

INCH-POUND

MIL-PRF-47257F (MI)
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SUPERSEDING
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PERFORMANCE SPECIFICATION COMPOUND, EPOXY, FILAMENT WINDING

This specification is approved for use by the US Army Research, Development, and Engineering Command and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

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

2. APPLICABLE DOCUMENTS

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

Comments, suggestions, or questions on this document should be addressed to RDMR-SET Redstone Arsenal, AL 35898-5000 or emailed to malinda.allcorn1@us.army.mil. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <https://assist.daps.dla.mil>.

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

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D1475	Density of Paint, Varnish, Lacquer, and Related Products, Test Methods for
ASTM D2291	Fabrication of Ring Test Specimens for Glass-Resin Composites, Standard Practice for

(Copies of these documents are available from www.astm.org or ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

2.3 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 First article. When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.3.

3.2 Materials. Materials shall consist of a) an epoxy resin, b) an anhydride hardener, c) an amine promoter, and d) fiberglass roving. Ingredients a, b, and c shall provide not more than 26 ± 4 percent of the product content by weight. (See 4.6.4.1 and 4.6.4.2).

3.2.1 Condition in container. The component materials in containers shall be free of gel particles and foreign matter that would adversely affect their intended purpose. Crystallization shall be eliminated by heating to not greater than 49 °C and stirring shall be acceptable (see 4.6.4). The resin content shall be marked on the container.

3.2.2 Storage life. The individual components shall be capable of meeting the requirements of this specification after storage in the original unopened containers at temperatures between 4 and 32 °C in no sunlight for not less than 12 months from the date of shipment (see 4.6.4.3).

3.2.3 Properties of the mixed compound. The mixed epoxy compound consisting of resin, hardener, and promoter and mixed to the manufacturer's specifications shall exhibit the following properties:

3.2.3.1 Viscosity. The viscosity, immediately after mixing, shall be within the range of 500 to 3000 milli pascal seconds (mPa·s) at 25 ± 1 °C (see 4.6.4.5).

3.2.3.2 Gel time. The mixed compound shall gel to a non-flowable solid in not greater than 1 hour at 149 ± 3 °C (see 4.6.4.6).

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3.2.3.3 Product. The impregnated roving product 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 or to the roll and no deterioration of the roving during removal (see 4.6.4).

3.3 Performance characteristics.

3.3.1 Product. The product shall be capable of meeting the requirements of this specification after storage at a temperature not greater than 10 °C for not less than 30 days, or after storage at room temperature (16 to 32 °C) for not less than 5 days (see 4.6.4.3).

3.3.2 Shear strength. After a cure time of not greater than 3 hours at 141 ± 3 °C, the shear strength of the material shall be not less than 5000 pounds per square inch (psi) at an ambient temperature of 25 ± 1 °C (see 4.6.4.7).

3.4 Warning label. Containers of the epoxy compound shall be marked with a warning label addressing hazards and cautions as identified by the component manufacturers.

3.5 Recycled, recovered, or environmentally preferable materials. Recycled, recovered, or environmentally preferable materials should be used to the maximum extent possible provided that the material meets or exceeds the operational and maintenance requirements, and promotes economically advantageous life cycle costs.

4. VERIFICATION

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

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

4.2 Inspection conditions. Unless otherwise specified, all inspections shall be performed in accordance with the test conditions specified in 4.3, 4.4, and 4.6.4.3

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 shall consist of a 1-quart kit containing all the component materials, or a 1-quart sample of each material except the amine promoter, which may be furnished as a 1-pint sample. The sample shall also consist of not less than 5 pounds of impregnated roving. First article inspection shall be as specified herein. Subsequent units shall 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 I shall be conducted in accordance with the corresponding test paragraphs.

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TABLE I. First article inspection.4.4 Conformance inspection.4.4.1 Lot formation.

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

Characteristics	Test paragraph
Storage life	4.6.4.3
Component materials	4.6.4.4
Specific gravity of epoxy resin	4.6.4.4
Specific gravity of anhydride hardener	4.6.4.4
Specific gravity of amine promoter	4.6.4.4
Viscosity of epoxy resin	4.6.4.5
Viscosity of anhydride hardener	4.6.4.5
Mixed compound	4.6.4.7
Shear	

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 component material shall be considered as a unit of product. Random selection of a container from each lot of component material shall be effected. From these sample component materials, sufficient quantities shall be randomly appropriated for the manufacture of sufficient fiberglass roving materials for subjection to the acceptance inspection as delineated in Table II. Failure of this sample to comply with the requirements specified in Table II shall be cause for lot rejection.

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TABLE II. Conformance Inspection.

Characteristics	Requirement paragraph	Test paragraph
Composition	3.2	4.6.4
Condition in Container	3.2.1	4.6.4
Mixed compound	3.2.3.1	4.6.4.5
Viscosity	3.2.3.2	4.5.4.6
Gel Time		

4.5 Inspection equipment. Inspection equipment for conducting examination and tests shall be as specified in the following 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)
- b. Altitude, facility ground
- c. Humidity, facility ambient up to 95 percent relative humidity.

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

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

4.6.3.1 Packaged product material. To prevent moisture condensation on the impregnated roving, all packages of material which are removed from the storage cooler (see 3.3.1) shall be allowed to warm to room temperature for a minimum of 2 hours prior to opening the sealed bag.

4.6.3.2 Shear specimens. The filament wound rings shall be prepared from the product 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 greater than 3 hours at 141 \pm 3 °C. Shear specimens shall be fabricated from the rings to the dimensions shown on figure 1.

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NOTE: Observe test precautions when handling the component or compounded materials during the following tests.

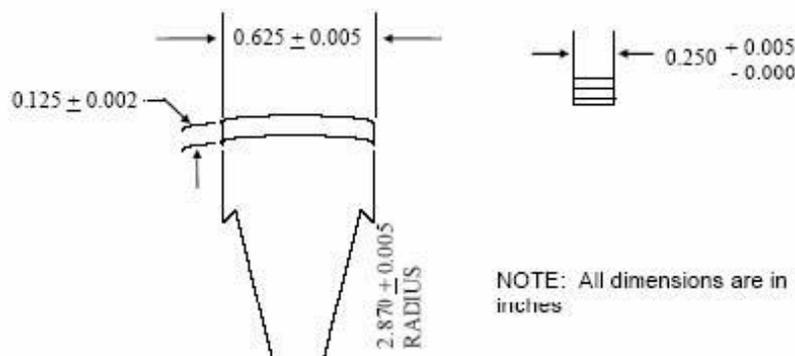


FIGURE 1. Shear test specimen.

4.6.4 Composition. The condition in containers shall be examined visually and shall conform to requirements of 3.2.1. The composition shall be certified by the contractor and shall conform to requirements of 3.2.2.

4.6.4.1 Resin content. The preferred procedure for determining the resin content of the product material shall be as follows:

- Weigh the empty impregnated roving spool (W_1) prior to installing on the roving impregnating machine.
- Weigh the dry spool of roving (W_2).
- After impregnating the roving, weigh the impregnated spool of (W_3), roving and the dry roving spool remainder (W_4)
- Calculate the resin content by the following formula:

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

Where R = resin percent content

W_1 = weight of empty impregnated roving spool

W_2 = weight of dry roving spool

W_3 = weight of impregnated roving spool

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W_4 = weight of dry roving spool remainder

Therefore, $W_2 - W_4$ = weight of dry roving

$W_3 - W_1$ = weight of impregnated roving

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

4.6.4.2 Resin content, alternate method. The alternate procedure for determining the resin content of the product material shall be as follows:

- a. Weigh the specimen on an analytical balance (W_5).
- b. Burn the specimen in a muffle furnace at 593 to 621 °C for 1 to 2 hours or until the specimen is white.
- c. Cool the specimen in a desiccator.
- d. Weigh the specimen again on an analytical balance (W_6).
- e. Calculate the resin content as follows:

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

Where R = percent of resin content

W_5 = weight of specimen before burning

W_6 = weight of specimen after burning

4.6.4.3 Storage life. The contractor's certification of compliance to the storage life requirements (see 3.2.2) is required.

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

4.6.4.5 Viscosity. The viscosity shall be determined in accordance with the following:

4.6.4.5.1 Apparatus.

- a. Brookfield Syncho-lectric Viscometer, Model L.V.F., Brookfield Engineering Company, Inc., 240 Cushing Street, Stoughton,

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Massachusetts, or equivalent.

