Society of Automotive Engineers, Inc.
400 COMMONWEALTH DRIVE, WARRENDALE, PA. 15096

AEROSPACE MATERIAL SPECIFICATION

AMS 3619A
Superseding AMS 3619
Issued 3-1-74
Revised 10-15-79

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 plastic 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 (AMS) shall apply. The applicable issue of other documents shall be as specified in AMS 2350.

2.1 SAE Publications: Available from Society of Automotive Engineers, Inc., 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

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 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 25/25C
ASTM D2344 - Apparent Horizontal Shear Strength of Reinforced Plastics by Short Beam Method

2.3 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 qualification 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 psi	(MPa)	Flexural Modulus psi	(MPa)
25°C ± 3 (77°F ± 5)				
Minimum Average	65,000	(448)	2,500,000	(17,250)
Individual Minimum	58,500	(403)	2,250,000	(15,500)
315°C ± 5 (600°F ± 9)				
Minimum Average	45,000	(310)	2,000,000	(13,800)
Individual Minimum	40,500	(279)	1,800,000	(12,400)
315°C ± 5 (600°F ± 9) after 100 hr at 315°C ± 5 (600°F ± 9)				
Minimum Average	35,000	(241)	2,000,000	(13,800)
Individual Minimum	31,500	(217)	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 psi	(MPa)	Compressive Modulus psi	(MPa)
25°C ± 3 (77°F ± 5)				
Minimum Average	45,000	(310)	2,750,000	(18,950)
Individual Minimum	40,500	(279)	2,475,000	(17,050)
315°C ± 5 (600°F ± 9)				
Minimum Average	35,000	(241)	2,250,000	(15,500)
Individual Minimum	31,000	(214)	2,025,000	(13,950)
315°C ± 5 (600°F ± 9) after 100 hr at 315°C ± 5 (600°F ± 9)				
Minimum Average	30,000	(207)	2,000,000	(13,800)
Individual Minimum	27,000	(186)	1,800,000	(12,400)

3.4.3 Short Beam Shear Strength: Shall be as follows, determined on 5 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 ± 9)		
Minimum Average	3500	(24.1)
Individual Minimum	3150	(21.7)

3.5 Quality: The product shall be uniform in quality and condition, clean, smooth, and free from foreign materials and from imperfections detrimental to fabrication, appearance, or performance of parts.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection: The vendor of the product shall supply all samples 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 perform such confirmatory testing as he deems 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 Qualification Tests: Tests to determine conformance to all technical requirements of this specification are classified as qualification tests and shall be performed on the initial shipment of resin to a purchaser, when a change in material or processing 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, qualification test material shall be submitted to the cognizant qualification 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: Each lot of resin shall be sampled at random to provide sufficient material to perform all required tests. The number of specimens for each test 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 continuous production run from the same batches of raw materials under the same fixed conditions and submitted for vendor's inspection at one time. A lot shall not exceed 1000 gal (3800 L) of resin and may be packaged in small quantities as noted in 5.1.1 under a basic lot approval as long as the lot identification is maintained.

4.3.1.2 A batch shall consist of 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) for resin 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 Qualification 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. Results of tests on production resin shall be essentially equivalent to those on the approved sample.

4.4.2 Vendor shall establish parameters for the control factors of processing which will produce resin meeting the technical requirements of this specification. These shall constitute the approved procedures and shall be used for manufacturing production resin. If necessary to make any change in parameters for the control factors of processing, vendor shall submit for reapproval a statement of the proposed changes in material or processing and, when requested, sample resin. Production resin made to the revised procedures shall not be shipped prior to receipt of reapproval.

4.4.2.1 Control factors for producing resin include, but are not limited to, the following:

Compound ingredients and proportions thereof within established limits
Sequence of mixing compound ingredients
Type of mixing equipment
Reaction time/temperature/pressure schedule
Methods of routine inspection

4.4.2.1.1 Any of the above control factors of processing for which parameters are considered proprietary by the vendor may be assigned a code designation. Each variation in such parameters shall be assigned a modified code designation.

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°C ± 5 (600°F ± 9) 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 ± 0.1 with 24 g ± 1 of acetone, followed by devolatilizing and curing as follows:

Time min. ± 0.1	Temperature °C ± 5	(°F ± 9)
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 122°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 ± 9) 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. (697 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°C ± 3 (360°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 °C ± 5	(°F ± 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 three copies of 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, material specification number and its revision letter, vendor's product designation, lot number, date of manufacture, and quantity.

4.6.1.1 A Material Safety Data Sheet conforming to AMS 2825 shall be supplied to each purchaser prior to, or concurrent with, the report of qualification test results or, if qualification testing be waived by purchaser, concurrent with the first shipment of resin for production use. Each request for modification of formulation shall be accompanied by a revised data sheet for the proposed formulation.