

MIL-C-47257B(MI)
 20 November 1987
 SUPERSEDING
 MIL-C-47257A(MI)

MILITARY SPECIFICATION

COMPOUND, EPOXY, FILAMENT WINDING

This specification is approved for use within the U.S. Army Missile Command, Department of the Army, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers an epoxy resin-hardener compound used in combination with fiberglass roving in the fabrication of filament wound structures.

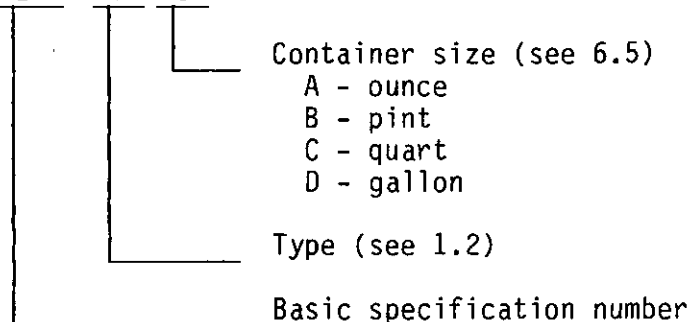
1.2 Classification. The epoxy compound shall be of the following types:

Type I - Impregnating compound.

Type II - Compound impregnated into fiberglass roving.

1.3 Military part number. The military part number shall consist of the letter B, the basic number of this specification, a number identifying type, and a letter identifying container size.

Example: B47257 - I - C



Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, U.S. Army Missile Command, ATTN: AMSMI-RD-SE-TD-ST, Redstone Arsenal, AL 35898-5270, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

AREA CMPS

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2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. The following specifications and standards form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation.

SPECIFICATIONS

MILITARY

MIL-R-60346	Roving, Glass, Fibrous (For Prepreg Tape and Roving, Filament Winding, and Pultrusion Applications)
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STANDARDS

FEDERAL

FED-STD-376	Preferred Metric Units for General Use by the Federal Government
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(Copies of specifications and standards required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting activity.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted shall be those listed in the issue of the DODISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS shall be the issue of the nongovernment documents which is current on the date of the solicitation.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D 1045	Sampling and Testing Plasticizers Used in Plastics, Methods of
ASTM D 1475	Density of Paint, Varnish, Lacquer, and Related Products, Test Methods for
ASTM D 1652	Epoxy Content of Epoxy Resins, Test Methods for
ASTM D 2073	Total, Primary, Secondary, and Tertiary Amine Values of Fatty Amines, Amidoamines and Diamines by Referee Potentiometric Method, Test Methods for

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ASTM D 2291	Fabrication of Ring Test Specimens for Glass-Resin Composites, Practice for
ASTM D 2393	Standard Test Method for Viscosity of Epoxy Resins and Related Components
ASTM D 2471	Standard Test Method for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins
ASTM D 3951	Commercial Packaging, Practice for

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

(Nongovernment standards and other publications are normally available from the organizations which prepare or which distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein (except for associated detail specifications, specification sheets or MS standards), the text of this specification shall take precedence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 First article. When specified in the contract or purchase order, a sample shall be subjected to first article inspection (see 4.3 and 6.2).

3.2 Material.

3.2.1 Composition and condition in container.

3.2.1.1 Type I material.

3.2.1.1.1 Composition. Type I compound materials shall consist of an epoxy resin, an anhydride hardener (maleic anhydride adduct of methylcyclopentadiene or methyltetrahydrophthalic anhydride) and an amine promoter (benzyltrimethylamine (BDMA)). The materials shall be furnished as individual ingredients. These ingredients, when mixed, can be used as an impregnant for fiberglass roving in filament winding applications (see 4.6.4.1).

3.2.1.1.2 Condition in container. The component materials in containers shall be free of gel particles and foreign matter that would adversely affect their intended purpose. Crystallized masses which can readily be reconstituted by heating to 49° Celsius (C) (120° Fahrenheit (F)) and stirring shall be acceptable (see 4.6.4.1).

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3.2.1.2 Type II material.

3.2.1.2.1 Composition. Type II compound shall consist of the type I material impregnated into a glass roving conforming to MIL-R-60346. The resin content of the type II material shall be 26 ± 4 percent (see 4.6.4.1.1).

3.2.1.2.2 Condition in container. The impregnated roving shall be evenly wound on a tube with negligible free liquid compound present. The material shall be readily removable from the container (aluminum alloy foil or polyethylene bag) with negligible adhesion to the container and no deterioration of the roving during removal (see 4.6.4.1).

