 - Tape, Pressure-Sensitive, Adhesive (Cellophane and Cellulose Acetate)
- O-C-303 - Chromium Trioxide, Technical
- O-O-670 - Orthophosphoric (Phosphoric) Acid, Technical
- P-S-651 - Sodium Orthosilicate, Technical
- TT-P-143 - Paint, Varnish, Lacquer, and Related Materials; Packaging, Packing, and Marking of

## MILITARY

- MIL-C-10578 - Corrosion Removing and Metal Conditioning Compound (Phosphoric Acid Base)

## STANDARDS

## FEDERAL

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

## 3. REQUIREMENTS

3.1 Materials.

3.1.1 Type I varnish shall be based on a straight thermosetting phenol-formaldehyde varnish free from rosin or rosin derivatives, but may include the use of other materials to improve the leveling, flexibility and adhesion of the coating.

3.1.2 Type II varnish shall be a straight thermosetting phenol-formaldehyde varnish free from added plasticizers, rosin or rosin derivatives.

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3.1.3 Type III varnish shall be based on a mixture of bisphenol epoxide and phenol-formaldehyde resins. It may include other materials to improve the leveling of coating.

### 3.2 Solvents.

3.2.1 Thinner. A thinner shall be supplied with the varnish so that the varnish can be reduced to the proper usable viscosity prior to application. The varnish and thinner delivered as a lot shall be furnished in quantities proportional to the ratio recommended by the manufacturer to produce the specified film thickness after baking.

3.2.1.1 Type I and II varnish, when diluted with supplied thinner to a viscosity of 30 centipoises at  $75^{\circ} \pm 2^{\circ}\text{F}$ , shall have a minimum solids content of 29 percent.

3.2.1.2 Type III varnish, when diluted with supplied thinner to a viscosity of 30 centipoises at  $75^{\circ} \pm 2^{\circ}\text{F}$ , shall have a minimum solids content of 24 percent.

3.2.2 Compositions G and L. The solvent, used with the resin and the solvent supplied as the thinner, shall be of such a nature that they will not constitute an undue hazard to personnel in application of the varnish. Evidence to this effect shall be subject to review by departmental medical authority.

3.2.3 Composition L. The solvent used with the resin and the solvent supplied as the thinner shall meet the requirements listed below, when tested as described in 4.4.5.

- (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.
- (d) Ethyl benzene and ketones: 20 percent maximum
- (e) Total of a + b + d: 20 percent maximum

### 3.3 Color.

3.3.1 Types I and III. Types I and III materials shall be clear or the natural color of the varnish. There shall be no pigment added. The use of soluble organic dyes is permissible.

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3.3.2 Type II. No color requirements applicable. Material may be clear or pigmented.

3.4 Solids content. The minimum solids content for types I and III varnish, as received, and then tested in accordance with 4.4.4, shall be 35 percent.

3.5 Absence of film defects. The varnish shall produce a smooth, continuous film with no visible pinholes, craters, or blisters when tested as specified in 4.4.2.

3.6 Film adhesion and flexibility. The dried film on metal shall be tough, elastic and shall not chip, flake, scale, or show poor adhesion to SAE 1010 steel panels when tested as specified in 4.4.3.

3.7 Resistance to chemicals and solvents. The varnish when applied to steel panels as specified in 4.4.1.1 and baked as prescribed in each of the three schedules listed in 4.4.1.2 shall not be softened or otherwise deteriorated when immersed in toluene, glacial acetic acid, ammonium hydroxide, ethyl alcohol or ether and tested as specified in 4.4.6. In addition, type II varnish shall not be softened, blistered, or otherwise deteriorated by boiling bis-(beta chloroethyl) ether when tested as specified in 4.4.6.

3.8 Corrosion resistance. There shall be no rusting of the panels, nor peeling or undercutting of the coating when subjected to salt spray test for 250 hours as specified in 4.4.7.

3.9 Abrasion resistance.

3.9.1 Types I and III materials. When tested as specified in 4.4.8, the maximum weight of coating removed by 500 cycles shall not exceed 8 milligrams and the coating shall not be abraded through to the metal.

3.9.2 Type II material. The diameter of the area abraded through to the steel when tested as specified in 4.4.8 shall not exceed 4 millimeters.

3.10 Impact resistance. Panels coated with type III varnish shall not flake, crack or lift when tested as specified in 4.4.9. This requirement does not apply to types I and II.

