

# **Measurement Good Practice Guide No. 64**

## **Fibre-Reinforced Plastic Composites - Qualification of Composite Materials**

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### **Abstract:**

Both composites suppliers and end-users are affected by the high cost of composite materials qualification. Qualifying a product against different user specifications, introducing new materials and finding data for materials selection and preliminary design can all be prohibitively costly due to repetition of testing.

This Measurement Good Practice Guide presents a Standard Qualification Plan (SQP) aimed at reducing the substantial costs involved in qualifying fibre-reinforced plastic composite materials. The SQP satisfies the minimum common requirements necessary to allow initial material selection, quality control and preliminary design to be undertaken. Specifically, it provides guidance on test method selection, panel and specimen preparation and machining, specimen sampling, conditioning and test environments, and statistical analysis of measured data. The results of an associated round-robin validation exercise are briefly presented demonstrating the suitability and robustness of test methods included in the SQP.

Collaboration with and support from national regulatory authorities such as the CAA is required, to encourage widespread use of the SQP. In addition, international collaboration (i.e. with the FAA) is key for harmonisation of qualification procedures.

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- Composites Testing Laboratory Ltd
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- Ford Motor Company Ltd
- Gearing Scientific
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- QinetiQ
- Rolls-Royce plc
- St. Bernard Composites Ltd
- Slingsby Aviation Ltd
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# **Fibre Reinforced Plastic Composites - Qualification of Composite Materials**

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# Nomenclature

## Test Methods

|      |                                   |
|------|-----------------------------------|
| SQP  | Standard qualification plan       |
| EQP  | Extended qualification plan       |
| ILSS | Interlaminar shear strength       |
| CAI  | Compression-after-impact          |
| DSC  | Differential scanning calorimetry |
| DMA  | Dynamic mechanical analysis       |

## Standards Organisations

|         |                                                                                                  |
|---------|--------------------------------------------------------------------------------------------------|
| AITM    | Airbus Industries Test Method                                                                    |
| ASTM    | American Society for Testing and Materials (produce ASTM standards)                              |
| BSI     | British Standards Institution (produce BS standards)                                             |
| CEN     | Comité Européen de Normalisation / European Committee for Standardisation (produce EN standards) |
| CENELEC | European Committee for Electrotechnical Standardisation                                          |
| IEC     | International Electrotechnical Commission                                                        |
| ISO     | International Organisation for Standardisation (produce ISO standards)                           |
| SACMA   | Suppliers of Advanced Composite Materials Association                                            |
| VAMAS   | Versailles Project on Advanced Materials and Standards                                           |

## ISO Standards

|      |                                     |
|------|-------------------------------------|
| WD   | working draft                       |
| NWI  | new work item                       |
| CD   | committee draft                     |
| DIS  | draft international standard        |
| FDIS | formal draft international standard |

## Others

|         |                                                                              |
|---------|------------------------------------------------------------------------------|
| $S_r$ - | standard deviation of test results obtained under repeatability conditions   |
| $S_R$ - | standard deviation of test results obtained under reproducibility conditions |
| $r$ -   | repeatability limit at 95 % confidence                                       |
| $R$ -   | reproducibility limit at 95 % confidence                                     |

# **1 Scope**

- 1.1 This guide details a general procedure for the initial qualification of composite materials in order to allow quality control, material selection and preliminary design to be undertaken.
- 1.2 The Standard Qualification Plan (SQP) presented in this guide contains the minimum common test requirements for initial materials qualification. Further test requirements are encompassed in an Extended Qualification Plan (EQP), which contains options to meet specific requirements. It will be of interest to the composites community in general and of particular use to material suppliers, end-users and designers.
- 1.3 This guide does not provide guidance on a full qualification programme, but is a standard building block for the initial stage of a qualification or certification programme.
- 1.4 This guide is generally applicable to preimpregnated (prepreg) fibre-reinforced thermoset and thermoplastic material systems. Individual standards referred to provide more details as to which classes of materials are suitable. It provides guidance on test method selection, panel and specimen preparation and machining, specimen sampling, conditioning and test environments, and statistical analysis of measured data.
- 1.5 The main text of this guide details the objectives and formulation of the SQP/EQP as well as the SQP/EQP itself. Details of an interlaboratory round-robin validation exercise, to assess the robustness and suitability of test methods included in the SQP, are given in the text and Appendix 1.

## 2. Introduction

Both composites suppliers and end-users are affected by the high cost of composite materials qualification. Qualifying a product against different user specifications, introducing new materials and finding data for materials selection and preliminary design can all be prohibitively costly due to repetition of testing. For example, one qualification programme for a new product cost £2 million to repeat ten times for different customers, rather than £130,000 if one qualification could have been accepted by all. An estimate of a 40% increase in design cost was also given due to the delayed availability of design data.

This guide details a Standard Qualification Plan (SQP) formulated by the National Physical Laboratory (NPL) that is aimed at reducing the substantial costs involved in qualifying materials on behalf of suppliers, designers and end-users of fibre-reinforced plastic composite materials. The SQP is aimed at satisfying the minimum common requirements necessary to allow initial material selection, quality control and preliminary design to be undertaken. Further qualification requirements, not met by the minimum SQP, are included in an Extended Qualification Plan (EQP). Figure 1 shows a schematic of the different stages of testing involved in component certification (typical of the aerospace industry, but applicable to other sectors) from coupon level up to full component testing. The SQP/EQP does not constitute a full qualification programme, but is intended to act as a building block

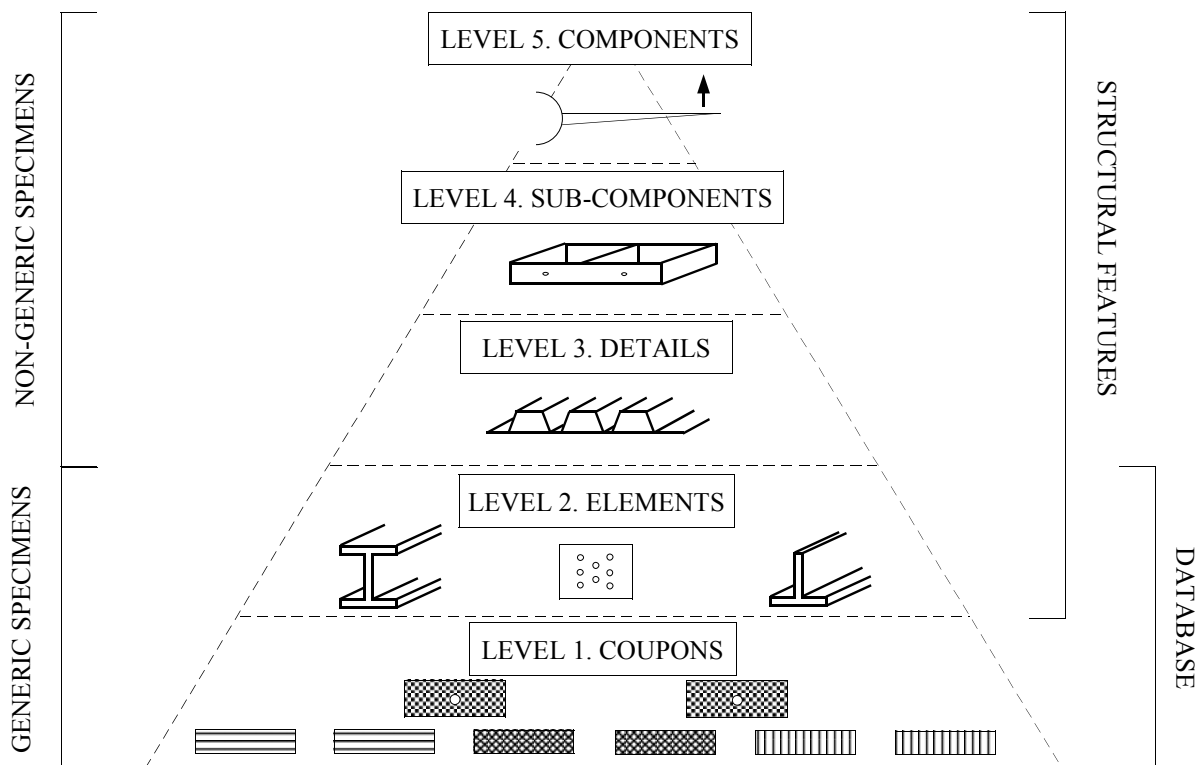


**Figure 1 : The certification pyramid [1]**

forming the initial stage of such a qualification or certification programme and is considered suitable for covering Level 1 tests of the certification pyramid (and possibly some Level 2 element tests). Level 1 and 2 type testing is mainly performed through the use of standard specimens providing generic data suitable for the population of shared material property databases.

The recent publication of a test plate manufacture standard (ISO/FDIS 1268), a suite of harmonised ISO test methods (mechanical, thermal and physical) and a data-sheet database standard (ISO 10350-2) has made the realisation of the SQP feasible. The following sections detail the process followed during the formulation of this draft procedure.

## **2.1 Industrial Consultation**

To determine which test methods should be included in the SQP/EQP, a survey was carried out by requesting several organisations in the composites industry (material suppliers, end-users, designers and certification bodies e.g. the Civil Aviation Authority [CAA]), to rank individual test methods with regard to their importance in materials qualification. The survey was performed using a detailed questionnaire followed by a number of ‘follow-up’ industrial visits in order to ensure that the SQP had a suitable content and format to be readily accepted and used by industry. Visits to discuss the requirements in more detail were made to the CAA, three Formula 1 manufacturers, two aerospace companies, a design consultancy and a pre-preg manufacturer.

## **2.2 Current Qualification Initiatives**

These requirements were compared with existing qualification initiatives (or those currently being developed) mostly driven by aerospace industry certification requirements in order to determine a suitable content, format and style for the SQP. The best known publicly available procedures are given in MIL-HDBK-17-1E [2], and are based on testing 30 specimens from 5 batches of material. Companies such as Airbus Industries also have well developed material qualification documentation [3, 4]. In the USA, the Advanced General Aviation Transport Experiments (AGATE) consortium have recently released a document under the title of ‘Material Qualification and Equivalency for Polymer Matrix Composite Material Systems’ [5], with the purpose of providing information and procedures for producing repeatable base composite material properties for primary and secondary aircraft structures. The AGATE consortium is a cost-sharing industry-university-government partnership initiated by the National Aeronautics and Space Administration (NASA) to create the technological basis for revitalisation of the US general aviation industry. It has more than 70 members from industry, universities, the Federal Aviation Administration (FAA), and other government agencies. The AGATE report considers a reduced, 18 specimen qualification plan (6 panels x 3 specimens) and an extended 55 specimen approach that uses, confusingly, alternatively 5 and 6 repeat sets of specimens from a total of 5 batches x 2 panels each.

The approach set out in the SQP procedure detailed in this guide is somewhat similar to that used in the AGATE initiative, but is based on measurements of 30 specimens (3 batches x 2 panels x 5 specimen sets). It does not prescribe a full materials qualification programme, nor is it solely intended for use by the aerospace industry. The 30 specimen approach can be shown statistically to be the preferred minimum number of measurements requirement [6]. In addition, the AGATE initiative uses mainly ASTM and SACMA test methods, whereas the SQP recommends, where available and suitable, ISO test standards.

## 2.3 Content and Format

After completing the initial industrial consultation process, a draft SQP document was written [7]. This section provides justification on the content and format of the draft SQP/EQP which is not included in the procedure itself.

Based on the results of the industrial consultation exercise and review of other documents, test methods identified as the highest priority formed the SQP and those of lower priority formed the EQP. All of the physical/chemical property tests that were surveyed have been included in the SQP, as it was considered that the vast majority of these properties were already released with the supplier's materials data. The cured mechanical property test methods selected for the SQP consist of tensile, compressive, shear and flexural tests (in addition to fibre and resin volume fraction), giving ultimate strength, failure strain, elastic modulus and Poisson's ratio data. Additional test methods deemed non-essential for initial materials qualification, but can be used to generate specific, application dependent data, were grouped into the EQP.

