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NCAMP Material <u>Procurement</u> Specification This specification is generated and maintained in accordance with NCAMP Standard Operating Procedures, NSP 100

Resin Specification: Syensqo Toughened Epoxy Resin (formerly Solvay)

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REVISIONS

Revision	Date	Description	
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1. SCOPE:

1.1 Form:

This specification and its associated detail specifications establish the requirements for resin intended to be used with dry fibers in an infusion process. Dry fibers may include plain weave, uni-tape, or non-crimp or unidirectional woven dry reinforcements. The detail specification shall provide specific requirements for the resin.

1.2 Application:

The resin is intended for use in fabrication of structures in support of the aerospace industry. The resin is designed specifically for resin infusion processes. The resin may be used alongside the qualified NCAMP fiber/fabric material specification and approved NCAMP process specification to produce laminates that will meet approved NCAMP laminate acceptance specification.

The use of this specification does not guarantee material or structural performance. Material users should be actively involved in evaluating material performance and quality including, but not limited to, performing regular purchaser quality control tests, performing periodic equivalency/additional testing, conducting statistical process control and conducting regular supplier audits if deemed relevant.

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 all 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.

1.4 Rounding of Values:

The following applies to all specified limits or requirements in this specification. For purposes of determining conformance with this specification, an observed value or a calculated value shall be rounded "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding method of ASTM E29.

1.5 Qualified Products:

This specification requires qualified products which are listed in the Qualified Products List (QPL) of each material slash sheet. In accordance with the requirements of this specification, the specific components and source(s) that make up the resin shall be considered qualified materials. Any materials not listed on the QPL of the detail

specification are not qualified. In addition, the production of the qualified products is controlled by an NCAMP approved process control document (PCD).

1.6 Detail Specification:

This base specification contains basic and unprocessed/uncured resin material requirements that apply to every product. The material slash sheet contain additional or superseding properties and requirements that apply to a specific product.

There shall be a base and detail specification for each class and style of fiber/reinforcement fabric, and a combined cured laminate acceptance specification. The fiber/fabric and resin specifications are intended to be used in tandem (with the referenced Process Specification) to produce the combined cured laminate. Use of non-referenced constituent materials are outside the scope of these specifications.

1.7 Change Control Approval:

The resin shall be produced in accordance with a manufacturer Process Control Document (PCD) that is reviewed and approved by NCAMP. Formal change notification and approval is required before a change may be implemented. DOT/FAA/AR-06/25 can be used as a guide to determine changes that require change notification. In general, Level 0 and level 1 are minor changes and level 2 through level 4 are considered major changes. Minor changes should be communicated to NCAMP using a change notice to ensure proper material change traceability. NCAMP approval is required for major changes, and is granted through Advance Change Notices (ACN). Prior to implementing a major change, the material supplier shall contact NCAMP with the following information:

- a. A detailed description of the change,
- b. A draft test plan to substantiate that the change will not affect the material properties, and
- c. A list of material users

NCAMP staff will communicate the proposed change(s) and obtain necessary suggestions and/or approvals from the material users and certification agencies. In general, FAA/DOT/AR-06/25 will be used as guidelines. The ACN along with test results will be reviewed by the material users and certification agencies. NCAMP staff will sign the ACN when a consensus is reached.

2 APPLICABLE DOCUMENTS

The latest issue of the NCAMP publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order unless otherwise specified. When a referenced document has been canceled and no superseding document has been specified, the last published issue of that document shall apply.

