

MIL-C-16555D
~~24 February 1975~~
SUPERSEDING
MIL-C-16555C
10 February 1961

PROPOSED MILITARY SPECIFICATION

COATING COMPOUND, STRIPPABLE, SPRAYABLE

*This specification is approved for use by all departments and agencies of
the Department of Defense.*

1. SCOPE

*1.1 **Scope.** This specification covers a strippable coating compound for application by spraying on painted or unpainted metal surfaces to seal openings and provide protection against corrosion. This specification meets Air Pollution Regulations (Rule 66).

1.2 **Classification.** Coating compound shall be of the following types of colors as specified:

- Type I Aluminum or aluminum gray
- Type II, Class 1—Olive drab
- Type II, Class 2 Marine Corps green

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

Federal

- | | |
|-----------|-------------------------------------|
| *QQ-S-698 | Steel, Sheet and Strip, Low Carbon |
| TT-E-485 | Enamel, Semi-Gloss, Rust-Inhibiting |
| TT-E-529 | Enamel, Alkyd, Semi-Gloss |

FSC 8030

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*TT-P-1757 Primer Coating, Zinc-Chromate, Low-Moisture Sensitivity.

PPP-D-729 Drums, Shipping and Storage, Steel, 55-Gallon

PPP-D-760 Drums and Pails, Metal (5 and 16.64 Gallon)

Military

MIL-S-20166 Steel Structural Shapes, Weldable Medium Carbon

STANDARDS

Federal

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

FED-STD-313 Symbols for Packages and Containers for Hazardous Chemicals and Materials

FED-STD-595 Colors

FED-STD-601 Rubber: Sampling and Testing

Military

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

MIL-STD-129 Marking for Shipment and Storage

MIL-STD-147 Palletized Unit Loads (40" X 48" 4-way partial and 4-way pallets)

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

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

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Uniform Freight Classification Rules

(Application for copies should be addressed to the Tariff Publishing Officer, Room 1106, 222 Riverside Plaza, Chicago, Ill. 60606.)

National Motor Freight Classification Rules

(Application for copies should be addressed to Issuing Officer, American Trucking Association, 1616 P St. N.W., Washington, D.C. 20036.)

***3. REQUIREMENTS**

3.1 **Materials and composition.** The coating compound shall consist of vinyl resins, plasticizers, solution and film stabilizers, pigments, fire retardants and solvents formulated to meet all applicable requirements of this specification. It shall not contain chlorinated paraffins, rosin, or rosin derivatives. The composition shall be as shown in table I.

Table I

COMPOSITION

Ingredient	Percent by weight	
	Type I	Type II
Nonvolatile material, min.	30	34
Volatile solvent, max.	70	66

Composition of Nonvolatile Content
Percent by weight, minimum

Vinyl resin	-	50
Vinyl resin and plasticizer	93	78
Pigment ¹	-	12
Aluminum powder	2	-
Titanium dioxide	0.5	-
Blue lead and other pigments	2	-
Fire retardants	-	1.5
Stabilizers	-	3.0

¹The pigment in type II shall contain at least 70 percent lead chromate by weight, equivalent to 8.4 percent of the nonvolatile content

3.2 Color

3.2.1 **Type I.** Type I coating compound shall be aluminum or aluminum gray in color.

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3.2.2 **Type II.** Type II, class 1 shall be olive drab and shall closely match standard color chips 14087 or 14064 of FED-STD-595. Type II, class 2 shall be marine corps green and shall closely match standard color chip 14052 of FED-STD-595. Lack of gloss shall not be cause for rejection.

3.3 **Viscosity.** When tested as specified in 4.4.1.1, the viscosity of the coating solution shall be as follows:

<u>Viscosity, Krebs Units</u>			
<u>Type</u>	<u>Temperature</u>	<u>Minimum</u>	<u>Maximum</u>
I and II	25° ± 1° C (77° ± 2° F)	60	80
I	10° ± 1° C (50° ± 2° F)	70	100

3.4 **Gelation at low temperature.** When tested as specified in 4.4.1.2 at low-temperature storage the compound shall be restorable to a free-flowing consistency for spraying by manual or mechanical stirring at 77° ± 2° F.

3.5 **Separation of pigment.** After the coating compound has been stored in a closed container at room temperature for 10 days, the pigment shall not separate from the solution in such a manner as to prevent restoration of the solution to consistency for satisfactory spraying by manual stirring at 77° ± 2° F.

3.6 **Sprayability.** The compound shall be supplied in a consistency satisfactory for application with conventional pressure-type spray equipment when tested under the conditions specified in 4.4.1.3 for the preparation of test specimens. Any clogging of the spray gun due to presence of lumps of undissolved or inadequately dispersed material shall be cause for rejection.

3.7 **Flammability.** When tested as specified in 4.4.1.4, test specimens of the dried coating shall cease to burn or glow within 5 seconds after the source of ignition is removed.

3.8 **Tensile strength.** When tested as specified in 4.4.1.5, the tensile strength of the coating shall be not less than 500 pounds per square inch.

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3.9 **Ultimate elongation.** When tested as specified in 4.4.1.5, the ultimate elongation of the coating shall be not less than 200 percent.