For all properties, where available and suitable, the preferred test methods are ISO standards. Some of the 'building block' test methods in the EQP are not ISO standards, but are ISO new work items (NWI) being led and validated [8] by the National Physical Laboratory (NPL) on behalf of the UK (e.g. open and filled-hole tension/compression, pin bearing etc.). In other cases, such as the compression-after-impact (CAI) test, there are no standardised versions of the test method available. This is currently an area of work under the G7/8 VAMAS (Versailles Project on Advanced Materials and Standards) international pre-standards programme. As this document is initially intended to be used for carbon fibre preimpregnated material systems, the conditioning and test temperatures/relative humidities are typical of material applications. The test temperatures and conditioning regimes selected (room temperature and/or 1000 hours at 70°C/85% relative humidity), are typical of those used for aerospace qualification requirements but have been chosen to be in-line with standard ISO test temperatures [9].

The format of the prescribed qualification regime is based on the lay-out of ISO 10350-2 - 'Plastics - Acquisition and presentation of comparable single-point data'.

## **2.4 Finalisation of SQP**

The draft SQP was made widely available to industry by means of direct distribution to over 100 UK organisations with an interest in composite material qualification and representing a range of sectors (e.g. aerospace, automotive, construction, marine, design consultancies, research establishments, test houses, regulatory authorities and material suppliers). In addition, the draft document was posted on the NPL website for free download. Over 100 downloads from the website were recorded from organisations across the globe. Appropriate mechanisms were put in place to encourage organisations to provide feedback on the draft SQP.

In general, the industrial feedback received was very positive and the overall message was that the SQP provides a useful and useable framework for initial materials qualification. Salient comments that were received and action taken as a result are detailed in Table 1. Where considered appropriate, the draft SQP document was modified to reflect industrial feedback. The finalised SQP procedure is presented in Section 3.

**Table 1:** Industrial Feedback on Draft Standard Qualification Plan

| Feedback from industry                                                                                                                                                                                                                  | Action taken/comment                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <ul style="list-style-type: none"> <li>Glass transition temperature measured by dynamic mechanical analysis (DMA) for cured resin - <i>not included in draft SQP</i></li> </ul>                                                         | <ul style="list-style-type: none"> <li>ISO/CD 6721-11 ‘Plastics - Determination of dynamic mechanical properties - Part 11: Glass transition temperature’ - <i>now included in finalised SQP</i> (see Section 3) and was one of the tests selected for the associated round-robin exercise (see Section 4)</li> </ul>                                                                                                                                                                                                                                                                                                                          |
| <ul style="list-style-type: none"> <li>Cured ply thickness is an important manufacturing issue effecting suitability of tooling-test method - <i>not included in draft SQP</i>.</li> </ul>                                              | <ul style="list-style-type: none"> <li>ISO/DIS 16012.2 ‘Plastics - Determination of the linear dimensions of specimens’ - <i>now included in finalised SQP</i></li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| <ul style="list-style-type: none"> <li>Exposure to generic types of fuel, hydraulic oil and solvents - <i>not included in draft SQP</i></li> </ul>                                                                                      | <ul style="list-style-type: none"> <li>It was considered that this level of conditioning would be more suited for inclusion in a full materials qualification programme rather than in a plan allowing initial material selection, qualification and design.</li> </ul>                                                                                                                                                                                                                                                                                                                                                                        |
| <ul style="list-style-type: none"> <li>Conditioning and/or test temperatures and humidity levels used by industry not always in agreement with those specified by the draft SQP</li> </ul>                                              | <ul style="list-style-type: none"> <li>No generic qualification plan will meet all conditioning and test requirements. Temperature and humidity levels specified for conditioning and testing are typical of those used for aerospace qualification requirements but have been chosen to be in-line with standard ISO specifications. Future work is planned to develop equivalent qualification plans for other industrial sectors (e.g. automotive, construction, etc.) using different material types and formats. Appropriate temperature and humidity levels for conditioning and testing will be specified for these sectors.</li> </ul> |
| <ul style="list-style-type: none"> <li>Tests on 5 batches of material are commonly required by the aerospace industry - will need to obtain acceptance from industry that 3 batches is sufficient for qualification purposes</li> </ul> | <ul style="list-style-type: none"> <li>A 30 specimen approach (3 batches) can be statistically shown to be the minimum number of measurements required [6]</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |

### 3. Finalised SQP Procedure

The finalised SQP procedure is presented in the following 'boxed' pages.

#### **Version 1.1**

April 2003

#### **Fibre-reinforced plastic composites – Standard qualification plan (SQP) for preimpregnated (prepreg) composite materials**

##### **Introduction**

This document has been prepared to provide suppliers, designers and end-users of fibre-reinforced plastic composite materials, an initial qualification framework aimed at reducing the substantial costs involved in qualifying materials against different specifications. Indeed, the cost associated with qualifying materials can prevent the use of new materials in certain applications. In addition, designers and end-users often find that appropriate data for materials selection and preliminary design are not available. It is envisaged that widespread use of this document for initial qualification, will lead to a reduction in qualification costs and increased availability of materials data.

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## Glossary of terms and symbols

|                   |                                                       |
|-------------------|-------------------------------------------------------|
| $V_r$             | resin volume fraction                                 |
| $V_f$             | fibre volume fraction                                 |
| $\sigma_{Mt}$     | maximum value of tensile stress                       |
| $E_t$             | tensile Young's modulus                               |
| $\epsilon_{Mt}$   | maximum value of tensile strain                       |
| $\nu$             | Poisson's ratio                                       |
| $\sigma_{Mc}$     | maximum value of compressive stress                   |
| $E_c$             | compressive Young's modulus                           |
| $\epsilon_{Mc}$   | maximum value of compressive strain                   |
| $G$               | shear modulus                                         |
| $\tau_M$          | maximum value of shear stress                         |
| $\gamma_M$        | maximum value of shear strain                         |
| $\sigma_{Mf}$     | maximum value of flexural stress                      |
| $E_f$             | flexural modulus                                      |
| $\sigma_{OHT}$    | open-hole tensile strength                            |
| $K_{OHT}$         | open-hole tensile strength concentration factor       |
| $\sigma_{FHT}$    | filled-hole tensile strength                          |
| $K_{FHT}$         | filled-hole tensile strength concentration factor     |
| $\sigma_{OHC}$    | open-hole compressive strength                        |
| $K_{OHC}$         | open-hole compressive strength concentration factor   |
| $\sigma_{FHC}$    | filled-hole compressive strength                      |
| $K_{FHC}$         | filled-hole compressive strength concentration factor |
| $F_M$             | maximum force                                         |
| $l_M$             | deflection at maximum force                           |
| $E_M$             | energy to maximum force                               |
| $E_P$             | puncture energy                                       |
| $l_P$             | puncture deflection                                   |
| $A_D$             | impact damage area                                    |
| $\sigma_{MCAI}$   | maximum value of compressive stress after impact      |
| $\epsilon_{MCAI}$ | maximum value of compressive strain after impact      |
| $G_{IC}$          | Mode I fracture toughness                             |
| $G_{IIC}$         | Mode II fracture toughness                            |

*N.B. Additional subscripts apply to some of the quantities above to indicate directions (see clause 4.3)*

## NPL Version 1.1

### **Fibre-reinforced plastic composites – Standard qualification plan (SQP) for preimpregnated (prepreg) composite materials**

#### **1 Scope**

**1.1** This document specifies a procedure for the initial qualification of composite materials in order to allow quality control, material selection and preliminary design to be undertaken. It does not constitute a full qualification programme, rather, ideally, a standardised building block forming the initial stage of a qualification or certification programme.

**1.2** The procedure is generally suitable for preimpregnated (prepreg) fibre-reinforced thermoset and thermoplastic material systems. Individual standards referred to in this document provide more details as to which classes of materials are suitable.

**1.3** The draft procedure comprises a Standard Qualification Plan (SQP) which contains the minimum common test requirements for initial materials qualification. Further test requirements are encompassed in an Extended Qualification Plan (EQP), which contains options to meet specific requirements.

**1.4** Annex A details a standard format for the release of data produced when using this document. Annex B provides a recommended procedure for the determination of B - and A - basis design allowables.

*It is envisaged that material suppliers will adopt this procedure for initial material qualification and supply release material data in the format given in Annex A.*

#### **2 Normative references**

The following standards contain provisions, which through reference in this text, constitute provisions of this procedure. At the time of publication (May 2003), the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this procedure are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC/CENELEC and ISO/CEN maintain registers of currently valid International Standards.

ISO 291 *Plastics - Standard atmospheres for conditioning and testing*

BS EN ISO 527-4 *Plastics - Determination of tensile properties - Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites*

BS EN ISO 527-5 *Plastics - Determination of tensile properties - Part 5: Test conditions for unidirectional fibre-reinforced plastic composites*

ISO 1268 *Plastics - Preparation of glass fibre reinforced, resin bonded, low-pressure laminated plates or panels for test purposes*

ISO 1183 *Plastics - Methods for determining the density and relative density of non-cellular plastics*

ISO 1675 *Plastics - Liquid resins - Determination of density by the pycnometer method*

ISO 2818 *Plastics - Preparation of test specimens by machining*

ISO 5893 *Rubber and plastics test equipment - Tensile, flexural and compression types (constant rate of traverse) - Description*

ISO 6603-2 *Plastics - Determination of puncture impact behaviour of rigid plastics - Part 2: Instrumented impact testing*

ISO/CD 6721-11 *Plastics - Determination of dynamic mechanical properties - Part 11: Glass transition temperature*

ISO 10119 *Carbon fibre - Determination of density*

ISO 10352 *Fibre-reinforced plastics - Moulding compounds and prepregs - Determination of mass per unit area*

ISO 11357-1 *Plastics - Differential scanning calorimetry (DSC) - Part 1: General*

ISO 11357-2 *Plastics - Differential scanning calorimetry (DSC) - Part 2: Determination of glass transition temperature*

ISO 11359-2 *Plastics - Thermomechanical analysis (TMA) - Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*

ISO 11667 *Fibre-reinforced plastics - Moulding compounds and prepregs - Determination of resin reinforced-fibre and mineral-filler content - Dissolution methods*

BS EN ISO 14125 *Fibre-reinforced plastic composites - Determination of flexural properties*

BS EN ISO 14126 *Fibre-reinforced plastic composites - Determination of compressive properties in the in-plane direction*

ISO 14127 *Composites - Determination of the resin, fibre and void content for composites reinforced with carbon fibre*

BS EN ISO 14129 *Fibre-reinforced plastic composites - Determination of the in-plane shear stress/shear strain response, including the in-plane shear modulus and strength, by the plus or minus 45 degree tension test method*

BS EN ISO 14130 *Fibre-reinforced plastic composites - Determination of apparent interlaminar shear strength by short-beam method*

ISO 15024 *Fibre-reinforced plastic composites - Determination of mode I interlaminar fracture toughness, GIC, for unidirectionally reinforced materials*

ISO 15034 *Composites - Prepregs - Determination of resin flow*

ISO 15040 *Composites - Prepregs - Determination of gel time*

ISO/DIS 16012.2 *Plastics - Determination of the linear dimensions of specimens*

EN 821-1 - *Advanced technical ceramics - Monolithic ceramics - Thermo-physical properties - Part 1: Determination of thermal expansion*

EN 2378 - *Aerospace series - Test method for the determination of water absorption by immersion in demineralised water*

EN 2558 - *Carbon fibre preimpregnates - Determination of the volatile content*

EN 2559 - *Carbon fibre preimpregnates - Determination of the resin and fibre content and the mass of fibre per unit area*

prEN 2823 - *Aerospace series - Fibre reinforced plastics - Determination of the effect of exposure to humid atmosphere on physical and mechanical characteristics*

EN 3615 - *Aerospace series - Fibre reinforced plastics - Procedure for the determination of the conditions of exposure to humid atmosphere and the determination of moisture absorption*

prEN 3783 - *Aerospace series - Fibre reinforced plastics - Expression of mechanical characteristics as a function of fibre content*

ISO New work item 712 - UK lead - *Fibre-reinforced plastic composites - Determination of the open-hole (notched), and filled-hole, tensile strength (similar to ASTM D5766, EN6035 and AITM 1.0007)*

ISO New work item 711 - UK lead - *Fibre-reinforced plastic composites - Determination of the open-hole (notched), and filled-hole, compressive strength (similar to EN6036 and AITM 1.0008)*

ISO New work item 714 - UK lead - *Fibre-reinforced plastic composites - Determination of the pin bearing strength (similar to ASTM D5961, EN 6037 and AITM 1.0009)*

### **3 Principle**

To provide a qualification plan based on accepted international test methods and specified material preparation and batch test requirements.

