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AEROSPACE MATERIAL SPECIFICATION

AMS 3099

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Revised

CURING AGENT, SOLID AROMATIC AMINE
4,4' -Diaminodiphenyl Sulfone

1. SCOPE: This specification covers an aromatic amine curing agent, used with epoxy resins, in the form of a light brown, nearly odorless powder.

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 the 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

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

ASTM E203 - Test for Water Using Karl Fischer Reagent
ASTM E324 - Relative Initial and Final Melting Points and the Melting Range of Organic Chemicals

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 4,4' -diaminodiphenyl sulfone, an aromatic amine, formed by reacting chlorobenzene and sulfur trioxide and subsequently reacting the compound with ammonia.

3.2 Properties: Curing agent shall conform to the following requirements, determined in accordance with specified test methods:

3.2.1	Melting Point	170° - 180°C (340° - 355°F)	ASTM E324
3.2.2	Assay (dry basis)	96 - 100%	4.5.1
3.2.3	Water Content, max	1%	ASTM E203

3.3 Quality: The product, as received by purchaser, shall be uniform in quality and condition, and free from foreign materials 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 melting point (3.2.1), assay (3.2.2), and water content (3.2.3) 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 curing agent 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.

4.2.2.1 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 curing agent 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 curing agent 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. An inspection lot shall not exceed 500 lb (225 kg) and may be packaged in small quantities under the basic lot approval provided lot identification is maintained.

4.3.1.2 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 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 curing agent shall be approved by purchaser before curing agent for production use is supplied, unless such approval be waived by purchaser. Results of tests on production curing agent 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 curing agent which are essentially the same as those used on the approved sample curing agent. 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 curing agent. Production curing agent made by the revised procedure shall not be shipped prior to receipt of reapproval.

4.5 Test Methods:

4.5.1 Assay (dry basis): Assay shall be determined, on a dry basis, in accordance with 4.5.1.1.

4.5.1.1 Perchloric-Glacial Acetic Acid Method:

4.5.1.1.1 Equipment: The following equipment, or its equivalent, shall be used:

Calomel reference electrode.

Glass electrode, all purpose.

Beckman zeromatic pH meter.

4.5.1.1.2 Reagents:

Acetic acid, glacial, reagent grade.

Acetic anhydride, reagent grade.

Potassium acid phthalate, primary standard.

Perchloric acid, reagent grade.

Acetonitrile, reagent grade.

4.5.1.1.3 Perchloric Acid Solution (0.1 Normal):

4.5.1.1.3.1 Preparation: Place approximately 250 mL of glacial acetic acid in a 1000 mL volumetric flask. Add 8 to 9 mL of 70% perchloric acid or 10 to 11 mL of 60% perchloric acid to the flask and mix. Add 15 mL of acetic anhydride, dilute to volume with glacial acetic acid, and mix. Stopper the flask and let stand for not less than 8 hours. Weigh 0.45 to 0.50 g, to the nearest 0.1 mg of dried potassium acid phthalate into a 250 mL beaker. Add 50 to 100 mL of glacial acetic acid, stir and heat gently until all of the potassium acid phthalate has dissolved. Cool to room temperature, immerse the electrodes in the sample, and titrate potentiometrically with the perchloric acid solution. Run a blank determination.

4.5.1.1.3.2 Calculation:

$$\text{Normality of perchloric acid solution} = \frac{(W)\ (4.897)}{(V_1 - V_2)}$$

Where: W = weight of potassium acid phthalate, g

V_1 = volume of perchloric acid solution required to titrate sample, mL

V_2 = volume of perchloric acid solution required to titrate blank, mL

4.897 = reciprocal of milliequivalent weight of potassium acid phthalate

4.5.1.1.3.3 Normality: The normality between duplicate standardizations shall not differ more than 0.0001.

4.5.1.1.4 Assay Procedure: Weigh to the nearest 0.1 mg a 2 to 4 milliequivalent sample into a 250 mL beaker. Add 50 to 100 mL glacial acetic acid and warm the solution, if necessary, to completely dissolve the sample. A suggested practice is to pre-dissolve the 4,4' -diaminodiphenyl sulfone in a small quantity of acetonitrile and then add the glacial

4.5.1.1.4 (Continued):

acetic acid to the acetonitrile solution. After the sample has dissolved, discontinue warming and let the solution cool to room temperature. Place the electrodes in the solution and titrate potentiometrically with the standardized perchloric acid solution. Run a blank determination and calculate as follows:

$$\text{Assay (dry basis), percent} = \frac{(V_1 - V_2)\,(N)\,(0.124)}{W - (W)\,\frac{\text{(percent water from 3.2)}}{100}} \times 100$$

Where: V_1 = volume of perchloric acid solution required to titrate sample, mL

V_2 = volume of perchloric acid solution required to titrate blank, mL

N = normality of perchloric acid solution

W = weight of 4,4' -diaminodiphenyl sulfone sample, g

4.5.1.1.4.1 Report the average of not less than 2 determinations.

4.6 Reports: The vendor of the curing agent shall furnish with each shipment a report showing the results of tests for melting point, assay content, and water content and stating that the product conforms to the other technical requirements of this specification. This report shall include the purchase order number, AMS 3099, vendor's product designation, lot number, 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 curing agent for production use. Each request for modification of curing agent formulation shall be accompanied by a revised data sheet for the proposed formulation.

4.7 Resampling and Retesting: If any specimen used in the above tests fails to meet the specified requirements, disposition of the curing agent 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 curing agent represented and no additional testing shall be permitted. Results of all tests shall be reported.

5. PREPARATION FOR DELIVERY:

5.1 Packaging and Identification: