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Superseding AMS3619C

Resin, Polyimide, Laminating
High Temperature Resistant, 315 °C (599 °F)

RATIONALE

This document has been revised to remove specific military requirements described in former sections 4.2.2.1, 5.1.5, and 8.3. MIL-STD-2073-1 was also deleted from section 2.3 since it was referenced in section 5.1.5.

1. SCOPE

1.1 Form

This specification covers a single-component, heat-reactive, thermosetting aromatic system which thermally cures to form a polyimide polymer structure.

1.2 Application

This product has been used typically as a resin matrix for fiber-reinforced-composite parts requiring good strength after long-term exposure up to 315 °C (599 °F) and after short-term exposure up to 370 °C (698 °F), but usage is not limited to such applications.

1.3 Safety-Hazardous Materials

While the materials, methods, applications, and processes described or referenced in this specification may involve the use of hazardous materials, this specification does not address the hazards which may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

2. APPLICABLE DOCUMENTS

The issue of the following documents in effect on the date of the purchase order forms a part of this specification to the extent specified herein. The supplier may work to a subsequent revision of a document unless a specific document issue is specified. When the referenced document has been cancelled and no superseding document has been specified, the last published issue of that document shall apply.

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2.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or 724-776-4970 (outside USA), www.sae.org.

AMS2825 Material Safety Data Sheets

AMS3824 Cloth, Glass, Finished for Resin Laminates

2.2 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org.

ASTM D 695 Compressive Properties of Rigid Plastics

ASTM D 790 Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials

ASTM D 1824 Apparent Viscosity of Plastisols and Organosols at Low Shear Rates by Brookfield Viscometer

ASTM D 1963 Specific Gravity of Drying Oils, Varnishes, Resins, and Related Materials at 25/25 °C

ASTM D 2344 Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short Beam Method

3. TECHNICAL REQUIREMENTS

3.1 Material

Shall be an uncured, single-component, heat-reactive, thermosetting, aromatic polymer, completely dissolved in a solvent system of N-methylpyrrolidone (NMP), which cures to form a polyimide polymer structure.

3.2 Storage Life

The resin solution, when stored in airtight containers at not higher than 7 °C (45 °F), shall meet the requirements of 3.3 and 3.4 when tested at any time up to 6 months from date of manufacture.

3.3 Properties of Uncured Product

The product, as received, shall conform to the following requirements; tests shall be performed on the product supplied and in accordance with specified test methods:

3.3.1 Viscosity

Shall be 30 to 70 poises (3 to 7 Pa·s), determined in accordance with ASTM D 1824 on a Brookfield Viscometer, Model LVF, or equivalent, except that a No. 3 spindle at 12 rpm, shall be used.

3.3.2 Resin Solids

Shall be 48.0 to 53.0% by weight, determined in accordance with 4.5.1.

3.3.3 Specific Gravity

Shall be 1.15 to 1.18, determined in accordance with ASTM D 1963 using a Hubbard-type pycnometer, or equivalent.

3.3.4 Infrared Spectrogram

The infrared (IR) transmission spectrogram of a resin film, determined in accordance with 4.5.2, shall be developed during initial preproduction testing. Subsequent resin batches may be tested to this IR signature as agreed upon by purchaser and vendor.

3.4 Properties of Cured Product

The product, combined with woven glass cloth in accordance with 4.5.3, shall conform to the following requirements; tests shall be performed in accordance with specified test methods on specimens cut from a test laminate panel prepared as specified in 4.5.3. Specimens shall be tested at the temperatures specified after being held at the test temperature for not less than 30 minutes prior to testing.

3.4.1 Flexural Strength and Flexural Modulus

Shall be as shown in Table 1, determined in accordance with ASTM D 790:

TABLE 1 - FLEXURAL STRENGTH AND MODULUS

Test Temperature	Flexural Strength ksi	Flexural Strength MPa	Flexural Modulus Msi	Flexural Modulus GPa
25 °C ± 3 (77 °F ± 5)				
Minimum Average	65.0	448	2.50	17.2
Individual Minimum	58.5	403	2.25	15.5
315 °C ± 5 (599 °F ± 9)				
Minimum Average	45.0	310	2.00	13.8
Individual Minimum	40.5	279	1.80	12.4
315 °C ± 5 (599 °F ± 9) after 100 hours at 315 °C ± 5 (599 °F ± 9)				
Minimum Average	35.0	241	2.00	13.8
Individual Minimum	31.5	217	1.80	12.4