### **4 Definitions**

For the purposes of this document the following definitions apply. Where indicated, definitions of specific quantities can be found in related standards.

#### **4.1 Preimpregnate (prepreg)**

Ready to mould or cure material in sheet form which may be tow, tape, cloth, or mat impregnated with resin. It may be stored before use (normally refrigerated for thermoset based systems).

#### **4.2 Batch**

A quantity of material formed during the same process and having identical characteristics throughout. For prepregs, laminae, and laminates, material made from one batch of fibre and one batch of resin.

#### **4.3 Specimen coordinate axes**

Where the material contains a known axis of preferred fibre orientation, then this is defined as the 1-axis (or direction). For materials prepared as test plates, the in-plane direction transverse to the 1-axis is defined as the 2-axis. Where any direction of preferred orientation is not known, the 1-axis is the production direction of the composite or the reinforcement (e.g. warp directions for fabrics).

For unidirectional specimens cut parallel to the “1”-direction, results are identified by the subscript “11” (e.g.  $E_{11}$ ). Similarly, for specimens cut parallel to the “2”-direction results are identified by the subscript “22” (e.g.  $E_{22}$ ).

For multidirectional laminates and fabrics, results are identified by the subscripts “XX” and “YY” for specimens cut parallel to the X and Y directions respectively. The X, Y and Z coordinate system for any material can be equated to the “1”-, “2”- and “3”- directions.

#### 4.4 A - basis value

A statistically based material property; a 95% lower confidence bound on the first percentile of a specified population of measurements. Also a 95% lower tolerance bound for the upper 99% of a specified population.

#### 4.5 B - basis value

A statistically based material property; a 95% lower confidence bound on the tenth percentile of a specified population of measurements. Also a 95% lower tolerance bound for the upper 90% of a specified population.

### 5 Test methods

Tables 1 and 2 detail the test methods to be used in the SQP, and Table 3 the test methods to be used in the EQP.

**Table 1:** Standard test methods for physical/chemical properties included in the SQP

| Property                                                                                | Number          |
|-----------------------------------------------------------------------------------------|-----------------|
| Mass per unit area of moulding compounds and preregs                                    | ISO 10352       |
| Resin and fibre content, and the mass of fibre per unit area                            | EN 2559         |
| Resin reinforced-fibre and mineral-filler content for moulding compounds and preregs    | ISO 11667       |
| Gel time for preregs                                                                    | ISO 15040       |
| Resin flow for preregs                                                                  | ISO 15034       |
| Glass transition temperature                                                            | ISO 11357-2     |
| Volatile content for carbon fibre preregs                                               | EN 2558         |
| Density of carbon fibre                                                                 | ISO 10119       |
| Differential scanning calorimetry (DSC)                                                 | ISO 11357-1     |
| Density of liquid resin                                                                 | ISO 1675        |
| Coefficient of thermal expansion                                                        | EN 821-1        |
| Coefficient of linear thermal expansion and glass transition temperature                | ISO 11359-2     |
| Water absorption by immersion in demineralised water                                    | EN 2378         |
| Conditions of exposure to humid atmosphere and the determination of moisture absorption | EN 3615         |
| Dynamic mechanical analysis (DMA)                                                       | ISO/CD 6721-11  |
| Cured ply thickness                                                                     | ISO/DIS 16012.2 |

**Table 2:** Standard test methods for cured mechanical properties included in the SQP

| Property                                                                             | Number          |
|--------------------------------------------------------------------------------------|-----------------|
| Density and relative density of non-cellular plastics                                | ISO 1183        |
| Resin, fibre and void content for composites reinforced with carbon fibre            | ISO 14127       |
| Tensile properties for isotropic and orthotropic fibre-reinforced plastic composites | BS EN ISO 527-4 |
| Tensile properties for unidirectional fibre-reinforced plastic composites            | BS EN ISO 527-5 |
| Compressive properties in the in-plane direction                                     | BS EN ISO 14126 |
| In-plane shear stress/shear strain response by $\pm 45^\circ$ tension test           | BS EN ISO 14129 |
| Apparent interlaminar shear strength by the short-beam method                        | BS EN ISO 14130 |
| Flexural properties                                                                  | BS EN ISO 14125 |

**Table 3:** Standard test methods for cured mechanical properties included in the EQP

| Property                                                   | Number/status                     |
|------------------------------------------------------------|-----------------------------------|
| Open-hole (notched), and filled-hole, tensile strength     | ISO New work item - 712 - UK lead |
| Open-hole (notched), and filled-hole, compressive strength | ISO New work item - 711 - UK lead |
| Pin bearing strength                                       | ISO New work item - 714 - UK lead |
| Puncture impact behaviour of rigid plastics                | ISO 6603-2                        |
| Compression-after-impact                                   | VAMAS study (similar to EN 6038)  |
| Mode I fracture toughness, $G_{IC}$                        | ISO 15024 (DIS ballot)            |
| Mode II fracture toughness, $G_{IIC}$                      | VAMAS study, (4ENF)               |

## 6 Test matrices and specimen sampling

The prescribed qualification test regime (using the test methods listed in Section 5), is detailed in Tables 4-6. The tables set out the numbers of batches of specimens per test method, per test condition.

Specimens shall be prepared and tested from either 1 or 3 batches of material depending on the criticality of the data (i.e. the importance of the data for use in design, material selection etc). Specimens shall be taken from 3 batches of material in order for measured values to be as representative as possible of the material being tested and these batches shall be taken from the production of the material over an extended timescale. In addition, to account for the processing variability associated with panel manufacture, two panels shall be fabricated from each material batch, using independent lay-up and cure processes. Five specimens per panel shall be extracted for each test method (*N.B. some tests use < 5 specimens*), giving a total of 30 specimens per property for a particular test environment. In a similar manner, those properties measured using only 1 batch of material shall be determined from 10 specimens prepared from 2 panels (prepared using independent lay-up and cure processes)

Specimen selection and traceability (as described above) is illustrated in Figure 1.

*NOTE - An independent lay-up and cure process shall be used for panels from the same material batch, but only two independent cure processes are required to fabricate all 6 panels, i.e. one cure process for panels 1, 3 and 5; and one for 2, 4 and 6.*

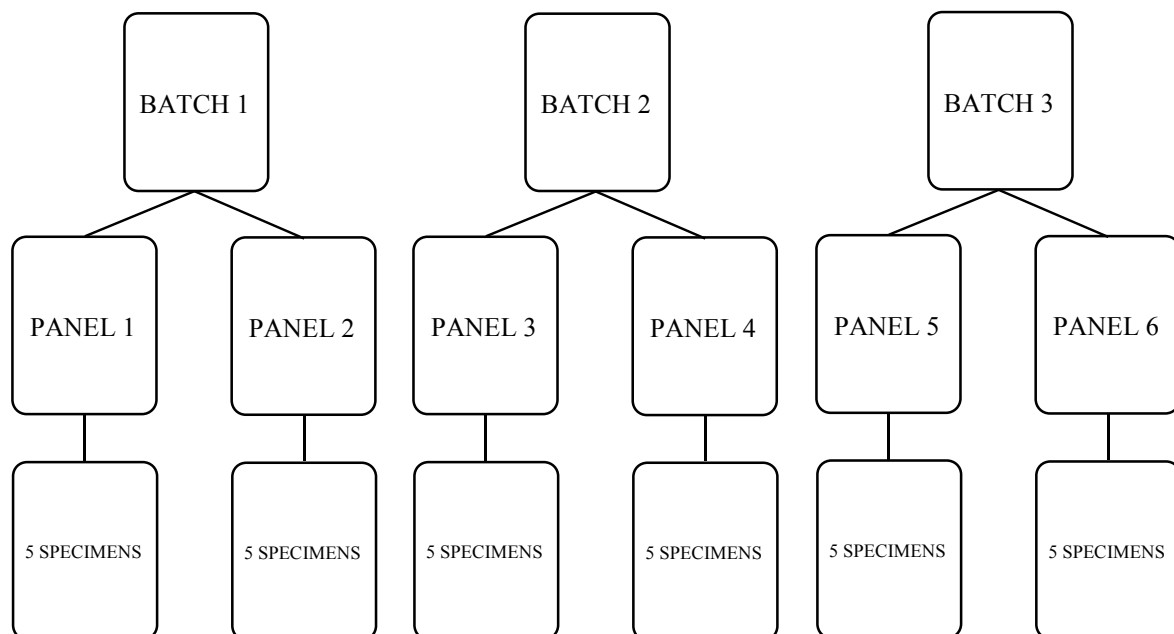


**Figure 1:** Batch, panel and specimen traceability

**Table 4: Standard Qualification Plan - Physical/chemical properties - Batch requirements**

| Property                               | Standard                   | Unit              | N <sup>o</sup> of batches | Test conditions and supplementary instructions                                                           |
|----------------------------------------|----------------------------|-------------------|---------------------------|----------------------------------------------------------------------------------------------------------|
| <b>UNCURED PREPREG PROPERTIES</b>      |                            |                   |                           |                                                                                                          |
| 1 Mass per unit area                   | ISO 10352                  | g/m <sup>2</sup>  | 3                         |                                                                                                          |
| 2 Fibre mass per unit area             | EN 2559                    | g/m <sup>2</sup>  | 3                         | Principally for carbon-fibre reinforced plastics, but also applicable to glass fibre-reinforced plastics |
| 3 Resin and fibre volume fraction      | ISO 11667                  | %                 | 3                         |                                                                                                          |
| 4 Gell time                            | ISO 15040                  | Minutes           | 3                         |                                                                                                          |
| 5 Resin flow                           | ISO 15034                  | % weight change   | 3                         |                                                                                                          |
| 6 Glass transition temperature         | ISO 11357-2                | °C                | 3                         |                                                                                                          |
| 7 Percentage of volatile content       | EN 2558                    | %                 | 3                         | Glass fibre prepreps – BS EN 2330                                                                        |
| 8 Density of fibre                     | ISO 10119                  | g/cm <sup>3</sup> | 3                         | Carbon fibre                                                                                             |
| 9 Analysis by DSC                      | ISO 11357-1                | Variable          | 3                         |                                                                                                          |
| 10 Density of resin                    | ISO 1675                   | g/cm <sup>3</sup> | 3                         |                                                                                                          |
| <b>CURED PROPERTIES</b>                |                            |                   |                           |                                                                                                          |
| 11 Coefficient of thermal expansion    | CEN EN821-1 or ISO 11359-2 | %/°C              | 1                         | CEN EN821-1 or ISO 11359-2 can be used here                                                              |
| 12 Moisture uptake                     | EN 3615                    | %                 | 1                         | Conditioning in 70°C/85% atmosphere                                                                      |
| 13 Moisture uptake                     | EN 2378                    | %                 | 1                         | Immersion in water at 70°C                                                                               |
| 14 Glass transition temperature by DMA | ISO/CD 6721-11             | °C                | 3                         |                                                                                                          |
| 15 Cured ply thickness                 | ISO/DIS 16012.2            | mm                | 3                         |                                                                                                          |