3.2.2 Storage life.

3.2.2.1 Type I. The individual components of type I compound shall be capable of meeting the requirements of this specification after storage in the original unopened containers at temperatures between 4 and 32°C (30 and 90°F) in the absence of sunlight for not less than 12 months from the date of shipment (see 4.6.4.2).

3.2.2.2 Type II (impregnated roving). Impregnated roving, packaged as required in 5.1, shall be capable of meeting the requirements of this specification after storage in a cooler maintained at a temperature not greater than 10°C (50°F) for not less than 30 days, or after storage at room temperature for not less than 5 days (see 4.6.4.2).

3.2.3 Physical properties of individual components. The physical properties of the epoxy resin, anhydride hardener and amine promoter shall be as specified in table I.

TABLE I. Physical properties of component materials

Property	Epoxy resin		Anhydride		Amine	
	Min.	Max.	Min.	Max.	Min.	Max.
Specific gravity 25/25°C (77/77°F)	1.14	1.19	1.19	1.25	0.90	0.910
Viscosity (centipoises)	10,000	16,000	175	225		
Epoxide equivalent weight (gm. eq.)	180	195				
Amine value					9.0	
Refractive index			1.503	1.506		

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3.2.4 Properties of the mixed compound. The type I mixed compound (see 6.4) shall exhibit the following properties:

3.2.4.1 Viscosity. The viscosity, after mixing, shall be within the range of 500 to 3000 centipoises (cps) at $25 \pm 1^\circ\text{C}$ ($77 \pm 2^\circ\text{F}$) (see 4.6.4.4).

3.2.4.2 Pot life. After 24 hours at room temperature, the viscosity of the mixed compound shall not exceed 5000 cps at an ambient temperature of $25 \pm 1^\circ\text{C}$ ($77 \pm 2^\circ\text{F}$) (see 4.6.4.8).

3.2.4.3 Gel time. The mixed compound shall gel to a non-flowable solid in not more than 1 hour at $149 \pm 3^\circ\text{C}$ ($300 \pm 5^\circ\text{F}$) (see 4.6.4.9).

3.3 Performance characteristics.

3.3.1 Interlaminar shear strength. After a cure time of not more than 3 hours at $141 \pm 3^\circ\text{C}$ ($285 \pm 5^\circ\text{F}$), the interlaminar shear strength of type II material shall be not less than 5000 pounds per square inch (psi) at an ambient temperature of $25 \pm 1^\circ\text{C}$ ($77 \pm 2^\circ\text{F}$) (see 4.6.4.10).

3.4 Workmanship. The workmanship shall be such as to ensure a product which is uniform and in accordance with this specification. All the materials shall be free of dirt, foreign matter, or other contaminants (see 4.8).

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 Responsibility for compliance. All items must meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling in quality conformance does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to acceptance of defective material.

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

- a. First article inspection (see 4.3).
- b. Quality conformance inspection (see 4.4).

4.3 First article inspection. Unless otherwise specified in the contractual document (see 6.2), a sample of each type of material, manufactured under the same conditions as those proposed for subsequent production, shall be subjected to first article inspection. The sample for type I material shall consist of a one quart (0.94 liters) kit containing all the component materials, or a one quart sample of each material except the amine promoter which may be furnished as a one pint (0.47 liters) sample. For type II material, the sample shall consist of a minimum of 5 pounds (2.26 kilograms) of impregnated roving. First article inspection shall be as specified herein. Subsequent units will not be considered for acceptance until Government approval of the first article sample has been obtained. Units subjected to first article inspection shall have successfully passed the quality conformance inspection. Testing of the first article sample to determine compliance with the characteristics listed in table II shall be conducted in accordance with the corresponding test paragraphs.

TABLE II. First article inspection.

Characteristics	Requirement paragraph	Test paragraph
Storage life (type I or II)	3.2.2	4.6.4.2
Component materials (type I)		
Specific gravity of epoxy resin	Table I	4.6.4.3
Specific gravity of anhydride hardener	Table I	4.6.4.3
Specific gravity of amine promoter	Table I	4.6.4.3
Viscosity of epoxy resin	Table I	4.6.4.4
Viscosity of anhydride hardener	Table I	4.6.4.4
Epoxide equivalent weight of epoxy resin	Table I	4.6.4.5
Amine equivalent of amine promoter	Table I	4.6.4.6
Refractive index of anhydride hardener	Table I	4.6.4.7
Mixed compound		
Pot life (type I)	3.2.4.2	4.6.4.8
Interlaminar shear (type I or II)	3.3.1	4.6.4.10

4.4 Quality conformance inspection.

4.4.1 Lot formation.

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4.4.1.1 Component material. A lot shall consist of all the component material manufactured in one continuous operation by the same process by the same manufacturer in accordance with this specification and submitted for inspection at one time.

