#### MILITARY SPECIFICATION

# COATING, PROTECTIVE, SOLVENT REMOVABLE, FOR MICROCIRCUITS

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

#### 1. SCÔPE

- 1.1 Scope. This specification covers solvent-soluble conformal coatings which are suitable for application to monolithic and hybrid microcircuits. The purpose of the coatings is to provide a dielectric barrier to the circuitry relative to particulate contamination. This document does not constitute either an U.S. Air Force or a DoD endorsement of coatings for protection against particulate contamination in microcircuits. The use of such coatings must be approved by the procuring activity.
- 1.2 Classification. The conformal coatings shall be based on one of the following types, as specified:
  - Type I Solvent-soluble block copolymer of silicone and organic resins.
     Type II Solvent-soluble block copolymer of halocarbon resins.
  - 2. APPLICABLE DOCUMENTS
  - 2.1 Government documents.
- 2.1.1 Specifications and standards. Unless otherwise specified, the following specifications and standards of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: AFWAL/MLSE, Wright-Patterson AFB, OH 45433 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

#### **SPECIFICATIONS**

#### FEDERAL

BB-N-411 PPP-C-96 PPP-C-2020	<ul> <li>Nitrogen, Technical.</li> <li>Can, Metal, 28 Gage and Lighter.</li> <li>Chemical, Liquid, Dry, and Paste, Packaging of.</li> </ul>
PPP-P-704	- Pail, Metal (Shipping, Steel, 1 Through 12 Gallon).

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MIL-M-38510	- Microcircuit, General Specification for.
MIL-C-81302	- Cleaning Compound, Solvent,
	Trichlorotrifluoroethane

#### **STANDARDS**

#### **FEDERAL**

FED-STD-141	<ul> <li>Paint, Varnish, Lacquer, and Related Materials,</li> </ul>
	Methods of Inspection, Sampling and Testing.
FED-STD-313	- Material Safety Data Sheets Preparation and the
	Submission of

#### MILITARY

MIL-STD-129	- Marking for Shipment and Storage.
MIL-STD-202	- Test Methods for Electronic and Electrical
	Component Parts.
MIL-STD-883	- Test Methods and Procedures for
	Microelectronics.
MIL-STD-45662	- Calibration Systems Requirements.

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

2.1.2 Other Government documents, drawings, and publications. The following other Government documents form a part of this specification to the extent specified herein.

# DEPARTMENT OF HEALTH, EDUCATION AND WELFARE

Federal Hazardous Substances Labeling Act.

(Application for copies should be addressed to the U.S. Department of Health, Education and Welfare, Food and Drug Administration, Washington, D.C. 20203.)

## DEPARTMENT OF DEFENSE (DD)

DD Form 1813 - Material Safety Data Sheet.

#### DEPARTMENT OF LABOR

OSHA Form 20 - Material Safety Data Sheet.

(Copies of DD Form 1813 should be obtained from the local Contract Administration Services Component. Copies of OSHA Form 20 should be obtained from the cognizant regional office of the Bureau of Labor Standards.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

## AMERICAN SOCIETY FOR TESTING MATERIALS (ASTM)

ASTM D 149	- Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commer- cial Power Frequencies, Tests for.
ASTM D 150	- A-C Loss Characteristics and Permittivity (Dielectric
73111 0 130	Constant) of Solid Electrical Insulating Materials,
	Tests for.
ASTM D 257	- D-C Resistance or Conductance of Insulating
	Materials, Tests for.
ASTM D 1644	- Nonvolatile Content of Varnishes, Tests for.
ASTM D 2267	- Aromatics in Light Naphthas, Reformates, and Gaso-
	lines by Gas Chromatography, Test for.
ASTM D 3850	- Rapid Thermal Degradation of Solid Electrical
	Insulating Materials by Thermogravimetric Method,
	Test for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

#### 3. REQUIREMENTS

3.1 <u>Materials</u>. The coatings formulated from synthetic resins, elastomers, and solvents shall be clear (unpigmented). Materials not definitely specified shall be of the quality regularly used in the commercial processing of microcircuits.