**Table 5:** Standard Qualification Plan - Cured mechanical properties - Batch requirements

| Property |                                 | Symbol            | Standard                                         | Unit | Test Condition |     |      |             |     |      | Test conditions and supplementary instructions                                                                                                                                                                               |  |
|----------|---------------------------------|-------------------|--------------------------------------------------|------|----------------|-----|------|-------------|-----|------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--|
|          |                                 |                   |                                                  |      | DRY            |     |      | 70°C/85% RH |     |      |                                                                                                                                                                                                                              |  |
|          |                                 |                   |                                                  |      | -55°C          | RT  | 70°C | 125°C*      | RT  | 70°C |                                                                                                                                                                                                                              |  |
| 16       | Fibre and resin volume fraction | V <sub>f</sub>    | ISO 14127 and ISO 1183                           | %    |                | 3   |      |             |     |      | ISO 1183 for density                                                                                                                                                                                                         |  |
|          |                                 | V <sub>r</sub>    |                                                  |      | 3              |     |      |             |     |      |                                                                                                                                                                                                                              |  |
| 17       | Tension                         | σ <sub>Mt11</sub> | Unidirectional<br>BS EN ISO 527-5                | MPa  | 1              | 3   | 1    |             |     | 1    | Refer to standard for specimen dimensions and test details.<br><br>() – not required for balanced fabrics                                                                                                                    |  |
|          |                                 | σ <sub>Mt22</sub> |                                                  | MPa  | 1              | 1   | (1)  |             |     | (1)  |                                                                                                                                                                                                                              |  |
|          |                                 | E <sub>t11</sub>  |                                                  | GPa  | 1              | 3   | 1    |             |     | 1    |                                                                                                                                                                                                                              |  |
|          |                                 | E <sub>t22</sub>  |                                                  | GPa  | 1              | 1   | (1)  |             |     | (1)  |                                                                                                                                                                                                                              |  |
|          |                                 | ε <sub>Mt11</sub> |                                                  | %    | 1              | 3   | 1    |             |     | 1    |                                                                                                                                                                                                                              |  |
|          |                                 | ε <sub>Mt22</sub> | %                                                | 1    | 1              | (1) |      |             | (1) |      |                                                                                                                                                                                                                              |  |
|          |                                 | ν <sub>12</sub>   |                                                  |      | 1              |     |      |             |     |      |                                                                                                                                                                                                                              |  |
|          |                                 | ν <sub>13</sub>   |                                                  |      | 1              |     |      |             |     |      |                                                                                                                                                                                                                              |  |
|          |                                 | ν <sub>21</sub>   |                                                  |      | 1              |     |      |             |     |      |                                                                                                                                                                                                                              |  |
|          |                                 | ν <sub>23</sub>   |                                                  |      | 1              |     |      |             |     |      |                                                                                                                                                                                                                              |  |
| 18       | Compression                     | σ <sub>Mc11</sub> | ISO 14126<br>unidirectional,<br>multidirectional | MPa  | 1              | 3   | 1    |             |     | 3    | Refer to standard for specimen dimensions and test details. Type A, B <sub>1</sub> and B <sub>2</sub> specimens, testing by Method 1 (shear loading) or Method 2 (end loading)<br><br>() – not required for balanced fabrics |  |
|          |                                 | σ <sub>Mc22</sub> |                                                  | MPa  |                | 1   | (1)  |             |     | 1    |                                                                                                                                                                                                                              |  |
|          |                                 | E <sub>c11</sub>  |                                                  | GPa  | 1              | 3   | 1    |             |     | 3    |                                                                                                                                                                                                                              |  |
|          |                                 | E <sub>c22</sub>  |                                                  | GPa  |                | 1   | (1)  |             |     | 1    |                                                                                                                                                                                                                              |  |
|          |                                 | ε <sub>Mc11</sub> |                                                  | %    | 1              | 3   | 1    |             |     | 3    |                                                                                                                                                                                                                              |  |
|          |                                 | ε <sub>Mc22</sub> |                                                  | %    |                | 1   | (1)  |             |     | 1    |                                                                                                                                                                                                                              |  |
| 19       | Shear ±45° tension              | τ <sub>M12</sub>  | BS EN ISO 14129                                  | MPa  | 1              | 3   | 1    | 1           | 1   | 1    |                                                                                                                                                                                                                              |  |
|          |                                 | γ <sub>M12</sub>  |                                                  | %    | 1              | 3   | 1    | 1           | 1   | 1    |                                                                                                                                                                                                                              |  |
|          |                                 | G <sub>12</sub>   |                                                  | GPa  | 1              | 3   | 1    | 1           | 1   | 1    |                                                                                                                                                                                                                              |  |
| 20       | ILSS – through thickness        | τ <sub>M1</sub>   | BS EN ISO 14130                                  | MPa  | 1              | 3   | 1    | 1           | 3   | 1    | 3                                                                                                                                                                                                                            |  |
|          |                                 | τ <sub>M2</sub>   |                                                  | MPa  |                | 1   | 1    |             |     |      |                                                                                                                                                                                                                              |  |
| 21       | Flexural                        | σ <sub>Mf11</sub> | BS EN ISO 14125                                  | MPa  |                | 3   |      |             |     |      | Refer to standard for specimen dimensions and test details. Provide details of which method used: Method A (3 point flexure) and Method B (4 point flexure)                                                                  |  |
|          |                                 | σ <sub>Mf22</sub> |                                                  | MPa  |                | 3   |      |             |     |      |                                                                                                                                                                                                                              |  |
|          |                                 | E <sub>f11</sub>  |                                                  | MPa  |                | 3   |      |             |     |      |                                                                                                                                                                                                                              |  |
|          |                                 | E <sub>f22</sub>  |                                                  | MPa  |                | 3   |      |             |     |      |                                                                                                                                                                                                                              |  |
|          |                                 | G <sub>13</sub>   |                                                  | GPa  |                | 3   |      |             |     |      |                                                                                                                                                                                                                              |  |

N.B. \* - for 180°C cure resins only

**Table 6:** Extended Qualification Plan – Cured mechanical properties - Batch requirements

| Property | Symbol                       | Standard    | Unit             | Test Condition |      |      |        |    |      | Test conditions and supplementary instructions |                                                                                                                                                                                                                                                                     |
|----------|------------------------------|-------------|------------------|----------------|------|------|--------|----|------|------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
|          |                              |             |                  | DRY            |      |      |        |    |      |                                                |                                                                                                                                                                                                                                                                     |
|          |                              |             |                  | 70°C/85% RH    |      |      |        |    |      |                                                |                                                                                                                                                                                                                                                                     |
|          |                              |             |                  | -55°C          | RT   | 70°C | 125°C* | RT | 70°C |                                                |                                                                                                                                                                                                                                                                     |
| 22       | Open/filled hole tension     | ISO NW1-UK  | MPa              | 1(1)           | 3(1) |      |        |    |      | 3(1)                                           | Quasi-isotropic lay-up. N.B. $K_{OHT}$ and $K_{FHT}$ are dimensionless stress concentration factors calculated using $\sigma_{OHT}$ , $\sigma_{FHT}$ and the unnotched tensile strength (using BS EN ISO 527-5 and determined from bracketed number of batches)     |
|          |                              |             | MPa              | 1(1)           | 3(1) |      |        |    |      | 3(1)                                           |                                                                                                                                                                                                                                                                     |
| 23       | Open/filled hole compression | ISO NW1-UK  | MPa              | 1(1)           | 3(1) |      |        |    |      | 3(1)                                           | Quasi-isotropic lay-up. N.B. $K_{OHC}$ and $K_{FHC}$ are dimensionless stress concentration factors calculated using $\sigma_{OHC}$ , $\sigma_{FHC}$ and the unnotched compressive strength (using BS EN ISO 14126 and determined from bracketed number of batches) |
|          |                              |             | MPa              | 1(1)           | 3(1) |      |        |    |      | 3(1)                                           |                                                                                                                                                                                                                                                                     |
| 24       | Pin-bearing                  | ISO NW1-UK  | MPa              | 1              | 3    |      |        |    |      | 3                                              | Quasi-isotropic lay-up<br>N.B. plain pin bearing i.e. un-torqued                                                                                                                                                                                                    |
| 25       | Instrumented Impact          | ISO 6603-2  | N                |                | 3    |      |        |    |      | 3                                              | Quasi-isotropic lay-up.<br>Refer to standard for additional information required with results e.g. specimen size, support parameters, failure criterion etc.                                                                                                        |
|          |                              |             | mm               |                |      |      |        |    |      |                                                |                                                                                                                                                                                                                                                                     |
|          |                              |             | J                |                |      |      |        |    |      |                                                |                                                                                                                                                                                                                                                                     |
|          |                              |             | J                |                |      |      |        |    |      |                                                |                                                                                                                                                                                                                                                                     |
|          |                              |             | mm               |                |      |      |        |    |      |                                                |                                                                                                                                                                                                                                                                     |
| 26       | Compression-after-impact     | VAMAS study | mm <sup>2</sup>  |                | 3    |      |        |    |      | 3                                              | Quasi-isotropic lay-up                                                                                                                                                                                                                                              |
|          |                              |             | MPa              |                |      |      |        |    |      |                                                |                                                                                                                                                                                                                                                                     |
|          |                              |             | %                |                |      |      |        |    |      |                                                |                                                                                                                                                                                                                                                                     |
| 27       | Mode I fracture toughness    | ISO 15024   | J/m <sup>2</sup> |                | 3    |      |        |    |      | 1                                              | Unidirectional lay-up, ISO 15024 at DIS ballot                                                                                                                                                                                                                      |
| 28       | Mode II fracture toughness   | VAMAS study | J/m <sup>2</sup> |                | 3    |      |        |    |      | 1                                              | Unidirectional lay-up                                                                                                                                                                                                                                               |

N.B. \* - for 180°C cure resins only

## 7 Panel and specimen preparation and machining

Specimens shall be cut from test plates manufactured according to the fabrication method given in ISO 1268 for the material under test. Machining shall be performed in accordance with ISO 2818 (additional guidance is given in [10]), as applicable, and the dimensions of the specimen shall comply with those given in the relevant test method standard (see recommended standards in Tables 1-3).

## 8 Test requirements

The test methods, test conditions and units specified in Tables 4-6 shall be used. Specific details on preparing and testing specimens are given in the individual test methods. Where the test method has different procedures for different types of reinforcement or matrix, the appropriate procedure shall be used for the material under test.

Where required (see Tables 4-5) measurements of a property shall be made along the 1 and 2 axes. Where required (indicated in Tables 5-6), specimens shall be tested at temperatures other than ambient after conditioning at  $23 \pm 2^\circ\text{C}/50 \pm 5\%$  relative humidity (RH) (DRY) and/or 1000 hours at  $70^\circ\text{C}/85\%\text{RH}$ . For tests at non-ambient conditions and/or after conditioning, reference should be made to prEN 2823 and prEN 3615.

## 9 Presentation of results

The presentation of data shall be as set out in Report Sheets 1-4 of Annex A, in addition to the information required by Section 7 where appropriate. This should include the matrix resin used, the type and form of the reinforcing fibres; and the processing method used to produce the test specimens.