2.1 NCAMP Publications:

NPS 82401	Fabrication of NMS 241 Qualification, Equivalency, and Acceptance Test Panels for Vacuum Assisted Resin Transfer Molding of Carbon Fiber Reinforcements with Syensqo PRISM™ EP2400 toughened epoxy resin	
NMS 241R/1	NCAMP Material Procurement Specification Syensqo PRISM™ EP 2400 Toughened Epoxy Resin (formerly Solvay)	
NMS 241F	NCAMP Material Procurement Specification Fabric Specification Tenax [™] Dry Intermediate Modulus Carbon Fiber Reinforcements	
NMS 241F/1	NCAMP Material Procurement Specification. Tenax™ Dry Reinforcement (Carbon Fiber) Class 1, Style BA, Grade 380	
NMS 241F/2	NCAMP Material Procurement Specification. Tenax™ Dry Reinforcement (Carbon Fiber) Class 1, Style BD, Grade 380	
NMS 241F/3	NCAMP Material Procurement Specification. Tenax™ Dry Reinforcement (Carbon Fiber) Class 2, Style UD, Grade 190	
NMS 241	NCAMP Material Acceptance Specification. Vacuum Assisted Resin Transfer Molded Laminates Using Dry Reinforcement Fabric and Resin	
NMS 241/1	NCAMP Material Acceptance Specification Oven Cure of VARTM Processed Dry Reinforcements with Toughened Epoxy Resin - Tenax [™] Biaxial DRNF with Syensqo PRISM [™] EP2400 Resin	
NMS 241/2	NCAMP Material Acceptance Specification Oven Cure of VARTM Processed Dry Reinforcements with Toughened Epoxy Resin - Tenax [™] Bidiagonal DRNF with Syensqo PRISM [™] EP2400 Resin	

NMS 241/3NCAMP Material Acceptance Specification
Oven Cure of VARTM Processed Dry Reinforcements with
Toughened Epoxy Resin – Tenax™ UD Woven DRWF with Syensqo
PRISM™ EP2400 Resin

2.2 SACMA Publications

(available from American Composites Manufacturers Association, 1010 N Glebe Rd., Suite 450, Arlington, VA 22201, <u>http://www.acmanet.org</u>):

SACMA SRM 25R-94 Onset Temperature and Peak Temperature for Composite System Resins Using Differential Scanning Calorimetry (DSC)

2.3 ASTM Publications

(available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, <u>http://www.astm.org</u>)

ASTM D792	Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
ASTM D3171	Standard Test Method for Constituent Content of Composite Materials
ASTM D3418	Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry
ASTM D4287	Standard Test Method for High-Shear Viscosity Using a Cone/Plate Viscometer
ASTM D7028	Standard Test Method for Glass Transition Temperature (DMA Tg) of Polymer Matrix Composites by Dynamic Mechanical Analysis (DMA)
ASTM E29	Using Significant Digits in Test Data to Determine Conformance with Specifications
ASTM E168	General Techniques of Infrared Quantitative Analysis
ASTM E1252	Standard Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis

2.4 ISO Publications:

ISO 9000	Quality Management Systems	
ISO 9001	O 9001 Quality Management Systems: Requirements	
AS9100	Quality Management Systems - Requirements for Aviation,	
	Space, and Defense Organizations.	

2.5 Additional Publications:

29 CFR 1910.1200	Hazard Communication, Occupational Safety and Health		
	Standards		
DOT/FAA/AR-06/25	Preliminary Guidelines and Recommendations for the		
	Development of Material and Process Specifications for Carbon		
	Fiber-Reinforced Liquid Resin Molded Materials		
MIL-D-3464	Desiccants, Activated, Bagged, Packaging Use and Static		
	Dehumidification		
CMH-17	Composite Materials Handbook (formerly MIL-HDBK-17)		

3 Resin Description

The infusion resin detailed in this specification is a single part, 180°C (356°F) curing, toughened liquid epoxy resin system.

Additional material description is described in the material slash sheet governing the resin used.

4 TECHNICAL REQUIREMENTS:

4.1 Detail Specification:

The requirements for a specific resin product shall consist of all requirements specified herein in addition to requirements specified in the applicable detail specification. In case of conflict between requirements of this basic specification and an applicable detail specification, requirements of the detail specification shall govern.