- 3.2 Volatile content. Type I coatings and the thinners furnished with these coatings may contain solvents that are photochemically reactive because of the solubility requirements of the type I resins. Where resin solubilities permit, the use of nonphotochemically reactive solvents shall be required. Nonphotochemically reactive solvents are defined in 3.3. The solvent composition of types I and II coatings shall be determined by the use of a gas chromotograph or other suitable device in accordance with ASTM D 2267. The shipping container shall be marked relative to the photochemical reactivity of the volatile content (see 5.2).
- 3.3 Nonphotochemically reactive solvents. A nonphotochemically reactive solvent shall be any solvent combination with an aggregate of less than 20 percent of its total volume composed of the chemical compounds classified below and shall not exceed any of the following individual volume percentage composition limitations, determined in accordance with 3.2.
  - a. A combination of hydrocarbon, alcohols, aldehydes, esters, ethers, or ketones having an olefinic or cycloolefinic type unsaturation: 5 percent.
  - b. A combination of aromatic compounds with eight or more carbon atoms to the molecule, except ethylbenzene: 8 percent.
  - c. A combination of ethylbenzene, ketones having branched hydrocarbon structures, trichloroethylene, or toluene: 20 percent.

# 3.4 <u>Coating solution properties</u>.

- 3.4.1 Storage life. The material as furnished by the manufacturer shall meet the requirements specified herein when tested at any time up to 12 months after receipt by purchaser provided the material has been stored in the original unopened containers at a temperature not exceeding either  $39^{\circ}\text{C}$   $(100^{\circ}\text{F})$ , or the highest temperature recommended by the manufacturer. When temperatures above these limits are expected either in storage or shipping, provisions for refrigeration shall be made. Storage life shall be determined in accordance with 4.4.2.
- 3.4.2 <u>Appearance</u>. The material shall be homogeneous, clear, and free of particles when inspected as specified in 4.4.3.
- 3.4.3 Nonvolatile content. The nonvolatile content shall be within 1 percentage point of the value certified by the manufacturer. The nonvolatile content shall be determined in accordance with 4.4.4.
- 3.4.4 <u>Ionic content</u>. The ionic content for both type I and II coating shall be  $\leq 10$  parts per million as NaCl, determined in accordance with 4.4.5.

# 3.5 Coating application requirements.

- 3.5.1 Working properties and appearance. Spray application parameters shall be furnished by the manufacturer. The applied, dried film shall be free from bubbles, pinholes, white spots, blisters, wrinkles, cracks and peeling when visually examined in accordance with 4.4.3. The dried coating shall not mask any identification markings. All circuitry must be visible after coating has been applied and dried.
- 3.5.2 Coating cure time. The coating materials when applied in accordance with 4.4.1.1 shall be dried to full hardness under the time and temperature conditions recommended by the supplier. The selected drying schedule shall be chosen such that the microcircuit assembly is not exposed to a temperature exceeding that of any prior assembly process.
- 3.5.3 Coating thickness. Dry film thickness on hybrid circuits shall be 0.0127 to 0.0381 millimeter (mm) (0.0005 to 0.0015 inch) for conformance to 4.4.1.1. Thick film specimens for bulk electrical property testing shall have a dry film thickness of 0.5 to 1.27 mm (0.020 to 0.050 inch). Thick film specimens are prepared in accordance with 4.4.1.2.

# 3.6 Properties of dried coatings.

3.6.1 <u>Electrical properties</u>. Electrical properties shall be as specified in table I, determined by the test method paragraph specified therein.