3.4.2 Compressive Strength and Compressive Modulus

Shall be as shown in Table 2, determined in accordance with ASTM D 695:

TABLE 2 - COMPRESSIVE STRENGTH AND MODULUS

Test Temperature	Compressive Strength ksi	Compressive Strength MPa	Compressive Modulus Msi	Compressive Modulus GPa
25 °C ± 3 (77 °F ± 5)				
Minimum Average	45.0	310	2.750	19.0
Individual Minimum	40.5	279	2.475	17.1
315 °C ± 5 (599 °F ± 9)				
Minimum Average	35.0	241	2.250	15.5
Individual Minimum	31.0	214	2.025	13.0
315 °C ± 5 (599 °F ± 9) after 100 hours at 315 °C ± 5 (599 °F ± 9)				
Minimum Average	30.0	207	2.000	13.8
Individual Minimum	27.0	186	1.800	12.4

3.4.3 Short Beam Shear Strength

Shall be as shown in Table 3, determined on five specimens per test in accordance with ASTM D 2344 except that the test specimens shall be flat:

TABLE 3 - SHORT BEAM SHEAR STRENGTH

Test Temperature	Short Beam Shear Strength psi	Short Beam Shear Strength MPa
25 °C ± 3 (77 °F ± 5)		
Minimum Average	5000	34.5
Individual Minimum	4500	31.0
315 °C ± 5 (599 °F ± 9)		
Minimum Average	3500	24.1
Individual Minimum	3150	21.7

3.5 Quality

The product, as received by purchaser, shall be uniform in quality and condition, smooth, and free from foreign materials and from imperfections detrimental to usage of the product.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection

The vendor of the product shall supply all samples for vendor's tests and shall be responsible for the performance of all required tests. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the product conforms to specified requirements.

4.2 Classification of Tests

4.2.1 Acceptance Tests

Viscosity (3.3.1), resin solids (3.3.2), specific gravity (3.3.3), and quality (3.5) are acceptance tests and shall be performed on each lot.

4.2.2 Preproduction Tests

Tests for all technical requirements are preproduction tests and shall be performed prior to or on the initial shipment of resin to a purchaser, when a change in ingredients and/or processing requires reapproval as in 4.4.2, and when purchaser deems confirmatory testing to be required.

4.3 Sampling and Testing

Shall be as follows:

4.3.1 For Acceptance Tests

Sufficient product shall be taken at random from each lot to perform all required tests. The number of determinations for each requirement shall be as specified in the applicable test procedure or, if not specified therein, not less than three.

- 4.3.1.1 A lot shall be all resin produced in a single production run from the same batches of raw materials under the same fixed conditions and presented for vendor's inspection at one time. A lot shall not exceed 1000 gallons (3785 L) of resin.
- 4.3.1.2 A batch shall be the quantity of material run in a reactor or mixer at one time.
- 4.3.1.3 When a statistical sampling plan has been agreed upon by purchaser and vendor, sampling shall be in accordance with such plan in lieu of sampling as in 4.3.1 and the report of 4.6 shall state that such plan was used.

4.3.2 For Preproduction Tests

As agreed upon by purchaser and vendor.

4.4 Approval

- 4.4.1 Sample resin shall be approved by purchaser before resin for production use is supplied, unless such approval be waived by purchaser. Results of tests on production resin shall be essentially equivalent to those on the approved sample.
- 4.4.2 Vendor shall use ingredients, manufacturing procedures, processes, and methods of inspection on production resin which are essentially the same as those used on the approved sample resin. If necessary to make any change in ingredients, in type of equipment for processing, or in manufacturing procedures, vendor shall submit for reapproval a statement of the proposed changes in ingredients and/or processing and, when requested, sample resin. Production resin made by the revised procedure shall not be shipped prior to receipt of reapproval.

4.5 Test Methods

4.5.1 Resin Solids Content

Resin solids shall be determined by heating a sample of as-received resin in an aluminum weighing dish in a forced draft oven at $315\text{ }^{\circ}\text{C} \pm 5$ ($599\text{ }^{\circ}\text{F} \pm 9$) for 15 minutes ± 1 , and calculating the weight of solids remaining as a percentage of the initial sample weight.

4.5.2 Infrared Spectrogram

An infrared transmission spectrogram shall be prepared covering wave lengths from 2.5 to 16 microns (4000 to 625 reciprocal centimeters) on cured resin films of sufficient thickness to produce major spectral bands with transmission approximating 40%.