4.2 Other Material Requirements:

The general definition for material batch (or lot) can be described as a quantity of material produced at the same time and under the same conditions from a well-defined collection of raw materials. The quantity of material must have minimal variation in properties throughout to be considered a unique batch (or lot) per DOT/FAA/AR-06/25. The description of a resin batch (or lot) is dependent on its mixing process and section 4.8.2 of the same document provides additional details. Constituent raw ingredients that are sourced to produce the resin shall be controlled within an NCAMP approved PCD. Any changes to the epoxy resin system or the infusion method used since qualification shall be re-approved by NCAMP. Specific details of the changes shall be documented.

Note: To maintain consistency with other documents relevant to the NMS 241 family, "lot" shall be used in reference to material batch defined above.

4.3 Resin Physical and Chemical Property Requirements:

4.3.1 Uncured Resin physical and chemical properties:

Each lot of neat resin shall be tested and meet the physical property requirements of Table 1 as specified below. Specific property requirement can be found in the respective NMS 241R slash sheet.

Property	Test Method ⁽¹⁾	Number of Replicates per lot
Volatile content	ASTM D3530	3
Min Viscosity	ASTM D2196/D4473	3
Viscosity at 100°C (212°F) at 0 hours (as received) and after 2 hours	ASTM D4287	3
Gel Time 70°C to gel at 2°C/min	87-AN-P82-04 ⁽²⁾	3
DSC for Cure Kinetics, heat reaction, onset temp, Peak Exotherm Temp	ASTM D3418	1
High-Pressure Liquid Chromatography (HPLC)	SACMA SRM 20R-94	1
Spectrophotometry (IR)	ASTM E168 ASTM E1252	1

⁽¹⁾ Specific procedures should be identical to those used in the original material qualification program.

⁽²⁾ Standard Operating Procedure for TA Instruments Ares-G2 viscometer.

4.3.2 Cured Resin physical and chemical properties:

Each lot of resin shall be tested and meet the cured resin chemical property requirements of Table 2 as specified below. Specific property requirement can be found in the respective NMS 241R slash sheet.

Table 2 – Cured Neat Resin Physical and Chemical Properties	

Property	Test Method ⁽¹⁾	Number of Replicates per lot
Density	ASTM D792	3
Glass Transition Temperature by DMA	ASTM D7028	3

⁽¹⁾ Specific procedures should be identical to those used in the original material qualification program.

4.4 Resin: Visual and Physical Requirements:

4.4.1 General:

The resin shall be uniform in quality and shall not contain contaminant or indication of being tampered. Any non-conformity shall not be detrimental to handling, infusion, cure, or physical and chemical properties.

4.4.2 Material Characteristics:

As a reactive resin, the material may be prone to exotherm if the appropriate processing time and temperature are not followed. Care shall be taken to ensure that manufacturer recommended processing time and temperature is followed.

4.4.3 Containers:

The total required quantity shall be specified by the purchaser. Typical weight of the canisters are 10 kg (22 lbs) or 200 kg (485 lbs).

4.4.4 Resin not conforming to requirements:

Non-conformance shall be documented and communicated to the purchaser. If accepted by the purchaser and product will not cause a loss in performance, the product may be shipped and used. Otherwise the non-conforming materials shall be logged and marked for discard.

4.5 Storage and Handling Requirements for Resin Component:

4.5.1 Storage Life and Conditions:

Resin Storage Life is 365 days from date of manufacture, when stored at -18°C (0°F) or below in a sealed container. Resin shall be shipped within 3 months from date of manufacture unless otherwise approved by the customer. Out life is 28 days when stored at 23°C (73°F) or below.

4.5.2 Thawing and Handling:

Allow the resin container to come to ambient temperatures prior to use. Preheat the resin to 60°C (140°F) in its container for transfer to the resin pot.

4.5.3 Shelf life extension:

Shelf life extension is not done for this material and is beyond the scope of this specification. All material used to fulfill this specification shall be within its shelf life.