3.10 **Low-temperature flexibility.** When tested as specified in 4.4.1.6, the coating shall not crack or break.

3.11 **Low-temperature impact resistance.** When tested as specified in 4.4.1.7, before and after accelerated weathering, the coating shall withstand the impact of a freely falling 28-gram steel ball dropped from a height of 30 inches. The impact shall produce no cracks in the coating.

3.12 **Water extractable matter.** When tested as specified in 4.4.1.8, the weight loss of the coating shall be not more than 0.20 percent of its original weight.

3.13 **Volatile matter.** When tested as specified in 4.4.1.9, the weight loss of the coating shall be not more than 1.5 percent of its original weight.

3.14 **Resistance to heat.** When tested as specified in 4.4.1.10, the coating shall show no shrinkage, separation from the panel(s), bubbles, darkening, or other visible evidence of degradation. The panel surface in contact with the coating shall show no blackening, pits, or other visible evidence of corrosion. The coating, after cooling to room temperature, shall not crack or break on being peeled back from the panel(s).

3.15 **Water vapor transmission.** When tested as specified in 4.4.1.11, the water vapor transmission rate after exposure to accelerated weathering, as specified in 4.4.1.13, shall be not more than 1.2 grams per 100 square inches per 24 hours.

3.16 **Salt spray resistance.** When tested as specified in 4.4.1.12, the test specimen shall show no evidence of corrosion, and the coating shall show no pinholes or separation from the metal.

3.17 **Accelerated weathering.** When tested as specified in 4.4.1.13, the coating shall show no visible evidence of degradation or shrinkage and shall be strippable from the panel(s) in one continuous sheet. The panel surface in contact with the coating shall show no evidence of corrosion. The coating after exposure to accelerated weathering shall meet the requirements for tensile strength, ultimate elongation, low-temperature flexibility,

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low-temperature impact resistance, water vapor transmission, and adhesion to steel, and painted surface as specified herein, except that specimens from the 1500-hour test shall have an ultimate elongation not less than 85 percent of that of the unweathered specimens. The adhesion to the test panel shall not be greater than 3.25 pounds per inch width for the type II material.

3.18 Adhesion.

*3.18.1 **Adhesion to steel.** When tested as specified in 4.4.1.14, the adhesion of the coating to the surface of an unpainted steel (conforming to QQ-S-698) panel shall be not less than 0.50 pound and not more than 2.25 pounds per inch width.

3.18.2 **Adhesion to painted surface.** When tested as specified in 4.4.1.15, the adhesion of the coating to the surface of a painted panel shall be not less than 0.50 pound and not more than 2.25 pounds per inch width. Stripping of the coating shall not lift or otherwise damage the painted surface.

3.19 **Shrinkage.** When tested as specified in 4.4.1.15.4, the coating shall not bridge the angle formed by the two legs of the angle iron nor pull loose from either the inside or outside flat surfaces.

*3.20 **Solvent.** The solvent when tested as specified in 4.4.2.5 and 4.4.2.6 shall consist of methyl ethyl ketone, or methyl ethyl ketone and toluene. Volatile stabilizers shall be considered as solvent constituents. The use of small amounts of methyl isobutyl ketone is permitted. Toluene plus methyl isobutyl ketone shall not exceed 20 percent by volume of the solvent content.

3.21 **Toxicity.** The vapor shall not cause serious injury or undue discomfort to workmen during application (see 6.3).

3.22 **Workmanship.** The coating compound shall be intimately compounded and free of particles of undissolved resin or undispersed pigments, to meet all the requirements of this specification.

*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 prescribed requirements.

***4.2 Quality conformance inspection.** Quality conformance inspection shall consist of all the examinations and tests of 4.3 and 4.4. Failure of the coating compound to conform to any of the requirements of this specification shall be cause for rejection of the lot represented. Coating compound which has been rejected may, at the option of the procuring activity, be reworked or replaced to correct the defects, and submitted for retest. Full particulars concerning previous rejection and the action taken to correct the defects found in the original shall be furnished to the procuring activity. Coating compound rejected after retest shall not be resubmitted without the specific approval of the procuring activity.

4.2.1 Sampling.

4.2.1.1 Lot. For purposes of sampling, a lot shall consist of all the coating compound manufactured at one time from one batch of constituents and submitted for delivery at one time.

4.2.1.2 Sampling for inspection of filled containers. A random sample of filled containers shall be selected from each lot in accordance with MIL-STD-105 at inspection level I and acceptable quality level 2.5 percent to verify compliance with all stipulations of this specification regarding fill, closure, marking, and other requirements not involving tests.

4.2.1.3 Sampling for tests. A 5-gallon sample shall be selected from each lot in accordance with method 1021 of FED-STD-141. The sample shall be forwarded to the laboratory designated by the procuring activity for lot acceptance testing.

4.2.1.4 When requested by the contractor, samples shall be selected in accordance with 4.2.1.3 with the following exception, a 30-gallon sample (six 5-gallon containers) shall be taken instead of a 5-gallon sample. Each 30-gallon sample shall be divided into three equal parts, one part to be delivered to the testing laboratory, one part to be held by the contractor, and one part to be held by the procuring activity in case of dispute.

