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MILITARY HANDBOOK

POLYMER MATRIX COMPOSITES

VOLUME I. GUIDELINES

DEPARTMENT OF DEFENSE
UNITED STATES OF AMERICA

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29 February 1988

DEPARTMENT OF DEFENSE
WASHINGTON, D.C. 20025

MIL-HDBK-17B: Volume I
Polymer Matrix Composites

1. Military Handbook 17 (Part 1) provides guidelines and material properties for polymer (organic) matrix composites materials. This handbook encompasses, but is not limited to, polymeric composites intended for aircraft and aerospace vehicles and military combat vehicle applications. Structural sandwich composites are covered in MIL-HDBK-23. Metal matrix composites (MMC), ceramic matrix composites (CMC), and carbon/carbon composites (C/C) will be covered in separate military handbooks as developments occur.

2. This standardization handbook has been developed and is being maintained as a joint effort of the Department of Defense and the Federal Aviation Administration.

3. The information contained in this handbook was obtained from materials producers, industry, reports on Government-sponsored research, the open literature, and by contact with research laboratories and those who participate in the MIL-HDBK-17 coordination activity.

4. All information and data contained in this handbook have been coordinated with industry and the U.S. Army, Navy, Air Force, NASA, and Federal Aviation Administration prior to publication.

5. Every effort has been made to reflect the latest information on polymeric composites. The handbook is continually reviewed and revised to insure its completeness and currentness. Users of this document are encouraged to report any errors discovered and recommendations for changes or additions to Department of the Army, U.S. Army Laboratory Command, Materials Technology Laboratory, ATTN: SLCMT-OM, MIL-HDBK-17 Coordinator, Arsenal St., Watertown, MA 02172-0001. Documentation for the Secretariat should be directed to Materials Sciences Corporation, MIL-HDBK-17 Secretariat, Gwynedd Plaza II, Spring House, PA 19477.

6. Copies of this document and revisions thereto may be obtained from the Naval Publications and Forms Center, 5801 Tabor Avenue, Philadelphia, Pennsylvania 19120.

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1. GENERAL INFORMATION

1.1 Introduction. The standardization of a statistically-based mechanical property data base, procedures used, and overall material guidelines for characterization of composite material systems is recognized as being beneficial to both manufacturers and governmental agencies. It is also recognized that a complete characterization of the capabilities of any engineering material system is primarily dependent on the inherent material physical and chemical composition which precede, and are independent of, specific applications. Therefore, at the material system characterization level, the data and guidelines contained in this handbook are applicable to military and commercial products and provide the technical basis for establishing statistically valid design values acceptable to certificating or procuring agencies.

This handbook specifically provides statistically-based mechanical property data on current and emerging polymer matrix composite materials, provides guidelines for the analysis and presentation of data, and provides fabrication and characterization documentation to ensure repeatability of results or reliable detection of differences. The primary focus of MIL-HDBK-17 in the overall characterization/design procedure as commonly applied to composites is shown in Figure 1.1.

The data contained herein, or appearing in approved items in the minutes of MIL-HDBK-17 coordination meetings are acceptable to the Army, the Navy, the Air Force, and the Federal Aviation Administration. Approval by the certificating or procuring agency must be obtained for the use of data or guidelines not contained herein.

This standardization handbook has been developed and is maintained as a joint effort of the Department of Defense and the Federal Aviation Administration. It is oriented toward the standardization of methods used to develop and analyze mechanical property data on current and emerging composite materials.

MIL-HDBK-17 will ultimately be divided into three volumes. The first volume is oriented toward guidelines for data development and analyses. Chapter 2 provides guidelines for the generation of material properties, including the qualification of alternate materials and pooling of data from different sources. Chapters 3 through 5 define acceptable procedures for the evaluation of composite constituents including reinforcement fibers, resins, and prepreg materials. Chapter 6 addresses acceptable procedures for the evaluation of lamina and laminate materials. Procedures for analyzing composite structural details, and in particular, bolted joints are presented in Chapter 7. Chapter 8 of Volume I addresses the analysis and presentation of composite material property data. Important references are cited at the end of each chapter and appendix.

The second volume of MIL-HDBK-17 will provide a compilation of statistically-based material properties for current and emerging composite materials. The first two chapters of this volume will address matrix material and reinforcement fiber constituent properties. The remaining chapters will address composite system properties and they will be defined according to the primary reinforcement fiber types. Glass fiber, aramid fiber, carbon fiber, and boron fiber composite properties will be covered in separate chapters.

Volume III will provide guidelines for the application of the data which are presented in Volume II. Chapter 1 addresses typical composite materials and