Electrical property	Requirements		Test method paragraph
	Type I	Type II	paragrapii
Volume resistivity, ohm/cm	$\geq 1 \times 10^{14}$	$\geq 1 \times 10^{11}$	4.4.6
Surface resistivity, ohm	$\geq$ 1 x $10^{12}$	$\geq$ 1 × 10 <sup>13</sup>	4.4.7
Dielectric constant, at 10 <sup>3</sup> Hz	≤ 2.75	≤ 3.75	4.4.8
Dissipation factor, at 10 <sup>3</sup> Hz	≤ 0.0005	≤ 0.060	4.4.9
Dielectric strength, volts per mm	≥ 23,600	≥ 31,500	4.4.10
(V/mil)	(≥ 600)	(≥ 800)	

TABLE I. Electrical properties.

<sup>3.6.2</sup> Solvent removability. The coatings shall be solvent removable and shall leave no evidence of coating residue, determined in accordance with 4.4.11.

3.6.3 Thermal stability. Loss of weight after exposure to elevated temperature shall not exceed the value specified in table II, determined in accordance with 4.4.12.

Test temperature	Weight loss, %		
rest temperature	Type I	Type II	
200°C (390°F)	≤ 0.40	≤ 0.30	
240°C (465°F)	≤ 0.70	≤ 0.50	
300°C (575°F)	≤ 1.75	≤ 1.25	

TABLE II. Weight loss.

- 3.6.4 Thermal shock resistance. The wire bonds on the test specimens shall exhibit a bond strength of not less than 3.0 grams (g) after temperature cycling, and a bond strength of not less than 2.5 g after coating removal, determined in accordance with 4.4.13. If bond failures occur in the control specimens, the test shall be repeated one time.
- 3.6.5 <u>Microcircuit compatibility</u>. Coated microcircuits shall meet the endpoint electrical parameters specified in the applicable specification for the microcircuit, determined in accordance with 4.4.14.

#### 4. QUALITY ASSURANCE PROVISIONS

- 4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor 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.
- 4.1.1 <u>Test equipment and inspection facilities</u>. Test and measuring equipment and inspection facilities of sufficient accuracy, quality, and quantity to permit performance of the required inspection shall be established and maintained by the supplier. The establishment and maintenance of a calibration system to control the accuracy of the measuring and test equipment shall be in accordance with MIL-STD-45662.
- 4.1.2 <u>Inspection conditions</u>. Unless otherwise specified herein, all inspection shall be performed in accordance with the test conditions specified in the "GENERAL REQUIREMENTS" of MIL-STD-202 and the requirements of MIL-STD-883.

- 4.2 Classification of inspection. The tests required herein are classified as either quality conformance tests or quality acceptance tests.
- 4.2.1 Quality conformance. To demonstrate quality conformance, the manufacturer (see 4.1) shall conduct all the tests required herein for his certification.
- 4.2.2 <u>Quality acceptance</u>. Test requirement paragraphs 3.4.2, 3.4.3, 3.4.4, 3.5.2, and 3.6.2 shall be used by the procuring agency for lot acceptance inspections.
- 4.3 <u>Material safety data sheets</u>. For each coating material the manufacturer shall furnish Material Safety Data Sheets, DD Form 1813 or OSHA Form 20, or a reasonable facsimile thereof, which state the pertinent toxicological data and formulations required to evaluate the safety of the material for the proposed use. Material safety data sheets shall be prepared and submitted to the procuring activity in accordance with FED-STD-313.

