

INCH-POUND

MIL-DTL-450D

27 January 2011

SUPERSEDING

MIL-C-450C

5 February 1971

DETAIL SPECIFICATION
COATING COMPOUND, BITUMINOUS SOLVENT TYPE,
BLACK (FOR AMMUNITION)

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers an asphalt compound for coating the surfaces of ammunition cavities prior to filling with explosives. It provides for an additional composition of material suitable for use in areas with specific air pollution regulations (see 6.1 and 6.4).

1.2 Classification. The coating covered by this specification will be of the following compositions and types, as specified (see 6.2).

Composition G - General use (all types)

Type I - Low solids (for spray application)

Type II - Medium solids (for spray or brush application)

Type III - Heavy paste

Composition L - Limited use (all types)

Type I - Low solids (for spray application)

Type II - Medium solids (for spray or brush application)

Type III - Heavy paste

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AMSC N/A

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2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3, 4, or 5 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3, 4, or 5 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

DEPARTMENT OF DEFENSE STANDARDS

MIL-STD-286 Propellants, Solid: Sampling, Examination and Testing

MIL-STD-650 Explosive: Sampling, Inspection and Testing

FEDERAL SPECIFICATIONS

TT-N-95 Naphtha; Aliphatic

FEDERAL STANDARDS

FED-STD-141 Paint, Varnish, Lacquer and Related Materials; Methods of Inspection, Sampling, and Testing

(Copies of these documents are available online at <https://assist.daps.dla.mil/quicksearch/> or from the Standardization Documents Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

INTERNATIONAL STANDARDIZATION AGREEMENTS

NORTH ATLANTIC TREATY ORGANIZATION

STANAG 4556 Explosives: Vacuum Stability Tests

(Copies of this document is available from NATO/MAS, Bvd Leopold 111, 1110 Brussels, BE online at <https://assist.daps.dla.mil/quicksearch/> or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

2.3 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents are those cited in the solicitation or contract.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM A 623	Standard Specification for Tin Mill Products, General Requirements
ASTM D 95	Standard Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
ASTM D 217	Standard Test Methods for Cone Penetration of Lubricating Grease
ASTM D 244	Standard Test Methods and Practices for Emulsified Asphalts
ASTM D 609	Standard Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products
ASTM D 1200	Standard Test Method for Viscosity by Ford Viscosity Cup
ASTM D 1308	Standard Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes
ASTM D 2369	Standard Test Method for Volatile Content of Coatings
ASTM D 3925	Standard Practice for Sampling Liquid Paints and Related Pigmented Coatings

(Copies of these documents are available online at <http://www.astm.org> or from the American Society for Testing and Materials Customer Service, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

2.4 Order of precedence. Unless otherwise noted herein or in the contract, in the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 First article. When specified (see 6.2), a sample (see 6.3) shall be subjected to first article inspection in accordance with 4.2.

3.2 Color. The color shall be black at a dry film thickness producing complete hiding and varying shades of brown in thinner film as verified by visual inspection.

3.3 Composition. The coating compound shall consist of one or more grades of natural or petroleum asphalts in a solvent and shall contain no drying oils, resins, or pigments.

3.3.1 Solvent.

3.3.1.1 Composition G. The solvent used in the formulation of the coating compound shall be a low boiling, fast evaporating aliphatic naphtha, TT-N-95.

3.3.1.2 Composition L. The solvent used in the formulation of the coating compound shall be the same as 3.3.1.1, except that it shall conform to the requirements of Table I when tested as specified in 4.4.19.

Table I. Composition L solvent requirements.

Material ¹	Maximum allowable percent by volume
(a) Solvents (hydrocarbons, alcohols, etc.) having an olefinic or cycloolefinic type of unsaturation	Negative test (less than 1%)
(b) A combination of any aromatic hydrocarbons having eight or more carbon atoms per molecule (except ethylbenzene)	8
(c) Ethylbenzene and toluene	20
(d) Total (b) and (c) above	20

¹The test (a) for olefinic and cycloolefinic compounds will be negative for solvents containing less than 1% of these compounds.

3.4 Quantitative requirements. When tested as specified in 4.4, the coating compound shall conform to the quantitative requirements of Table II.

Table II. Quantitative requirements.