Where appropriate, certain data values required by Report Sheets 2-3 of Annex A shall be normalised with respect to a nominal fibre volume fraction of 60%. The normalisation procedure is detailed in prEN 3783. All mechanical strength and stiffness data shall be normalised except:

- $90^\circ$  (transverse) tension of unidirectional laminates
- bearing strength
- $90^\circ$  compression of unidirectional laminates
- strain energy release rates
- interlaminar shear
- Poisson's ratio
- in-plane shear
- impact data

## 10 Precision and statistical analysis

The level of precision required of the data released in the SQP/EQP is specific to the application of the material. As set out in Section 6, each property at a specific environmental condition will be determined from the results of 10 or 30 individual specimens (i.e. 1 or 3 batches of material, 2 panels per batch, 5 specimens per panel). The results detailed in Report Forms 1-3, Annex A shall be given as mean and standard deviation values calculated for 10 or 30 specimens per test condition (i.e. 1 or 3 batches, respectively) and after normalisation (where applicable) with respect to fibre volume fraction.

For calculation of design allowable values, such as A- and B- basis values, individual test results for all specimens shall be listed separately for subsequent data analysis. A procedure for calculating A - and B - basis values is detailed in Annex B.

*Note. This procedure is based on the statistical analysis procedures detailed in [2], which are used in [5].*

## 11 Test report

In addition to completed Report Sheets 1-4 (Annex A), the test report shall include the following information:

- a) a reference to this procedure;
- b) complete identification of the material tested, including type, source, manufacturer's code number and test plate manufacture details, where these are known;
- c) the date of measurements;

And, in addition, for each data set:

- d) the method of preparing the specimens;
- e) the specimen dimensions;
- f) the test conditions and conditioning procedures;
- g) the number of specimens tested;
- h) the accuracy grading of test machine used (see ISO 5893);
- i) the individual test results;

- j) the mean and standard deviations (95% confidence level);
- k) statement as to whether any test specimens from a data set have been rejected and, if so, the reasons;
- l) any operation not specified in this or referenced documents, as well as any incident likely to have affected the results.

| Report Sheet 1: Standard Qualification Plan - Physical/chemical properties |                                     |                            |                   |                              |          |
|----------------------------------------------------------------------------|-------------------------------------|----------------------------|-------------------|------------------------------|----------|
| Annex A                                                                    |                                     |                            |                   |                              |          |
| Property                                                                   | Standard                            | Unit                       | Mean, $\mu$       | Standard deviation, $\sigma$ | Comments |
| UNCURED PROPERTIES                                                         |                                     |                            |                   |                              |          |
| 1                                                                          | Mass per unit area                  | ISO 10352                  | g/m <sup>2</sup>  |                              |          |
| 2                                                                          | Fibre mass per unit area            | EN 2559                    | g/m <sup>2</sup>  |                              |          |
| 3                                                                          | Resin and fibre volume fraction     | ISO 11667                  | %                 |                              |          |
| 4                                                                          | Gell time                           | ISO 15040                  | Minutes           |                              |          |
| 5                                                                          | Resin flow                          | ISO 15034                  | % weight change   |                              |          |
| 6                                                                          | Glass transition temperature        | ISO 11357-2                | °C                |                              |          |
| 7                                                                          | Percentage of volatile content      | EN 2558                    | %                 |                              |          |
| 8                                                                          | Density of fibre                    | ISO 10119                  | g/cm <sup>3</sup> |                              |          |
| 9                                                                          | Analysis by DSC                     | ISO 11357-1                | Variable          |                              |          |
| 10                                                                         | Density of resin                    | ISO 1675                   | g/cm <sup>3</sup> |                              |          |
| CURED PROPERTIES                                                           |                                     |                            |                   |                              |          |
| 11                                                                         | Coefficient of thermal expansion    | CEN EN821-1 or ISO 11359-2 | %/°C              |                              |          |
| 12                                                                         | Moisture uptake                     | EN 3615                    | %                 |                              |          |
| 13                                                                         | Moisture uptake                     | EN 2378                    | %                 |                              |          |
| 14                                                                         | Glass transition temperature by DMA | ISO/CD 6721-11             | °C                |                              |          |
| 15                                                                         | Cured ply thickness                 | ISO/DIS 16012.2            | mm                |                              |          |

**Report Sheet 2: Standard Qualification Plan - Cured mechanical properties**

| Property | Symbol                          | Standard                                                                                                                                                                                         | Unit                                       | Test Condition |     |    |   |      |   |             |   |    |   |      |   | Comments |  |
|----------|---------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------|----------------|-----|----|---|------|---|-------------|---|----|---|------|---|----------|--|
|          |                                 |                                                                                                                                                                                                  |                                            | DRY            |     |    |   |      |   | 70°C/85% RH |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | -55°C          |     | RT |   | 70°C |   | 125°C       |   | RT |   | 70°C |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | μ              | σ   | μ  | σ | μ    | σ | μ           | σ | μ  | σ | μ    | σ |          |  |
| 16       | Fibre and resin volume fraction | V <sub>f</sub><br>V <sub>r</sub>                                                                                                                                                                 | %                                          |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
| 17       | Tension                         | σ <sub>M11</sub><br>σ <sub>M22</sub><br>E <sub>t11</sub><br>E <sub>t22</sub><br>ε <sub>M11</sub><br>ε <sub>M22</sub><br>V <sub>12</sub><br>V <sub>13</sub><br>V <sub>21</sub><br>V <sub>23</sub> | ISO 14127 and ISO 1183                     | %              | MPa |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            |                | MPa |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 | GPa                                                                                                                                                                                              |                                            |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 | GPa                                                                                                                                                                                              |                                            |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 | %                                                                                                                                                                                                |                                            |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 | %                                                                                                                                                                                                |                                            |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
| 18       | Compression                     | σ <sub>Mel1</sub><br>σ <sub>Mel22</sub><br>E <sub>c11</sub><br>E <sub>c22</sub><br>ε <sub>Mel1</sub><br>ε <sub>Mel22</sub>                                                                       | ISO 14126 unidirectional, multidirectional | MPa            |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | MPa            |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | GPa            |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | GPa            |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | %              |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | %              |     |    |   |      |   |             |   |    |   |      |   |          |  |
| 19       | Shear ±45° tension              | τ <sub>M12</sub><br>γ <sub>M12</sub><br>G <sub>12</sub>                                                                                                                                          | MPa                                        |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  | %                                          |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  | GPa                                        |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
| 20       | ILSS – through thickness        | τ <sub>M1</sub><br>τ <sub>M2</sub>                                                                                                                                                               | MPa                                        |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  | MPa                                        |                |     |    |   |      |   |             |   |    |   |      |   |          |  |
| 21       | Flexural                        | σ <sub>M11</sub><br>σ <sub>M22</sub><br>E <sub>t11</sub><br>E <sub>t22</sub><br>G <sub>13</sub>                                                                                                  | BS EN ISO 14125                            | MPa            |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | MPa            |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | MPa            |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | MPa            |     |    |   |      |   |             |   |    |   |      |   |          |  |
|          |                                 |                                                                                                                                                                                                  |                                            | GPa            |     |    |   |      |   |             |   |    |   |      |   |          |  |

**Report Sheet 3: Extended Qualification Plan – Cured mechanical properties**

| Property | Symbol                       | Standard    | Unit | Test Condition |   |      |   |       |   |             |   |      |   |   |   | Comments |  |
|----------|------------------------------|-------------|------|----------------|---|------|---|-------|---|-------------|---|------|---|---|---|----------|--|
|          |                              |             |      | DRY            |   |      |   |       |   | 70°C/85% RH |   |      |   |   |   |          |  |
|          |                              |             |      | RT             |   | 70°C |   | 125°C |   | RT          |   | 70°C |   |   |   |          |  |
|          |                              |             |      | μ              | σ | μ    | σ | μ     | σ | μ           | σ | μ    | σ | μ | σ |          |  |
|          |                              |             |      | -55°C          |   |      |   |       |   |             |   |      |   |   |   |          |  |
| 22       | Open/filled hole tension     | ISO NW1-UK  | MPa  |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
|          |                              |             | MPa  |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
| 23       | Open/filled hole compression | ISO NW1-UK  | MPa  |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
|          |                              |             | MPa  |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
| 24       | Pin-bearing                  | ISO NW1-UK  | MPa  |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
| 25       | Instrumented Impact          | ISO 6603-2  | N    |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
|          |                              |             | mm   |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
|          |                              |             | J    |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
|          |                              |             | J    |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
|          |                              |             | mm   |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
| 26       | Compression-after-impact     | VAMAS study | mm²  |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
|          |                              |             | MPa  |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
|          |                              |             | %    |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
| 27       | Mode I fracture toughness    | ISO 15024   | J/m² |                |   |      |   |       |   |             |   |      |   |   |   |          |  |
| 28       | Mode II fracture toughness   | VAMAS study | J/m² |                |   |      |   |       |   |             |   |      |   |   |   |          |  |

**Report Sheet 4: Standard Qualification Plan - Material record sheet**

|                                      |  |                |  |
|--------------------------------------|--|----------------|--|
| <b>Material Record Sheet</b>         |  | N°:            |  |
| Material code:                       |  |                |  |
| Fibre type:                          |  | Fibre code:    |  |
| Fibre format:                        |  |                |  |
| Fibre supplier:                      |  |                |  |
| Resin type:                          |  | Resin code:    |  |
| Resin supplier:                      |  |                |  |
| Prepreg type:                        |  | Prepreg code:  |  |
| Prepreg supplier:                    |  | Ply-thickness: |  |
| Manufacturing process:               |  |                |  |
| Test plates lay-up(s) and thickness: |  |                |  |
| Cure conditions:                     |  |                |  |
| Post-cure conditions:                |  |                |  |
| Manufacturing date(s)                |  |                |  |
| Specimen machining method(s)         |  |                |  |
| Test machine grades(s)               |  |                |  |
| Comments:                            |  |                |  |
| Signature                            |  | Date:          |  |

## Annex B

This Annex details a procedure used for calculating A - and B - basis design allowables. The approach is based on the methods given in [2, 5, 11]. The user is referred to these documents for further information on the approach used. The flow chart in Figure B1 provides a step by step guide to the statistical analysis process.

It has been assumed that the data produced approximates to a normal distribution. If the assumption of normality is not accepted or disproven, then an alternative distribution (e.g. Weibull) may be used. In general the Weibull distribution tends to give more conservative values.

The statistical analysis procedure is based on the assumption that pooling data sets is justifiable when variability across data sets (for different environmental test conditions) for a particular test and property is comparable and the failure modes for each data set do not significantly change.