4.6 Environmental, Health, and Safety:

- 4.6.1 Equipment, materials, solutions, and emissions (if applicable) shall be controlled, handled, used, and disposed of in accordance with all local, State, and Federal Government Safety, Health, and Environmental Affairs (SHEA).
- 4.6.2 The delivered constituent materials shall fulfill the local requirements of the health and safety laws of the country of the purchaser. When processing the materials in the composite shop, there shall be no health hazards or emissions that require special measures to be taken to protect the environment.
- 4.6.3 The manufacturer shall inform the purchaser about the safe handling procedures of the material. The Material Safety Data Sheet (MSDS) or Product Information on Safe Handling (PISH) shall be made available to the purchaser.

4.7 Defects During Usage:

4.7.1 Undocumented defects or non-conformance in excess, as defined by this specification, found in the materials after acceptance shall be discussed with the supplier and may be a cause for rejection. If rejected the defective material shall be returned to the supplier. Defects caused by the shipper/carrier and user mishandling or improper storage are not the responsibility of the supplier and shall not be cause for rejection back to the supplier.

4.8 Qualification Requirements:

Materials supplied against this specification have been qualified in accordance with an NCAMP test plan which includes testing of cured laminate panels using reinforcement materials.

4.9 Material Re-Qualification and Equivalency:

- 4.9.1 If any change occurs relevant to this specification or the PCD, NCAMP reserves the right to require a re-qualification by the resin manufacturer to validate that the changed material is equivalent to the material in the initial qualification. The extent of the re-qualification program will depend on the nature of the change of the material or the material processing. DOT/FAA/AR-06/25 provides guidance in this area.
- 4.9.2 Equivalency testing is limited to the evaluation of minor changes in a material's constituents, manufacturing process, or fabrication (e.g. curing) process used with a material. Significant changes to the material will require a full qualification program and a separate specification.
- 4.9.3 It is the responsibility of the material supplier to conduct testing to demonstrate that the current material, when processed to the baseline process specification, will generate composite properties statistically equivalent to the properties of the original materials.

4.10 Process Control Document:

- 4.10.1 The supplier shall prepare and control a Process Control Document (PCD). The PCD shall be considered proprietary and shall be protected in accordance with disclosure agreements signed by the supplier and NCAMP. The established Process Control Document (PCD) shall be presented to NCAMP upon request. NCAMP shall treat any information contained in the PCD as proprietary.
- 4.10.2 Changes to the PCD of a qualified material (as defined by DOT/FAA/AR-06/25) are subject to the written approval of NCAMP. Such changes may require substantial testing.

4.11 Traceability:

Each individual material and its constituents as defined by the PCD shall be identifiable at all stages of manufacture and delivery. The material manufacturer shall present evidence of the material traceability upon request.

4.12 Manufacturer's Responsibility:

- 4.12.1 The manufacturer is responsible for the development and manufacture of the constituent material submitted in accordance with this specification. Quality control by the manufacturer shall be in accordance with this specification.
- 4.12.2 Changes to the resin supplied against this specification require review and approval by NCAMP in accordance the NCAMP ACN process. Any testing required to validate the changes or adjustment of manufacturing materials, techniques and/or procedures is the manufacturer's responsibility.

4.13 Quality Management System:

The manufacturer's quality system shall be approved as defined in ISO 9000, ISO 9001, AS 9100 or equivalent.

5 QUALITY ASSURANCE:

5.1 Responsibility for Inspection:

Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all lot release test requirements for the resin product. Requirements are detailed in the procurement specification slash sheets.

The supplier may use their own facilities or any commercial laboratory acceptable to NCAMP. The purchaser or NCAMP reserves the right to perform additional tests to assure that the material furnished conforms to the prescribed requirements.

5.2 Classification of Tests and Inspections:

5.2.1 Qualification Tests:

The preproduction tests performed for material qualification are those tests performed on representative samples of each specific form of material to establish a qualified product in accordance with this specification. Qualification testing shall be in accordance with an NCAMP test plan.