Requirements	Minimum	Maximum	Test Method
Total solids, percent by weight of compound:			4.4.2
Type I	36	40	
Type II	45	55	
Type III	79	85	
Water, percent by weight of compound	-	0.5	4.4.3
Mineral matter, percent by weight of compound	-	1	4.4.4
Manganese, percent by weight of compound	-	0.05	4.4.5
Lead, percent by weight of compound	-	0.02	4.4.6
Insoluble in carbon disulfide, percent by weight of compound	-	0.5	4.4.7
Acidity (as sulfuric acid), percent by weight of compound	-	0.01	4.4.8
Alkalinity (as sodium hydroxide), percent by weight of compound	-	0.01	4.4.8
Viscosity:			4.4.9
Type I - No. 4 Ford cup, seconds	15	28	
Type II - No. 4 Ford cup, seconds	120	190	
Type III - Penetration test value, tenths of mm (see 4.4.9)	150	250	
Drying time:			4.4.10
Dust free			
Type I, minutes	-	5	
Type II, minutes	-	5	
Type III, hours	-	8	
Free from after-tack			
Type I, hours	-	0.5	
Type II, hours	-	1	
Type III, hours	-	24	

3.5 Qualitative requirements.

3.5.1 Storage properties. When tested as specified in 4.4.11, the coating compound shall show no livering, thickening, or settling.

3.5.2 Dilution stability (Type II only). When tested as specified in 4.4.12, the coating compound shall remain stable and show no evidence of precipitation.

3.5.3 Brushing properties (Type II only). When tested as specified in 4.4.13, the coating compound, as packaged, shall be capable of being brushed out to a smooth film, free from discontinuities or other defects.

3.5.4 Spraying properties, appearance when dry (Types I and II only). When tested as specified in 4.4.14, the coating compound shall have satisfactory spraying properties in every respect. The dry film shall present a smooth, glossy appearance, free from irregularities and rough particles.

3.5.5 Water resistance. A film of the coating compound prepared and tested as specified in 4.4.15 shall withstand immersion in distilled water without blistering, whitening, softening, or no more than a slight dulling.

3.5.6 Acid resistance. A film of coating compound prepared and tested as specified in 4.4.16 shall withstand the action of picric, sulfuric, nitric, and hydrochloric acids without disintegration, browning, or dulling. There shall be no etching of the metal underneath the coating compound.

3.5.7 Heat resistance. A film of coating compound prepared and tested as specified in 4.4.17 shall not sag or flow.

3.5.8 Flexibility. A film of coating compound prepared and tested as specified in 4.4.18 shall be tough, elastic, and shall withstand bending without cracking or flaking.

3.6 First article only requirements.

3.6.1 Reactivity. When subjected to the vacuum stability test as specified in 4.4.20, the reactivity of the compound with the following explosives shall not exceed 2.0 milliliters (mL) of gas over and above that generated by the controls:

- a. Composition A-3
- b. Composition B-type explosive (1)
Either Composition B-4, Composition B, or Cyclotol
- c. Composition C-4
- d. Octol
- e. TNT or Tritonal (1)
- f. HBX-type explosive (1)
Either HBX-1, HBX-3, or H-6

3.6.2 Ignition. When tested as specified in 4.4.21, mixtures of the dried compound and the explosives listed in Table III shall give ignition temperatures for consecutive tests at or above those shown.

Table III. Ignition temperature.

Explosives	Minimum
Composition B	175 °C
TNT	200 °C
Composition A-3	190 °C

3.7 Workmanship. The asphaltic compound ingredients shall be processed in a manner that will produce the high quality material necessary to meet the requirements of this specification. The finished product shall be homogeneous and free from a foam-like texture on its surface or other defects that could adversely affect its intended use.

4. VERIFICATION

4.1 Classification of inspections. The inspection requirements specified herein are classified as follows:

- a. First article inspection (see 4.2)
- b. Conformance inspection (see 4.3).

4.2 First article inspection. The first article inspection shall consist of all tests of this specification as described in 4.4. The first article sample shall be examined and tested for all requirements of this specification. The procuring activity reserves the right to accept the manufacturer's certification of testing, when available, in lieu of Government testing. Unless otherwise specified in the contract, a first article inspection shall be required if:

- a. There has been more than 12 months since production of the latest accepted lot
- b. There has been a relocation of the production facility
- c. There has been a major change in personnel or procedures
- d. There has been a change in the source of supplies for the materials used in the manufacture of the coating compound.

4.2.1 First article sample. When specified, a test report from the manufacturer or a commercial laboratory shall be forwarded to the procuring activity before the first article sample (see 6.3) is tested showing the following:

- a. Formula number of the compound
- b. Formulation
- c. Composition of the coating compound including:
 - (1) The identification of ingredient samples by specific chemical name and trade name
 - (2) Laboratory data showing complete test results required by this specification except reactivity and ignition.

4.2.1.1 Sample identification. The first article samples shall consist of four 1-quart samples of the compound, selected as required in accordance with ASTM D 3925. The samples shall be forwarded to the agency responsible for first article testing as designated in the solicitation or contract (see 6.3). The samples shall be plainly identified by securely attached durable tags or labels marked with the following information:

- a. Sample for first article inspection
- b. COATING COMPOUND, BITUMINOUS SOLVENT TYPE, BLACK (FOR AMMUNITION)
- c. Name and address of manufacturer
- d. Location and identification of the plant which produced the samples
- e. Manufacturer's identification
- f. Date of manufacture
- g. First article sample batch or lot number
- h. Submitted by (name) (date) for first article inspection in accordance with the requirements of MIL-DTL-450 under authorization of (reference authorizing letter) (see 6.3).