# 4.4 Test methods.

## 4.4.1 Test specimens.

- 4.4.1.1 Thin film specimens, 0.0127 to 0.0381 mm (0.0005 to 0.0015 inch) thick. Thin film specimens with final dry film thickness of 0.0127 to 0.0381 mm (0.0005 to 0.0015 inch) shall be prepared by spray coating the material on a suitable substrate. The maximum temperature in the drying schedule of the coated microcircuit shall not exceed that of the highest temperature used in prior microcircuit assembly processing. The elevated temperature exposure shall be in a vented explosion-proof oven (see 6.4).
- 4.4.1.2 Thick film specimens, 0.5 to 1.27 mm (0.020 to 0.050 inch) thick. Thick film specimens with final dry film thickness of 0.50 to 1.27 mm (0.020 to 0.050 inch) shall be prepared by casting the material on a sheet of clean polytetrafluoroethylene, air drying at room temperature for not less than seven days, followed by heating to  $50^{\circ}\text{C}$  ±1 (122°F ±2) and holding at temperature for 24 hours ±0.25, raising temperature to  $150^{\circ}\text{C}$  ±5 (302°F ±9) and holding at temperature for not less than 1.0 hour. The elevated temperature exposures shall be in a vented explosion-proof oven. (See 6.4).
- 4.4.1.3 Glass panels. Two clear glass panels or microscope slides, approximately  $\frac{1}{1}$  by 25 by 75 mm (0.040 by 1.0 by 3.0 inch) shall be prepared in accordance with method 2021 of FED-STD-141 and coated in accordance with 4.4.1.1.
- 4.4.2 Storage life. A container of material in the original unopened container shall be stored under the conditions specified in 3.4.1. At the end of the storage period, the material shall meet the requirements specified in 3.4.2, 3.4.3, 3.4.4, 3.5.2, and 3.6.2.

- 4.4.3 Appearance. The material shall be visually examined in the container for conformance to 3.4.2. The dried coating prepared in accordance with 4.4.1.3 shall conform to 3.5.1.
- 4.4.4 Nonvolatile content. The nonvolatile content shall be determined in accordance with ASTM D 1644 for conformance to 3.4.3.
- 4.4.5 <u>Ionic content</u>. The ionic content shall be determined by the following procedure:
  - a. A magnetic stirrer is placed in a clean ion-free polyethylene bottle or Erlenmeyer flask. A total of 50 milliliters (mL) of high purity trichlorotrifluoroethane (MIL-C-81302) is pipetted into the container and stirring is initiated. After stirring for several minutes, 100 mL of deionized water is pipetted into the container under continuous stirring. After separation of the two liquids, the conductivity of the water is measured. This process is repeated three additional times using fresh trichlorotrifluoroethane each time. The average of the four measurements is recorded as the blank. The conductivity of the blanks shall be less than 0.5 micromhos/centimeters (µmho/cm) and the difference between the high and low values shall not exceed 0.05 µmho/cm.
  - b. The container is then weighed to the nearest 0.01 g and approximately 15 g of sample added and weighed to the nearest 0.01 g. The stirrer, 50 mL trichlorotrifluoroethane, and 100 mL of deionized water are added as before and the conductivity again measured after two minutes of stirring.
  - c. The content of soluble ionizable material as sodium chloride in the dried coating is calculated using the following equation:

$$\frac{47.0 (L_2 - L_1)}{WF}$$
 = parts per million as NaCl

where:  $L_2$  = conductance value of sample ( $\mu$ mho/cm)

 $L_1$  = conductance value of blank ( $\mu$ mho/cm)

W = weight of sample in grams

F = weight fraction of solids (from 4.4.4)

47.0 = grams of NaCl per mho/cm

d. The factor 47.0 can be obtained experimentally or calculated as follows:

 $\frac{A}{L_S} = 47.0 \text{ g NaC1 in 100 mL water per mho/cm}$ where: A = 2.923 x  $10^{-3}$  g NaC1 in 100 mL 0.0005N NaC1  $L_S = 6.225 \times 10^{-5} \text{ mho/cm} = \text{specific conductance}$ of 0.0005N NaC1