5.2.2 Lot Release Tests:

Lot release tests shall be those tests performed by the supplier on representative samples taken from each production lot of each type of material submitted by the supplier for acceptance under contract or purchase order. Data and certification of data generated shall accompany each shipment of material. Lot release test of the raw constituents may be repeated by the purchaser if desired (optional).

5.2.3 Additional Testing:

The purchaser reserves the right to perform additional testing to confirm the supplier's certification data, and to approve incoming material for use in the fabrication of production parts. Each lot of resin product may be examined by the purchaser for appearance, color uniformity, imperfections which would be detrimental for use in the fabrication of parts, and for quality of workmanship.

5.2.4 Laminate Inspection Tests:

The laminate inspection tests shall be those tests performed by the purchaser or approved test lab on representative samples taken from each production lot of each type of material delivered by the supplier. Resin is accepted if it meets the physical and chemical property requirements specified in Section 4.3.

Prior to use/application of the resin and fiber/fabric combination, the purchaser shall be responsible for the specification limits specified in the laminate acceptance specification.

This shall be conducted on the cured laminates produced when the resin is processed with the specified fiber/fabric (detailed in a separate specification) in accordance with the NCAMP Process Specification.

5.3 Supplier Statistical Process Control (SPC):

The supplier shall establish and maintain procedures and requirements for an SPC system based on Key Characteristics (KC) and Controlled Process Parameters (CPP). The KC are the material properties required for lot release per 4.4.1. The KC monitoring, typically using control charts, must be provided to material users, certification agencies, and NCAMP staff upon request. The CPP monitoring must also be provided to material users, certification agencies, and NCAMP staff upon request, but proprietary information may be coded or normalized. Alternatively, supplier may send the KC data to NCAMP for inclusion in the NCAMP's control charts which are available to the public.

5.4 **Product Certification:**

5.4.1 Lot Release Tests:

The following lot release tests shall be performed on each lot of the resin product as specified in this section and the detail specification.

- 5.4.2 Resin Physical and Chemical Properties: Test in accordance with the requirements of Table 1 and Table 2 and the detail specification. Shall be performed by the material supplier.
- 5.4.3 Certification of Conformance

The supplier shall furnish with each material shipment supplied against this specification one copy of a Certification of Conformance including certified test reports, confirming that all the material in the shipment complies with the requirements of this specification. The Certificate of Conformance shall include the following information:

- a. Manufacturer's identification.
- b. Manufacturer's material designation.
- c. Specification number, title, revision.
- d. Purchase order number.
- e. Date of resin manufacture.
- f. Results of lot release tests, including actual individual test data and average values.
- g. Results of any retests.
- h. Material lot numbers.
- 5.4.4 Records:

The following records shall be available for inspection by NCAMP and purchasers upon request.

- 5.4.4.1 The supplier shall keep on permanent file all records pertaining to the qualification of the candidate material.
- 5.4.4.2 The supplier shall keep the following records on file, for each material lot, for a minimum period of 7 years:
 - a. Full resin raw material constituent traceability.
 - b. All records pertaining to raw material receiving inspection and certification, inprocess records, and product testing specified in the supplier PCD.
 - c. All records pertaining to the SPC requirements which are specified in the supplier PCD.

5.5 Receiving Inspection:

Before the materials are accepted, the purchaser shall perform the following:

- 5.5.1 Verification: Material shall be inspected to assure that:
 - a. The material identification is correct.
 - b. The quantity is correct.
 - c. The required test data is received.
 - d. The Certificate of Conformance is received.
 - e. Each shipment of <u>resin</u> meets the requirements for storage temperature between the point when the purchaser assumes ownership and the point when the material is received.

(i) the purchaser shall document the procedure used to verify temperature exposure.

(ii) If any exposure exceed the maximum handling and mechanical life conditions, the material shall be rejected.