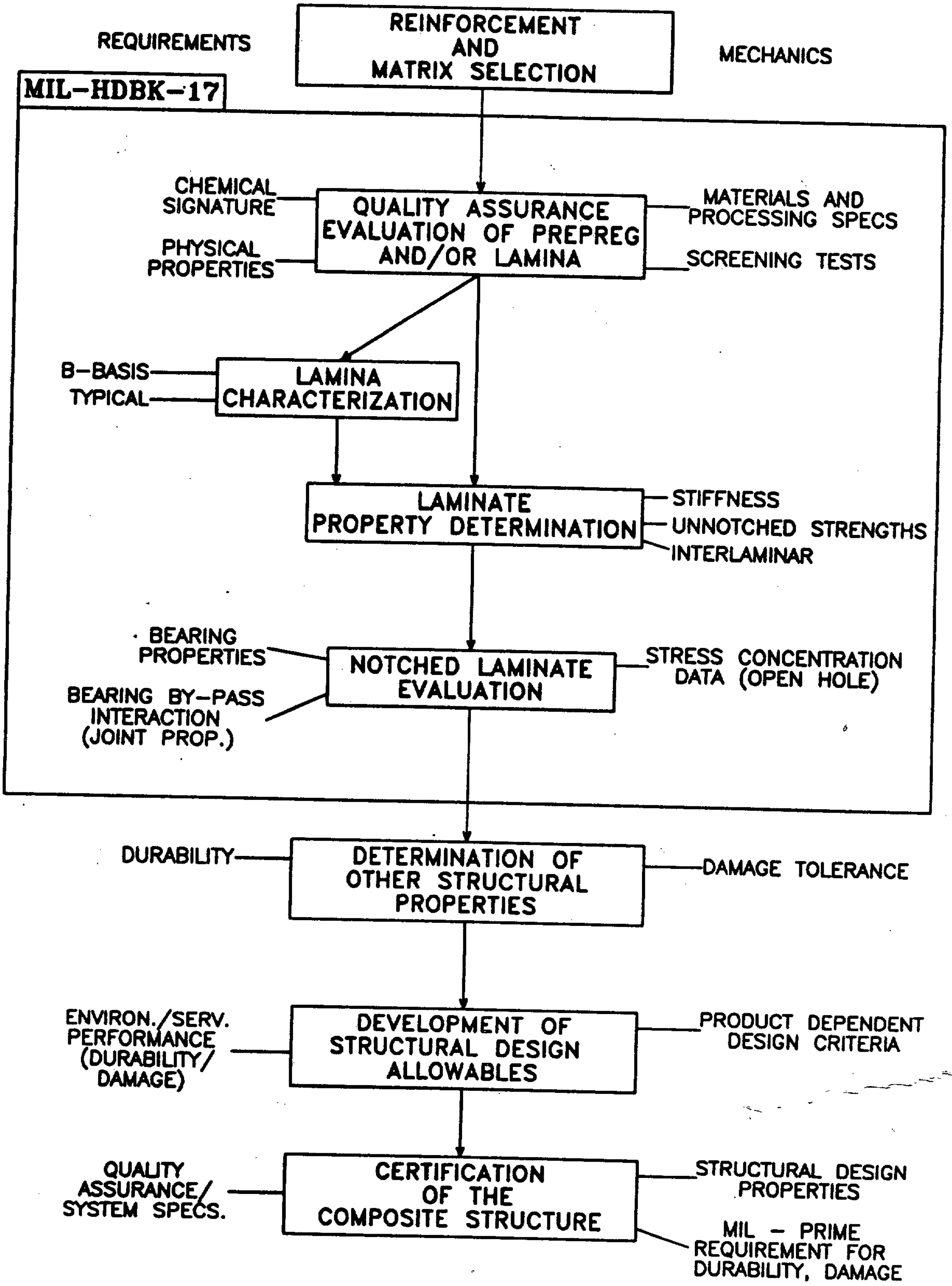


Figure 1.1 Focus of MIL-HDBK-17.

processes. The matrix materials and reinforcement fibers of interest in MIL-HDBK-17 are reviewed, as are the typical product forms. Fabrication methods are also covered for information purposes. Chapter 2 reviews important issues related to quality control in the production of composite materials. Recommended manufacturing inspection procedures are reviewed, along with techniques for material property verification and statistical quality control. Chapter 3 addresses the design and analysis of composite material systems. It provides an overview of the current techniques and describes how the various constituent properties reported in MIL-HDBK-17 are used in the design and analysis of a composite system.

The remainder of Chapter 1 of this volume provides additional information of importance to any user of MIL-HDBK-17. The purpose and scope of the document are defined and some comments are provided on its use and limitations. The accepted system of symbols, abbreviations, and units are also reviewed, and a large collection of terms pertinent to this handbook are defined.

1.2 Purpose. The purpose of this handbook is to provide a standard source of statistically-based mechanical property data for current and emerging composite materials. In order to serve this purpose the handbook must provide specific guidelines on how the necessary data should be developed and analyzed. Documentation requirements on the fabrication and characterization of these composites must also be clearly defined.

Twice yearly MIL-HDBK-17 coordination meetings are held for the specific purpose of reviewing and approving new guidelines and data proposals. These meetings consist of representatives from the DOD, FAA, and industry. Materials which are approved and included in this handbook or the minutes of the MIL-HDBK-17 coordination meetings are acceptable to the Army, Navy, Air Force, and Federal Aviation Administration and considered effective on the date approved by MIL-HDBK-17 coordination committee. The use of data or guidelines that are not approved and therefore not included in this document must be approved by the appropriate certification or procurement agency.

1.3 Scope. MIL-HDBK-17 will ultimately be published in three volumes, and will serve as a source for the following:

Volume I -	Provides guidelines for the characterization of composite material systems to be used in aerospace vehicles and structures. Composite material systems must normally be evaluated in accordance with these, or equivalent guidelines, in order to be considered acceptable by government certification and procuring agencies.
Volume II -	Will provide a compilation of statistically-based mechanical property data for current and emerging composite material systems used in the aerospace industry. B-basis strength and strain-to-failure values will be presented along with related data.
Volume III -	Will provide information regarding materials and fabrication procedures, quality control, and design and analysis.

Specifically, Volume I provides guidelines for the physical, chemical, and mechanical characterization of composite materials. These guidelines address characterization of the fiber, matrix, and prepreg materials, which are the primary constituents in a typical composite system. The guidelines also address the characterization of the composite system, with particular emphasis on lamina properties, rather than laminate properties. Recommendations on the evaluation of bolted joints in composite materials are provided. Volume I also provides guidelines for the statistical analysis and presentation of data.

Volume II will provide statistically-based mechanical property data for composite material systems used in the aerospace industry. Strength and strain-to-failure properties will be reported either in terms of B-values or S-values (see Section 1.7). Stiffness properties will generally be reported as typical values. The specific statistical significance of each of these quantities will be defined in the first chapter of Volume II. Physical, chemical, and mechanical properties of the composite constituents--the fibers, matrix material and prepreg--will be reported where applicable. Later chapters will include data summaries for the various composite systems. Individual chapters focus on particular reinforcement fibers.

Volume III provides guidelines for the design and analysis of composite materials. The chapters in Volume III address related topics of 1) Materials and Processes, 2) Quality Control of Production Composites, and 3) Design and Analysis. This information is included primarily for background information and are not offered for regulatory purposes.

Statistically-based strength properties will be defined for each composite material system over a range of potential usage conditions. The intent will be to provide data at the upper and lower limits of the potential environmental conditions for a particular material, so that applications issues do not govern the mechanical property characterizations. If data are also available at intermediate environmental conditions, they will also be used to more exactly define the relationship between the mechanical properties and the effect of the environment on those properties. The statistically-based strength data which are available will be tabulated in Volume II. These data will be useful as a starting point for establishing structural design allowables when stress and strength analysis capabilities permit lamina level margin of safety checks. Depending on the application, some structural design allowables will have to be determined empirically at the laminate and composite level, since MIL-HDBK-17 does not provide these data.

Additional information and properties will be added as they become available and are demonstrated to meet the guideline criteria. Typical property values, as well as S-values (see definitions) will be included if they meet the approval of the MIL-HDBK-17 Coordination Group.

When the guidelines or data requirements of MIL-HDBK-17 cannot be followed, the certifying or procuring government agency should be contacted to determine data requirements and other documentation which may be necessary to justify data values proposed or used by the manufacturer.

1.4 Use of the document and limitations. The information contained in MIL-HDBK-17 is obtained from materials producers and fabricators, the aerospace industry, reports on government-sponsored research, the open literature, and by contact with research laboratories and those who participate in the MIL-HDBK-17 coordination activity. All of the information and data contained in this document have been coordinated with representatives from industry and the Army, Navy, Air Force, and Federal Aviation Administration prior to publication. Every effort has been made to reflect the latest information on composite materials and structural details for aerospace vehicles and structures. The handbook is continually reviewed and revised to insure its completeness and to keep it as current as possible.

All data included herein are based on test specimens only. Test specimens dimensions conform with those specified for the particular test method which is used. Standard test methods are recommended where possible (ASTM standards are the primary source). The designer and all other users must be responsible for any translation of the data contained herein to other coupon dimensions, temperature, humidity, and other environmental conditions not covered in this document. Problems such as scale up effects and the influence of the test method selected on properties are also not addressed in this document. The manner in which S-basis values are used is also up to the discretion of the designer. In general, decisions concerning which properties to use for a specific application or design are the responsibility of the designer and are outside the scope of this handbook.