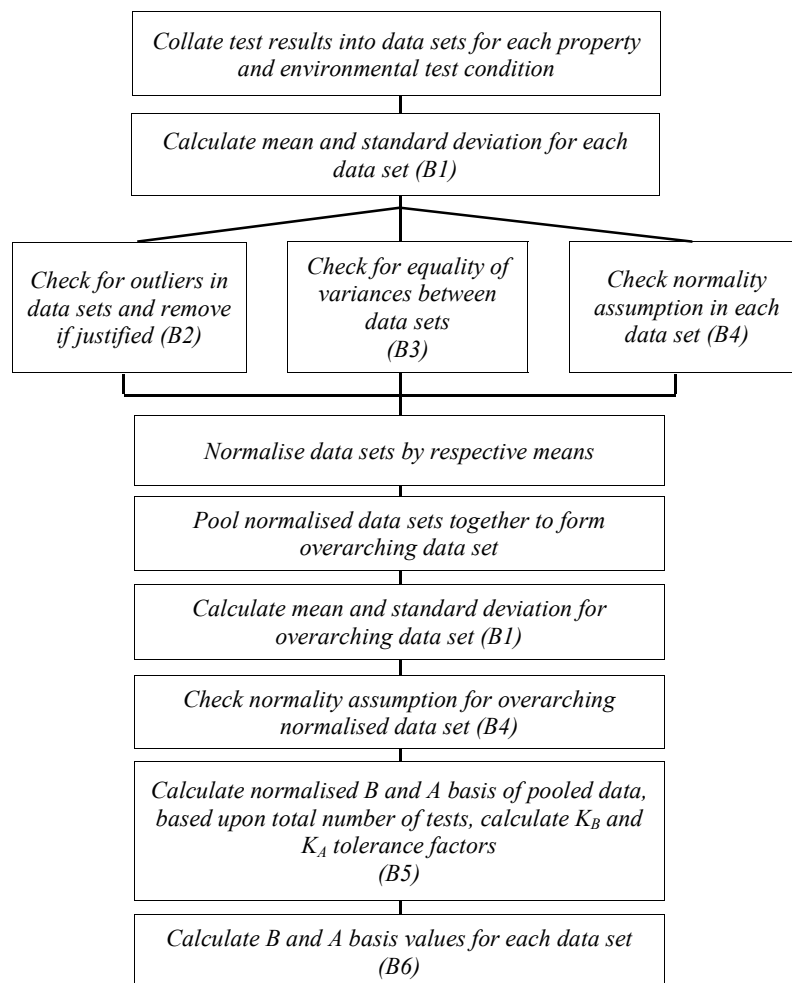


**Figure B1:** Step by step procedure for calculating B - and A - basis allowables for data provided in the SQP

## B1 Calculation of Mean and Standard Deviation

The mean and standard deviations for each data set are calculated using (1) and (2) respectively:

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i \quad (1)$$

$$s^2 = \frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2 \quad (2)$$

(where  $\bar{x}$  = data set mean,  $x_i$  = individual observation in a data set,  $n$  = number of observations in a data set,  $s$  = standard deviation of data set)

## B2 Detection of Outliers

Each data set shall be examined for the presence of outliers. The Maximum Normed Residual (MNR) method shall be used for quantitative screening of outliers. Equation (3) shows that the MNR quantity is the maximum absolute deviation from the data set mean divided by the data set standard deviation. This quantity is calculated for each observation in each data set and the resulting values compared to a critical value calculated for a data set size,  $n$ , using equation (4):

$$MNR = \frac{\max_i |x_i - \bar{x}|}{s} \quad i=1, 2, \dots, n \quad (3)$$

$$C = \frac{n-1}{\sqrt{n}} \sqrt{\frac{t^2}{n-2+t^2}} \quad (4)$$

(where  $t$  = the  $[1-\alpha/[2n]]$  quantile of the  $t$ -distribution with  $n-2$  degrees of freedom and  $\alpha$  is the significance level ( $\alpha=0.05$  for the MNR test))

If all of the MNR values calculated are smaller than the  $C$  value calculated in (4) then no outliers are detected. However, if there are MNR values greater than  $C$ , then the largest value of MNR is declared an outlier and can be expelled from the data set (see [2] for detail on expelling outliers). If an outlier is removed then the process is repeated until no more outliers are detected.

### B3 Assessment of Equality of Variances Between Data Sets

The assessment of whether data set variances differ significantly is performed in a 2-stage procedure.

Firstly, Levene's test for determining whether the data set variances of k data sets differ significantly is used. This test is performed using (5):

$$w_{ij} = |x_{ij} - x_{mi}| \quad (5)$$

(where  $w_{ij}$  = transformed data set,  $x_{ij} = j^{\text{th}}$  observation in the  $i^{\text{th}}$  data set,  $x_{mi}$  is the median for data set I)

Secondly, an F-test is performed on the transformed data to test the assumption that the population from which the k data sets were drawn have the same mean. The F-statistic is calculated using (6):

$$F = \frac{\sum_{i=1}^k n_i (\bar{w}_i - \bar{w})^2 / (k-1)}{\sum_{i=1}^k \sum_{j=1}^{n_i} (w_{ij} - \bar{w}_i)^2 / (n-k)} \quad (6)$$

(where  $w_i$  = the average of the  $n_i$  values in the  $i^{\text{th}}$  data set,  $w$  = the average of all the  $n$  observations (i.e. total number of observations when all the data sets are pooled),  $k$  = the number of data sets,  $n$  = the total number of observations,  $n_i$  = is the number of observations in the  $i^{\text{th}}$  group)

The F values calculated in (6) are then compared to a critical F value ( $F_{\text{critical}}$ ) for the  $(1-\alpha)$  quantile of the F-distribution having  $k-1$  numerator and  $n-k$  degrees of freedom. Typically,  $\alpha = 0.05$  is used. The  $F_{\text{critical}}$  value can be approximated from (7):

$$F_{\text{critical}} = \exp \left[ 2\delta \left\{ 1 + \frac{z^2 - 1}{3} - \frac{4\sigma^2}{3} \right\} + 2\sigma z \sqrt{1 + \frac{\sigma^2(z^2 - 3)}{6}} \right] \quad (7)$$

Where:

$$\delta = 0.5 \left\{ \frac{1}{\gamma_2 - 1} - \frac{1}{\gamma_1 - 1} \right\}$$

$$\sigma^2 = 0.5 \left\{ \frac{1}{\gamma_2 - 1} + \frac{1}{\gamma_1 - 1} \right\}$$

$$z = 1.645$$

$\gamma_1$  is the numerator degrees of freedom (k-1)

$\gamma_2$  is the denominator degrees of freedom (n-k)

If the calculated F-statistic, F, is less than the value of  $F_{\text{critical}}$  then the variances of the data sets are not significantly different.

## B4 Assessment of Normality Assumption

The assumption of normality is tested by means of a graphical method. This test consists of arranging all observations ( $x_1, x_2, x_3, \dots, x_n$ ) in non-decreasing order and then plotting  $x_g$  against:

$$P_g = \frac{\left( g - \frac{3}{8} \right)}{\left( n + \frac{1}{4} \right)} \quad (8)$$

(where  $P_g$  = the probability of survival of the  $x_g$  observation,  $x_g$  = the value of the  $g^{\text{th}}$  observation when sorted in non-decreasing order,  $n$  = the number of observations)

If the points obtained form a more or less straight line, a normal distribution can be assumed.

## B5 Calculation of $K_B$ and $K_A$

The one-sided B - and A - basis tolerance factors for the normal distribution for each class  $j$  that is based upon the number of samples in the pooled data set  $N$  and the number of samples in each class  $n_j$  (N.B.  $n_j$  is a sub-set of  $N$ ). The B - basis tolerance factor (number of standard deviations)  $(k_B)_j$  may be approximated by (9):

$$(k_B)_j = z_B \sqrt{\frac{f}{Q}} + \sqrt{\frac{1}{c_B n_j} + \left(\frac{b_B}{2c_B}\right)^2} - \frac{b_B}{2c_B} \quad (9)$$

(where  $z_B$  is the standard normal random variable - in the case of B-basis calculation  $z_B$  is taken as 1.28115 (90% probability)).

The coefficients  $b_B$  and  $c_B$  are given by the following

$$b_B(f) = 1.1372 \frac{1}{\sqrt{f}} - 0.49162 \frac{1}{f} + 0.18612 \frac{1}{f\sqrt{f}} \quad (10)$$

$$c_B(f) = 0.36961 + 0.0040342 \frac{1}{\sqrt{f}} - 0.71750 \frac{1}{f} + 0.19693 \frac{1}{f\sqrt{f}} \quad (11)$$

Where  $f = N-2$  is the degrees of freedom for the variance. In the case of  $f \geq 3$ ,  $Q$  may be approximated by:

$$Q = f - 2.327\sqrt{f} + 1.138 + 0.6057 \frac{1}{\sqrt{f}} - 0.3287 \frac{1}{f} \quad (12)$$

For  $f = 2$ , the exact value of  $Q$  may be used as  $Q=0.05129$ . The above approximations are accurate to within 1.2% of the tabulated values for B-basis calculations.

Similarly, the A - basis tolerance factor  $(k_A)_j$  may be approximated by (13):

$$(k_A)_j = z_A \sqrt{\frac{f}{Q}} + \sqrt{\frac{1}{c_A n_j} + \left(\frac{b_A}{2c_A}\right)^2} - \frac{b_A}{2c_A} \quad (13)$$

and

$$b_A(f) = 2.0643 \frac{1}{\sqrt{f}} - 0.95145 \frac{1}{f} + 0.51251 \frac{1}{f\sqrt{f}} \quad (14)$$

$$c_A(f) = 0.36961 + 0.0026958 \frac{1}{\sqrt{f}} - 0.65201 \frac{1}{f} + 0.011320 \frac{1}{f\sqrt{f}} \quad (15)$$

Where  $f = N-2$  is the degrees of freedom for the variance. In the case of  $f \geq 3$ ,  $Q$  may be approximated by (12). For  $f=2$ , the exact value of  $Q$  may be used as  $Q=0.05129$ . The above approximations are accurate to within 0.9% of the tabulated values for A - basis calculations.

## B6 Calculation of B - and A - Basis Values

The normal distribution B - and A - basis allowables are calculated by using the pooled mean, standard deviation and tolerance factors for each class  $j$  via (16) and (17):

$$B_j = \bar{x} - (k_B)_j s \quad (16)$$

$$A_j = \bar{x} - (k_A)_j s \quad (17)$$

These values are then used to multiply the mean strengths calculated for each class of data to finally give the B - and A - basis values for each class.

## 4 Round-Robin Validation Exercise

A round-robin exercise was undertaken with the aim of increasing confidence in the data measured by any organisation using the SQP/EQP and associated test methods. Results from the round-robin were analysed according to ISO 5725 [12] (using 95% confidence limits) in order to assess the repeatability (measure of within site scatter) and reproducibility (measure of between site scatter) of 5 test methods selected from the SQP and to generate materials data for possible future inclusion in a materials database. Tests were performed on two carbon fibre-epoxy pre-preg materials.

### 4.1 Participants

The following organisations participated in the round-robin programme:

- Advanced Composites Group Ltd.
- Aerostructures Hamble Ltd
- Bureau Veritas
- Composites Testing Laboratory Ltd
- Ford Motor Company Ltd
- Gearing Scientific
- Kingston University
- Motor Industry Research Association
- National Physical Laboratory
- Perkin Elmer Thermal Analysis Solutions
- Slingsby Aviation Ltd
- Triton Technology Ltd

### 4.2 Test Standards and Properties Measured

The test standards used for the round-robin validation exercise were:

- BS EN ISO 527-5 Plastics - Determination of tensile properties - Part 5: Test conditions for unidirectional fibre-reinforced plastic composites (see also Part 1),
- BS EN ISO 14126 Fibre-reinforced plastic composites - Determination of compressive properties in the in-plane direction,
- BS EN ISO 14125 Fibre-reinforced plastic composites - Determination of flexural properties,
- BS EN ISO 14130 Fibre-reinforced plastic composites - Determination of apparent interlaminar shear strength (ILSS) by short-beam method,
- ISO/CD 6721-11 Plastics - Determination of dynamic mechanical properties - Part 11: Glass transition temperature,

The four mechanical test methods selected are recommended tests in the SQP. The dynamic mechanical analysis (DMA) test was included in this round-robin, as it was a test recommended to be included following industry review of the draft document. All specimens were of unidirectional (UD) lay-up and the following properties were measured by participants; maximum value of tensile stress ( $\sigma_{Mt11}$ ), tensile modulus ( $E_{t11}$ ), Poisson's ratio ( $\nu_{12}$ ), maximum value of compressive stress ( $\sigma_{Mc11}$ ), compressive modulus ( $E_{c11}$ ), maximum value of interlaminar shear stress ( $\tau_{M1}$ ), maximum value of flexural strength ( $\sigma_{Mf11}$ ), flexural modulus ( $E_{f11}$ ), glass transition temperature ( $T_g$ , plus  $T_{onset}$ ,  $T_{loss}$ , and  $T_{\tan \delta}$ ).