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4.3 Inspection of filled containers. Each sample filled container selected in accordance with 4.2.1.2 shall be examined for defects of the container and the closure, evidence of leakage, and unsatisfactory markings. Each sample filled container shall also be weighed to determine the amount of the contents. Any container in the sample having one or more defects, or which is under required fill, shall be rejected; and if the number of defective containers in any sample exceeds the acceptance number for the appropriate sampling plan of MIL-STD-105, the lot represented by the sample shall be rejected. Rejected lots may be resubmitted for acceptance tests provided that the contractor has removed or repaired all nonconforming containers.

4.4 Test methods.

4.4.1 Physical test methods.

4.4.1.1 Viscosity. The viscosity of the coating compound shall be determined as specified in method 4281 of FED-STD-141 with the following addition. The viscosity of type I shall also be determined at $10^{\circ} \pm 0.25^{\circ} \text{C}$ ($50^{\circ} \pm 0.5^{\circ} \text{F}$).

4.4.1.2 Gelation at low temperature. A specimen of coating compound in a tightly sealed metal container shall be stored at $-20^{\circ} \pm 2^{\circ} \text{F}$ for 16 hours. The specimen shall then be allowed to come to room temperature, after which it shall be examined for evidence of gelation. If there is evidence of gelation, the specimen shall be mechanically stirred for not more than 5 minutes at $77^{\circ} \pm 2^{\circ} \text{F}$ to determine compliance with 3.4.

4.4.1.3 Preparation of test samples.

***4.4.1.3.1 Steel panels.** Except as otherwise indicated herein, all steel test panels 12 inches square shall be prepared from annealed steel conforming to QQ-S-698, 0.035-inch thick, polished on one side to a bright finish with No. 280 grit emery cloth. The panels shall then be rinsed in successive baths of ethyl alcohol (95 percent) and naphtha and then allowed to dry thoroughly.

4.4.1.3.2 Spray equipment. The spray equipment shall consist of a commercial type, pressure-feed spray cup and spray gun. The spray gun shall have a solution-adjusting screw for controlling the amount of compound passing through the spray nozzle, a spreader adjustment valve for controlling the width of spray fan, an air cap for adjustment of a horizontal or vertical spray pattern, and a fluid spray nozzle having a diameter of 0.070 ± 0.001 inch.

4.4.1.3.3 Spraying procedure. Prior to the spraying operation, the coating compound shall be thoroughly mixed at $77^{\circ} \pm 2^{\circ}$ F. Test panels prepared as specified in 4.4.1.3.1 shall be placed in a horizontal position at $77^{\circ} \pm 2^{\circ}$ F. The compound shall be sprayed onto the polished surface to yield a dry coating 0.035 ± 0.005 inch thick, using the equipment specified in 4.4.1.3.2. The spray operation shall be carried out in 150 ± 30 seconds for each 12- by 12-inch panel. The spray equipment shall be adjusted to emit a wet spray which will produce a smooth set continuous coating with no evidence of sagging or running. The coating on the test panel shall be dried for 24 hours at $77^{\circ} \pm 2^{\circ}$ F and then for 72 hours at $140^{\circ} \pm 2^{\circ}$ F in a circulating air oven. Any tendency of the coating to shrink or pull loose from the panel surface during the drying period shall be prevented by the use of clamps, suitable adhesive, or adhesive tape applied to the edges of the surface.

4.4.1.4 Flammability. Three test specimens shall be prepared by cutting 0.5- by 4-inch strips from a sheet of coating prepared as specified in 4.4.1.3. Prior to the test, the specimens shall be conditioned at $140^{\circ} \pm 2^{\circ}$ F for 24 hours in a circulating air oven. The specimen shall be gripped at one end with a pair of tongs, hooks, or clamps and allowed to hang in a vertical position. The free end of the specimen shall be ignited with a bunsen burner flame, the flame then removed, and the specimen observed for continued burning, to determine compliance with 3.7.

4.4.1.5 Tensile strength and ultimate elongation. Using a die conforming to Die No. II in method 4111 of FED-STD-601, five dumbbell test specimens of coating shall be cut from each of three sheets of coating prepared as specified in 4.4.1.3. The tensile strength and ultimate elongation of the test specimens shall be determined as specified in method 4111 and method 4121, respectively, of FED-STD-601; the distance between benchmarks in method 4121 shall be 2.000 ± 0.003 inches. Specimens shall be maintained at $77^{\circ} \pm 2^{\circ}$ F for at least 2 hours before and during the test. In lieu of the median values specified, the average values of the 15 determinations shall be taken as the tensile strength and ultimate elongation. If an individual result is more than 10 percent lower than the average, this result shall be discarded and a new specimen tested.

4.4.1.6 Low-temperature flexibility. Six test specimens, two from each of three separate panels, shall be prepared by cutting 1- by 6-inch strips from a sheet of coating prepared as specified in 4.4.1.3. These specimens and a mandrel 1 inch in diameter shall be conditioned at $-30^{\circ} \pm 2^{\circ}$ F for 2 hours. The test specimens shall then be bent 180° within 2 seconds around the mandrel in the cold chamber. The test specimens shall then be examined for evidence of cracking or breaking, to determine compliance with 3.10.