4.3 Conformance inspection. For each inspection lot of material submitted for acceptance, conformance inspection shall consist of all the examinations and tests required in 4.4 except reactivity (see 4.4.20) and ignition (see 4.4.21). Failure of a test sample to comply with any of the requirements of this specification shall result in the rejection of the inspection lot of material represented.

4.3.1 Batch. A batch is defined as that quantity of material that has been manufactured by some unit chemical or physical mixing process intended to make the final product substantially uniform.

4.3.2 Inspection lot. An inspection lot shall consist of the coating compound produced by one manufacturer, at one plant, from the same materials, and under the same manufacturing conditions provided the operation is continuous. In the event the process is a batch operation, each batch shall constitute an inspection lot.

4.3.3 Sampling. A 10-pound sample shall be taken at random from each inspection lot in accordance with ASTM D 3925.

4.4 Test methods.

4.4.1 Test conditions. Ensure the routine and referee testing conditions are in accordance with section 9 of FED-STD-141, except as otherwise specified herein.

4.4.2 Total solids.

4.4.2.1 Method. Conduct test in accordance with ASTM D 2369 Method B using toluene as the solvent.

4.4.3 Water.

4.4.3.1 Method. Conduct test in accordance with ASTM D 244, sections 4 through 10, using toluene as the solvent with the exceptions noted herein. Mix the sample with 75 mL of solvent. Make the connections between the flask, trap, and condenser by means of standard taper joints according to Figure 1 of ASTM D 95. Regulate the heat applied so that condensate falls from the end of the condenser at a rate of one to two drops per second.

4.4.4 Mineral matter.

4.4.4.1 Equipment.

- a. Crucible
- b. Hotplate
- c. Hood
- d. Muffle furnace
- e. Desiccator
- f. Scale

4.4.4.2 Method.

- a. Transfer approximately 25 grams (g) of the compound to a tared crucible and weigh accurately to 1 milligram (mg).
- b. Evaporate the low boiling solvent on a hotplate and under a hood.
- c. Transfer the crucible with the viscous solid to a muffle furnace and gradually ignite to a dull, red heat in order to avoid spattering until the residue is free from carbon.
- d. Cool in a desiccator and weigh.

- e. Reserve the residue for manganese determination.

4.4.4.3 Calculation of test results. Calculate the percentage of mineral matter as follows:

$$\text{Percent of mineral matter} = \frac{100A}{W}$$

Where:

A = weight of residue (g)

W = weight of sample (g).

4.4.5 Manganese.

4.4.5.1 Equipment.

- a. 250-mL beaker
- b. Hotplate
- c. Acid-washed Celite Analytical Filtering Aids (CAFA)
- d. Glass microfiber filter
- e. Burette

4.4.5.2 Reagents.

- a. Nitric acid
- b. Distilled water
- c. Lead peroxide
- d. N/10 oxalic acid

4.4.5.3 Method.

- a. Dissolve the ash obtained from 4.4.4 in 10 mL of concentrated nitric acid and filter if necessary.
- b. Dilute to 100 mL with distilled water and transfer a 20-mL aliquot of the solution to a 250-mL beaker. Retain the remaining 80 mL of the solution for lead determination.
- c. Add 1 g of lead peroxide and boil.
- d. Note if manganese is present by the development of a red coloration in the solution.

e. If the presence of manganese is indicated, filter using acid-washed CAFA and glass microfiber filter. Titrate the clean filtrate with N/10 oxalic acid until the red coloration disappears.

4.4.5.4 Calculation of test results. Calculate the percentage of manganese in the sample as follows:

$$\text{Percent of manganese} = \frac{5.495VN}{W}$$

Where:

V = volume of N/10 oxalic acid used (mL)

N = normality of oxalic acid

W = weight of sample in 4.4.4 (g).

4.4.6 Lead.

4.4.6.1 Equipment.

- a. Beaker
- b. Hotplate
- c. Gooch crucible or equivalent
- d. Oven
- e. Scale

4.4.6.2 Reagents.

- a. Ammonium hydroxide
- b. Acetic acid
- c. Sodium or potassium dichromate
- d. Water
- e. Alcohol

4.4.6.3 Method.

a. Transfer the remaining 80-mL portion of the solution obtained as described in 4.4.5 to a beaker and evaporate to 5 mL.