The data which are tabulated in this handbook are intended as an aid in assigning property values to a material. In specific cases where it is necessary or preferable to develop strength properties superior to those in this handbook acceptance of such values must be obtained from the appropriate procurement or certification agency. The applicability and interpretation of specific provisions of this handbook must also be defined by the appropriate procurement, regulatory, or certification agency.

Reference information which is cited in this handbook may not comply in every respect with the guidelines or other criteria specified in this document. References are provided at the end of each chapter primarily as a source of additional information in a given subject area.

The use of tradenames and proprietary product names does not constitute an endorsement of those products by the Government.

1.5 Approval procedures. The MIL-HDBK-17 Coordination Group is a joint government-industry activity that meets twice yearly. At each meeting, this group acts upon proposed changes or additions to the document that are submitted by any of the working groups. The agenda is usually mailed to attendees four weeks prior to the meeting date, and the minutes four weeks following the meeting. Requests for consideration of material for inclusion in the handbook should be submitted to the appropriate working group and the Secretariat well in advance of the mailing date.

Attachments containing proposed changes or additions to the document shall include specific notation of the changes or additions to be made and adequate documentation of supporting data and analytical procedures. Reproducible copies of drawings or photographs intended for inclusion in the document shall be furnished to the Secretariat.

Requests for inclusion of data in MIL-HDBK-17 should be submitted to the Secretariat with the documentation specified in Section 8.1.4. Following analysis and review of the data by the Secretariat, the data will be presented at the next meeting of the Coordination Group.

The choice of new materials to be included herein is governed by the MIL-HDBK-17 Coordination Group. Practical considerations preclude inclusion of all advanced composite materials that may be of interest. Reasonable attempts will be made to add new materials of interest in a timely manner.

1.6 Symbols, abbreviations, and systems of units. This section defines the symbols and abbreviations which are used within MIL-HDBK-17 and describes the system of units which is maintained. Common usage is maintained where possible. References 1.6(a), 1.6(b), and 1.6(c) served as primary sources for this information.

1.6.1 Symbols and abbreviations. Both subscripts and superscripts are used to identify the various composite material quantities and parameters properly. The detailed rules for subscript and superscripts usage are detailed as follows:

- The symbols f and m, when used as either subscripts or superscripts, always denote fiber and matrix, respectively.
- The type of stress (e.g., cy - compression yield) is always used in the superscript position.
- Direction indicators (e.g., x, y, z, L, T, etc.) are always used in the subscript position.
- Ordinal indicators of laminae sequence (e.g., 1, 2, 3, etc.) are used in the superscript position and must be parenthesized to distinguish them from mathematical exponents.
- Other indicators may be used in either subscript or superscript position, as appropriate for clarity.
- Compound symbols (i.e., basic symbol plus indicators) which deviate from these rules are shown in their specific form in the following list.

The following general symbols and abbreviations are considered standard for use in MIL-HDBK-17. Where exceptions are made, they are noted in the text and tables.

A	- area (m^2,in^2)
a	- (1) length dimension (mm,in)
	- (2) acceleration (m/sec^2,ft/sec^2)
b	- width dimension (mm,in), e.g. the width of a bearing or compression panel normal to load, or breadth of beam cross-section
C	- column buckling fixity coefficient
$\bar{c}$	- (1) specific heat (kJ/kg °C,BTU/lb °F)
	- (2) honeycomb sandwich core depth (mm,in)
CF	- centrifugal force (N,lbf)
CG	- center of mass; "center of gravity"; also, frequency, area of volume centroid
℄	- centerline
D	- (1) diameter (mm,in)
	- (2) stiffness (N-m,lbf-in)
E	- Young's modulus (GPa,Msi)
E'	- storage modulus
E''	- loss modulus
E'_c	- Young's modulus of honeycomb core normal to sandwich plane (GPa,Msi)
e	- edge distance (mm,in)
F	- allowable stress (MPa,ksi)
f	- applied stress (MPa,ksi)

$F_{0.70}, F_{0.85}$ - stress levels at intercepts of stress-strain curve with secants whose slopes are 0.70E and 0.85E, respectively (MPa,ksi)

G - shear modulus (MPa,ksi)

g - acceleration of gravity

G'_{cx} - shear modulus of sandwich core along X axis (MPa,ksi)

G'_{cy} - shear modulus of sandwich core along Y axis (MPa,ksi)

h - height dimension (mm,in) e.g. the height of a beam cross section. Also, sometimes used for thickness.

H/C - honeycomb (sandwich)

I - area moment of inertia (mm^4,in^4)

K - (1) dielectric constant

- (2) general coefficient

k - (1) general coefficient

- (2) coefficient of thermal conductivity (W/m °C,BTU/ft^2/hr/in/°F)

K_t - (1) geometrical stress concentration factor

- (2) t_w/c ratio in H/C sandwich

K_s - plate or cylinder shear buckling coefficient

K_x, K_y - plate or cylinder compression buckling coefficient

L - cylinder, beam, or column length (mm,in)

L' - effective column length (mm,in)

M - moment (N-m,in-lbf)

m - (1) mass (kg,lb)
- (2) number of half wave lengths

M.S. - margin of safety

MW - molecular weight

MWD - molecular weight distribution

N - (1) number of fatigue cycles
- (2) number of laminae in a laminate
- (3) distributed in-plane forces on a panel (lbf/in)

n - (1) number of times in a set
- (2) number of half or total wavelengths

NA - neutral axis

P - applied load (N,lbf)

p - normal pressure (Pa,psi)

Q - area static moment (mm^3,in^3)

q - shear flow (N/m,lbf/in)

R - (1) radius (mm,in)
- (2) algebraic ratio of minimum load to maximum load in cyclic loading
- (3) ratio of applied to allowable stress

r - radius (mm,in)

s - (1) arc length (mm,in)
- (2) H/C sandwich cell size

T - (1) temperature (°C,°F)
- (2) torque (N-m,in-lbf)

T_d - thermal decomposition temperature (°C,°F)

T_g - glass transition temperature (°C,°F)

T_m - melting temperature (°C,°F)

t - (1) thickness (mm,in)

- (2) time (s)

V - (1) volume (mm^3,in^3)

- (2) shear force (N,lbf)

W - weight (N,lbf)

α - coefficient of thermal expansion (m/m/°C,in/in/°F)

γ - shear strain (m/m,in/in)

Δ - difference (used as prefix to quantitative symbols)

δ - elongation or deflection (mm,in)

ϵ - strain (m/m,in/in)

η - plasticity reduction factor

$[\eta]$ - intrinsic viscosity

η^* - dynamic complex viscosity

ν - Poisson's ratio

ρ - density (kg/m^3,lb/in^3)

ρ'_c - H/C sandwich core density (kg/m^3,lb/in^3)

Σ - total

σ - (1) applied axial stress (MPa,ksi)

- (2) standard deviation

σ_{ij}, τ_{ij} - stress in j direction on surface whose outer normal is in i direction (i, j = 1, 2, 3 or x, y, z)

T - applied shear stress (MPa,ksi)

1.6.1.1 Constituent properties. The following symbols apply specifically to the constituent properties of a typical composite material.