4.4.1.7 Low-temperature impact. Three circular test specimens slightly over 6 inches in diameter shall be cut from sheets of coating prepared as specified in 4.4.1.3. The test specimens shall be supported on 6-inch-diameter embroidery hoops, with the edges of the specimens well secured to the hoops. The specimens shall be maintained at $-12^{\circ} \pm 2^{\circ}$ F for

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2 hours before and during the test. With the test specimen in a horizontal position, a 3/4-inch steel ball weighing 28 grams shall be dropped freely from a height of 30 inches above the test specimen so that the ball strikes the center of the surface. This same procedure shall also be used to determine the low-temperature impact resistance of coating which has been exposed to accelerated weathering as specified in 4.4.1.13, except that the test specimens shall be maintained at $-7^{\circ} \pm 2^{\circ}$ F for 2 hours before and during the test.

4.4.1.8 Water extractable matter. Three 3- by 3-inch test specimens shall be cut from a sheet of coating prepared as specified in 4.4.1.3. The test specimens shall be conditioned for 1 hour in a forced draft oven at $140^{\circ} \pm 2^{\circ}$ F, cooled to room temperature in a desiccator, and immediately weighed on an analytical balance. Each of the specimens shall then be immersed in a pint jar of distilled water previously heated on $140^{\circ} \pm 2^{\circ}$ F. The jars shall be sealed and maintained at $140^{\circ} \pm 5^{\circ}$ F for 24 hours. The specimens shall then be removed, rinsed with distilled water, conditioned at $140^{\circ} \pm 2^{\circ}$ F for 24 hours, cooled to room temperature in a desiccator, and immediately reweighed. The percentage weight loss of each specimen shall be calculated and the results of the three determinations averaged, to determine compliance with 3.12.

4.4.1.9 Volatile matter. Three 1- by 3-inch test specimens taken from three separate sheets of coating shall be prepared as specified in 4.4.1.3. The test specimens shall be conditioned for 1 hour in an air circulating oven at $140^{\circ} \pm 2^{\circ}$ F, cooled to room temperature in a desiccator, and immediately weighed on an analytical balance. The specimen shall then be hung vertically by a small hook or clamp in a forced draft oven maintained at $220^{\circ} \pm 5^{\circ}$ F for 5 hours. Upon removal from the oven, the specimens shall be cooled to room temperature in a desiccator and immediately reweighed. The percentage weight loss of each specimen shall be calculated and the results of the three determinations averaged, to determine compliance with 3.13.

4.4.1.10 Resistance to heat. A coated panel prepared as specified in 4.4.1.3 shall be trimmed with a sharp knife so that the coating shall be the same size as the panel. The coated panel shall then be placed in an air circulating oven maintained at $380^{\circ} \pm 5^{\circ}$ F for 1 hour. The coating shall then be examined for visible evidence of separation from the panel surface or edges. Before the coated panel has cooled thoroughly, one end of the coating shall be peeled back to allow examination of the contracting surfaces. The coating may be peeled back by inserting a spatula under the coating and prying it back. Both surfaces of the coating shall be examined for bubbles, darkening, or other visible evidence of degradation. The steel surface in contact with the coating shall be examined for blackening, pits, or other visible evidence of corrosion. After cooling to room temperature, the coating shall be peeled back and examined for compliance with 3.14 with regard to cracking and breaking while being peeled back.

4.4.1.11 Water vapor transmission (type 1).

4.4.1.11.1 **Preparation of test dish.** The test dish for the water vapor transmission test shall have a test area approximately 5 inches in diameter. The dish shall be filled with anhydrous calcium sulfate of approximately No. 8 sieve size to be in contact with the test specimen of coating placed over the desiccant. Three circular test specimens of diameter slightly greater than that of the test area of the dish shall be cut from a sheet of coating prepared as specified in 4.4.1.3 and subsequently exposed to accelerated weathering as specified in 4.4.1.13. The test specimen shall be centered over the opening of the dish and clamped in position by the ring or flange. A circular metal template 1/8 inch thick, with the edge beveled to an angle of about 45°, shall be used in sealing the edge of the specimen. The smaller diameter of the template shall be equal to the diameter of the test area of the specimen. A thin film of petrolatum shall be applied to the beveled edge of the template, and any petrolatum that may have been deposited on the lower surface of the template shall be wiped off. The template shall be centered exactly over the test specimen and dish opening. Molten wax consisting of 60 percent amorphous wax and 40 percent refined crystalline paraffin wax shall be flowed into the annular space surrounding the beveled edge of the template. As soon as the wax has cooled and solidified, the template shall be removed with a twisting motion. Loose or superfluous wax shall be removed from the dish.

4.4.1.11.2 **Humidity cabinet.** The humidity cabinet shall be a General Foods type or equivalent which shall provide a relative humidity of 90 to 95 percent at a temperature of $100^{\circ} \pm 2^{\circ}$ F, with no condensation on the test specimens or in the space where the test specimens are placed. The air circulation over the test specimens shall be negligible.