4.4.1.2 Compounded material. A lot shall consist of all the compound material mixed in a continuous operation by the same process by the same compounder using the same specified lot of component materials in accordance with this specification and submitted for inspection at one time.

4.4.2 Sampling for examination. Each container of type I component material shall be considered as a unit of product. Random selection of a container from each lot of type I component material shall be effected. From these sample component materials sufficient quantities shall be randomly appropriated for the manufacture of sufficient type II compound materials for subjection to the acceptance inspection as delineated in table III. Failure of this sample to comply with the requirements specified in table III, quality conformance, shall be cause for lot rejection.

TABLE III. Quality conformance inspection.

Characteristics	Requirement paragraph	Test paragraph
Composition and condition in container	3.2.1	4.6.4.1
Mixed compound		
Viscosity	3.2.4.1	4.6.4.4
Gel Time	3.2.4.3	4.6.4.9
Workmanship	3.4	4.8
Preservation, packaging, unitization and marking	5.1	4.7

4.5 Inspection equipment. The inspection equipment for conducting examination and tests shall be as specified in the applicable test methods and procedures paragraphs.

4.6 Test methods and procedures.

4.6.1 Test conditions. Unless otherwise specified herein, the following conditions shall be used as a basis to establish performance requirements:

- a. Temperature, room ambient (16 to 32°C) (60 to 90°F).
- b. Altitude, facility ground.
- c. Humidity, facility ambient up to 95 percent relative humidity.

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4.6.2 Test sequence. Test sequence within each classification of inspection (first article or quality conformance) shall be at the option of the supplier.

4.6.3 Test specimen preparation. Unless otherwise specified herein, specimens shall be prepared as follows:

4.6.3.1 Packaged type II material. To prevent moisture condensation on the impregnated roving, all packages of type II material which are removed from the storage cooler (see 3.2.2.2) shall be allowed to warm to room temperature for a minimum of 2 hours prior to opening the sealed bag.

4.6.3.2 Interlaminar shear specimens. The filament wound rings shall be prepared from type II material in accordance with ASTM D 2291. If supplied as type I material, the components shall be mixed and type II material prepared. The filament wound rings shall be cured for not more than 3 hours at $141 \pm 3^\circ\text{C}$ ($285 \pm 5^\circ\text{F}$). Interlaminar shear specimens shall be fabricated from the rings to the dimensions shown in figure 1.