3.11 Thermoplasticity. The finish shall not soften or stick when tested at 600 F as specified in 4.4.10.

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3.12 Viscosity. Types I and III varnishes shall have a minimum viscosity of 40 centipoises at  $75^{\circ} \pm 2^{\circ}$ F as received. The viscosity shall be determined as specified in 4.4.11.

3.13 Shelf life. When tested as specified in 4.4.12, there shall be no more than a 15 percent change in viscosity after 90 hours at  $140^{\circ}$ F nor separation of any component.

3.14 Workmanship. The ingredients of the varnish shall be thoroughly mixed to form a homogeneous product free of visual dirt, grit, water, or other foreign materials. The unpigmented varnish shall have no sediment or solid particles.

#### 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 supplies and services conform to specified requirements.

4.2 Sampling, inspection and testing. Unless otherwise specified in the contract or order, 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 and shall consist of tests for all requirements specified herein.

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4.4 Test procedures.

4.4.1 Preparation of test panels. Test panels of cold rolled SAE 1010 steel of the sizes required for the applicable test shall be cleaned and pretreated in accordance with the following schedule:

- (a) Degrease in trichloroethylene at room temperature.
- (b) Dip for 5 minutes in alkaline cleaner solution (4 to 6 oz/gal.) at temperature of 190° F min. The alkaline cleaner shall comply with P-S-651, type II, to which shall be added 3.5 percent min. by weight of Nacconal NR or equal (see 6.5).
- (c) Rinse in water at temperature of 160°-180° F.
- (d) Dip in 20 percent by weight phosphoric acid complying with MIL-C-10578, type V, for 1 minute at 170°-190° F.
- (e) Rinse in water 160°-180° F.
- (f) Dip in solution maintained at a pH value of from 2.0 to 5.0 by the addition of a sufficient amount of chromic acid which shall comply with the requirements of O-C-303 (approximately .08 ounces per gallon) or a mixture of phosphoric acid which shall comply with the requirements of O-O-670 and chromic acid (approximately .04 ounces of each per gallon).

4.4.1.1 Film thickness. The types I and III coatings shall be applied by spray, dip, or other methods according to class in such a manner that the thickness of the film after baking shall be not less than 0.0004 inch nor more than 0.0006 inch, except as specified in 4.4.2. Type II coatings shall be applied by either dip, spray, or other methods in such a manner that the thickness of the film after baking shall be not less than 0.0004 inch nor more than 0.0006 inch for clear coatings and not less than 0.0010 inch nor more than 0.0015 inch for pigmented films. The panels shall be coated in an atmosphere as free as possible from dirt, dust or laboratory fumes.

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4.4.1.2 Three sets of coated panels shall be prepared and baked in accordance with the following schedules:

- (a) For the length of time and the temperature normally recommended by the finish manufacturer. (All tests).
- (b) For the same length of time but at a temperature of 25 degrees Fahrenheit lower than (a) above. (All tests except 4.4.2 and 4.4.9).
- (c) For the same length of time but at a temperature 25 degrees Fahrenheit higher than (a) above. (All tests except 4.4.2 and 4.4.9).

4.4.2 Absence of film defects. Two test panels, 4 by 6 by .025 inches in size, prepared as specified in 4.4.1, shall be spray coated or dipcoated to a dry film thickness of 0.0005 plus or minus 0.0001 inch for types I and III materials. The thickness of type II coatings shall be in accordance with 4.4.1.1. The panels shall be prebaked at  $212 \pm 4^{\circ}\text{F}$  for 20 minutes and then baked according to the manufacturer's recommended baking schedule. The panels shall be examined visually for defects specified in 3.5.

4.4.3 Film adhesion and flexibility. Test panels, 4 by 6 by 0.0125 inches in size, shall be prepared and coated as specified in 4.4.1 and 4.4.1.1. A minimum of two coated panels for each of the three schedules specified in 4.4.1.2 shall be tested for film adhesion and flexibility by rapidly bending each coated panel around a mandrel 0.50 plus or minus 0.01 inch in diameter for type I material, 1.00 plus or minus 0.01 inch in diameter for type II material and 0.125 plus or minus 0.005 inch in diameter for type III material. The film shall be examined for compliance with 3.6. A strip of transparent, pressure-sensitive, adhesive cellophane tape conforming to L-T-90 shall then be pressed down on the bend in the coated panel and stripped off. None of the phenolic coating shall be lifted off by the adhesive tape.