- b. Make the solution slightly alkaline by adding ammonium hydroxide.
- c. Acidify with acetic acid and add an excess.
- d. Bring to the boiling point and add 10 to 15 mL of a 10 percent solution of sodium or potassium dichromate.
- e. Boil the solution and allow to stand for 16 hours.
- f. Note if lead is present as indicated by a yellow precipitate.
- g. If lead is present, filter the solution through a tared Gooch crucible (or equivalent) and wash the precipitate with water and alcohol.
- h. Dry the crucible and contents at approximately 110 °C (230 °F) for 1 hour, cool, and weigh.

4.4.6.4 Calculation of test results. Calculate the increase in weight of the crucible as lead in the sample as follows:

$$\text{Percent of lead} = \frac{80.1A}{W}$$

Where:

A = weight of residue (g)

W = weight of original sample (g).

4.4.7 Insoluble matter (in carbon disulfide).

4.4.7.1 Equipment.

- a. Scale
- b. 150-mL beaker
- c. Steam bath
- d. Gooch crucible
- e. Oven

4.4.7.2 Reagents.

- a. Carbon disulfide

4.4.7.3 Method.

- a. Weigh accurately about 2 g of the sample into a 150-mL beaker.
- b. Evaporate the volatile solvent on a steam bath.
- c. Add 50 mL of carbon disulfide and agitate until solids are broken up and all lumps disappear.
- d. Cover and set aside for 15 minutes.
- e. Filter through a tared Gooch crucible.
- f. Wash down the sides of the beaker and the Gooch crucible with a small portion of carbon disulfide until the filtrate is clear.
- g. Draw air through the Gooch crucible for about 10 minutes to remove the carbon disulfide.
- h. Dry the crucible and contents for about 20 minutes in an oven at 105 to 110 °C.
- i. Cool and weigh.

4.4.7.4 Calculation of test results. Calculate the percentage of matter insoluble in carbon disulfide as follows:

$$\text{Percent of insoluble matter} = \frac{100A}{W}$$

Where:

A = weight of residue (g)

W = weight of sample (g).

4.4.8 Acidity or alkalinity.**4.4.8.1 Equipment.**

- a. Scale
- b. 150-mL flask
- c. Stopper
- d. 250-mL flask
- e. Water bath

- f. Funnel
- g. Filter paper
- h. 150-mL beaker

4.4.8.2 Reagents.

- a. Distilled water
- b. N/100 sulfuric acid
- c. N/100 sodium hydroxide
- d. Phenolphthalein indicator

4.4.8.3 Method.

- a. Quickly weigh 10.0 ± 0.5 grams of the sample in a clean, dry 150-mL flask and stopper the flask.
- b. Add 150 mL of distilled water and 10 to 12 drops of phenolphthalein indicator to a 250-mL flask. Distilled water is acidic and is neutralized to the first perceptible pink color with N/100 sodium hydroxide.
- c. Carefully add 100 mL of the neutralized distilled water to the flask containing the sample, and stopper the flask.
- d. Bring flask to 38 °C in a water bath, then shake gently and swirl for 5 minutes.
- e. Let flask stand for 15 minutes.
- f. Filter solution through a funnel fitted with fluted filter paper. Collect the filtrate into a clean, dry 150-mL beaker.
- g. Transfer 80 mL of filtrate to a clean, dry 150-mL beaker.
- h. When testing for alkalinity, if the solution has not changed color, sample is free of alkalinity. If solution changes color, titrate solution with N/100 sulfuric acid until the solution first turns colorless.
- i. When testing for acidity, add 2 to 5 drops of phenolphthalein indicator to beaker and titrate solution with N/100 sodium hydroxide to the first pink color.

4.4.8.4 Calculation of test results. Calculate the alkalinity as weight percent sodium hydroxide as follows:

$$\text{Alkalinity as wt. \% sodium hydroxide} = \frac{[(V_A * N)(8/V_B)]}{W}$$

Where:

V_A = volume of sulfuric acid needed to titrate sample (mL)
 V_B = volume of filtrate selected for titration (mL)
 N = normality of sulfuric acid
 W = weight of sample (g).

Calculate the acidity as weight percent sulfuric acid as follows:

$$\text{Acidity as wt. \% sulfuric acid} = \frac{[(V_A * N)(4.9/V_B)]}{W}$$

Where:

V_A = volume of sodium hydroxide needed to titrate sample (mL)
 V_B = volume of filtrate selected for titration (mL)
 N = normality of sodium hydroxide
 W = weight of sample (g).

4.4.9 Viscosity.

4.4.9.1 Equipment.

- a. Penetrometer and penetrometer cone with plunger assembly
- b. Cylindrical container

4.4.9.2 Methods.

a. Method 1 (Ford cup, Types I and II only). Conduct test in accordance with ASTM D 1200 using a No. 4 Ford cup.

b. Method 2 (Penetration, Type III only).

(1) Determine the penetration test value of Type III coating compound using the penetrometer and the penetrometer cone as described in ASTM D 217 with plunger assembly (total moving weight) weighing 150 g.

