

**RESIN, POLYIMIDE, LAMINATING
High Temperature Resistant, 315°C (600°F)**

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: Primarily as a resin matrix for fiber-reinforced-composite parts requiring good strength after long-term exposure up to 315°C (600°F) and after short-term exposure up to 370°C (700°F).

2. APPLICABLE DOCUMENTS: The following publications form a part of this specification to the extent specified herein. The latest issue of Aerospace Material Specifications shall apply. The applicable issue of other documents shall be as specified in AMS 2350.

2.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096.

2.1.1 Aerospace Material Specifications:

AMS 2350 - Standards and Test Methods

AMS 2825 - Material Safety Data Sheets

AMS 3824 - Cloth, Type "E" Glass, Finished for Resin Laminates

SAE Technical Board Rules provide that: "This report is published by SAE to advance the state of technical and engineering sciences. The use of this report is entirely voluntary, and its applicability and suitability for any particular use, including any patent infringement arising therefrom, is the sole responsibility of the user."

AMS documents are protected under United States and international copyright laws. Reproduction of these documents by any means is strictly prohibited without the written consent of the publisher.

2.2 ASTM Publications: Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

- ASTM D695 - Compressive Properties of Rigid Plastics
- ASTM D790 - Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials
- ASTM D1824 - Apparent Viscosity of Plastisols and Organosols at Low Shear Rates by Brookfield Viscometer
- ASTM D1963 - Specific Gravity of Drying Oils, Varnishes, Resins, and Related Materials at 25/25°C
- ASTM D2344 - Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short Beam Method

2.3 U.S. Government Publications: Available from Commanding Officer, Naval Publications and Forms Center, 5801 Tabor Avenue, Philadelphia, PA 19120.

2.3.1 Military Standards:

MIL-STD-794 - Parts and Equipment, Procedures for Packaging and Packing of

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 - 70 poises (3 - 7 Pa·s), determined in accordance with ASTM D1824 on a Brookfield Viscometer, Model LVF, except that a No. 3 spindle at 12 rpm, or equivalent, shall be used.

3.3.2 Resin Solids: Shall be 48.0 - 53.0% by weight, determined in accordance with 4.5.1.

3.3.3 Specific Gravity: Shall be 1.15 - 1.18, determined in accordance with ASTM D1963 using a Hubbard-type pycnometer.

3.3.4 Infrared Spectrogram: The infrared 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 I. R. 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 min. prior to testing.

3.4.1 Flexural Strength and Flexural Modulus: Shall be as follows, determined in accordance with ASTM D790:

Test Temperature	Flexural Strength		Flexural Modulus	
	psi	MPa	psi	MPa
25°C + 3 (77°F + 5)				
Minimum Average	65,000	450	2,500,000	17,250
Individual Minimum	58,500	405	2,250,000	15,500
315°C + 5 (600°F + 10)				
Minimum Average	45,000	310	2,000,000	13,800
Individual Minimum	40,500	280	1,800,000	12,400
315°C + 5 (600°F + 10) after T00 hr at 315°C ± 5 (600°F + 9)				
Minimum Average	35,000	240	2,000,000	13,800
Individual Minimum	31,500	215	1,800,000	12,400

3.4.2 Compressive Strength and Compressive Modulus: Shall be as follows, determined in accordance with ASTM D695:

Test Temperature	Compressive Strength		Compressive Modulus	
	psi	MPa	psi	MPa
25°C + 3 (77°F + 5)				
Minimum Average	45,000	310	2,750,000	18,950
Individual Minimum	40,500	280	2,475,000	17,050
315°C + 5 (600°F + 10)				
Minimum Average	35,000	240	2,250,000	15,500
Individual Minimum	31,000	215	2,025,000	13,950
315°C + 5 (600°F + 10) after T00 hr at 315°C ± 5 (600°F + 10)				
Minimum Average	30,000	205	2,000,000	13,800
Individual Minimum	27,000	185	1,800,000	12,400

- 3.4.3 Short Beam Shear Strength: Shall be as follows, determined on five specimens per test in accordance with ASTM D2344 except that the test specimens shall be flat:

Test Temperature	Short Beam Shear Strength	
	psi	MPa
25°C + 3 (77°F + 5)		
Minimum Average	5000	34.5
Individual Minimum	4500	31.0
315°C + 5 (600°F + 10)		
Minimum Average	3500	24.0
Individual Minimum	3150	21.5

- 3.5 Quality: The product, as received by purchaser, shall be uniform in quality and condition, clean, 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 performing all required tests. Results of such tests shall be reported to the purchaser as required by 4.6. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the product conforms to the requirements of this specification.