- 4.4.6 <u>Volume resistivity</u>. The volume resistivity, using thick film specimens prepared in accordance with 4.4.1.2, shall be determined in accordance with ASTM D 257 for conformance to table I.
- 4.4.7 <u>Surface resistivity</u>. The surface resistivity, using thick film specimens prepared in accordance with 4.4.1.2, shall be determined in accordance with ASTM D 257 for conformance to table I.
- 4.4.8 <u>Dielectric constant</u>. The dielectric constant, using thick film specimens prepared in accordance with 4.4.1.2, shall be determined in accordance with ASTM D 150 for conformance to table I.
- 4.4.9 <u>Dissipation factor</u>. The dissipation factor of the material, using thick film specimens prepared in accordance with 4.4.1.2, shall be determined in accordance with ASTM D 150 for conformance to table I.
- 4.4.10 Dielectric strength. The dielectric strength, using thick film specimens prepared in accordance with 4.4.1.2, shall be determined in accordance with ASTM D 149 for conformance to table I.
- 4.4.11 Solvent removability. Two clear glass panels shall be prepared in accordance with 4.4.1.3. The panels shall be placed in a Soxhlet extractor containing trichlorotrifluoroethane solvent (MIL-C-81302), subjected to four extraction cycles, and examined visually under 10 times magnification for conformance to 3.6.2.
- 4.4.12 Thermal stability. The thermal stability shall be determined on three specimens of approximately 10 milligrams each of film material produced as specified in 4.4.1.2 and peeled from the substrate, and in accordance with ASTM D 3850. Tests shall be performed by heating the specimens from room temperature to not less than  $350^{\circ}\text{C}$  ( $650^{\circ}\text{F}$ ) at a heating rate not to exceed  $10^{\circ}\text{C}$  ( $18^{\circ}\text{F}$ ) per minute in a nitrogen atmosphere (BB-N-411, type I, class 1, grade B) with 20 mL per minute nitrogen flow. The temperatures noted in table II shall be marked on the temperature/weight loss curve developed for each specimen.
- 4.4.13 Thermal shock resistance. Test specimens shall be prepared using silicon devices containing not less than 15 aluminum metallized bonding pads (may be nonfunctional devices). The devices shall be adhesive bonded into packages containing not less than 15 leads. Gold wires 0.0254 mm (0.001 inch)

in nominal diameter shall be thermosonically or thermocompression bonded from the package leads to the device bonding pads. A total of eight specimens shall be prepared. Five wires on each specimen shall be tested in tension to failure in accordance with MIL-STD-883, method 2011, condition C. The wires selected for destructive test shall be from identical locations on each specimen and should be representative of the bonding configuration (length and dress) contained on the specimens. The remaining wires shall be proof-loaded (nondestructively tested) with a 3 g load. If any of the wires on the specimen show bond strength less than 3.0 g, a new specimen shall be prepared. Four of the specimens shall then be spray coated and dried in accordance with 4.4.1.1. If the coating forms bridges between adjacent wires or from the wires to the device or package, the coating shall be removed using trichlorotrifluoroethane (MIL-C-81302) in a vapor degreaser or Soxhlet extractor. The specimens may then be recoated in accordance with 4.4.1.1. The four uncoated control specimens shall be subjected to the same cleaning and drying processes as the coated specimens. The coated specimens and uncoated control specimens shall then be thermally cycled in accordance with MIL-STD-883, method 1010, condition C, for 100 cycles in a dry nitrogen atmosphere. In order to prevent frost damage, the test environment shall be nitrogen gas ambient with a dewpoint of less than -65°C (-85°F). An additional five wires on each specimen shall be tested in tension to determine the bond strength. The coating shall then be removed from each specimen as specified above and the remaining wires on each specimen shall be tested in tension to determine the bond strength to establish compliance with 3.6.4.

4.4.14 Microcircuit compatibility. Five microcircuits (equivalent to those to be procured) shall be coated as specified in 4.4.1.1 and hermetically sealed in accordance with the applicable assembly specification. The microcircuits shall be screen tested in accordance with the applicable procurement specification defining microcircuit performance or to a minimum level of class B. Circuits failing the screen tests may be reworked in accordance with MIL-M-38150 provisions. For acceptance after rework, the microcircuits shall be subjected to and pass the complete screening sequence specified above. After passing the screening tests, the five coated microcircuits shall be steady state life tested in accordance with MIL-STD-883, method 1005, for not less than 1000 hours at 125°C (260°F). After passing the steady state life test, the five microcircuits shall be tested in accordance with MIL-STD-883, method 1018 and pass the requirements as specified in test methods 5005 or 5008, and shall conform to 3.6.5.