(2) Take precautions in carrying out this procedure to eliminate, as far as possible, error due to the volatilization of solvent and inclusion of air bubbles.

- (3) Fill a cylindrical container, at least 3 inches in diameter, with the sample to a depth of at least 3 inches.
- (4) Bring the sample to 77 ± 1 °F and level the exposed surface.
- (5) Level the penetrometer.
- (6) Place the container on the penetrometer table so that the approximate center of the exposed surface lies beneath the tip of the cone.
- (7) Adjust the height of the penetrometer table and plunger assembly until the tip of the cone just touches the surface of the sample.
- (8) Release the plunger assembly and allow to remain free for 5 seconds.
- (9) Read the penetrometer [depth of penetration expressed in tenths of millimeter (mm)].
- (10) Raise the plunger assembly, level the exposed surface of the sample, and repeat the test as directed above.
- (11) Report the average of 10 tests if the mean deviation of the first five values exceeds 3 percent.

4.4.10 Drying time.

4.4.10.1 Equipment.

- a. Film applicator (see Table IV).

Table IV. Film applicator.

Type	Film applicator (in)	Gap clearance (in)
I	0.0030	0.0060
II	0.0020	0.0040
III	To produce a 0.015 inch wet film thickness	

4.4.10.2 Methods.

a. Method 1 (Dust-free). Determine dust-free time in accordance with method 4061.3 of FED-STD-141 using the film applicator specified in Table IV. Observe for compliance with Table II.

b. Method 2 (Free from after-tack). Determine free from after-tack time in accordance with method 4061.3 of FED-STD-141 using the film applicator specified in Table IV. Observe for compliance with Table II.

4.4.11 Storage properties.

4.4.11.1 Equipment.

- a. 8-ounce bottle
- b. Stopper

4.4.11.2 Method.

- a. Put approximately 4 ounces of the coating compound as packaged into an 8-ounce bottle.
- b. Stopper and allow to stand 24 hours at room temperature.
- c. Observe for compliance with 3.5.1.

4.4.12 Dilution stability (Type II only).

4.4.12.1 Method. Conduct the test in accordance with method 4203.2 of FED-STD-141. Reduce one part of the package material with one part of aliphatic naphtha thinner and observe for compliance with 3.5.2.

4.4.13 Brushing properties (Type II only).

4.4.13.1 Equipment.

- a. 1 ½-inch brush
- b. 4- by 12-inch steel panel

4.4.13.2 Method.

a. Using a 1 ½-inch brush, apply the packaged material quickly on a solvent-cleaned, 4- by 12-inch steel panel and observe for compliance with 3.5.3.

4.4.14 Spraying properties, appearance when dry (Types I and II only).

4.4.14.1 Method. Conduct the test in accordance with method 4331.2 of FED-STD-141. Spray the Type I material as packaged. Reduce two volumes of the Type II material with a maximum of one volume of aliphatic naphtha thinner and spray. Observe for compliance with 3.5.4.

4.4.15 Water resistance.

4.4.15.1 Equipment.

- a. 3-inch by 5-inch steel panel
- b. Film applicator

4.4.15.2 Reagents.

- a. Naptha
- b. Ethylene glycol monoethyl ether
- c. Wax
- d. Distilled water

4.4.15.3 Method.

- a. Prepare one 3-inch by 5-inch steel panel in accordance with Procedure C or D of ASTM D 609. The steel shall be of Types 1 or 2 steel conforming to ASTM D 609. The cleaning solvent shall be a mixture of three volumes of naphtha and one volume of ethylene glycol monoethyl ether.
- b. Using a film applicator that will deposit a dry film thickness between 0.0009 and 0.0011 inch, draw down a 2-inch-wide film of the coating compound on the clean steel panel.
- c. Air dry for 24 hours at 23 ± 1 °C.
- d. Coat all exposed metal surfaces with wax or other suitable coating, and immerse for 18 hours in distilled water at 23 ± 1 °C in accordance with ASTM D 1308.
- e. At the end of the test period, remove the panel and inspect for compliance with 3.5.5.

4.4.16 Acid resistance.

4.4.16.1 Equipment.

- a. Steel panels (see 4.4.15)
- b. Watch glass

4.4.16.2 Reagents.

- a. Picric acid
- b. Sulfuric acid
- c. Nitric acid
- d. Hydrochloric acid

4.4.16.3 Method.

- a. Prepare four steel panels as in 4.4.15.
- b. Apply saturated picric acid, sulfuric acid (specific gravity, 1.30), nitric acid (specific gravity, 1.22), and hydrochloric acid (specific gravity, 1.09) to the panels as in ASTM D 1308.
- c. Place 3 or 4 drops of each acid on the test coating on separate panels and cover with a watch glass approximately 1.5 inches in diameter. The watch glass shall be at least ¼ inch from the coating edge.
- d. At the end of six hours, rinse the acid from the panel and inspect for compliance with 3.5.6.
- e. Remove the coating compound from the panel, and check for etching of the metal.

