

AEROSPACE MATERIAL SPECIFICATION

SAE

AMS 3701B

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Superseding AMS 3701A

Submitted for recognition as an American National Standard

(R) EPOXY RESIN, TETRAGLYCIDYL METHYLENEDIANILINE (TGMDA) 10,000 to 14,000 Centipoise (10 to 14 Pa-s) Viscosity

1. SCOPE:

1.1 Form:

This specification covers a prepolymer in the form of a liquid.

1.2 Application:

This product has been used typically in the manufacture of high-temperature composite matrices and adhesive resins, 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 following publications form a part of this specification to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order.

2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 2825 Material Safety Data Sheets

ARP1610 Physical-Chemical Characterization Techniques, Epoxy Adhesive and Prepreg Resin Systems

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2.2 ASTM Publications:

Available from ASTM, 1916 Race Street, Philadelphia, PA 19103-1187.

ASTM D 1652 Epoxy Content of Epoxy Resins

ASTM D 1726 Hydrolyzable Chlorine Content of Liquid Epoxy Resins

ASTM D 2393 Viscosity of Epoxy Resins and Related Components

2.3 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

MIL-STD-2073-1 DOD Materiel, Procedures for Development and Application of Packaging Requirements

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 conform to the requirements shown in Table 1, determined in accordance with specified test methods.

TABLE 1 - Maximum Impurities

Paragraph	Impurity	Requirement	Test Method
3.2.1	Hydrolyzable Chlorine	0.35%	ASTM D 1726
3.2.2	Volatile Content	0.7%	4.5.1
3.2.3	Acetone Solubles	0.5%	4.5.2

3.3 Properties:

Resin shall conform to the requirements, shown in Table 2, determined in accordance with specified test methods.

TABLE 2 - Properties

Paragraph	Property	Requirement	Test Method
3.3.1	Viscosity at 50 °C (122 °F)	10,000 to 14,000 centipoise (10 to 14 Pa·s)	ASTM D 2393
3.3.2	Epoxy Content	0.75 to 0.95 equivalent per 100 grams	ASTM D 1652
3.3.3	Molecular Weight Distribution	400 to 800 \bar{M}_w	4.5.3
3.3.4	Thermal Analysis by DSC		4.5.4
	T_{OS}	262 to 282 °C (504 to 540 °F)	
	T_{PEAK}	282 to 302 °C (540 to 576 °F)	

3.4 Quality:

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

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection:

The manufacturer of resin shall supply all samples for required tests and shall be responsible for performing all required tests. 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: Impurities (3.2), viscosity (3.3.1), and epoxy content (3.3.2) are acceptance tests and shall be performed on each lot.

4.2.2 Preproduction Tests: 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: 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 manufacturer's inspection at one time.
- 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 A statistical sampling plan, acceptable to purchaser, may be used in lieu of sampling as in 4.3.1.

4.3.2 For Preproduction Tests: Shall be acceptable to purchaser.

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 Manufacturer 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. If necessary to make any change in ingredients, in type of equipment for processing, or in manufacturing procedures, manufacturer 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:

Tests to determine conformance to specified requirements shall be conducted as follows:

- 4.5.1 Volatile Content: Shall be determined by accurately weighing (W_1), to the nearest 0.0001 gram, four samples weighing not less than 1.0 gram each. Using porcelain crucibles (preferably covered) previously brought to constant weight by igniting at $845\text{ }^\circ\text{C} \pm 25$, ($1553\text{ }^\circ\text{F} \pm 45$), dry samples in a circulating-air oven at $175\text{ }^\circ\text{C} \pm 5$ ($347\text{ }^\circ\text{F} \pm 9$) for 15 minutes ± 1 . Cool in a desiccator and reweigh (W_2). Calculate the individual results of the four determinations using Equation 1.

$$\text{Volatile Content, \%} = \frac{(W_1 - W_2)}{W_1} \times 100 \quad (\text{Eq.1})$$

- 4.5.1.1 Report both the individual results and the arithmetic mean.
- 4.5.2 Acetone Solubility: 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 approximately 1 gram, to the nearest 0.0001 gram (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 two minutes. Time starts when the acetone starts to boil. After two minutes 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 $175\text{ }^\circ\text{C} \pm 5$ ($347\text{ }^\circ\text{F} \pm 9$) for not less than 1 hour or until the weight is constant. Remove and place in a desiccator.
- 4.5.2.4 After 10 minutes, remove specimens from the desiccator and weigh to the nearest 0.0001 gram (W_3).
- 4.5.2.5 Calculate the individual results for acetone solubility content using Equation 2.

$$\text{Acetone Soluble, \% by weight} = \frac{(W_2 - W_3)}{W_2} \times 100 \quad (\text{Eq.2})$$

where:

W_2 = Original volatile-free weight of the specimen, grams
 W_3 = Final weight of specimen, grams

- 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 ARP1610. 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 Differential Scanning Calorimeter (DSC): Shall be used to measure the starting temperature and the exotherm peak temperature for the self-curing reaction in accordance with ARP1610. The temperatures obtained for a heating rate of 5 C degrees per minute (9 F degrees per minute) shall be reported.

4.6 Reports:

The supplier 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. This report shall include the purchase order number, lot number, AMS 3701B, manufacturer's identification, date of manufacture, and quantity.