E_f - Young's modulus of filament material (MPa,ksi)

E_m - Young's modulus of matrix material (MPa,ksi)

E^g_L - Young's modulus of impregnated glass scrim cloth in the filament direction or in the warp direction of a fabric (MPa,ksi)

E^g_T - Young's modulus of impregnated glass scrim cloth transverse to the filament direction or to the warp direction in a fabric (MPa,ksi)

G_f - shear modulus of filament material (MPa,ksi)

G_m - shear modulus of matrix (MPa,ksi)

$G^g{}_{LT}$ - shear modulus of impregnated glass scrim cloth (MPa,ksi)

ℓ - filament length (mm,in)

α_f - coefficient of thermal expansion for filament material (m/m/°C,in/in/°F)

α_m - coefficient of thermal expansion for matrix material (m/m/°C,in/in/°F)

α_L^g - coefficient of thermal expansion of impregnated glass scrim cloth in the filament direction or in the warp direction of a fabric (m/m/°C,in/in/°F)

α_T^g - coefficient of thermal expansion of impregnated glass scrim cloth transverse to the filament direction or to the warp direction in a fabric (m/m/°C,in/in/°F)

ν_f - Poisson's ratio of filament material

ν_m - Poisson's ratio of matrix material

ν_{LT}^g - glass scrim cloth Poisson's ratio relating to contraction in the transverse (or fill) direction as a result of extension in the longitudinal (or warp) direction

ν_{TL}^g - glass scrim cloth Poisson's ratio relating to contraction in the longitudinal (or warp) direction as a result of extension in the transverse (or fill) direction

$\bar{\sigma}$ - applied axial stress at a point, as used in micromechanics analysis (MPa,ksi)

$\bar{\tau}$ - applied shear stress at a point, as used in micromechanics analysis (MPa,ksi)

1.6.1.2 Laminae and laminates. The following symbols, abbreviations, and notations apply to composite laminae and laminates. At the present time the focus in MIL-HDBK-17 is on laminae properties. However, commonly used nomenclature for both laminae and laminates are included here to avoid potential confusion.

A_{ij} (i,j = 1,2,6) - extensional rigidities (N/m,lbf/in)

B_{ij} (i,j = 1,2,6) - coupling matrix (N,lbf)

C_{ij} (i,j = 1,2,6) - elements of stiffness matrix (Pa,psi)

D_x, D_y - flexural rigidities (N-m,lbf-in)

D_{xy} - twisting rigidity (N-m,lbf-in)

D_{ij} (i,j = 1,2,6) - flexural rigidities (N-m,lbf-in)

E_L, E_α - Young's modulus of lamina parallel to filament or warp direction (GPa,Msi)

E_T, E_β - Young's modulus of lamina transverse to filament or warp direction (GPa,Msi)

E_x - Young's modulus of laminate along X reference axis (GPa,Msi)

E_y - Young's modulus of laminate along Y reference axis (GPa,Msi)

G_{LT}, $G_{\alpha\beta}$ - shear modulus of lamina in LT or $\alpha\beta$ plane (GPa,Msi)

G_{xy} - shear modulus of laminate in XY reference plane (GPa,Msi)

h_i - thickness of i^{th} ply or lamina (mm,in)

M_x, M_y, M_{xy} - bending and twisting moment components (N-m/m,in-lbf/in in plate and shell analysis)

n_f - number of filaments per unit length per lamina

Q_x, Q_y - shear force parallel to Z axis of sections of a plate perpendicular to X and Y axes, respectively (N/m,lbf/in)

Q_{ij} $(i,j = 1,2,6)$ - reduced stiffness matrix (Pa,psi)

u, v, w - components of the displacement vector (mm,in)

u_o, v_o, w_o - components of the displacement vector at the laminate's midsurface (mm,in)

V_v - void content (% by volume)

V_f - filament content or fiber volume (% by volume)

V_g - glass scrim cloth content (% by volume)

V_m - matrix content (% by volume)

V_x, V_y - edge or support shear force (N/m, lbf/in)

W_f - filament content (% by weight)

W_g - glass scrim cloth content (% by weight)

W_m - matrix content (% by weight)

W_s - weight of laminate per unit surface area (N/m^2, lbf/in^2)

α_L, α_α - lamina coefficient of thermal expansion along L or α axis (m/m/°C, in/in/°F)

α_T, α_β - lamina coefficient of thermal expansion along T or β axis (m/m/°C, in/in/°F)

α_x - laminate coefficient of thermal expansion along general reference X axis (m/m/°C, in/in/°F)

α_y - laminate coefficient of thermal expansion along general reference Y axis (m/m/°C, in/in/°F)

α_{xy} - laminate shear distortion coefficient of thermal expansion (m/m/°C, in/in/°F)

θ - angular orientation of a lamina in a laminate, i.e., angle between L and X axes (°)

λ_{xy} - product of ν_{xy} and ν_{yx}

ν_{LT}, $\nu_{\alpha\beta}$ - Poisson's ratio relating contraction in the T or β direction as a result of extension in the L or α direction.

ν_{TL}, $\nu_{\beta\alpha}$ - Poisson's ratio relating contraction in the L or α direction as a result of extension in the T or β direction.

ν_{xy} - Poisson's ratio relating contraction in the y direction as a result of extension in the x direction

ν_{yx} - Poisson's ratio relating contraction in the x direction as a result of extension in the y direction

ρ_c - density of a single lamina (kg/m^3, lb/in^3)

$\bar{\rho}_c$ - density of a laminate (kg/m^3,lb/in^3)

ϕ - (1) general angular coordinate, (°)
- (2) angle between X and load axes in off-axis loading (°)

1.6.1.3 Subscripts. The following subscript notations are considered standard in MIL-HDBK-17.

a - adhesive, or alternating stress

c - composite system, specific filament/matrix composition. Composite as a whole, contrasted to individual constituents. Also, sandwich core when used in conjunction with prime superscript(′)

cf - centrifugal force

eff - effective

eq - equivalent

f - filament

g - glass scrim cloth

i - i^{th} position in a sequence

L, T, z - laminae natural orthogonal coordinates (L is filament or warp direction)

m - matrix

max - maximum

min - minimum

n - n^{th} (last) position in a sequence

s - symmetric

st - stiffener

t - value of parameter at t

w - sheet

x, y, z - general coordinate system

α, β, z - laminae natural orthogonal coordinates (α is filament or warp direction)

Σ - total, or summation

o - initial or reference datum

$(\ )_T$ - format for indicating specific temperature associated with term in parentheses. RT-room temperature (21°C,70°F); all other temperatures in °F unless specified.