4.4.1.11.3 **Test procedure.** Two assembled test dishes prepared as specified in 4.4.1.11.1 shall be conditioned in a humidity cabinet conforming to 4.4.1.11.2 for 16 to 24 hours. The assembled dishes shall then be removed from the cabinet, cooled in a desiccator for one-half hour, and immediately weighed on an analytical balance. The dishes can be fitted diagonally on the balance pan or suspended from the balance beam by a hook. The assembled dishes shall then be returned to the humidity cabinet for 72 hours, after which they shall be cooled in a desiccator for one-half hour and immediately reweighed. The gain in weight shall be used to calculate the water vapor transmission rate in grams of water per 100 square inches of test area per 24 hours per 0.035 \pm 0.005 inch thick:

$$\text{Water vapor transmission rate} = \frac{\text{weight gain in grams}}{\text{test area in square inches}} \times \frac{100}{3}$$

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4.4.1.12 **Resistance to salt spray test.** Three 4- by 6-inch panels shall be spray coated on all surfaces with the barrier material (the edges shall be dip coated to ensure proper sealing). After air drying for 24 hours at room conditions, and after conditioning for 72 hours at $60^{\circ} \pm 2^{\circ} \text{ C}$ ($140^{\circ} \pm 5^{\circ} \text{ F}$), the film of one specimen (control) shall be stripped and examined for corrosion. Evidence of corrosion shall be cause for rejection. If the control panel shows no corrosion, the two remaining panels shall be subjected to a 20 percent salt (NaCl) spray (fog) test for 168 hours. The panels shall be positioned 15° from the vertical, polished side up, parallel to the horizontal flow of the fog through the chamber. On removal from the fog chamber, the specimens shall be rinsed under tap water and dried. The film shall be stripped from the panels and the polished side of the panel examined for corrosion.

4.4.1.13 **Accelerated weathering.** Ten coated test panels (3 × 6 inches) prepared as specified in 4.4.1.3 shall be subjected to accelerated weathering as specified in method 6152 of FED-STD-141. Five panels shall be tested for 200 hours and five for 1500 hours. After each test, the weathered coatings shall be examined for visible evidence of degradation, and the panel surface in contact with the coating shall be examined for evidence of corrosion. The adhesion test samples and the coating stripped from the panels shall be tested for compliance with 3.17.

4.4.1.14 **Adhesion to steel.** Two test strips of coating 1 inch wide shall be cut through to the metal from the central portion of each of three coated test panels prepared as specified in 4.4.1.3. The strips shall be peeled back approximately 4 inches. The test panels so prepared shall be maintained at $77^{\circ} \pm 2^{\circ} \text{ F}$ for 24 hours before and during the adhesion determination. The stripped end of the test panel shall be clamped in one grip of a suitable tensile machine so that the center line of the grip lines up with the center line of the strip to be pulled. The loose end of the strip shall be bent back and fastened to the other grip of the testing machine at an angle as close to 180° as possible. The grips shall then be separated at a rate of 3 inches per minute until at least 2 inches of the strip are peeled from the panel. The maximum force necessary to peel each of the six test specimens shall be observed; momentary increases in tension that do not persist for more than a few seconds, however, shall be ignored. The average force necessary to strip the six specimens shall be reported as the adhesion value in pounds per inch of width.

4.4.1.15 Adhesion to painted surfaces.

4.4.1.15.1 **Cleaning of test panels.** This procedure shall be used in cleaning and painting the steel panels used to determine adhesion to painted surface and shrinkage 4.4.1.15.2 and 4.4.1.15.3. The panels shall be buffed lightly, using steel wool and lacquer thinner. Rust and corrosion spots shall be removed by sanding with No. 240 sandpaper or emery cloth. The panels shall be cleaned with lacquer thinner and a soft bristle brush and wiped dry with a clean cloth.

4.4.1.15.2 Painting of test panels.

*4.4.1.15.2.1 **Type I.** Two coats of zinc chromate primer conforming to TT-P-1757 type I, shall be applied to the cleaned panel by spraying at $77^{\circ} \pm 5^{\circ}$ F and a relative humidity of 50 ± 5 percent in a well-ventilated room. The primer shall then be allowed to dry for 24 hours after each coat. Then, two coats of enamel conforming to TT-E-529, class B, shall be applied over the primer by spraying, allowing 24 hours drying time between coats. During the painting operation, all excess paint accumulated on the lower edges of the panels shall be carefully removed. After the panels have received their final coat of paint, they shall be allowed to dry for 48 hours at $77^{\circ} \pm 5^{\circ}$ F and 50 ± 5 percent relative humidity in a vertical position in a well-ventilated room but out of drafts and the direct rays of the sun. The painted panels shall then be placed in an oven and baked for 8 hours at 300° F. The dry film thickness for the four coats (primer and paint) shall be not less than 3 mils.

4.4.1.15.2.2 **Type II.** Test panels shall be spray coated with a 0.001-inch coating of TT-E-485, type IV, enamel. The enamel shall be air dried at room temperature for 75 to 96 hours.