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4.4.4 Solids content. Weigh to the nearest milligram a small disposable aluminum dish approximately 2 inches in diameter, that has been provided with a tared aluminum cover. (These aluminum dishes may be obtained from Fisher Scientific Co., catalog number 8-732). Weigh into the dish a very small sample of the varnish that does not exceed 0.3 gram in weight. Dissolve in 2 ml. of reagent grade toluene and dry the dish for 30 minutes in a gravity convection oven at  $105^{\circ} \pm 2^{\circ}\text{C}$ . Determinations shall be run in duplicate. Upon cooling, reweigh to the nearest 0.1 milligram and calculate the percent total solids:

$$\text{Percent solids} = \frac{\text{weight of residue}}{\text{weight of sample}} \times 100$$

4.4.5 Solvent analysis for composition L.

4.4.5.1 Separation of volatile portion. Pour about 15 grams of the varnish into a 50 ml. 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. The receiver consists of a test tube (20 x 150mm) 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  $160^{\circ}\text{C}$ . Raise the oil bath until the oil reaches the sample level. Reduce the pressure slowly to 10 mm of mercury. After all the 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.

4.4.5.2 Determination of aromatic hydrocarbons.

4.4.5.2.1 Qualitative test for aromatic hydrocarbons. Place 5 ml. of the distillate in a 10 ml. glass stoppered graduate. Add 5 ml. of 86 percent sulfuric acid slowly while the graduate is being cooled with tap water. After the acid has been added, shake vigorously for 2 minutes, then allow the layers to separate. If there is no separation of layers, it can be assumed that no aromatic hydrocarbons are present. If there is an upper layer, determine the aromatic hydrocarbons quantitatively as described in 4.4.5.2.2.

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4.4.5.2.2 Quantitative 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 percent 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	70

4.4.5.2.2.1 Aromatic and oxygenated solvents - procedure A. Install the 6-ft. column and follow the operating conditions described above. Inject about 3 micro-liters of the isolated distillate and scan the chromatogram. The aliphatic solvents will emerge within 1 minute and the complete chromatogram should develop in about 5 minutes. From the position of the peaks observed on the chromatogram, select an internal standard that will be free of interference, such as cyclopentanol or cyclohexanol. Add 0.6 ml. of internal standard to 3 ml. of the distillate, analyze according to the above procedure. Peaks emerging after 1 minute are aromatic solvents along with any oxygenated solvents that may be present. Calculate the percent of aromatic and oxygenated solvents as follows:

$$\text{Percent aromatic and oxygenated solvents, } \frac{v}{v} = \frac{20* \times A}{1.02** \times B}$$

where A = area of aromatic and oxygenated solvents.

B = area of internal standard

\* - is percent of internal standard added

\*\* - is correction factor if cyclopentanol is used. If another internal standard is used, calibrate to determine the correction factor.

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

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4.4.5.2.2 Total aromatic content - procedure B. Proceed as in the qualitative test for aromatic hydrocarbons (see 4.4.5.2.1). Remove as much of the top layer as possible and wash it with distilled water. Carefully pipet 3 ml. of the washed solvent into a small flask followed by 0.6 ml. of the internal standard. Mix and analyze according to procedure A. Calculate the percent of aromatics after acid treatment in the same manner as in procedure A and the percent of total aromatic solvents as follows:

$$\text{Percent total aromatic solvents, v/v} = \frac{B \times (100 - A)}{100 - B}$$

where A = percent of aromatic and oxygenated solvents from procedure A  
B = percent of aromatic solvents after acid treatment

NOTE: If the total aromatic content of the solvent is between 8 percent and 20 percent, continue with the following procedure.

4.4.5.2.2.3 Toluene and ethyl benzene - procedure C. Install the 18-ft. column and follow the operating conditions described for that column. Add 0.3 ml. of high purity benzene to the 3 ml. sample used in procedure A. If the results of procedures A and B indicated the presence of oxygenated solvents, treat this sample with 85 percent sulfuric acid (use 3 ml. acid) as described in procedure B. Inject about 3 micro-liters of sample and allow the chromatograph to develop until all 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 70°C. Calculate the percent of toluene and ethylbenzene as follows:

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

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

where \* = the correction factor for the detector response.  
\*\* = 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.