4.4.17 Heat resistance.

4.4.17.1 Equipment.

- a. 3-inch by 5-inch steel panel
- b. Oven

4.4.17.2 Reagents.

- a. Naphtha

4.4.17.3 Method.

- a. Mask off half of a 3-inch by 5-inch steel panel, and spray the coating compound on the uncovered portion to a dry film thickness between 0.0009 and 0.0011 inch. Type II and Type III compounds should be thinned with low boiling, fast evaporating naphtha to a suitable spraying viscosity.
- b. Air dry for 24 hours at 23 ± 1 °C.

c. Remove the masking tape, and heat in an oven at approximately 100 °C in a vertical position. The coated end of the panel shall be uppermost with the dividing line horizontal.

d. At the end of 1 hour, remove from the oven and examine for compliance with 3.5.7.

4.4.18 Flexibility.

4.4.18.1 Equipment.

a. Tin plated steel panel (tin panel). The tin panel shall be made by plating Type 3 steel conforming to ASTM D 609. The tin coating shall conform to the dimensional and chemical composition requirements of ASTM A 623 and be of class A2, grade 1, 31 gage ICL tinplate.

b. Film applicator

c. 1/8-inch-diameter mandrel

4.4.18.2 Reagents.

a. Naptha

b. Ethylene glycol monoethyl ether

4.4.18.3 Method.

a. Prepare one tin panel in accordance with Procedure C or D of ASTM D 609, using a 3:1 volumetric mixture of naptha and ethylene glycol monoethyl ether as the cleaning solvent.

b. Using a film applicator that will deposit a dry film thickness of 0.0009 to 0.0011 inch, draw down a 2-inch-wide film of coating compound on the clean tin panel.

c. Air dry for 24 hours under referee conditions.

d. Place the panel with the coated side up on a 1/8-inch-diameter mandrel at a point equidistant from the top and bottom edges of the panel.

e. Bend the panel double in approximately one second and examine for compliance with 3.5.8.

4.4.19 Solvent analysis for Composition L coating compound.

4.4.19.1 Separation of volatile portion.

4.4.19.1.1 Equipment.

- a. Large test tube (22 mm by 175 mm)
- b. Antibumping stones or Berl saddles
- c. 2-hole rubber stopper
- d. Glass delivery tube (5-mm diameter) with stopcock
- e. Test tube (20 mm by 150 mm) with sidearm
- f. Dry ice-acetone bath
- g. Silicone oil bath
- h. Vacuum pump

4.4.19.1.2 Reagents.

- a. Tricresyl phosphate

4.4.19.1.3 Method.

- a. Pour approximately 15 g of coating material into a large test tube (22 mm by 175 mm).
- b. Add 10 mL of tricresyl phosphate and several antibumping stones or Berl saddles.
- c. Fit a 2-hole rubber stopper into the mouth of the test tube to accommodate a stopcock and a glass delivery tube (5-mm diameter) which is attached to another test tube (20 mm by 150 mm) to serve as receiver. The latter test tube should have a sidearm for attaching a vacuum pump. The glass delivery tube should reach 1 inch from the bottom of the receiver tube.
- d. Immerse the receiver in a dry ice-acetone bath.
- e. Preheat a silicone oil bath to 160 °C. Raise the oil bath until the oil reaches the sample level.
- f. Reduce the pressure slowly to 10 mm of mercury. After all the solvent has distilled, carefully release the vacuum using the stopcock that is connected to the sample tube.

g. Reserve the collected distillate for the aromatic solvent determination and qualitative test for olefinic compounds.

4.4.19.2 Determination of aromatic hydrocarbons.

4.4.19.2.1 Equipment.

a. Gas chromatograph equipped with a thermal conductivity detector

b. The chromatographic column used shall be 18 feet of ½-inch copper tubing with 35 percent by weight of N, N-bis (2-cyanoethyl) formamide on 60- to 80-mesh Chromosorb P. The column may be prepared in two 9-foot sections and joined together, if preferred. The packings should be re-sieved before preparing the column. The gas chromatograph shall operate under the following conditions:

Detector cell temperature, °C	300
Detector cell current, ma	150
Injection port temperature, °C	300
Helium flow at exit, cc/minute	110
Column temperature, °C	100

4.4.19.2.2 Reagents.

a. Benzene or internal standard

4.4.19.2.3 Method.

a. Add 0.5 mL of internal standard (benzene) to 5 mL of the distillate from 4.4.19.1. Mix thoroughly.

b. Inject about 3 microliters into the gas chromatograph. If aliphatic naphtha solvent is used, toluene, ethylbenzene, and the xylene will be the only aromatic solvents present. Extraneous peaks emerging after benzene, which do not coincide with the above-mentioned aromatics, are to be grouped together and calculated as C₈ and higher aromatic solvents. This would include any oxygenated solvents that may be present.