4.4.1.15.3 **Adhesion to painted surfaces.** Six test panels shall be prepared from cold rolled steel 0.035 inch thick. The panels shall be cleaned as specified in 4.4.1.15.1 and painted as specified in 4.4.1.15.2.1 for type I and 4.4.1.15.2.2 for type II. The painted surface shall be sprayed with coating compound as specified in 4.4.1.3 to yield a dry coating 0.035 ± 0.005 inch thick. The adhesion test shall be carried out as specified in 4.4.1.14 on three of these panels and on the other three after the panels have been subjected to 1500 hours of accelerated weathering (4.4.1.13).

4.4.1.15.4 **Shrinkage.** Test specimens shall be prepared on the painted surfaces of an angle iron. A section 8 inches long shall be cut from a standard 4- by 4-inch steel angle iron conforming to MIL-S-20166. All burrs and sharp edges shall be removed and then the angle iron shall be cleaned and painted according to the procedures described in 4.4.1.15.1 and 4.4.1.15.2.1 or 4.4.1.15.2.2 as applicable. The four flat surfaces shall be spray coated with a film 0.035 ± 0.005 inch thick. When testing for type II, the material shall be applied drier during the first two passes than the succeeding ones. The film shall be dried for 96 hours at room temperature during which time they shall be periodically examined for evidence of bridging.

4.4.2 Chemical analysis.

4.4.2.1 **Nonvolatile content.** The nonvolatile content of the coating compound shall be determined in accordance with method 4041 of FED-STD-141, with the following two

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exceptions: two samples of 2 to 3 grams shall be used, and the sample shall be heated for 2 hours, cooled in a desiccator, reweighed, and then heated for 1-hour periods until the loss in weight between two successive weighings is equal to or less than 1 percent. If the two determinations differ by more than 1 percent, they shall be repeated.

4.4.2.2 Isolation of vehicle. Three grams of thoroughly mixed coating compound, accurately weighed, shall be thinned with approximately one-half its volume of methyl isobutyl ketone. The resulting mixture shall be super centrifuged until clear. The isolated vehicle shall be run off from the bottom of the centrifuge bowl into a small Erlenmeyer flask, which shall be immediately stoppered. Every precaution shall be taken to prevent evaporation of the solvent from the vehicle.

4.4.2.3 Vinyl resin content. The entire 4.4.2.2 quantity of clear vehicle isolated as specified in 4.4.2.2 shall be placed in a 250-ml Erlenmeyer flask, 25 ml of methyl isobutyl ketone added, and the solution mixed thoroughly. With constant swirling, 100 ml of 95 percent ethyl alcohol shall be added to the flask. The mixture shall be filtered using a weighed fritted-glass crucible of coarse porosity containing an additional mat of asbestos fiber, and the precipitate in the crucible shall be washed with 95 percent ethyl alcohol. The crucible shall be dried at 60° C for 3 hours, cooled in a desiccator, weighed, and then heated for 1-hour periods at the same temperature (60° C) until the loss in weight between two successive weighings is less than 1 percent. The percentage of vinyl resins in the nonvolatile content of the coating compound shall be calculated from the weight of precipitate, the original sample weight, and the percent nonvolatile (4.4.2.1), as follows:

$$\text{Percent vinyl resin} = \frac{\text{weight of precipitate} \times 10.000}{\text{weight of original sample} \times \text{percent nonvolatile}}$$

4.4.2.4 Lead chromate content. (Type II only). The lead chromate content of the dry coating shall be determined in accordance with method 7111, par. 3, of FED-STD-141, except that a 1-gram sample of dry coating, accurately weighed, shall be used and the following procedure followed in lieu of the specified digestion with concentrated sulfuric acid: A 1-gram sample of coating, accurately weighed, shall be placed into a 40-ml nickel crucible, and the crucible ignited at a dull red heat until all organic matter is destroyed. The crucible shall be half-filled with sodium hydroxide pellets which shall be fused at a moderate temperature for approximately 10 minutes. The crucible shall be allowed to cool at room temperature until the fusion mixture just begins to solidify. The mixture shall be immediately poured into a 600-ml pyrex beaker, into which the crucible shall also be placed. After sufficient cooling, 300 ml of water shall be added and the beaker heated until the fusion mixture dissolves. The crucible shall be removed from the beaker and washed with hot water so that the wash water is added to the solution in the beaker. The solution

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shall be slowly neutralized with 5 N nitric acid, adding an excess of 15 to 20 ml. Heat to boiling and continue the analysis as specified, beginning with paragraph 3.2.1 of method 7111 of FED-STD-141. Calculate the lead chromate content as a percentage of the dry coating (nonvolatile) to determine compliance with 3.1.

4.4.2.5 **Toluene content.** The solvents may be collected by vacuum distillation as in method 5172 of FED-STD-141 and the toluene content determined as follows:

a. To 25 ml of the solvent in a 50-ml glass-stoppered graduated cylinder, add 25 ml of 85 percent phosphoric acid. Mix, cool to room temperature, and shake vigorously. Allow to stand 45 minutes; if toluene is present 80 percent of it will separate as the upper layer. Record the volume of the upper layer and correct by multiplying by the factor 1.25. Corrected volume shall not exceed 5 ml.

b. Pipet 2 or 3 ml of the upper layer into a small separatory funnel and wash twice with 50-100 ml of water. Draw off and discard as much water as possible, add a few grams of anhydrous sodium sulfate and shake. Pour out through the top of the funnel, 1 or 2 ml of the dried solvent, into a "percolator" type boiling point apparatus¹ and determine the boiling point. Solvent must boil at $110^{\circ} \pm 2^{\circ} \text{C}$.