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**4.4.5.3 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 decoloration 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 3.2.3 and 6.7).

**4.4.5.4 Test for ketones.**

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

**4.4.5.4.2 Procedure.** Pipette 1 ml. of reagent into a 20 by 170 mm test tube. Add 10 drops of distillate (see 4.4.5.1) and shake for 30 seconds. A yellow precipitate or cloud in the reagent layer indicates the presence of ketones. Run a blank using one milliliter of reagent and 10 drops of mineral spirits.

**4.4.6 Resistance to chemicals and solvents.** Test panels, 1 by 2-1/2 by 0.025 inches in size, shall be prepared and coated as specified in 4.4.1 and 4.4.1.1. A minimum of one coated panel for each of the three schedules specified in 4.4.1.2 shall be tested separately for resistance to the following chemicals and solvents by immersion. A separate panel shall be used for each individual test.

Boiling toluene	1 hour
Glacial acetic acid	1 hour at room temperature
Ammonium hydroxide (10 percent by weight)	1 hour at room temperature
Ethyl alcohol	1 hour at room temperature
Ethyl ether	1 hour at room temperature
Boiling bis-(beta chloroethyl) ether	2 hours (type II varnish only)

Coated panels which are to be immersed in acetic acid and ammonium hydroxide shall have their edges protected by dipping in molten paraffin prior to immersion. The paraffin shall not extend more than 1/8 inch onto the coated area of the panels.

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At the end of the specified test period, the film of the varnish shall not have softened, blistered or otherwise deteriorated. (In the immersion in boiling bis-(beta chloroethyl) ether, moderate discoloration shall not be considered as deterioration.)

**4.4.7 Corrosion resistance.** Test panels, 4 by 6 by 0.025 inches in size, shall be prepared and coated as specified in 4.4.1 and 4.4.1.1. A minimum of two coated panels for each of the three baking schedules specified in 4.4.1.2 shall be tested for corrosion resistance by scribing each panel along the two diagonals for a distance of 5 inches using a razor blade. The scribe marks shall completely penetrate the varnish film. Expose the panels for 48 hours to 5 percent salt spray in accordance with method 6061 of Fed. Test Method Std. No. 141. At the end of the test period, examine the panels visually for evidence of peeling or undercutting of the coating at the scribe marks or signs of rusting at points other than within 1/8 inch distance from the scribe marks or edges of the panels.

**4.4.8 Abrasion resistance.**

**4.4.8.1 Types I and III.** A minimum of two panels, 4 by 4 by .025 inches in size, for each of the three baking schedules specified in 4.4.1.2 shall be used. The panels shall be prepared and coated as specified in 4.4.1 and 4.4.1.1. The test shall be conducted in accordance with method 6192 of Fed. Test Method Std. No. 141 except that CS-10 calibrase wheels with 1000 gram load shall be used.

**4.4.8.2 Type II.** A minimum of two panels, 4 by 6 by .025 inches in size, for each of the three baking schedules specified in 4.4.1.2 shall be used. The panels shall be prepared and coated as specified in 4.4.1 and 4.4.1.1. The test shall be conducted in accordance with method 6191 of Fed. Test Method Std. No. 141 except that 5 liters of sand shall be used.

**4.4.9 Impact resistance (type III only).** Two test panels, 4 by 6 inches by 0.025 inch in size, shall be prepared and coated as specified in 4.4.1 and 4.4.1.1 and baked in accordance with 4.4.1.2(a). The coated panels shall be tested for impact resistance with an impact tester device similar to the Parlin-duPont or the Gardner Impact Tester (see 6.4). The impacting tool shall be of hard steel and shall have a rounded end with a 1/4 inch radius. The anvil shall have a 5/8 inch diameter hole placed so that the

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impacting tool will strike exactly in the center of the hole. The coated panel shall be placed over the anvil hole with the test surface facing downward or away from the impacting tool. A 30 inch pound impact shall be made on the panel. The film shall be examined on the convex surface for compliance with 3.10. A strip of transparent, pressure-sensitive adhesive cellophane tape conforming to L-T-90 shall be pressed down on the convex area of the impacted surface and then stripped off. None of the coating shall be lifted by the adhesive tape.