1.6.1.4 Superscripts. The following superscript notations are considered standard in MIL-HDBK-17.

b - bending (denotes bending modulus of rupture when used with minimum design property symbol, F)
br - bearing
bru - bearing ultimate
bry - bearing yield
c - compression or creep
ccr - compression buckling
cu - compression ultimate
cy - compression yield
e - elastic
f - filament
g - glass scrim cloth
is - interlaminar shear
isu - interlaminar shear ultimate
isy - interlaminar shear yield
(i) - i^{th} ply or lamina
lim - limit, used to indicate limit loading

m	matrix
p	plastic
pl	proportional limit
rup	rupture
s	shear
scr	shear buckling
sec	secant (modulus)
su	shear ultimate
sy	shear yield
T	temperature or thermal
t	tension
tan	tangent (modulus)
tu	tension ultimate
ty	tension yield
ult	ultimate, used to indicate ultimate loading
	secondary (modulus), or denotes properties of H/C core when used with subscript c

1.6.2 System of units. To comply with Department of Defense Directive 4120.18, "Metric System of Measurement," dated January 28, 1980, the data in MIL-HDBK-17 are generally presented in both the International System of Units (SI units) and the U. S. Customary (English) system of units. ASTM E-380, Standard for Metric Practice, provides guidance for the application for SI units which are intended as a basis for worldwide standardization of measurement units. Further guidelines on the use of the SI system of units and conversion factors are contained in the following publications:

(1) DARCOM P 706-470, Engineering Design Handbook: Metric Conversion Guide, July 1976.

(2) NBS Special Publication 330, "The International System of Units (SI)," National Bureau of Standards, 1986 edition.

(3) NBS Letter Circular LC 1035, "Units and Systems of Weights and Measures, Their Origin, Development, and Present Status," National Bureau of Standards, November 1985.

(4) NASA Special Publication 7012, "The International System of Units Physical Constants and Conversion Factors" (1964).

English to SI conversion factors pertinent to MIL-HDBK-17 data are contained in Table 1.6.2.

Table 1.6.2 English to SI conversion factors.

To convert from	to	Multiply by
Btu (thermochemical)/in^2-s	watt/meter^2 (W/m^2)	1.634 246 E+06
Btu-in/(s-ft^2-°F)	W/(m K)	5.192 204 E+02
degree Fahrenheit	degree Celsius (°C)	$T_C = (T_F - 32)/1.8$
degree Fahrenheit	kelvin (K)	$T_K = (T_F + 459.67)/1.8$
foot	meter (m)	3.048 000 E-01
ft^2	m^2	9.290 304 E-02
foot/second	meter/second (m/s)	3.048 000 E-01
ft/s^2	m/s^2	3.048 000 E-01
inch	meter (m)	2.540 000 E-02
in.^{2}	meter^2 (m^2)	6.451 600 E-04
in.^{3}	m^3	1.638 706 E-05
kilogram-force (kgf)	newton (N)	9.806 650 E+00
kgf/m^2	pascal (Pa)	9.806 650 E+00
kip (1000 lbf)	newton (N)	4.448 222 E+03
ksi (kip/in^2)	MPa	6.894 757 E+00
lbf-in	N-m	1.129 848 E-01
lbf-ft	N-m	1.355 818 E+00
lbf/in^2 (psi)	pascal (Pa)	6.894 757 E+03
lb/in^3	kg/m^3	2.767 990 E+04
Msi (10^6 psi)	GPa	6.894 757 E+00
pound-force (lbf)	newton (N)	4.488 222 E+00
pound-mass (lb avoirdupois)	kilogram (kg)	4.535 924 E-01
torr	pascal (Pa)	1.333 22 E+02

*The letter "E" following the conversion factor stands for exponent and the two digits after the letter "E" indicate the power of 10 by which the number is to be multiplied.

1.7 Definitions. The following definitions are used within MIL-HDBK-17. This glossary of terms is not totally comprehensive but it does represent nearly all commonly used terms. Where exceptions are made, they are noted in the text and tables.

For ease of identification the definitions have been organized alphabetically.

A-Basis (or A-Value) -- The mechanical property value above which 99 percent of the population of values is expected to fall, with a confidence of 95 percent. Note - A-values are not currently presented in MIL-HDBK-17. This is a 95% lower confidence limit on the first percentile.

A-Stage -- An early stage in the reaction of thermosetting resins in which the material is still soluble in certain liquids and may be liquid or capable of becoming liquid upon heating. (Sometimes referred to as resol.)

Absorption -- A process in which one material (the absorbent) takes in or absorbs another (the absorbate).

Accelerator -- As applicable to an epoxy resin system, accelerators are added to an epoxy resin - curing agent mixture to speed up a sluggish reaction. (Accelerators are added in small amounts, non-stoichiometrically, which have been determined empirically to give the best results.)

Accuracy -- The degree of conformity of a measured or calculated value to some recognized standard or specified value. Accuracy involves the systematic error of an operation.

Addition Polymerization -- Polymerization in which monomers are linked together without the splitting off of water or other simple molecules.

Adhesion -- The property denoting the ability of a material to resist delamination or separation into two or more layers.

Adhesive -- A substance capable of holding two materials together by surface attachment. In the handbook, the term is used specifically to designate structural adhesives, those which produce attachments capable of transmitting significant structural loads.

ADK -- Notation used for the k-sample Anderson-Darling statistic, which is used to test the hypothesis that k batches have the same distribution.

Aging -- The effect on materials of exposure to an air environment for an interval of time.

Anelasticity -- A characteristic exhibited by certain materials in which strain is a function of both stress and time, such that, while no permanent deformations are involved, a finite time is required to establish equilibrium between stress and strain in both the loading and unloading directions.

Angleply -- Same as Crossply.

Anisotropic -- Not isotropic; having mechanical and/or physical properties which vary with direction relative to natural reference axes inherent in the material.

Aramid -- A manufactured fiber in which the fiber-forming substance consisting of a long-chain synthetic aromatic polyamide in which as least 85% of the amide (-CONH-) linkages are attached directly to two aromatic rings.

Areal Weight of Fiber -- The weight of fiber per unit area of prepreg. This is often expressed as grams per square meter. See Table 1.6.2 for conversion factors.

Artificial Weathering -- Exposure to laboratory conditions which may be cyclic, involving changes in temperature, relative humidity, radiant energy and any other elements found in the atmosphere in various geographical areas.

Aspect Ratio -- In an essentially two-dimensional rectangular structure (e.g., a panel), the ratio of the long dimension to the short dimension. However, in compression loading, it is sometimes considered to be the ratio of the load direction dimension to the transverse dimension. Also, in fiber micro-mechanics, it is referred to as the ratio of length to diameter.

Autoclave -- A closed vessel for producing an environment of fluid pressure, with or without heat, to an enclosed object which is undergoing a chemical reaction or other operation.