(iii) For exposures that exceed the storage temperature and are less than the maximum handling and mechanical life exposure conditions the purchaser may reject the material. If the purchaser accepts the material the time exceeding the storage temperature shall be deducted from the handling life for the material in that shipment.

5.5.2 Resin Product Material Testing:

The purchaser may choose to repeat the supplier lot release tests on the materials as part of the receiving inspection tests of each material lot. As use and confidence increase, the receiving inspection testing may be modified based on proven performance in cooperation with the material supplier, customer, and appropriate certification agency.

5.5.3 Cured Laminate Material Testing:

Upon verification and receipt of the constituent materials, the purchaser shall process the materials in accordance with the applicable NCAMP process specification to fabricate the panels defined in NMS 241. The combination of fiber/fabric and resin materials shall be accepted only when the cured laminate tests per NMS 241 meet the requirements of that specification.

This shall be the sole responsibility of the purchaser and resulting data shall be reviewed internally or with the appropriate certification agency. Conformance of processed laminate panel shall be in accordance with the appropriate NCAMP Material Acceptance Specification.

5.5.4 Re-Testing:

One retest is allowed for each test property. Additional re-test(s) is allowed only when one or more of the following conditions exist:

- a. The initial test was performed with significant deviation to the appropriate procedure. Significant deviations are those expected to affect the measured response.
- b. In the course of sample preparation, there was an occurrence known to cause or contribute to the observed test result(s).
- c. Standard statistical analysis procedures establish the suspect individual data point(s) as an outlier and there is a probable, if not provable, relationship to a deviation from required procedure.

6 Material Shipping:

6.1 Packaging:

- 6.1.1 The canisters shall be packed in a shipping container that will be acceptable for safe transportation by common carriers and shall include a packing list. It shall be secured to prevent damage/unnecessary movement during shipment. A data logger shall be used to record temperature and humidity exposure of the shipment to properly record the shelf life of the product.
- 6.1.2 A suitable label accompanying the material that is supplied shall be clearly marked with the following information:
 - a. Title, number and revision letter of this specification, (and the PCD if required by the detail specification).
 - b. Date of manufacture (or date of shipment if specified by the detail specification)
 - c. Quantity of material/product
 - d. Purchase order number and/or sales order number (not required on roll core)
 - e. Supplier's name
 - f. Supplier's lot number
 - g. Canister number (if applicable)
 - h. Material expiry
 - i. All material labeling shall comply with OSHA Hazard Communication, 29 CFR 1910.1200.
- 6.1.3 Shipping: The resin shall be maintained at or below 0°F during shipment. This can be achieved with use of dry ice or by refrigeration. A data logger shall be used to monitor temperature of the shipment and appropriate packaging and shipping shall be in accordance with supplier guidelines.

6.2 Receipt at Purchaser:

- 6.2.1 Store material at recommended storage temperature and humidity. The product shall be stored in a manner to prevent exposure to heat and UV light sources in an environment -18°C (~0°F) and below in its original sealed container.
- 6.2.2 If requested by the supplier and documented on the purchase order, retain temperature recording instruments (if used) for return to the supplier.

7 ACKNOWLEDGEMENT:

A vendor shall mention this specification number and the applicable detail specification number and their revision letters, if any, in all quotes and when acknowledging purchase orders.

8 **REJECTION**:

Product not conforming to this specification and the applicable detail specification, or to modifications authorized by purchaser, will be subject to rejection.

9 NOTES:

This section of the specifications is reserved for explanatory and other notes.

9.1 Definitions:

For definitions that are not provided in this specification or other applicable NCAMP specifications, the definitions in DOT/FAA/AR-06/25 shall apply. For definitions not provided in DOT/FAA/AR-06/25, the definitions in ASTM D3878 shall apply. For definitions not provided in ASTM D3878, the definitions in CMH-17 (formerly MIL-HDBK-17) shall apply. The document listed may or may not be used in their entirety.