- b. Water bath capable of maintaining a temperature of $25 \pm 0.1^\circ\text{C}$.
- c. A 600 ml beaker.

4.6.4.5.2. Procedure.

- a. Place the sample in the beaker and temperature condition it in the water bath to $25 \pm 0.1^\circ\text{C}$.
- b. Attach a No. 1 spindle to the viscometer.
- c. Lower the spindle into the sample so that the resin surface is at the machined groove in the spindle. Center the spindle in the beaker. Care must be exercised to prevent bubble entrapment on the spindle. Using the leveling device, level the viscometer.
- d. Depress the clutch at the back of the viscometer; turn the instrument on; then, release the clutch.
- e. Allow the spindle to rotate at 30 revolutions per minute (rpm), for approximately one minute. Record the approximate scale reading. Depress the clutch, and stop the motor. Obtain three readings and take the average.
- f. Multiplying by the appropriate factor (as given in the tables and instructions furnished with the viscometer), convert the average scale reading for the spindle used into centipoises.

4.6.4.6 Gel time. To ensure the gel time conforms to 3.2.3.2, the following gel time test method shall be conducted.

4.6.4.6.1. Volumes. Test sample volumes for thin-section applications, adhesives, tooling the surface coats, dip coatings, laminating materials, impregnants, caulking and sealing compounds, small-volume encapsulating and potting compounds shall be 15 mL (0.5 fluid oz). Working quantity shall be 60 mL (2 fluid oz).

4.6.4.6.2 Test apparatus. The following equipment shall be used to determine gel time.

- a. Sample container shall be an aluminum foil dish, approximately 7 cm (2.76 in.) in diameter and 1.4 cm (0.55 in.) deep.
- b. Wooden probes shall be applicator sticks approximately 0.24 cm (0.009 in.) in diameter by 15.2 cm (6.00 in.) long.
- c. Non-conducting surface shall be dry wood or corrugated casing.
- d. Temperature measuring device shall be any temperature recorder or indicator that uses expendable thermocouples and is accurate to approximately $\pm 1\%$ of scale.
- e. Temperature-controlled bath shall hold the temperature to within $\pm 0.1^\circ\text{C}$.
- f. Stop watch shall be used to determine the gel time.

4.6.4.6.3 Test procedure. The following test procedure shall be followed to ensure the gel time meets the requirements of 3.2.3.2 herein.

4.6.4.6.3.1 Conditioning. Place the sample in the aluminum foil dish in the temperature-controlled bath at the test temperature for not less than 4 hours prior to being tested, or for the length of time needed for all parts of the sample to reach the test temperature

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within 0.5 °C (1.0 °F). Condition all containers and probes to be used in the test at the test temperature at the same time.

NOTE: If experience has shown that less than 4 hours conditioning is required for all parts of the sample to reach the test temperature, record the time of conditioning.

4.6.4.6.3.2 Procedure. When all components have reached $149 \pm 3^{\circ}\text{C}$ (300°F), the following procedures shall be completed:

- a. Agitate each component slowly and separating with a stirring rod or mixing paddle for not less than 3 minutes, avoiding the entrapment of air.
- b. Combine the components to provide a working quantity of 60 mL (2 fluid oz).

NOTE: For accurate measurements of quantities of components, the individual components shall be weighed, using the specific gravities of the respective components to determine the weights needed to make up the required working volume.

- c. Start the stop watch and mix the components thoroughly for not less than 3 minutes, avoiding air entrapment by slow agitation with a stirring rod or mixing paddle. To avoid transfer of heat, do not hold the container by hand during the mixing operation. Record the start of mixing as the “starting time.”
- d. Transfer the test volume (15 mL (0.5 fluid oz)) of the mixed components immediately to an aluminum foil dish, which has been previously conditioned at the test temperature.
- e. Place the sample container on dry wood or corrugated casing in still air at the test temperature.

NOTE: Make a record of the description of the surface. If it is desirable, because of the nature of the application, to use a conducting surface of high heat capacity instead of a non-conducting surface. If, because of the nature of the application, it becomes desirable to measure gel time and peak exothermic temperature in a temperature-controlled bath, the conditions must be noted. WARNING – Gel times and peak exothermic temperatures observed in a temperature-controlled bath are functions of the total system, including the quantity and nature of the coolant, as well as the nature of all components inserted into the bath. These properties are not functions of the resin-curing agent system alone.

