AEROSPACE MATERIAL SPECIFICATION

AMS3729	REV. A

Issued 1985-10
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Reaffirmed 2014-08

Superseding AMS3729

Epoxy Resin Matrix, Thermosetting
Moderate Temperature Resistant, Unfilled

RATIONALE

AMS3729A has been reaffirmed to comply with the SAE five year review policy.

1. SCOPE:

1.1 Form:

This specification covers a heat-reactive, thermosetting, epoxy resin matrix in the form of a semi-solid. The resin matrix thermally cures to an epoxy polymer.

1.2 Application:

This product has been used typically in manufacture of moderate temperature, high strength composite prepreg materials, 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.

TO PLACE A DOCUMENT ORDER: **Tel: 877-606-7323 (inside USA and Canada)**
Tel: +1 724-776-4970 (outside USA)
Fax: 724-776-0790
Email: CustomerService@sae.org
SAE WEB ADDRESS: **http://www.sae.org**

SAE values your input. To provide feedback on this Technical Report, please visit
http://www.sae.org/technical/standards/AMS3729A

2.1 SAE Publications:

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

AMS 2825 Material Safety Data Sheets
AMS 3099 Curing Agent, Solid Aromatic Amine, 4,4'-Diaminodiphenyl Sulfone
AMS 3701 Epoxy Resin, Tetraglycidyl Methylenedianiline (TGMDA), 10,000 - 14,000 Centipoise Viscosity
AMS 3705 Epoxy Resin, Cycloaliphatic Liquid
AMS 3728 Epoxy Cresol Novolac Resin, Low Molecular Weight

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

2.2 ASTM Publications:

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

ASTM D 1652 Epoxy Content of Epoxy Resins

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 a thermosetting formulation conforming to the percentages by weight shown in Table 1.

TABLE I - Composition

Component	min	max
Tetraglycidyl Methylenedianiline, AMS 3701	54.5	58.5
Epoxy Resin, Cycloaliphatic Liquid, AMS 3705	8.0	10.0
Epoxy Cresol Novolac Resin, AMS 3728	7.5	9.5
4,4' - Diaminodiphenyl Sulfone, AMS 3099	23.0	27.0
Boron Trifluoride Amine Complex	0.8	1.6

3.2 Impurities:

Volatile content shall be not greater than 1.5%, determined in accordance with 4.5.1:

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	Epoxy Content (equivalent per 100 grams)	0.36 to 0.62	ASTM D 1652
3.3.2	4,4' - Diaminodiphenyl Sulfone	23.0 to 27.0%	4.5.2
3.3.3	Boron Trifluoride Amine Complex	0.8 to 1.6%	4.5.3
3.3.4	Epoxy Resin, Cycloaliphatic Liquid	8.0 to 10.0%	4.5.4
3.3.5	Thermal Analysis by Differential Scanning Calorimeter (DSC)	Tpeak 202 to 222 °C (396 to 432 °F)	4.5.5
3.3.6	Resin Rheology Flow Number	5.0 to 10.0 minutes/poise	4.5.6

3.4 Quality:

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

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection:

(R)

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: Tests for impurities (3.2) and epoxy content (3.3.1) 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:
(R)

Shall be as follows:

4.3.1 For Acceptance Tests: Each lot of resin shall be sampled at random to provide sufficient resin 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 When a statistical sampling plan has been agreed upon by purchaser and
(R) supplier, 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 supplier.

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:

Shall be as follows:

4.5.1 Volatile Content: Shall be determined by accurately weighing (W,) to the nearest 0.1 mg, four samples weighing not less than 1.0 gram each. Using porcelain crucibles (preferably covered) previously brought to constant weight by igniting at 845 °C ± 25 (1553 °F ± 45), dry samples in a circulating-air oven at 175 °C ± 5 (347 °F ± 9) for 15 minutes ± 1. Cool in a desiccator and reweigh (W_2). Calculate the individual results of the four determinations using Equation 1.

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

4.5.1.1 Report both the individual results and the arithmetic mean.

4.5.2 4,4' - Diaminodiphenyl Sulfone (DDS) Content: Shall be determined by infrared absorption spectroscopy (IR) in accordance with ARP1610. The percent by weight DDS shall be reported.

4.5.3 Boron Trifluoride Amine Complex Content: Shall be determined by atomic absorption (AA) in accordance with ARP1610. The percent by weight accelerator shall be reported.

4.5.4 Epoxy Cycloaliphatic Liquid Resin (ECLR) Content:

4.5.4.1 Weigh, to the nearest 0.1, 50, 100, 150, and 200 mg samples of the ECLR. Transfer the samples to 100 mL volumetric flasks and dilute to volume with chloroform.

