SAE The Engineering Resource For Advancing Mobility®
400 COMMONWEALTH DRIVE WARRENDALE PA 15096

AEROSPACE MATERIAL SPECIFICATION

AMS 3701

Issued 7-1-84
Revised

EPOXY RESIN, TETRAGLYCIDYL METHYLENEDIANILINE (TGMDA) 10,000 - 14,000 Centipoise Viscosity

1. SCOPE:

1.1 Form: This specification covers a prepolymer in the form of a liquid.

1.2 Application: Primarily for use in manufacture of high-temperature composite matrices and adhesive resins.

2. APPLICABLE DOCUMENTS: The following publications form a part of this specification to the extent specified herein. The latest issue of Aerospace Material Specifications and Aerospace Recommended Practices 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

2.1.2 Aerospace Recommended Practices:

ARP 1610 - Physio-Chemical Characterization Techniques, Epoxy Adhesive and Prepreg Resin Systems

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

ASTM D1652 - Epoxy Content of Epoxy Resins
ASTM D1726 - Hydrolyzable Chlorine Content of Liquid Epoxy Resins
ASTM D2393 - Viscosity of Epoxy Resins and Related Components

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 tetraglycidyl methylenedianiline (TGMDA), a single-component, tetrafunctional epoxy resin derived from the reaction of epichlorohydrin with methylene dianiline.

3.2 Impurities: Shall be as follows, determined in accordance with specified test methods:

3.2.1	Hydrolyzable Chlorine, max	0.35%	ASTM D1726
3.2.2	Volatile Content, max	0.7%	4.5.1
3.2.3	Acetone Solubles, max	0.5%	4.5.2

3.3 Properties: Resin shall conform to the following requirements, determined in accordance with specified test methods:

3.3.1	Viscosity at 50°C (122°F)	10,000 - 14,000 cps	ASTM D2393
3.3.2	Epoxy Content (equiv. per 100 g)	0.75 - 0.95	ASTM D1642
3.3.3	Molecular Weight Distribution		4.5.3
	$\overline{M}_W$	400 - 800	
3.3.4	Thermal Analysis by DSC		
	T_{OS}	262° - 282°C (502° - 540°F)	4.5.4
	T_{PEAK}	282° - 302°C (540° - 574°F)	

3.4 Quality: The resin, as received by purchaser, shall be uniform in quality and condition, clean, and free from foreign materials and contaminants detrimental to usage of the resin.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection: The vendor of resin 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 resin conforms to the requirements of this specification.

4.2 Classification of Tests:

4.2.1 Acceptance Tests: Tests to determine conformance to requirements for impurities (3.2), viscosity (3.3.1), and epoxy content (3.3.2) 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 or processing, or both, requires reapproval as in 4.4.2, and when purchaser deems confirmatory testing to be required.

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 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 continuous 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 may be packaged in smaller quantities under the basic lot approval provided 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 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 or 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: Tests to determine conformance to the requirements of this specification shall be conducted as follows:

4.5.1 Volatile Content: Shall be determined by accurately weighing (W_1), to the nearest 0.0001 g, four samples weighing not less than 1.0 g each. Using porcelain crucibles (preferably covered) previously brought to constant weight by igniting at 845°C ± 25, (1555°F ± 45), dry samples in a circulating-air oven at 175°C ± 5 (350°F ± 9) for 15 min. ± 1. Cool in a desiccator and reweigh (W_2). Calculate the individual results of the four determinations using the following equation:

$$\text{Volatile Content, \%} = \left(\frac{W_1 - W_2}{W_1}\right) \times 100$$

4.5.1.1 Report both the individual results and the arithmetic mean.

4.5.2 Acetone Soluble: Shall be determined on the four samples of 4.5.1 from which volatiles have been removed.

4.5.2.1 Record weight of volatile-free samples, each about 1 g, to the nearest 0.0001 g (W_2).

4.5.2.2 Place the samples in separate containers and extract the resin by heating in 50 mL of acetone for not less than 2 minutes. Time starts when the acetone starts to boil. After 2 min. of boiling, decant the solvent. Repeat the extraction for three complete wash cycles. Report the temperature used.

4.5.2.3 Dry the specimens by placing them in a circulating-air oven maintained at 175°C ± 5 (350°F ± 9) for not less than 1 hr or until the weight is constant. Remove and place in a desiccator.

4.5.2.4 After 10 min., remove specimens from the desiccator and weigh to the nearest 0.0001 g (W_3).

4.5.2.5 Calculate the individual results for acetone soluble content using the following equation:

$$\text{Acetone Soluble, \% by weight} = \left(\frac{W_2 - W_3}{W_2}\right) \times 100$$

where, W_2 = Original volatile-free weight of the specimen, g

W_3 = Final weight of specimen, g

4.5.2.6 Report both the individual results and the arithmetic mean (average results).

4.5.3 Molecular Weight Distribution: Shall be determined by gel permeation chromatography (GPC) in accordance with ARP 1610. Ratios of the peak heights of the higher molecular weight species to that of the TGMDA monomer shall be reported.

4.5.4 Thermal Analysis by DSC: Shall be used to measure the starting temperature and the exotherm peak temperature for the self-curing reaction in accordance with ARP 1610. The temperatures obtained for a heating rate of 5°C/min. shall be reported.

4.6 Reports:

4.6.1 The vendor of resin shall furnish with each shipment a report showing the results of tests for impurities and properties, including copies of thermograms and chromatograms, and stating that the product conforms to the other technical requirements of this specification. This report shall include the purchase order number, AMS 3701, 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 3701, contractor or other direct supplier of resin, vendor'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.