- f. Insert a thermocouple, or other temperature-measuring device, into the geometric center of the reacting mass, and record the observed temperature changes to the end of the test.
- g. Every 15 seconds probe the center surface of the reacting mass with the applicator stick perpendicular to the material surface.

NOTE: Although a mechanical gel time meter may be feasible

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in the larger sample sizes, the results obtained with the various mechanical gel time meters have not been consistent with results obtained with hand probing. Therefore, a record shall be kept concerning the use of a mechanical gel time meter.

- h. When the reacting material no longer adheres to the end of a clean probe, record the “gel time” as the elapsed time from the start of mixing.
- i. Continue recording the time and temperature until the temperature starts to drop. Record the highest temperature reached as the “peak exothermic temperature.” Record the “peak exothermic time” as the elapsed time from the start of mixing.

4.6.4.7 Shear strength. The shear strength of test specimens prepared as specified in 4.6.3.2 shall be determined as follows and shall meet the requirements of 3.3.2.

4.6.4.7.1 Equipment. The following equipment shall be used:

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

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

- a. Measure the thickness and width of the specimen to the nearest 0.001 inch at midpoint.
- b. Place the 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 per 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 the horizontal shear strength as follows:

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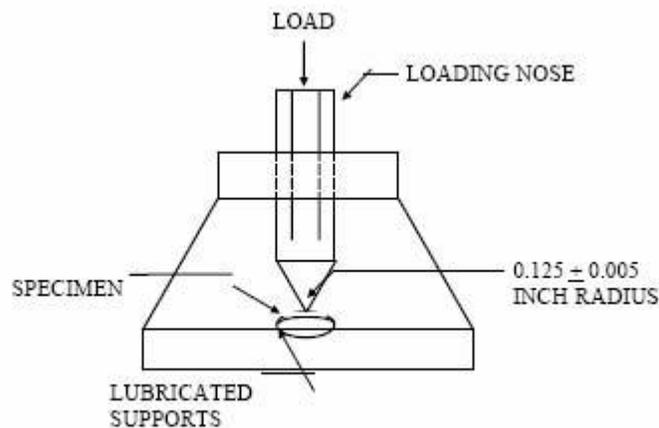
$$S_H = \frac{0.75 P_B}{bd}$$

Where: S_H = Apparent horizontal shear strength (psi)

P_B = Breaking load (pounds)

b = Width (inches)

d = Thickness (inches)



NOTE: Dimension is in inches.

FIGURE 2. Typical horizontal shear fixture.

4.6.4.7.3 Test measurements. The following test measurements 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
- h. Conditioning (if any)
- i. Resin content
- j. Specific gravity.

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5. PACKAGING

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

6. NOTES

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

6.1 Intended use. This 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 -54 to 121 °C.

6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number, and date of the specification.
- b. Issue of DoDISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced.
- c. Whether first article is required (see 3.1 and 4.3).
- d. Conditions for quality conformance inspection (see 4.4).
- e. First article sample size (see 4.3).
- f. Packaging requirements (see 5.1).

6.3 First article. When first article inspection is required, the contracting officer should provide specific guidance to offerors whether the item(s) should be a first article sample, a first production item, or a number of items to be tested as specified in 4.3. The contracting officer should also include specific instructions in acquisition documents regarding arrangements for examinations, approval of first article test results and disposition of first articles. Invitations for bids should provide that the Government reserves the right to waive the requirement for samples for first article inspection to those bidders offering a product which has been previously acquired or tested by the Government, and that bidders offering such products, who wish to rely on such production or test, must furnish evidence with the bid that prior Government approval is presently appropriate for the pending contract.

6.4 Material safety data sheets. Contracting officers will identify those activities requiring copies of completed material safety data sheets prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

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

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6.6 Subject term (keyword) listing.

Anhydride hardener
Resin-hardener
Amine promoter

6.7 Changes from previous issue. Test apparatus and procedures for gel time and peak exothermic temperature of reacting thermosetting resins were incorporated into 4.6.4.6 because ASTM D2471 was withdrawn without replacement.

Custodian:
Army - MI

Preparing Activity:
Army - MI

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Review activities:
Army - MR
DLA - GS

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