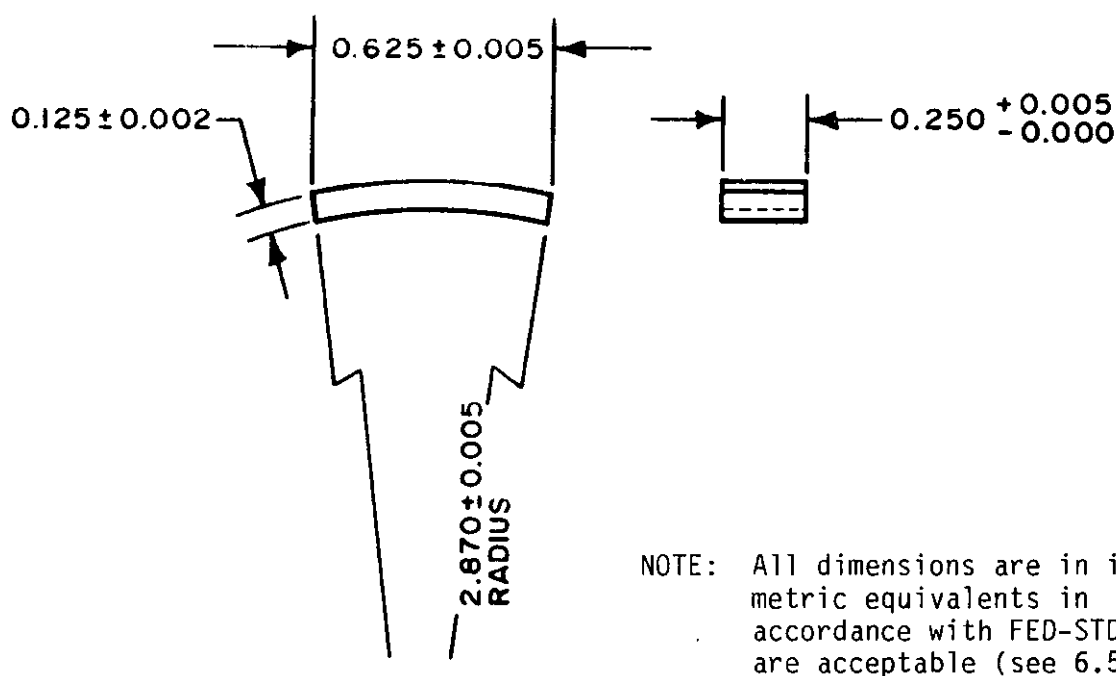


FIGURE 1. Interlaminar shear test specimen.

4.6.4 Tests precautions. Observe precautions (see 6.3) when handling the component or compounded materials.

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4.6.4.1 Composition and condition in container. The condition in containers shall be examined visually and shall conform to requirements of 3.2.1.1.2 or 3.2.1.2.2 as applicable. The composition shall be certified by the suppliers and shall conform to the requirements of 3.2.1.1.1 or 3.2.1.2.1 as applicable except that the resin content of type II material shall be determined by one of the following methods and shall conform to the requirements of 3.2.1.2.1.

4.6.4.1.1 Resin content. The preferred procedure for determining the resin content of type II material shall be as follows:

- a. Weigh empty impregnated roving spool (W_1) prior to installing on roving impregnating machine.
- b. Weigh dry spool of roving (W_4).
- c. After impregnating the roving, weigh the impregnated spool of roving (W_2), and the dry roving spool (W_4).
- d. Calculate resin content by the following formula:

$$R = 100 - 100 \frac{W_2 - W_4}{W_3 - W_1}$$

Where R = weight of impregnated roving spool

W_1 = weight of impregnated roving spool

W_2 = weight of dry roving and dry roving spool

W_3 = weight of impregnated roving and impregnated roving spool

W_4 = weight of dry roving spool

e. Record the impregnated roving spool number, net weight of impregnated roving, date, resin formula number, roving type, and percent resin content on a form affixed on the core of the impregnated roving spool.

4.6.4.1.2 Resin content, alternate method. The alternate procedure for determining the resin content of type II material shall be as follows, using pieces cut from the container in the finishing operations:

- a. Weigh specimen on an analytical balance (W_5).
- b. Burn specimen in a muffle furnace at 593 to 621°C (1100 to 1150°F) for 1 to 2 hours or until white.
- c. Cool specimen in a dessicator.
- d. Reweigh specimen on an analytical balance (W_6).

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e. Calculate resin content as follows:

$$R = \frac{W_5 - W_6}{W_6} \times 100$$

Where R = percent of resin content

W₅ = weight of specimen before burning

W₆ = weight of specimen after burning

4.6.4.2 Storage life. A sufficient amount of materials in the original unopened containers shall be stored under the conditions specified in 3.2.2. After the storage period, interlaminar shear specimens shall be prepared as specified in 4.6.3.2. The interlaminar shear specimens shall be tested as specified in 4.6.4.10 and shall conform to requirements of 3.3.1. The suppliers' certification of compliance to the storage life requirements (see 3.2.2) may be accepted during the storage period.

4.6.4.3 Specific gravity. The specific gravities of the epoxy resin, anhydride hardener, and amine promoter shall be determined in accordance with ASTM D 1475 and shall conform to table I.

4.6.4.4 Viscosity. The viscosities of the epoxy resin, the anhydride hardener, and the mixed compound shall be determined in accordance with ASTM D 2393 and shall conform to table I or 3.2.4.1, as applicable.

4.6.4.5 Epoxide equivalent weight. The epoxide equivalent weight of the epoxy resin shall be determined in accordance with ASTM D 1652 and shall conform to table I.

4.6.4.6 Amine value. The total amine value of the amine promoter (benzyltrimethylamine (BDMA)) shall be determined in accordance with ASTM D 2073 and shall conform to table I.

4.6.4.7 Refractive index. The refractive index of the anhydride hardener shall be determined in accordance with ASTM D 1045 and shall conform to table I.