Autoclave Molding -- A process similar to the pressure bag technique. The lay-up is covered by a pressure bag, and the entire assembly is placed in an autoclave capable of providing heat and pressure for curing the part. The pressure bag is normally vented to the outside.

B-Basis (or B-Value) -- The mechanical property value above which at least 90 percent of the population of values is expected to fall, with a confidence of 95 percent, specifically the same as A-Basis except on the tenth percentile.

B-Stage -- An intermediate stage in the reaction of a thermosetting resin in which the material softens when heated and swells when in contact with certain liquids but does not entirely fuse or dissolve. Materials are usually precured to this stage to facilitate handling and processing prior to final cure. (Sometimes referred to as resistol.)

Bag Molding -- A method of molding or laminating which involves the application of fluid pressure to a flexible material which transmits the pressure to the material being molded or bonded. Fluid pressure usually is applied by means of air, steam, water or vacuum.

Balanced Laminate -- A composite laminate in which all laminae at angles other than 0 degrees and 90 degrees occur only in ± pairs (not necessarily adjacent).

Batch (or Lot) -- In general, a quantity of material formed during the same process and having identical characteristics throughout. As applied to the handbook, a batch of prepreg is defined as a quantity which is produced from a single batch of matrix material and fiber. The prepreg batch is produced at one time in the same equipment under identical conditions.

Bearing Area -- The diameter of the hole multiplied by the thickness.

Bearing Load -- A compressive load on an interface.

Bearing Yield Strength -- The bearing stress at which a material exhibits a specified limiting deviation from the proportionality of bearing stress to bearing strain.

Bend Test -- A test of ductility by bending or folding, usually with steadily applied forces. In some instances the test may involve blows to a specimen having a cross section that is essentially uniform over a length several times as great as the largest dimension of the cross section.

Binder -- A bonding resin used to hold strands together in a mat or preform during manufacture of a molded object.

Binomial Random Variable -- The number of successes in independent trials where the probability of success is the same for each trial.

Birefringence -- The difference between the two principal refractive indices (of a fiber) or the ratio between the retardation and thickness of a material at a given point.

Bleeder Cloth -- A nonstructural layer of material used in the manufacture of composite parts to allow the escape of excess gas and resin during cure. The bleeder cloth is removed after the curing process and is not part of the final composite.

Bond -- The adhesion of one surface to another, with or without the use of an adhesive as a bonding agent.

Broadgoods -- A term loosely applied to prepreg material greater than about 12 inches in width, usually furnished by suppliers in continuous rolls. The term is currently used to designate both collimated uniaxial tape and woven fabric prepregs.

Buckling (Composite) -- A mode of failure characterized generally by an unstable material deflection due to compressive action on the structural element involved. In advanced composites, buckling may take the form not only of conventional general instability and local instability but also a micro-instability of individual fibers.

Bundle -- A general term for a collection of essentially parallel filaments or fibers.

C-Stage -- The final stage of the curing reaction of a thermosetting resin in which the material has become practically infusable and insoluble. (Normally considered fully cured and sometimes referred to as resite.)

Carbon Fibers -- Fibers made from a precursor by oxidation and carbonization, and not having a graphitic structure.

Caul Plates -- Smooth metal plates, free of surface defects, the same size and shape as a composite lay-up, used immediately in contact with the lay-up during the curing process to transmit normal pressure and to provide a smooth surface on the finished laminate.

Censoring -- Data is right (left) censored at M, if, whenever an observation is less than or equal to M (greater than or equal to M), the actual value of the observation is recorded. If the observation exceeds (is less than) M, the observation is recorded as M.

Chain-Growth Polymerization -- A chemical reaction in which polymer formation is initiated by a reactive species R* produced from some compound I termed an initiator. The reactive species may be a free radical, cation or anion. The reactive center, once produced, adds monomer units in a chain reaction and grows rapidly to a large size. High molecular weight polymer forms immediately with the molecular weight changing slightly, if at all, as the monomer concentration decreases steadily during the reaction.

Chromatogram -- A plot of detector response against peak volume of solution (eluate) emerging from the system for each of the constituents which have been separated.

Circuit -- The winding produced by a single revolution of a mandrel or form in filament winding.

Cocuring -- The act of curing a composite laminate and simultaneously bonding it to some other prepared surface during the same cure cycle (see Secondary Bonding).

Coefficient of Variation -- The ratio of the population (or sample) standard deviation to the population (or sample) mean.

Collimated -- Rendered parallel.

Compatible -- Descriptive term referring to different batches which may be treated as coming from the same population.

Composite Class -- As used in the handbook, a major subdivision of composite construction in which the class is defined by the fiber system and the matrix class, e.g., organic-matrix filamentary laminate.

Composite Material -- Composites are considered to be combinations of materials differing in composition or form on a macroscale. The constituents retain their identities in the composite; that is, they do not dissolve or otherwise merge completely into each other although they act in concert. Normally, the components can be physically identified and exhibit an interface between one another.

Compound -- An intimate mixture of polymer or polymers with all the materials necessary for the finished product.

Condensation Polymerization -- This is a special type of step-growth polymerization characterized by the formation of water or other simple molecules during the stepwise addition of reactive groups.

Confidence Coefficient -- See confidence interval.

Confidence Interval -- A confidence interval is defined by a statement of one of the following forms:

$$(1)\ P\{a<\theta\} \geq 1-\alpha$$
$$(2)\ P\{\theta<b\} \geq 1-\alpha$$
$$(3)\ P\{a<\theta<b\} \geq 1-\alpha$$

where $1-\alpha$ is called the confidence coefficient. A statement of type (1) or (2) is called a one-sided confidence interval and a statement of type (3) is called a two-sided confidence interval. In (1) a is a lower confidence limit and in (2) b is an upper confidence limit. With probability at least $1-\alpha$, the confidence interval will contain the parameter θ.

Constituent -- In general, an element of a larger grouping. In advanced composites, the principal constituents are the fibers and the matrix.

Continuous Filament -- A yarn or strand in which the individual filaments are substantially the same length as the strand.

Coupling Agent -- Any chemical substance designed to react with both the reinforcement and matrix phases of a composite material to form or promote a stronger bond at the interface. Coupling agents are applied to the reinforcement phase from an aqueous or organic solution or from a gas phase, or added to the matrix as an integral blend.

Crazing -- Apparent fine cracks at or under the surface of an organic matrix.

Creep -- The time dependent part of strain resulting from an applied stress.

Creep, Rate Of -- The slope of the creep-time curve at a given time.

Critical Value(s) -- If a sample statistic is outside the critical value(s), the statistical hypothesis under test is rejected.

Crossply -- Any filamentary laminate which is not uniaxial. Same as Angleply. In some references, the term crossply is used to designate only those laminates in which the laminae are at right angles to one another, while the term angleply is used for all others. In the handbook, the two terms are used synonymously. The reservation of a separate terminology for only one of several basic orientations is unwarranted because a laminate orientation code is used.