4.4.19.2.4 Calculation of test results. Calculate the percentage of aromatics as follows:

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

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

$$\% \text{ C}_8 \text{ aromatics except ethylbenzene, } v/v = \frac{(\text{area of xylene isomer peaks})(1.04) * (10)**}{(\text{area of benzene peak})}$$

**is the correction factor for the detector response*

*** is the percentage of internal standard added.*

4.4.19.3 Tests for olefinic and cycloolefinic compounds.

4.4.19.3.1 Equipment.

- a. Two test tubes

4.4.19.3.2 Reagents.

- a. Carbon tetrachloride
- b. Bromine
- c. Acetone
- d. Permanganate

4.4.19.3.3 Method.

- a. Take two test tubes and place two drops of the distillate from 4.4.20.1 in each.
- b. Dissolve the first sample in 1 mL of carbon tetrachloride, and add one drop of 1 percent bromine in carbon tetrachloride.
- c. Shake first sample and allow to sit for 5 minutes. A positive test is indicated by the complete absence of yellow color when observed against a white background.
- d. Dissolve the second sample in 1 mL of acetone, and add one drop of 1 percent permanganate solution (1 g of potassium permanganate crystals in 95 mL of acetone and 5 mL of water).
- e. Shake and allow to sit for 2 minutes. A positive test is indicated by the decolorization of the purple solution.

f. The solvent is considered to fail the test for olefinic and cycloolefinic compounds if either of the above tests is positive (see 3.3.1.2 and 6.4).

4.4.20 Reactivity. Determine the reactivity of the compound in contact with the explosives listed in 3.6.1 using the vacuum stability test.

4.4.20.1 Equipment (method 3 only).

- a. Glass plates
- b. Knife
- c. Glass rods
- d. Air-circulating oven/cabinet or fan
- e. 12 mesh or finer screen
- f. Desiccator
- g. Containers with stoppers or covers
- h. Heating tubes consistent with method 403.1.3 in MIL-STD-286
- i. Capillary tubes

4.4.20.2 Sample preparation.

- a. Pour a sufficient amount of coating compound on glass plates to provide 2.5 (N + 2) g of dried film (where N equals the number of explosives involved).
- b. Air dry the films under ambient conditions for 48 hours, then peel off with a sharp edged tool in strips approximately ½-inch wide.
- c. Suspend the strips on glass rods in an oven or cabinet with circulating air at 30 °C (86 °F) for another 48 hours. If an air-circulating oven is not available, place the suspended strips before a fan in a warm room for 48 hours. Ensure that the room in which this operation is conducted is free from acid, alkaline, or other contaminating fumes.
- d. Remove and cut the strips into approximately ¼-inch squares.
- e. Reduce the explosive materials to 12 mesh or finer by remote grinding or rasping, and dry for at least 24 hours in a desiccator over a desiccating agent. Keep both the coating film and the ground explosives dry in stoppered containers until ready for testing.

4.4.20.3 Methods.

a. Method 1. Conduct VTS test in accordance with MIL-STD-650, Method 503.1.1, except that the test sample size shall be at least 0.2 g. Horizontal capillary tube dimension may be 26 cm or 15 cm.

b. Method 2. Conduct VTS test by the pressure transducer method in accordance with STANAG 4556.

c. Method 3.

(1) Calibrate the necessary number of vacuum stability test assemblies in accordance with procedure 4.1 of method 403.1.3 in MIL-STD-286.

(2) Use $2N + 1$ (where N equals the number of explosives used) tubes similar to the heating tube portion of the apparatus in procedure 4.1 of method 403.1.3 in MIL-STD-286. For controls, add 2.6 g of the dried compound to each of two tubes and 2.5 g of each explosive to additional individual tubes.

(3) Place uniform mixtures of 2.5:2.5 g of the coating compound and each of the explosives specified in the test in single separate tubes. Obtain uniform distribution or mixing by carefully layering alternate portions of the dried coating with the explosive.

(4) After the addition of each portion, lightly tamp the mixture with a glass or metal rod to insure intimate contact of the materials.

(5) After all the samples have been prepared, connect the respective heating tubes with the capillary tubes as prescribed, and conduct the vacuum stability test according to method 403.1.3 of MIL-STD-286 at 100 ± 0.5 °C (212 ± 1 °F) for 48 hours.

(6) Take all readings at room temperature with the samples removed from the bath. Correct the readings of both the controls and the test samples to standard conditions of temperature and pressure, and check for compliance with 3.6.1.