#### 5. PACKAGING

- 5.1 Preservation. Preservation shall be level A or Commercial as specified (see 6.2).
- 5.1.1 <u>Level A.</u> Preparation for delivery shall be in accordance with PPP-C-2020, type II, class 1 or 2 as appropriate. Level A unit containers

shall be limited to pint (one-half liter [L]), quart (L), or gallon (4 L) cans conforming to II, as specified (see 6.2). Cans conforming to PPP-C-96 shall be exteriorly coated in accordance with Plan B with side seams stripped with a suitable corrosion-resistant coating. The 1-gallon (4 L) cans and the 5-gallon (20 L) pails for level A unit packaging shall be provided with galvanized or protectively coated wire handles. Smaller containers or kits may be used where appropriate to a manufacturing environment as requested by the procuring activity.

- 5.1.2 <u>Commercial</u>. Commercial preparation for delivery shall be in accordance with PPP-C-2020.
- 5.2 <u>Marking</u>. In addition to any special marking required by the contract or purchase order (see 6.2), each unit and shipping container shall be marked in accordance with MIL-STD-129 and the requirements of the Interstate Commerce Commission. Marking shall also include the following:
  - a. Specification number, type and photochemical reactivity (see 3.2).
  - b. Manufacturer's part number.
  - c. Manufacturer's lot number.
  - d. Name of manufacturer.
  - e. Name of contractor (if other than the manufacturer).
  - f. Date of manufacture (month and year).
  - g. Precautionary handling as follows:

    CAUTION: AVOID INHALATION OF FUMES OR EXCESSIVE SKIN CONTACT.

    KEEP AWAY FROM FACE AND EYES. WORK IN A WELL-VENTILATED AREA.

    CLEANSE SKIN THOROUGHLY WITH SOAP AND WATER AFTER CONTACT.

#### 6. NOTES

- 6.1 <u>Intended use</u>. The coating materials in conformance with this specification are intended for use as coatings to provide particulate-contamination protection to hermetically-sealed monolithic and hybrid microcircuits.
- 6.1.1 Procuring activity approval. The existence of this specification does not eliminate the necessity of proper particulate controls during microcircuit manufacture. Since the use of organic or semi-organic materials inside a microcircuit package has reliability implications, such as ionic or outgassing contamination, or both, the use of the coating materials specified herein must be approved by the procuring activity.

- 6.2 Ordering data. Procurement documents should specify the following:
  - Title, number, and date of this specification.
  - Type of coating required.
  - c. Size of unit containers (see 5.1.1).
  - d. Quantity of material desired.
  - e. Cure conditions (time/temperature) desired (see 3.5.2).
- 6.3 Ventilation. Coatings may contain toxic solvents and should be used with caution and in well-ventilated areas. Suppliers shall include a WARNING on the label of containers having material which may have a toxic effect on using personnel. Pertinent toxicological data is emphasized in the required material safety data sheets (see 4.3).
- 6.4 Oven atmosphere. A nitrogen atmosphere in a vented circulating oven with nitrogen flow of not less than 1 cubic foot per hour (0.028 m³/hour) for each cubic foot (0.028 m³) of oven volume, using commercial nitrogen, has been found satisfactory in lieu of an explosion-proof oven.
- 6.5 Use of Government data. When Government drawings, specifications, or other data are used for any purpose other than in conformance with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian:

Air Force - 20 Army - MR

Navy - EC

Other - NS

Preparing Activity: Air Force - 20

(Project No. 8030-0509)

Reviewers:

Air Force - 17, 19, 84, 99

Army - MI, ER

STAND	ARDIZATION DOCUMES (See Instruction)		NT PROPOSAL	
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