Cure -- To change the properties of a thermosetting resin irreversibly by chemical reaction, i.e., condensation, ring closure, or addition. Cure may be accomplished by addition of curing (cross-linking) agents, with or without catalyst, and with or without heat. Cure may occur also by addition, such as occurs with anhydrite cures for epoxy resin systems.

Cure Cycle -- The schedule of time periods at specified conditions to which a reacting thermosetting material is subjected in order to reach a specified property level.

Cure Stress -- A residual internal stress produced during the curing cycle of composite structures. Normally, these stresses originate when different components of a lay-up have different thermal coefficients of expansion.

Debond -- A deliberate separation of a bonded joint or interface, usually for repair or rework purposes. (see Disbond, Unbond).

Deformation -- The change in shape of a specimen caused by the application of a load or force.

Degradation -- A deleterious change in chemical structure, physical properties or appearance.

Delamination -- The separation of the layers of material in a laminate. This may be local or may cover a large area of the laminate. It may occur at any time in the cure or subsequent life of the laminate and may arise from a wide variety of causes.

Denier -- A direct numbering system for expressing linear density, equal to the mass in grams per 9000 meters of yarn, filament, fiber, or other textile strand.

Density -- The mass per unit volume.

Desorption -- A process in which an absorbed or adsorbed material is released from another material. Desorption is the reverse of absorption, adsorption, or both.

Deviation -- Variation from a specified dimension or requirement, usually defining the upper and lower limits.

Disbond -- An area within a bonded interface between two adherends in which an adhesion failure or separation has occurred. It may occur at any time during the life of the structure and may arise from a wide variety of causes. Also, colloquially, an area of separation between two laminae in the finished laminate (in this case the term "delamination" is normally preferred.) (See Debond, Unbond, Delamination.)

Distribution -- A formula which gives the probability that a value will fall within prescribed limits. (See normal, Weibull, and lognormal distributions).

Dry Fiber Area -- Area of fiber not totally encapsulated by resin.

Ductility -- The ability of a material to deform plastically before fracturing.

Elasticity -- The property of a material which allows it to recover its original size and shape immediately after removal of the force causing deformation.

Elongation -- The increase in gage length or extension of a specimen during a tension test, usually expressed as a percentage of the original gage length.

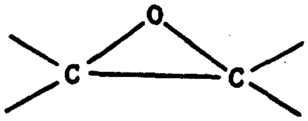
Eluate -- The liquid emerging from a column (in liquid chromatography).

Eluent -- The mobile phase used to sweep or elute the sample (solute) components into, through, and out of the column.

End -- A single fiber, strand, roving or yarn being or already incorporated into a product. An end may be an individual wrap yarn or cord in a woven fabric. In referring to aramid and glass fibers, an end is usually an untwisted bundle of continuous filaments.

Epoxy Equivalent Weight -- The number of grams of resin which contain one chemical equivalent of the epoxy group.

Epoxy Resin -- Resins which may be of widely different structures but are characterized by the presence of the epoxy group:



(The epoxy or epoxide group is usually present as a glycidyl ether, glycidyl amine, or as part of an aliphatic ring system. The aromatic type epoxy resins are normally used in composites.)

Extensometer -- A device for measuring linear strain.

Fabric, Nonwoven -- A textile structure produced by bonding or interlocking of fibers, or both, accomplished by mechanical, chemical, thermal, or solvent means, and combinations thereof.

Fabric, Woven -- A generic material construction consisting of interlaced yarns or fibers, usually a planar structure. Specifically, as used in this handbook, a cloth woven in an established weave pattern from advanced fiber yarns and used as the fibrous constituent in an advanced composite lamina. In a fabric lamina, the warp direction is considered the longitudinal direction, analogous to the filament direction in a filamentary lamina.

Fiber -- A general term used to refer to filamentary materials. Often, fiber is used synonymously with filament. It is a general term for a filament of finite length.

Fiber Content -- The amount of fiber present in a composite. This is usually expressed as a percentage volume fraction or weight fraction of the composite.

Fiber Count -- The number of fibers per unit width of ply present in a specified section of a composite.

Fiber Direction -- The orientation or alignment of the longitudinal axis of the fiber with respect to a stated reference axis.

Fiber System -- The type and arrangement of fibrous material which comprises the fiber constituent of an advanced composite. Examples of fiber systems are collimated filaments or filament yarns, woven fabric, randomly oriented short-fiber ribbons, random fiber mats, whiskers, etc.

Filament -- The smallest unit of a fibrous material. The basic units formed during spinning and which are gathered into strands of fiber, (for use in composites). Filaments usually are of extreme length and of very small diameter. Filaments normally are not used individually. Some textile filaments can function as a yarn when they are of sufficient strength and flexibility.

Filamentary Composites -- A major form of advanced composites in which the fiber constituent consists of continuous filaments. Specifically, a filamentary composite is a laminate comprised of a number of laminae, each of which consists of a nonwoven, parallel, uniaxial, planar array of filaments (or filament yarns) imbedded in the selected matrix material. Individual laminae are directionally oriented and combined into specific multiaxial laminates for application to specific envelopes of strength and stiffness requirements.

Filament Winding -- An automated process in which a continuous fiber bundle (or tape), either preimpregnated or wet impregnated with resin, are wound on a removable mandrel in a pattern.

Filament Wound -- Pertaining to an object created by the filament winding method of fabrication.

Fill -- Yarn oriented at right angles to the warp in a woven fabric.

Filler -- A relatively inert substance added to a material to alter its physical, mechanical, thermal, electrical, and other properties or to lower cost. Sometimes the term is used specifically to mean particulate additives.

Finish (or Size System) -- A material, with which filaments are treated, which contains a coupling agent to improve the bond between the filament surface and the resin matrix in a composite material. In addition, finishes often contain ingredients which provide lubricity to the filament surface, preventing abrasive damage during handling, and a binder which promotes strand integrity and facilitates packing of the filaments.

Flash -- Excess material which forms at the parting line of a mold or die, or which is extruded from a closed mold.

Fracture Ductility -- The true plastic strain at fracture.

Gage Length -- (1) The length of a specimen measured between the points of attachment to clamps while under uniform tension. (2) The original distance between extensometer probes prior to a tension or compression test.

Gage Length, True -- The precise length between well-defined bench marks located on the specimen while under known tension in the unsupported portion between the holding clamps and free from contact with any snubbing surfaces or other sources which could result in nonuniform strain.

Gel -- The initial jelly-like solid phase that develops during formation of a resin from a liquid. Also, a semi-solid system consisting of a network of solid aggregates in which liquid is held.

Gel Coat -- A quick-setting resin used in molding processes to provide an improved surface for the composite; it is the first resin applied to the mold after the mold-release agent.

Gel Point -- The stage at which a liquid begins to exhibit pseudo-elastic properties. (This can be seen from the inflection point on a viscosity-time plot.)

Gel Time -- The period of time from a pre-determined starting point to the onset of gelation (gel point) as defined by a specific test method.