4.5.4.2 Place two 2.0 mm potassium bromide (KBr) precision path length, matched liquid cells filled with chloroform in both the sample and reference beams of the infrared (IR) spectrophotometer. Scan the region from 1600 to 1800 cm^{-1} to ensure a clean baseline free of extraneous absorptions. For the Beckman IR-9 infrared spectrophotometer, instrumental parameters shown in Table 3 are suggested:

TABLE 3 - Instrumental Parameters

Parameter	Value
Scan range	1600 to 1800 cm^{-1}
Scan Speed	40 cm^{-1}/minute
Slit	Routine (2 times standard)
Gain	3%
Period	2
Scale	0 to 100% T
Reference	Chloroform

4.5.4.3 Replace the chloroform in the sample cell with a standard sample solution. Flush the sample cell 2 to 3 times with the solution to be analyzed. Record the spectrum from 1600 to 1800 cm-1 for each standard solution.

4.5.4.4 ECLR exhibits an absorption maximum at 1730 cm^{-1}. Construct a calibration curve on linear graph paper for percent ECLR by plotting the absorbance values for the standard solutions, minus the baseline absorbance, versus their concentration in mg/mL of ECLR.

4.5.4.5 Weigh, to the nearest 0.1 mg, a 2.5 gram sample of the epoxy resin matrix or adhesive. Transfer this sample to approximately 70 mL of chloroform. Thoroughly agitate the mixture in an ultrasonic bath for 15 minutes. Filter the mixture using a glass crucible, wash the crucible and contents with two 10 mL portions of fresh chloroform, and dilute the filtrate and washings to volume with chloroform in a 100 mL volumetric flask. Shake the solution to ensure adequate mixing.

4.5.4.6 Analyze the chloroform solution in accordance with 4.5.4.2 and 4.5.4.3.

4.5.4.7 Take the absorbance value of the sample from the IR spectra and determine the amount of ECLR in the solution from the calibration curve.

4.5.4.8 Record the ECLR value (mg/mL).

4.5.4.9 Calculate weight percent ECLR in the resin matrix using Equation 2.

$$\%ECLR = \frac{ECLR\ concentration\ mg/mL}{Sample\ concentration\ mg1mL} X\ 100 \qquad \text{(Eq.2)}$$

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

4.5.6 Resin Rheology, Flow Number:

4.5.6.1 Remove epoxy resin matrix from the freezer and allow to warm to room temperature before opening the package. Hydraulically press resin samples between teflon cloth to approximately 5 mil (0.13 mm) thickness and place uncovered in a desiccator at room temperature for 24 hours prior to testing.

4.5.6.2 After desiccation, cut 24-mm circular resin wafers for rheology tests.

4.5.6.3 Determine the flow characteristics of the resin using a Rheometric Dynamic Spectrometer Model RDS-7700, or equivalent. With the spectrometer in the parallel plate mode, the instrumental parameters and test conditions are shown in Table 4.

TABLE 4 - Instrumental Parameters and Test Conditions

Parameter	Value
Sample Plates	25 mm disposable
Gap Setting	0.5 mm ± 0.01 mm (0.02 inch ±0.0004)
Starting Temperature	50 °C (122 °F)
Strain	50%
Frequency	10 rads/second
Heating Rate	50 °C (122 °F) (start) to gel temperature at 1 C degree per minute (1.8 F degree per minute)
Program Plot	Viscosity versus time. Plot data to 10^3 poise.

4.5.6.4 Calculate flow number by integrating Equation 3.

$$\text{Flow Number} = \int_{t_o}^{t_{gel}} \frac{dt}{n} \quad \left(\frac{\text{minutes}}{\text{poise}}\right) \qquad \text{(Eq.3)}$$

where:

t = time (minutes)
n = time dependent viscosity (poise)
t_{gel} = t at n = 10^3 poise

4.5.6.5 Report the flow number as ($\frac{\text{minutes}}{\text{poise}}$).

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 resin conforms to the other technical requirements. This report shall include the purchase order number, lot number, AMS 3729A, manufacturer's product designation, date of manufacture, and quantity.

4.6.1 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.

4.7 Resampling and Retesting:
(R)

If any specimen used in the above tests fails to meet the specified requirements, disposition of the resin may be based on the results of testing three additional specimens for each original nonconforming specimen. Failure of any retest specimen to meet the specified requirements shall be cause for rejection of the resin represented. Results of all tests shall be reported.

5. PREPARATION FOR DELIVERY:

5.1 Packaging and Identification:

5.1.1 A lot of resin may be packaged in small quantities and delivered under the basic lot approval provided lot identification is maintained.

5.1.2 Resin shall be packaged in airtight containers and maintained at a temperature not higher than -18 °C (0 °F). Type and size of containers shall be agreed upon by purchaser and supplier.