### 4.3 Materials and Specimen Preparation

The materials tested in this round-robin were supplied by Hexcel Composites Ltd and SP Systems Ltd. Both materials were carbon fibre-epoxy pre-pregs and were cured at 120°C.

Test panels were manufactured at NPL according to the fabrication method given in ISO 1268 and following material supplier's guidance. All panels were autoclave cured. Specimens were extracted from test panels in accordance with ISO 2818. Machining operations were performed using a Bennett diamond grit coated circular saw, with water used as a coolant/lubricant. Specimens were machined to the dimensions and tolerances given in the appropriate test standards.

### 4.4 Test Results and Discussion

The round-robin results are fully reported in NPL Report MATC(D)128 "Towards a Standard Qualification Plan (SQP) for Composite Materials - Round-Robin Validation Exercise" [13]. Tables 2-3 show the values of repeatability and reproducibility for both materials, as percentages of the mean values obtained for the range of properties measured. These data are presented graphically in Figure 2. Mean values obtained by participating sites are detailed in Figures 1-10 of Appendix 1.

In general the results of the round-robin exercise showed that a range of test methods selected from the SQP document are suitably robust for inclusion in such a materials qualification plan. Details of a number of salient findings from the exercise now follow:

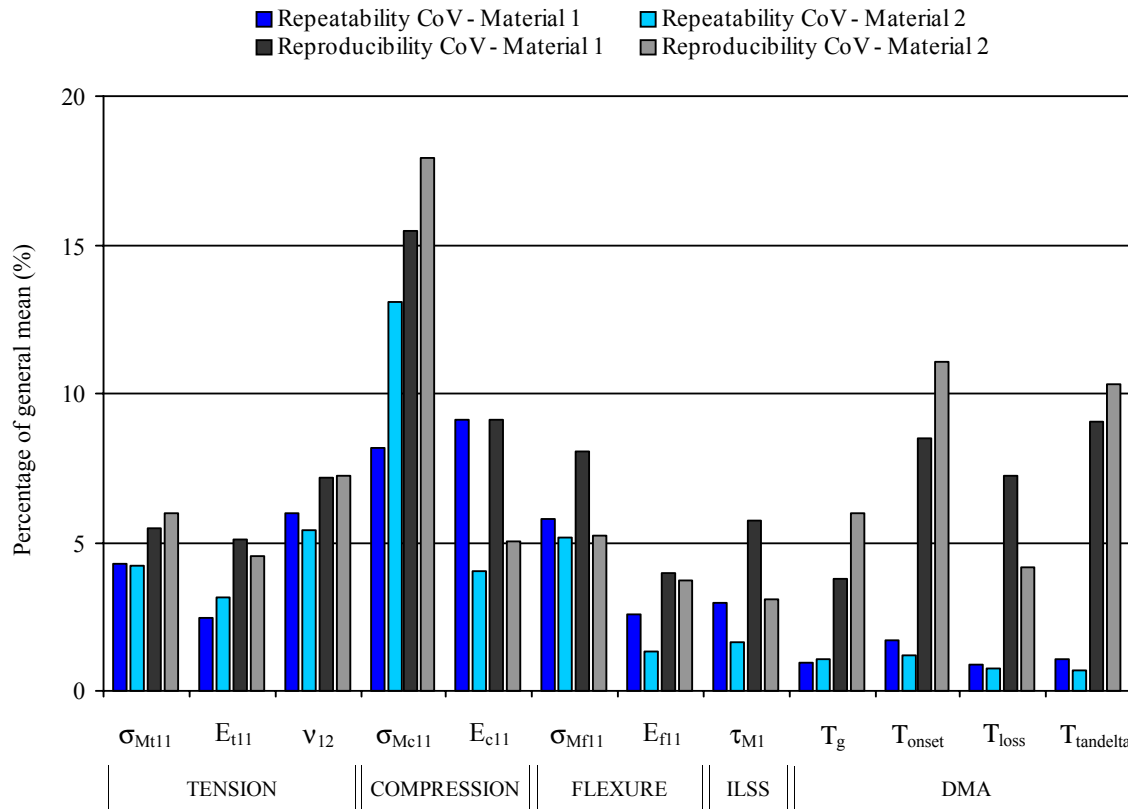
- Tensile (strength, modulus and Poisson's ratio), flexure (strength and modulus) and interlaminar shear strength (ILSS) tests all showed very good repeatability and reproducibility values (close to, or better than, 5% of their respective means).

**Table 2:** Repeatability and reproducibility values as percentages of mean value for Material 1

| Property    |                  | Value as Percentage of Mean |      |                            |      |
|-------------|------------------|-----------------------------|------|----------------------------|------|
|             |                  | Repeatability Conditions    |      | Reproducibility Conditions |      |
|             |                  | $S_r$                       | $r$  | $S_R$                      | $R$  |
| Tension     | $\sigma_{Mt11}$  | 4.27                        | 12.0 | 5.45                       | 15.2 |
|             | $E_{t11}$        | 2.46                        | 6.88 | 5.09                       | 14.2 |
|             | $\nu_{12}$       | 5.95                        | 17.0 | 7.14                       | 19.9 |
| Compression | $\sigma_{Mc11}$  | 8.18                        | 22.9 | 15.5                       | 43.3 |
|             | $E_{c11}$        | 9.10                        | 25.5 | 9.10                       | 25.5 |
| Flexure     | $\sigma_{Mf11}$  | 5.81                        | 16.3 | 8.05                       | 22.6 |
|             | $E_{f11}$        | 2.57                        | 7.21 | 3.97                       | 11.1 |
| ILSS        | $\tau_{M1}$      | 2.98                        | 8.36 | 5.72                       | 16.0 |
| DMA         | $T_g$            | 0.96                        | 2.67 | 3.76                       | 10.5 |
|             | $T_{onset}$      | 1.68                        | 4.70 | 8.50                       | 23.8 |
|             | $T_{loss}$       | 0.85                        | 2.37 | 7.24                       | 20.3 |
|             | $T_{tan\ delta}$ | 1.10                        | 3.07 | 9.05                       | 25.4 |

**Table 3:** Repeatability and reproducibility values as percentages of mean value for Material 2

| Property    |                  | Value as Percentage of Mean |      |                            |      |
|-------------|------------------|-----------------------------|------|----------------------------|------|
|             |                  | Repeatability Conditions    |      | Reproducibility Conditions |      |
|             |                  | $S_r$                       | $r$  | $S_R$                      | $R$  |
| Tension     | $\sigma_{Mt11}$  | 4.22                        | 11.8 | 6.00                       | 16.9 |
|             | $E_{t11}$        | 3.17                        | 8.92 | 4.51                       | 12.6 |
|             | $\nu_{12}$       | 5.44                        | 15.4 | 7.25                       | 19.9 |
| Compression | $\sigma_{Mc11}$  | 13.1                        | 36.5 | 17.9                       | 50.4 |
|             | $E_{c11}$        | 4.05                        | 11.4 | 5.06                       | 14.2 |
| Flexure     | $\sigma_{Mf11}$  | 5.15                        | 14.4 | 5.25                       | 14.7 |
|             | $E_{f11}$        | 1.32                        | 3.67 | 3.69                       | 10.4 |
| ILSS        | $\tau_{M1}$      | 1.61                        | 4.52 | 3.07                       | 8.62 |
| DMA         | $T_g$            | 1.08                        | 3.02 | 5.97                       | 16.7 |
|             | $T_{onset}$      | 1.18                        | 3.31 | 11.1                       | 31.1 |
|             | $T_{loss}$       | 0.73                        | 2.05 | 4.15                       | 11.6 |
|             | $T_{tan\ delta}$ | 0.72                        | 2.02 | 10.3                       | 28.9 |



N.B. CoV - Coefficient of variation

**Figure 2:** Repeatability and reproducibility standard deviations as percentages of mean values [13]

- The inherent variability of compression testing was reflected with fairly high values of repeatability and reproducibility for the compressive strength results. Compressive modulus results were lower i.e. less than 10% of the mean. However it is noted that the number of sites able to perform the compression tests was low. In addition, in order to ascertain whether Euler buckling has taken place during loading, it is essential that two strain measurements are made on opposite faces of each specimen. It is considered that further work on compression testing development and guidance is essential, especially for aspects such as effect of end-tab design (see [14]), strain measurement and testing of thick sections.
- The DMA results showed very low repeatability but comparatively high reproducibility values due to deficiencies in temperature measurement using various DMA equipment. It is noted that DMA is increasingly being considered as an alternative to quality control tests such as flexure and ILSS, however concern exists over using DMA for this purpose due to the high values of reproducibility presently inherent in the method, as verified in this round-robin exercise and in previous exercises organised by NPL [15].

- The use of accurate load and displacement monitoring equipment is essential for precision measurements. For measurement of displacement, at the very least the crosshead displacement should only be used in conjunction with a machine compliance correction, which may be non-linear. Clip gauge extensometers, strain gauges or linear variable differential transducers (LVDTs)/dial gauge indicators with a low spring stiffness are recommended. Specimen dimensions should always be measured to the level of accuracy required in the test standard as small errors in the measurement of dimensions can make large differences to the results i.e. thickness measurements are squared and cubed in calculations for flexural strength and modulus respectively.

## **5. Concluding Comments**

This Measurement Good Practice Guide details the development of a procedure for the initial qualification of preimpregnated fibre-reinforced thermoset and thermoplastic material systems.

The SQP procedure has been widely distributed to industry and has had positive feedback as being a useful and useable tool for composite materials qualification. The results of the associated round-robin validation exercise demonstrates the suitability and robustness of test methods included in the SQP, and it is anticipated that this will encourage the use of the procedure and increase confidence in the data measured by users. It is envisaged that the widespread use of this document for initial qualification, will not only lead to a reduction in qualification costs, but increase the availability of materials data and the use of composites in new applications. Drawing a parallel with the FAA approved qualification procedure (released by the AGATE consortium), discussions with the CAA [16] have indicated that a reduction in materials qualification testing and increased sharing of data between organisations is already evident.

Having produced an SQP procedure and obtained feedback from industry, areas of further work have been identified. One key area is to develop equivalent qualification plans for other industrial sectors using different material types and formats, and operating under different in-service conditions. In addition, further collaboration with and support from national regulatory authorities such as the CAA is required, to encourage widespread use of the SQP. International collaboration (i.e. with the FAA) is also key for harmonisation of qualification procedures.