4.6.4.8 Pot life. The pot life shall be determined by checking the viscosity of the mixed compound in accordance with ASTM D 2393 and shall meet the requirements of 3.2.4.2.

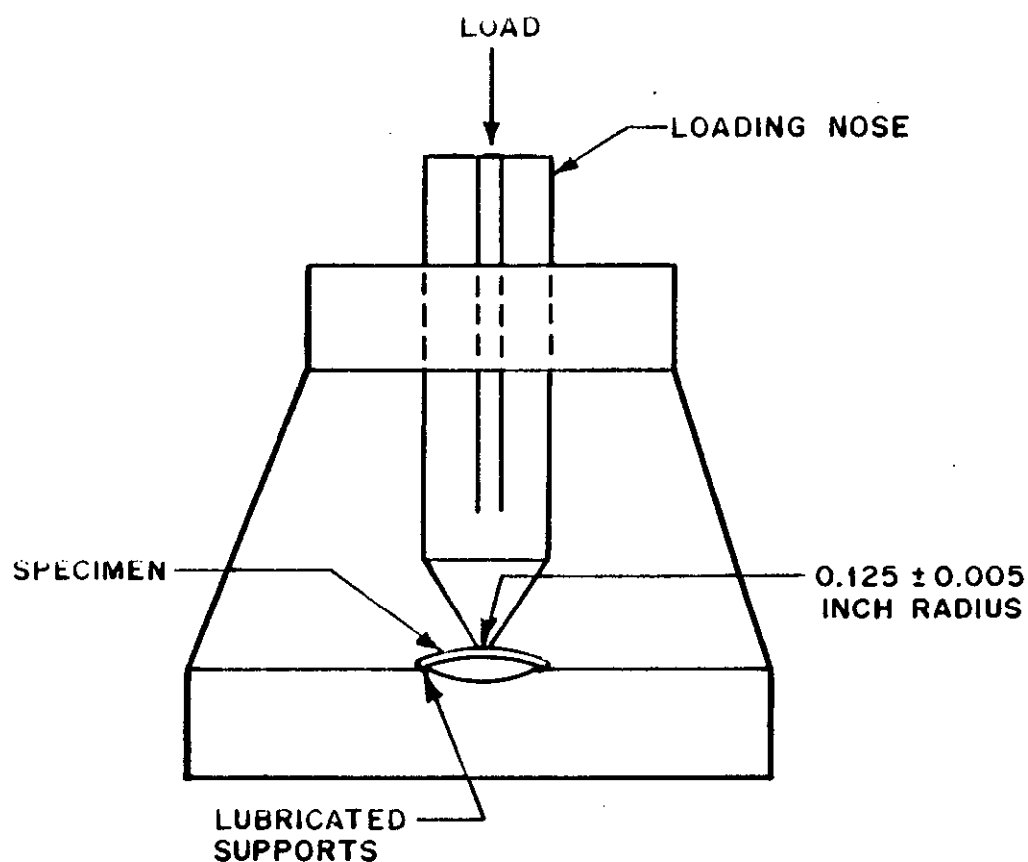
4.6.4.9 Gel time. The gel time shall be determined in accordance with ASTM D 2471 and shall conform to 3.2.4.3.

4.6.4.10 Interlaminar shear strength. The interlaminar shear strength of test specimens prepared as specified in 4.6.3.2 shall be determined as follows and shall meet the requirements of 3.3.1.

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4.6.4.10.1 Equipment. The following equipment shall be used:

- a. Compression testing machine.
- b. Horizontal shear fixture as shown in figure 2.
- c. Micrometer.



NOTE: Dimension is in inches; metric equivalent in accordance with FED-STD-376 is acceptable (see 6.5).

FIGURE 2. Typical horizontal shear fixture.

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4.6.4.10.2 Test procedure. The test for interlaminar shear strength shall be as specified below:

- a. Measure the thickness and width of the specimen to nearest 0.001 inch at midpoint.
- b. Place specimen in test fixture, convex surface up. Align the specimen so that its midpoint is centered under the loading nose.
- c. Apply the load at a crosshead speed of 0.05 inches minute.
- d. Record the load required to break the specimen. (A typical shear failure is characterized by a sharp, audible report.)
- e. A minimum of three specimens shall be tested from each ring.
- f. Values for properties at break shall not be calculated for any specimen that breaks at some obvious flaw, unless such flaw constitutes a variable being studied. Retest shall be made for any specimen which fails in any manner other than shear.
- g. Calculate horizontal shear strength as follows:

$$S_H = \frac{0.75 P_B}{bd}$$

Where: S_H = Apparent horizontal shear strength (psi)