4.4.2.6 **Methyl ethyl ketone content.**

4.4.2.6.1 **Reagents.**

a. Indicator solution. Mix together 10 ml pyridine, 5 ml of 0.04 percent bromphenol blue (in 95 percent ethanol), and 1.5 ml of 0.1 percent aqueous congo red solution, and dilute to 500 ml with 95 percent ethanol.

b. Alkali. Dissolve 20 grams of NaOH in 1 liter of absolute methanol, filter if cloudy before using. (Standardize against standard acid, using phenolphthalein as indicator.)

c. Hydroxylamine hydrochloride. Dissolve 3.5 grams of hydroxylamine hydrochloride in 16 ml of water and dilute to 100 ml with 95 percent ethanol.

d. The indicator solutions and hydroxylamine solutions used on any set of samples and blank, must be from the same source; i.e., be sure of an adequate supply of reagents before starting the analysis.

¹Shriner & Fuson, "Systematic Identification of Organic Compound," 2d Edition, page 92. (An electric heater may be substituted for the Bunsen burner.)

MIL-C-16555D**4.4.2.6.2 Procedure.**

a. To prepare the solvent for drawing of sample aliquots: Weigh a 100-ml volumetric glass-stoppered flask which contains 20-30 ml of 95 percent ethanol. By means of a pipet, add 10 ml of the solvent collected in 4.4.2.5, stopper, swirl and reweigh to determine sample weight. Dilute to mark with 95 percent ethanol.

b. In a 250-ml glass-stoppered Erlenmeyer flask, combine 25 ml of the hydroxylamine hydrochloride reagent and 100 ml of the indicator solution. Add exactly 10 ml (pipet) of the above prepared sample, stopper the flask and agitate frequently over a period of 5 minutes. Prepare a blank in which 10 ml of 95 percent ethanol are substituted for the 10 ml of sample. Titrate slowly and carefully, with the methanolic NaOH, against a bright light source, until each sample matches the color of the blank when viewed through the solution.

4.4.2.6.3 Calculation.

$$\text{Percent ketone} = \frac{\text{ml NaOH} \times \text{normality NaOH} \times 0.0721 \times 100}{\text{weight of sample} \times 0.1}$$

4.4.2.7 Rosin and rosin derivatives. Determine presence of rosin or rosin derivatives in accordance with method 5031 of FED-STD-141.

4.4.2.8 Total pigment content. Transfer a weighted portion of approximately 10 grams (weighed in a Lunge weighing bottle) to a pear-shaped centrifuge tube and dilute with methyl ethyl ketone to the full mark of the tube. Stopper with cork stopper and shake until all of the sample is in suspension. Centrifuge for 1 hour at 1500 rpm, then remove supernatant liquid by suction, using a suitable trap. Refill the tube to the halfway mark with methyl ethyl ketone and using a smooth glass stirring rod, break up the solids in the bottom of the tube, wash the stirring rod with methyl ethyl ketone from a wash bottle and fill to the full mark. Shake until all solids are in suspension and repeat the centrifuging as above. Repeat the above operation until all resins and plasticizers are removed (usually four or five times). After all resins and plasticizers are removed, as indicated by the nonstickiness of the solids, the operation is repeated one time using ethyl ether. The solids are jarred or bumped out of the lower stem while damp into the large part of the tube. The tube is allowed to air dry until ether fumes have left the tube, then the tube dried at 70° C for 16 hours.

$$\text{Percent total pigment content} = \frac{\text{weight of solids taken from tube}}{\text{percent of non-volatiles} \times \text{sample weight}} \times 100$$

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Percent resin and plasticizer = 100 minus percent total pigment content

Percent blue lead and other solids = percent total pigment content minus percent (Al plus TiO₂)

4.4.2.9 Titanium dioxide. Dissolve the solids taken from the centrifuge tube in 15 ml of 1:1 hydrochloride acid. Boil. Dilute with 50 ml hot water (reserve the filtrate for aluminum determinations). Wash the residue from the filter paper with hot water. Evaporate the solution obtained to dryness. COOL. Cautiously add 30 ml of concentrated sulphuric acid and 10 grams of ammonium sulphate. Boil 10 to 15 minutes until SO₃ fumes are evolved or until it is apparent that residue is composed of siliceous matter. Cool, dilute with 100 ml water, heat to boiling and filter. Wash the insoluble residue with 50 ml cold 5 percent (by volume) sulphuric acid. Add 20 ml water and 30 ml concentrated ammonium hydroxide to lower the acidity to approximately 5 percent by volume. Wash out a Jones reductor with 5 percent sulphuric acid and water leaving enough water to just cover the zinc. Empty the receiver and add 25 ml of freshly prepared ferric ammonium sulphate (100 grams/liter) and proceed as follows: (1) Run 50 ml of 5 percent by volume sulphuric acid through the reductor. (2) Run the prepared solution containing the titanium through the reductor. (3) Run through 100 ml 5 percent sulphuric acid followed by 100 ml of water. Release suction, wash tube which was immersed in the solution and titrate immediately with 0.1 N KMnO₄ (1 ml KMnO₄ = 0.008 gram TiO₂).