4.2 Classification of Tests:

- 4.2.1 Acceptance Tests: Tests to determine conformance to requirements for viscosity (3.3.1), resin solids (3.3.2), specific gravity (3.3.3), and quality (3.5) are classified as acceptance tests and shall be performed on each lot.

- 4.2.2 Preproduction Tests: Tests to determine conformance to all technical requirements of this specification are classified as preproduction tests and shall be performed prior to or on the initial shipment of resin to a purchaser, when a change in material, processing, or both requires reapproval as in 4.4.2, and when purchaser deems confirmatory testing to be required.

- 4.2.2.1 For direct U.S. Military procurement, substantiating test data and, when requested, preproduction test material shall be submitted to the cognizant agency as directed by the procuring activity, the contracting officer, or the request for procurement.

- 4.3 Sampling: 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 gal (3800 L) of resin and may be packaged and delivered in smaller quantities under the basic lot approval provided lot identification is maintained.

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 and acceptance quality level (AQL) have 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 4.6.1 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 material, processing, or both 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 $315^{\circ}\text{C} + 5$ ($600^{\circ}\text{F} + 10$) for 15 min. + 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 centimetres) on cured resin films of sufficient thickness to produce major spectral bands with transmissions approximating 40%.

- 4.5.2.1 A cured film of the resin shall be prepared by mixing 1.0 g + 1 of resin with 24 g + 1 of acetone, followed by devolatilizing and curing as follows:

Time min. + 0.1	Temperature	
	°C + 5	°F + 10
5	120	250
5	230	445
5	290	555

- 4.5.3 Test Laminate Preparation: A 12-ply test laminate panel, at least 8.0 x 12.0 in. (200 x 300 mm), shall be prepared from style 7781 glass cloth conforming to AMS 3824, 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 in such a manner that the resultant impregnated cloth shall contain 47% + 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°C + 3 (250°F + 5) until the volatile content has been reduced to 10 - 12% by weight. The volatile content of the impregnated cloth shall be determined on a single ply sample after heating at 315°C + 5 (600°F + 10) for 15 min. + 1.
- 4.5.3.2 Layup of Test Laminate Panel: Layup 12 plies of the B-staged impregnated material 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 in. (50 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 in. (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 in. (600 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°C + 3 (240°F + 5) in 20 - 30 min. with no pressure in the autoclave.
- 4.5.3.3.2 When the thermocouple in the laminate panel reaches 115°C + 3 (240°F + 5), apply 100 psi (690 kPa) autoclave pressure.
- 4.5.3.3.3 Continue to raise the autoclave air temperature to 180°C + 3 (360°F + 5) at a uniform rate to achieve 180°C (360°F) in 70 - 85 minutes.

- 4.5.3.3.4 Maintain the autoclave air temperature at $180^{\circ}\text{C} + 3$ ($360^{\circ}\text{F} + 5$) for not less than 2 hr after the thermocouple in the laminate panel reaches 170°C (340°F).
- 4.5.3.3.5 Cool the laminate panel to below 65°C (150°F) under at least 40 psig (275 kPag) 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 follows:

Time hr, ± 0.1	Post Cure Temperature	
	$^{\circ}\text{C} \pm 5$	$^{\circ}\text{F} \pm 9$
12	175	345
12	200	390
12	230	445
12	260	500
12	290	555
24	315	600

4.6 Reports:

- 4.6.1 The vendor of resin shall furnish with each shipment a report showing the results of tests to determine conformance to the acceptance test requirements and stating that the product conforms to the other technical requirements of this specification. This report shall include the purchase order number, AMS 3619B, vendor's product designation, lot number, date of manufacture, and quantity.
- 4.6.1.1 A material safety data sheet conforming to AMS 2825, or equivalent, shall be supplied to each purchaser prior to, or concurrent with, the report of preproduction test results or, if preproduction testing be waived by purchaser, concurrent with the first shipment of resin for production use. Each request for modification of resin formulation shall be accompanied by a revised data sheet for the proposed formulation.
- 4.6.2 The vendor of finished or semi-finished parts shall furnish with each shipment a report showing the purchase order number, AMS 3619B, contractor or other direct supplier of resin, supplier's product designation, part number, and quantity. When resin for making parts is produced or purchased by the parts vendor, that vendor shall inspect each lot of resin to determine conformance to the requirements of this specification and shall include in the report either a statement that the resin conforms or copies of laboratory reports showing the results of tests to determine conformance.