P_B = Breaking load (pounds)

b = Width (inches)

d = Thickness (inches)

4.6.4.10.3 Data. The following data shall be recorded:

- a. Date of test
- b. Material identification (cure, fabrication technique, etc.)
- c. Ultimate breaking load
- d. Horizontal shear strength
- e. Specimen dimensions
- f. Test temperature
- g. Rate of loading

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h. Conditioning (if any)

i. Resin content

j. Specific gravity

4.7 Preservation, packing unitization and marking. The inspector shall ascertain that the preservation, packing unitization, and marking of the material conform to this specification.

4.8 Workmanship. The material shall be visually examined to assure conformance to the workmanship requirement specified in 3.4.

5. PACKAGING

5.1 Preservation, packing, unitization, and marking. Preservation, packing, unitization and marking shall be in accordance with ASTM D 3951.

6. NOTES

6.1 Intended use. This material is intended for use as an impregnant for fiberglass filaments used in missile systems. The cured parts are satisfactory for use at temperatures from minus 54 to plus 121 degrees C (minus 65 to plus 250 degrees F).

6.2 Ordering data. Procurement documents should specify, but not be limited to, the following:

a. Military part number (see 1.3).

b. Whether a first article sample is required (see 3.1) and, if so pertinent details (see 4.3).

c. Requirements for certification of composition (see 4.6.4.1).

d. Packaging required, if other than commercial (see 5.1).

6.3 Handling precautions. The following precautions should be observed in the handling of the epoxy resin, anhydride hardener and amine promoter.

a. Avoid exposure of the skin with the epoxy resin as contact dermatitis may result.

b. Avoid skin contact with the hardening and promoting agents, as the materials can produce a sensitization dermatitis if proper handling procedures are not observed. The vapors are also irritating to the eyes and mucous membranes of the respiratory tract.

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c. Observe the following precautions when handling the materials:

- (1) Mix the components in an area with adequate ventilation to remove fumes.
- (2) Wear rubber gloves to prevent skin contact.
- (3) Wear goggles or eye shield to provide eye protection.
- (4) Confine curing operations to areas provided with exhaust ventilating facilities.

d. In case of accidental skin contact with the materials, the contaminated skin areas should be washed immediately with soap and water. To accomplish thorough cleansing, it may occasionally be necessary to use solvents. This practice should, however, be kept to a minimum. Contaminated clothing should be laundered before use. Meticulous housekeeping standards should also be observed. Strict supervision should be enforced to prevent the inadvertent contamination of non-operating areas; for example, the contamination of doorknobs, valves and handrails, by failure to remove contaminated gloves.

6.4 Type I compound. The formulation of type I compound is listed in table IV.

TABLE IV. Formulation of type 1 compound.

Components	Parts by weight
Epoxy resin	100 \pm 2 percent
Anydride hardener	80 \pm 2 percent
Amine promoter	1 \pm 10 percent

6.5 Metricalion. Metric equivalents, in accordance with FED-STD-376, are acceptable for use in this specification.

6.6 Changes from previous issue. Asterisks are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

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6.6 Subject term (key word) listing.

Composite

Compound

Epoxy

Filament winding

Custodian:
Army - MI

Review Activities:
Army - MR
DLA - GS

Preparing activity:
Army - MI

Project No. CMPS-A030

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions - Reverse Side)

1. DOCUMENT NUMBER MIL-C-47257B(MI)		2. DOCUMENT TITLE COMPOUND, EPOXY, FILAMENT WINDING	
3a. NAME OF SUBMITTING ORGANIZATION		4. TYPE OF ORGANIZATION (Mark one) <input type="checkbox"/> VENDOR <input type="checkbox"/> USER <input type="checkbox"/> MANUFACTURER <input type="checkbox"/> OTHER (Specify): _____	
b. ADDRESS (Street, City, State, ZIP Code)			
5. PROBLEM AREAS			
a. Paragraph Number and Wording:			
b. Recommended Wording:			
c. Reason/Rationale for Recommendation:			
6. REMARKS			
7a. NAME OF SUBMITTER (Last, First, MI) - Optional		b. WORK TELEPHONE NUMBER (Include Area Code) - Optional	
c. MAILING ADDRESS (Street, City, State, ZIP Code) - Optional		8. DATE OF SUBMISSION (YYMMDD)	

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