**4.4.10 Thermoplasticity.** Two test panels, free from defects, 4 by 6 by 0.025 inches in size, and two panels 1 by 2-1/2 by 0.025 inches in size for each baking schedule specified in 4.4.1.2, shall be used. Panels shall be prepared and coated as specified in 4.4.1 and 4.4.1.1. The edges of the 1 by 2-1/2 inch coated panels shall be broken down to the bare metal so that all burrs and varnish beads are completely removed. The test panels shall be placed film to film in an oven at  $600^{\circ} \pm 10^{\circ}\text{F}$ . These panels, with the smaller panel uppermost in each case, shall rest on a smooth flat steel plate previously brought to oven temperature. The steel plate shall be a minimum of 4 by 6 by 1/4 inches in size. To assure that the weights and steel plates have been brought to oven temperature, they shall be placed in the oven maintained at  $600^{\circ} \pm 10^{\circ}\text{F}$  for one hour prior to use. As soon as the test panels and plates have been placed in the oven, a 2-kilogram weight, previously brought to oven temperature, shall be placed on each of the smaller panels. Keep the panels in oven maintained at  $600^{\circ} \pm 10^{\circ}\text{F}$  for 15 minutes. Remove the weights from the panels and immediately reverse the position of the panels so that the larger panels are uppermost. The smaller panels shall immediately fall away from the larger panels.

**4.4.11 Viscosity.** The viscosity shall be determined by the Brookfield Viscosimeter, Model LVF, using the No. 1 spindle at 30 RPM at a temperature of  $75^{\circ} \pm 2^{\circ}\text{F}$ , or similar method.

**4.4.12 Shelf life.** A tall, wide mouth 8 oz. bottle of a kind which will pass the guard of a Brookfield Viscosimeter shall be filled with the varnish to within 1/8 inch of the top. Record the viscosity, then place the covered bottle in the oven at  $140^{\circ} \pm 3^{\circ}\text{F}$  for 90 hours. Remove bottle, allow to cool and again determine the viscosity.

$$\text{Percent change in viscosity} = \frac{(V_2 - V_1) 100}{V_1}$$

where  $V_1$  = original viscosity  
 $V_2$  = viscosity after 90 hours at  $140^{\circ}\text{F}$

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4.5 Preparation for shipment. Examination of the packing and marking for shipment shall be made for conformance to the requirements of section 5.

4.6 Rejection.

4.6.1 Test failures. A lot shall be rejected for failure to meet any of the test requirements when tested in accordance with 4.4.

5. PREPARATION FOR DELIVERY

5.1 Packaging, packing and marking shall be in accordance with TT-P-143. In addition, all containers shall be marked with (1) type and class of varnish, (2) batch number, (3) instructions as to thinning and viscosity for application, (4) baking cycles, and (5) date of manufacture.

6. NOTES

6.1 Intended use. Types I and III varnishes covered by this specification are intended for use as finishes on steel cartridge cases to protect them from atmospheric corrosion. Classes A and B of type I and classes A, B and C of type III may be used depending upon the type of equipment used to produce the finish on the cartridge cases. Type I is intended for use where the metal is not deformed or fabricated after coating. Type III is intended for use where the metal is deformed or fabricated after it is coated. Type II varnish covered by this specification is intended for use as a protective finish for lining munitions, chemical and other containers requiring special protection.

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) Type and class required (see 1.2).
- (c) Size containers (see 5.1).
- (d) Level of packaging and packing required (see 5.1).
- (e) The varnish should be purchased by volume, the unit being one U. S. liquid gallon of 231 cubic inches at 68°F (20°C).

6.3 The requirements for packaging and for packing and marking for shipment (see 5.1) specified herein apply to direct shipment to Government activities and apply also, where specified, to contracts or orders between the manufacturer and the Government prime contractor.

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6.4 Recommended impacting devices.

Gardner-Variable Impact Tester or  
Parlin-duPont Impact Tester

(References: Physical & Chemical, Examination of Paints, Varnishes,  
Lacquers, and Colors, Henry A. Gardner and G. G. Sward,  
Twelfth Ed. (1962) p. 146.

6.5 Nacconal NR, Sodium alkyl aryl sulfonate, is manufactured by National Aniline Div., Allied Chemical Corp., New York, N. Y. (see 4.4.1).

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

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

Custodian:  
Army - MR

Preparing activity:  
Army - MR

Review activities:  
Army - MR, MU, MD

Project No. 8010-A002

User activities:  
Army - MU

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