Calculation:

$$\frac{\text{ml KMnO}_4 \times \text{N KMnO}_4 \times .08 \times \text{percent total solids}}{\text{weight of solids from tube}} = \text{percent TiO}_2$$

4.4.2.10 Aluminum. Evaporate the filtrate reserved from the titanium residue separation to less than 100-ml volume, add 1 or 2 drops of phenol red, add ammonium hydroxide until color just changes from yellow to red then bubble hydrogen sulfide gas through the solution 15 minutes, filter to remove any lead, add indicator and neutralize. Filter through a tared, ignited Gooch crucible (asbestos filter bed) and wash with water to which has been added 1 or 2 drops of ammonium hydroxide. Dry crucible in oven at 105° C then ignite in a muffle furnace. Cool crucible in desiccator and weigh. Increase in weight is Al₂O₃.

$$\text{Percent aluminum} = \frac{\text{increase in weight of crucible} \times 0.5292 \times \text{Percent pigment content}}{\text{weight of sample from tube}}$$

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4.4.2.11 **Chlorinated paraffins.** The filtrate obtained in the vinyl resin determination as specified in 4.4.2.3 shall be transferred to a 500-ml flask, with the aid of 50 ml of absolute ethyl alcohol. To this solution shall be added 50 ml of 1.0 N alcoholic potassium hydroxide, and the mixture then refluxed for 1 hour and filtered. The precipitate shall not contain appreciable quantities of potassium chloride as indicated by a white precipitate formed by the addition of silver nitrate reagent solution to a nitric acid solution of the separated salts.

5. PREPARATION FOR DELIVERY

5.1 Packaging.

5.1.1 **Level A.** Unless otherwise specified, the coating compound shall be packaged in 5-gallon pails or 55-gallon drums. Five-gallon pails shall conform to PPP-D-760, type II. Exterior coating shall be olive drab or as specified in the contract or order. Fifty-five-gallon drums shall conform to PPP-D-729, type IV.

5.1.2 **Level C.** Coating compound shall be packaged in accordance with manufacturer's commercial practice.

5.2 Packing.

5.2.1 When specified containers of coating compound shall be palletized in accordance with MIL-STD-147.

5.2.2 **Levels A and B.** No overpacking of containers specified in 5.1.1 is required.

*5.2.3 **Level C.** The coating compound shall be packed to insure carrier acceptance and safe delivery to destination at lowest rates. Containers shall comply with Uniform Freight Classification Rules, or National Motor Freight Classification Rules.

*5.3 **Marking.** In addition to any special marking required by the contract or order, containers shall be marked in accordance with MIL-STD-129 and shall include the date of manufacture. Each container shall have affixed a warning label of appropriate size conforming to the requirements of FED-STD-313. Each container shall also have conspicuously marked on the cover as follows: "CAUTION! Reseal container immediately after withdrawals.

6. NOTES

6.1 **Intended use.** This material is intended for use as a strippable, sprayable, protective coating for application on painted or unpainted metal surfaces to seal openings and to provide protection to equipment while in storage or during shipment.

6.2 **Ordering data.** Procurement documents should specify the following:

- (a) Title, number, and date of this specification
- (b) Type and class of coating compound required (see 1.2)
- (c) Quantity
- (d) Additional packing, if required (see 5.2)
- (e) Level of packaging required.
- (f) Palletizing if required (see 5.2.1).

6.3 **Toxicity.** Any questions regarding toxicity should be referred to the cognizant departmental medical authority (see 3.21).

6.4 **Resins and plasticizers.** The following virgin unprocessed vinyl resins have been tested and found to be acceptable for formulating type II compounds: VYNS, VYHH, VMCH, Geon 400 X 74L, Geon 400 X 110, Exon 470, and Exon 450. A minimum polymeric plasticizer content of 50% of the total plasticizer has been found to be acceptable for formulating the type II compound.

6.5 **Caution.** The prospective user should test a specimen air dried painted surface before the coating is utilized on finished air dried painted surfaces of equipment. Saturate a cloth with methyl ethyl ketone and rub the painted surface for 1 minute to determine whether any deleterious effect such as wrinkling, peeling or other deterioration occurs other than a simple loss of gloss.

*6.6 **Application method.** Application shall be accomplished by spraying in multiple passes, with successive passes at approximately right angles, until a sufficient thickness of material is applied to provide a dry coating thickness of 0.035 to 0.040 inch.

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*6.7 The margins of this specification are marked with an asterisk to indicate where changes (additions, modifications, corrections, deletions) from the previous issue were made. This was done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in these notations. Bidders and contractors are cautioned to evaluate the requirements of this document based on the entire content irrespective of the marginal notations and relationship to the last previous issue.

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