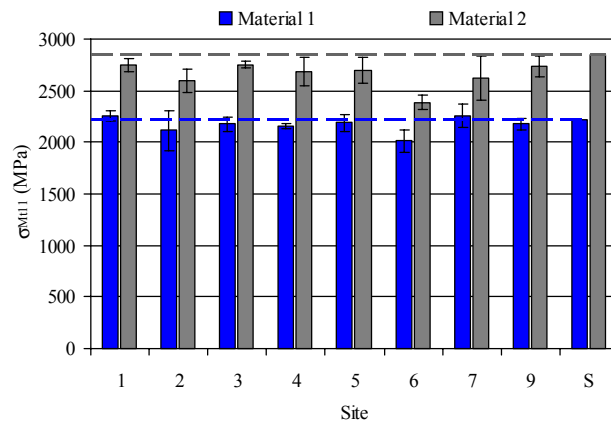
## References

1. Rouchon, J., "Certification of Large Aircraft Composite Structures, Recent Progress and New Trends in Compliance Philosophy", presented at the 17th ICAS, Stockholm, Sweden, 1990.
2. Military Handbook - Polymer Matrix Composites - Volume 1 - Guidelines for Characterisation of Structural Materials, MIL-HDBK-17-1E, Volume 1, 1996.
3. Airbus Industrie Material Specification (AIMS) 05-01-000 Part 1, 'Carbon fibre reinforced epoxy prepreg - Technical Specification', November 2000.
4. Airbus Industrie Material Specification (AIMS) 05-01-000 Part 2, 'Carbon fibre reinforced epoxy prepreg - Technical Specification - Qualification program/batch release testing - Unidirectional tape/180°C curing class structural material', January 2000.
5. U.S. Department of Transportation, Federal Aviation Administration, 'Material Qualification and Equivalency for Polymer Matrix Composite Material Systems', DOT/FAA/AR-00/47, Final Report, April 2001.
6. UKAS publication M3003, "The Expression of Uncertainty and Confidence in Measurement", Edition 1, December 1997.
7. Gower, M. R. L. and Sims, G.D., "Towards a Standard Qualification Plan (SQP) for Composite Materials - Initial Draft", NPL Report MATC(A)84, March 2002.
8. Niklewicz, J. and Sims, G.D., "Round-Robin Validation Exercise for Three Structural Test Methods (Open-Hole Tension, Open-Hole Compression and Pin-Bearing)", NPL Measurement Note CMMT(MN)43, November 1999.
9. Sims, G.D. and Nimmo, W., "Reference Temperatures for Thermal Testing of Polymer Matrix Composites", NPL Measurement Note CMMT(MN)18, August 1997.
10. Davies, A., Foreman, A., Shaw, R. and Sims, G.D., "Fibre Reinforced Plastic Composites - Machining of Composites and Specimen Preparation", NPL Measurement Good Practice Guide N° 38, 2001.
11. Shyprykevich, P., "The Role of Statistical Data Reduction in the Development of Design Allowables for Composites," Test Methods for Design Allowables for Fibrous Composites: 2<sup>nd</sup> Volume, ASTM STP 1003, C.C. Chamis ed., American Society for Testing and Materials, Philadelphia, PA, 1989, pp. 111-135.
12. ISO 5725-2:1994 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.
13. Gower, M. R. L. and Sims, G.D., "Towards a Standard Qualification Plan (SQP) for Composite Materials - Round-Robin Validation Exercise", NPL Report MATC(D)128
14. Niklewicz, J. and Sims, G.D., "Size Effects in Composite Materials", NPL Report MATC(A)74, January 2002.
15. Mulligan, D., Gnaniah, S. and Sims, G.D., "Thermal Analysis Techniques for Composites and Adhesives", NPL Measurement Good Practice Guide N° 32, 2000.
16. Private communications with Professor John Bristow, Civil Aviation Authority, March 2003.



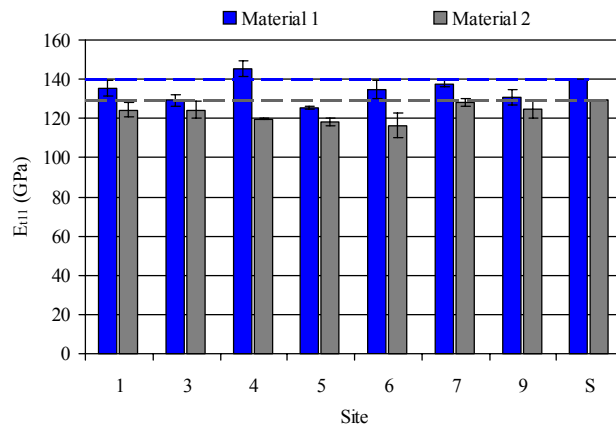
## Appendix 1: Round-Robin Results

**Figure 1: Tensile strength results ( $\sigma_{Mt11}$ )**



S = material supplier's data

**Figure 2: Tensile modulus results ( $E_{t11}$ )**



S = material supplier's data

**Figure 3: Poisson's ratio results ( $\nu_{12}$ )**

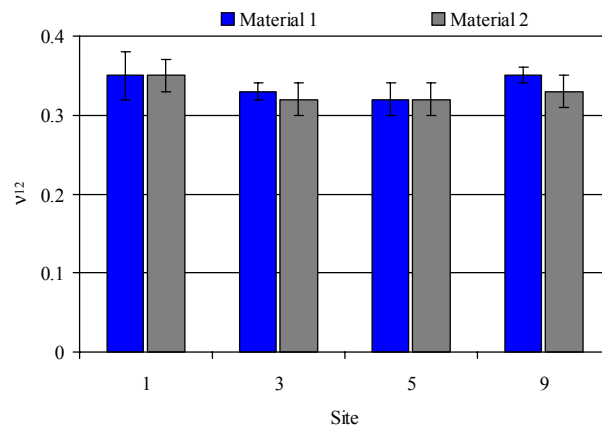
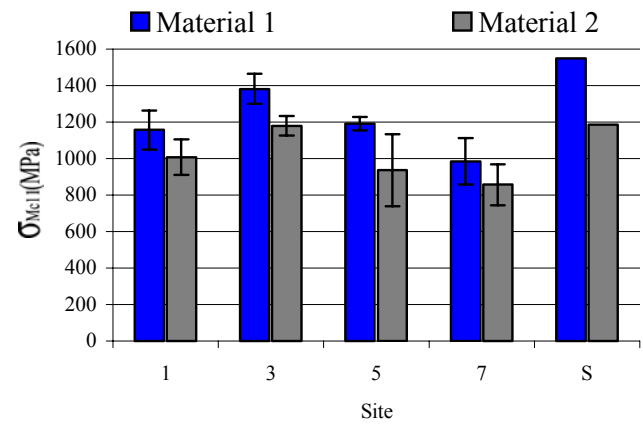
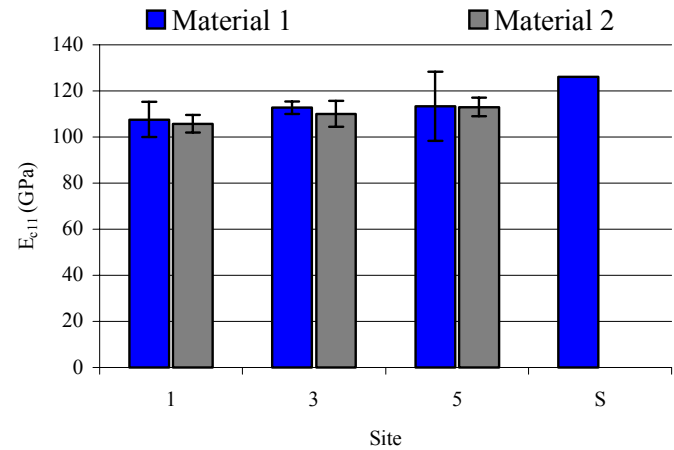


**Figure 4:** Compressive strength results ( $\sigma_{Mc11}$ )

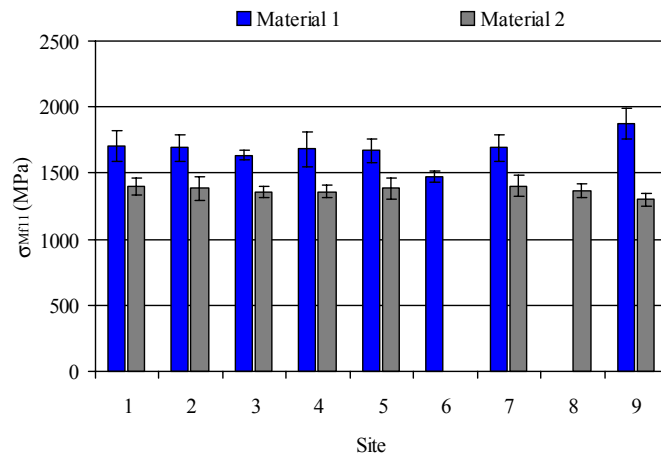
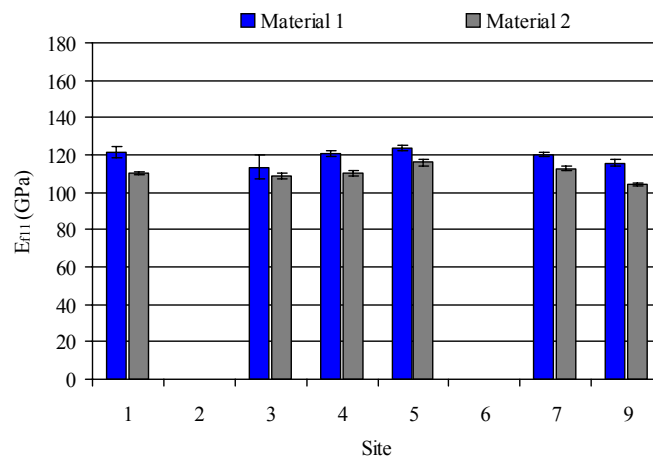
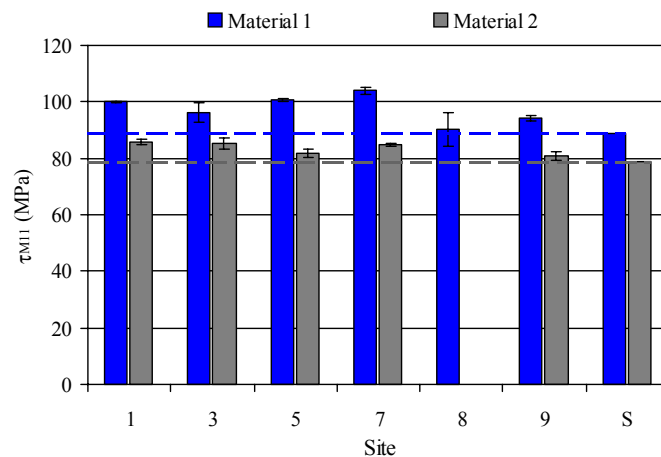


S = material supplier's data

**Figure 5:** Compressive modulus results ( $E_{c11}$ )



S = material supplier's data

**Figure 6:** Flexural strength results ( $\sigma_{Mf11}$ )**Figure 7:** Flexural modulus results ( $E_{f11}$ )**Figure 8:** Interlaminar shear strength results ( $\tau_{M1}$ )

S = material supplier's data

**Figure 9: DMA results for Material 1**

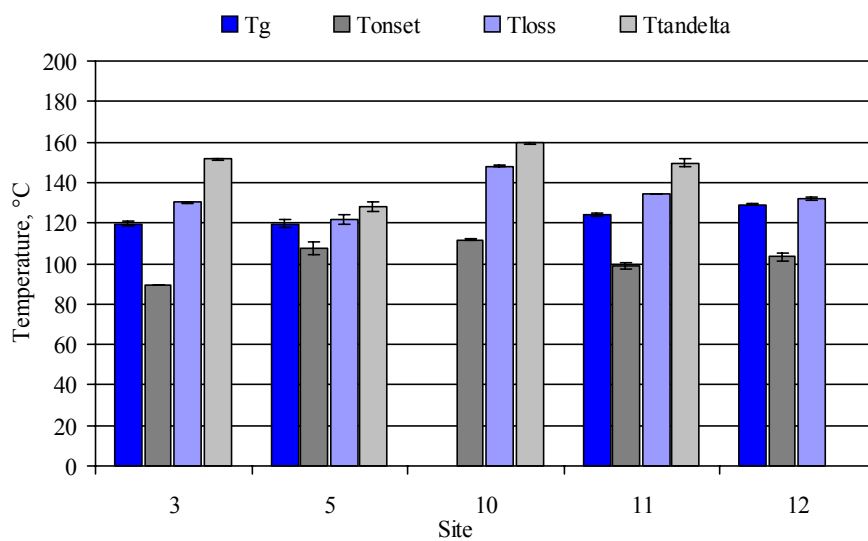


**Figure 10: DMA results for Material 2**