4.4.20.4 Calculation of test results. Calculate the reactivity of each of the explosive materials with the coating compound for compliance with 3.6.1 as follows:

$$\text{Reactivity in ml gas} = X - (Y + Z)$$

Where:

X = volume of gas produced by the mixture of explosive material and coating compound (mL)

Y = volume of gas produced by the explosive material alone (mL)

Z = volume of gas produced by the coating compound alone (mL).

4.4.21 Minimum ignition temperature.

4.4.21.1 Equipment (method 2 only).

- a. Glass heat resistant test tube
- b. Wood's metal bath containing a standardized thermometer
- c. Safety shield

4.4.21.2 Methods.**a. Method 1.**

(1) Mix 0.25 g of the explosive to be tested with 0.25 g of the dried film of the coating compound in a glass heat resistant test tube.

(2) Heat the test tube containing the mixture at a rate of 5 to 10 °C per minute in a wood's metal bath containing a standardized thermometer.

(3) Check the temperature at which fume-off or ignition occurs for compliance with 3.6.2. This test should be conducted behind a safety shield.

b. Method 2. Minimum ignition temperature for a 1:1 mixture of the dried coating compound and the explosive to be tested may be determined by instrumental methods using differential thermal analysis (DTA) or differential scanning calorimetry (DSC). From compounds of known reaction temperatures, such as ammonium nitrate or silver nitrate, construct a standard temperature curve, or calibrate the instrument as required, using a temperature programming rate of 10 to 15 °C per minute. Follow the instrument manufacturer's recommended explosive sample size. Maintaining the same instrumental conditions, determine the minimum ignition temperature for the mixture of coating and explosive being tested. The ignition of the sample is indicated by a sharp and sometimes violent exothermic peak at or near the ignition temperature of the particular explosive in the sample. Relate the indicated ignition of the sample thermogram to the standard temperature curve to determine the actual ignition temperature. Check the ignition temperature for compliance with 3.6.2.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of materiel is to be performed by DOD personnel or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Service or Defense Agency, or within the military service's system commands. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. This asphalt coating compound is intended for coating the interior surfaces of ammunition items such as bombs, shells, rockets, and mines prior to being filled with explosives. Since it was developed for use in military ammunition, there is no commercial application.

6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number and date of this specification
- b. If required, the specific issue of individual documents referenced (see 2.2.1, 2.2.2, and 2.3)
- c. Composition and type required
- d. Quantity in pounds (avoirdupois)
- e. Packaging requirements (see 5.1 and 6.5)
- f. Whether first article inspection is required and, if so, specify the test activity (see 3.1 and 4.2).

6.3 First article. When a first article inspection is required, the contracting officer should provide specific guidance to offeror(s) whether the sample(s) should be a preproduction sample, a first article sample, a first production item, a sample selected from the first production items, or a standard production sample from the contractor's current inventory. The contracting officer should also include specific instructions in acquisition documents regarding arrangements for examinations, approval of first article test results, and disposition of the first articles. Invitation(s) for bid(s) should provide that the U.S. Government reserves the right to waive the requirements for samples for first article inspection to those bidders offering a product which has been previously acquired or tested by the U.S. Government, and that bidders offering such products, who wish to rely on such production or test, must furnish evidence with the bid that prior U.S. Government approval is presently appropriate for the pending contract. Bidders should not submit alternate bids unless specifically requested to do so in the solicitation.

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

6.5 Packaging and marking.

6.5.1 Packaging. The material should be packaged in accordance with the manufacturer's best commercial practice. The exterior pack should meet all of the test requirements in accordance with the Code of Federal Regulations, Title 49 (49 CFR), including testing, and retesting as specified in Paragraph 178.601 (e) of 49 CFR.

6.5.2 Packing. The material, packaged as specified in 6.5.1, should be packed to ensure carrier acceptance and safe delivery to the destination at the lowest applicable rate.

6.5.3 Special marking. In addition to any special markings required by the contract or purchase order (see 6.2), all containers should be marked as required by the Code of Federal Regulation 49 CFR 100-199. In addition, shipments should be marked in accordance with Standard MIL-STD-129-1. Each container should be clearly labeled with the lot number and net weight of its contents.

6.7 Supersession data. This document supersedes MIL-C-450C dated 5 February 1971, MIL-C-450C Notice 1 dated 9 May 1996, MIL-C-450C Notice 2 dated 28 March 2001, QPL-450-11 Amendment 1 dated 14 November 1977, and QPL-450-11 dated 25 June 1975.

6.8 Subject term (key word) listing.

Ammunition items
Benzene
Carbon tetrachloride
Composition B
Composition C-4
HBX
TNT

6.9 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes

Custodians
Army – AR
Navy – OS
Air Force – 99

Preparing Activity:
Navy – OS
(Project 8030-2009-001)

Review activities:
Army - MR
Air Force - 84

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at <https://assist.daps.dla.mil/>.