- 4.5.2.1 A cured film of the resin shall be prepared by mixing 1.0 gram ± 0.1 of resin with 24 grams ± 1 of acetone, followed by devolatilizing and curing as shown in Table 4:

TABLE 4 - CURING CONDITIONS

Time minutes ± 0.1	Temperature $^{\circ}\text{C} \pm 5$	Temperature $^{\circ}\text{F} \pm 9$
5	120	248
5	230	446
5	290	554

4.5.3 Test Laminate Preparation

A 12-ply test laminate panel, at least 8.0×12.0 inches (203×305 mm), shall be prepared from style 7781 glass cloth conforming to AMS3824, with A-1100 soft aminosilane finish, impregnated with resin, and cured as specified in 4.5.3.3 and 4.5.3.4.

4.5.3.1 Resin Impregnation

The glass cloth shall be uniformly saturated with the as-received resin so that the resultant impregnated cloth shall contain $47\% \pm 3$ resin, calculated from the total impregnated cloth weight. The impregnated cloth shall then be B-staged in a forced-draft oven preheated to $120\text{ }^{\circ}\text{C} \pm 3$ ($248\text{ }^{\circ}\text{F} \pm 5$) until the volatile content has been reduced to 10 to 12% by weight. The volatile content of the impregnated cloth shall be determined on a single ply sample after heating at $315\text{ }^{\circ}\text{C} \pm 5$ ($599\text{ }^{\circ}\text{F} \pm 9$) for 15 minutes ± 1 .

4.5.3.2 Layup of Test Laminate Panel

Layup 12 plies of the B-staged impregnated cloth prepared in 4.5.3.1, unnested, with the warp parallel, and with each ply positioned in the layup so that the satin shafts of the warp always face the top of the layup. The layup shall be on a suitable released caul plate and shall be covered with one ply of release glass cloth and two plies of dry 181 type glass cloth extending approximately 2 inches (51 mm) beyond the laid up panel on all sides. A suitable bleeder mechanism shall be placed around the periphery and attached to a vacuum line. A thermocouple shall be placed between plies No. 6 and No. 7, approximately 1 inch (25 mm) from the edge of the laminate panel. The assembly shall be enclosed in a heat-resistant-film bag and a vacuum of not less than 24 inches (610 mm) Hg shall be applied.

4.5.3.3 Autoclave Cure

The assembly shall be placed in an autoclave, under vacuum, and cured as follows:

- 4.5.3.3.1 Raise the autoclave air temperature to $115\text{ }^{\circ}\text{C} \pm 3$ ($239\text{ }^{\circ}\text{F} \pm 5$) in 20 to 30 minutes with no pressure in the autoclave.
- 4.5.3.3.2 When the thermocouple in the laminate panel reaches $115\text{ }^{\circ}\text{C} \pm 3$ ($239\text{ }^{\circ}\text{F} \pm 5$), apply 100 psi (689 kPa) autoclave pressure.
- 4.5.3.3.3 Continue to raise the autoclave air temperature to $180\text{ }^{\circ}\text{C} \pm 3$ ($356\text{ }^{\circ}\text{F} \pm 5$) at a uniform rate to achieve $180\text{ }^{\circ}\text{C}$ ($356\text{ }^{\circ}\text{F}$) in 70 to 85 minutes.
- 4.5.3.3.4 Maintain the autoclave air temperature at $180\text{ }^{\circ}\text{C} \pm 3$ ($356\text{ }^{\circ}\text{F} \pm 5$) for not less than 2 hours after the thermocouple in the laminate panel reaches $170\text{ }^{\circ}\text{C}$ ($338\text{ }^{\circ}\text{F}$).
- 4.5.3.3.5 Cool the laminate panel to below $65\text{ }^{\circ}\text{C}$ ($149\text{ }^{\circ}\text{F}$) under at least 40 psi (276 kPa) autoclave pressure.
- 4.5.3.3.6 Release pressure, remove laminate panel from autoclave, and cool to room temperature. Mark warp direction on panel immediately after cure.

4.5.3.4 Postcure

The test laminate panel shall be postcured under restraint in successive steps as shown in Table 5:

TABLE 5 - POSTCURE CONDITIONS

Time hours ± 0.1	Post Cure Temperature $^{\circ}\text{C} \pm 5$	Post Cure Temperature $^{\circ}\text{F} \pm 9$
12	175	347
12	200	392
12	230	446
12	260	500
12	290	554
24	315	599