Glass -- An inorganic product of fusion which has cooled to a rigid condition without crystallizing. In the handbook, all reference to glass will be to the fibrous form as used in filaments, woven fabric, yarns, mats, chopped fibers, etc.

Glass Cloth -- Conventionally-woven glass fiber material (see Scrim).

Glass Fibers -- A fiber spun from an inorganic product of fusion which has cooled to a rigid condition without crystallizing.

Glass Transition -- The reversible change in an amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one.

Glass Transition Temperature -- The approximate midpoint of the temperature range over which the glass transition takes place.

Graphite Fibers -- Fibers made from a precursor by oxidation, carbonization, and graphitization process (which provides a graphitic structure).

Greige -- Fabric that has received no finish.

Hand lay-up -- A process in which components are applied either to a mold or a working surface, and the successive plies are built up and worked by hand.

Hardness -- Resistance to deformation; usually measured by indention. Types of standard tests include Brinell, Rockwell, Knoop, and Vickers.

Heat Cleaned -- Glass or other fibers which have been exposed to elevated temperatures to remove preliminary sizings or binders which are not compatible with the resin system to be applied.

Heterogeneous -- Descriptive term for a material consisting of dissimilar constituents separately identifiable; a medium consisting of regions of unlike properties separated by internal boundaries. (Note that all nonhomogeneous materials are not necessarily heterogeneous).

Homogeneous -- Descriptive term for a material of uniform composition throughout; a medium which has no internal physical boundaries; a material whose properties are constant at every point, i.e., constant with respect to spatial coordinates (but not necessarily with respect to directional coordinates).

Horizontal Shear -- Sometimes used to indicate interlaminar shear. This is not an approved term for use in this handbook.

Humidity, Relative -- The ratio of the pressure of water vapor present to the pressure of saturated water vapor at the same temperature.

Hybrid -- A composite laminate comprised of laminae of two or more composite material systems. Or, a combination of two or more different fibers such as carbon and glass or carbon and aramid into a structure (tapes, fabrics and other forms may be combined).

Hysteresis -- The energy absorbed in a complete cycle of loading and unloading.

Inclusion -- A physical and mechanical discontinuity occurring within a material or part, usually consisting of solid, encapsulated foreign material. Inclusions are often capable of transmitting some structural stresses and energy fields, but in a noticeably different manner from the parent material.

Integral Composite Structure -- Composite structure in which several structural elements, which would conventionally be assembled by bonding or with mechanical fasteners after separate fabrication, are instead laid up and cured as a single, complex, continuous structure; e.g., spars, ribs, and one stiffened cover of a wing box fabricated as a single integral part. The term is sometimes applied more loosely to any composite structure not assembled by mechanical fasteners.

Interface -- The boundary between the individual, physically distinguishable constituents of a composite.

Interlaminar -- Descriptive term pertaining to some object (e.g., voids), event (e.g., fracture), or potential field (e.g., shear stress) referenced as existing or occurring between two or more adjacent laminae.

Interlaminar Shear -- Shearing force tending to produce a relative displacement between two laminae in a laminate along the plane of their interface.

Intermediate Bearing Stress -- The bearing stress at the point on the bearing load-deformation curve where the tangent is equal to the bearing stress divided by a designated percentage (usually 4%) of the original hole diameter.

Intralaminar -- Descriptive term pertaining to some object (e.g., voids), event (e.g., fracture), or potential field (e.g., temperature gradient) existing entirely within a single lamina without reference to any adjacent laminae.

Isotropic -- Having uniform properties in all directions. The measured properties of an isotropic material are independent of the axis of testing.

Knuckle Area -- The area of transition between sections of different geometry in a filament wound part.

k-Sample Data -- A collection of data consisting of values observed when sampling from k batches.

Lamina -- A single ply or layer in a laminate made up of a series of layers.

Laminae -- Plural of lamina.

Laminate -- A product made by bonding together two or more layers or laminae of material or materials.

Laminate Orientation -- The configuration of a crossplied composite laminate with regard to the angles of crossplying, the number of laminae at each angle, and the exact sequence of the lamina lay-up.

Lattice Pattern -- A pattern of filament winding with a fixed arrangement of open voids.

Lay-up -- A process of fabrication involving the assembly of successive layers of resin-impregnated material.

Lognormal Distribution -- A probability distribution for which the probability that an observation selected at random from this population falls between a and b $(0 < a < b < \infty)$ is given by the area under the normal distribution between ℓog a and ℓog b. The common (base 10) or the natural (base e) logarithm may be used.

Lower Confidence Bound -- See Confidence Interval.

Macro -- In relation to composites, denotes the gross properties of a composite as a structural element but does not consider the individual properties or identity of the constituents.

Macrostrain -- The mean strain over any finite gage length of measurement which is large in comparison to the material's interatomic distance.

Mandrel -- A form fixture or male mold used for the base in the production of a part by lay-up or filament winding.

Mat -- A fibrous material consisting of randomly oriented chopped or swirled filaments loosely held together with a binder.

Material System -- A specific composite material made from specifically identified constituents in specific geometric proportions and arrangements and possessed of numerically defined properties.

Material System Class -- As used in this handbook, a group consisting of material systems categorized by the same generic constituent materials, but without defining the constituents uniquely; e.g., the graphite/epoxy class.

Matrix -- The essentially homogeneous material in which the fiber system of a composite is imbedded.

Mean -- See Sample Mean and Population Mean.

Mechanical Properties -- The properties of a material that are associated with elastic and inelastic reaction when force is applied, or the properties involving the relationship between stress and strain.

Median -- See Sample Median and Population Median.

Micro -- In relation to composites, denotes the properties of the constituents, i.e., matrix and reinforcement and interface only, as well as their effects on the composite properties.

Microstrain -- The strain over a gage length comparable to the material's interatomic distance.

Modulus, Initial -- The slope of the initial straight portion of a stress-strain or load-elongation curve.

Modulus of Rupture, in Bending -- The maximum tensile or compressive stress (whichever causes failure) value in the extreme fiber of a beam loaded to failure in bending. The value is computed from the flexure equation:

$$F^b = \frac{Mc}{I} \qquad 1.7(a)$$

where M = maximum bending moment computed from the maximum load and the original moment arm,
c = initial distance from the neutral axis to the extreme fiber where failure occurs,
I = the initial moment of inertia of the cross section about its neutral axis.

Modulus of Rupture, in Torsion -- The maximum shear stress in the extreme fiber of a member of circular cross section loaded to failure in torsion calculated from the equation:

$$F^s = \frac{Tr}{J} \qquad 1.7(b)$$

where T = maximum twisting moment,
r = original outer radius,
J = polar moment of inertia of the original cross section.

Modulus, Secant -- The ratio of change in stress to change in strain between two points on a stress-strain curve, particularly the points of zero stress and failure stress.

Modulus, Tangent -- The ratio of change in stress to change in strain derived from the tangent to any point on a stress-strain curve.

Modulus, Young's -- The ratio of change in stress to change in strain below the elastic limit of a material. (Applicable to tension and compression).

Modulus of Rigidity (also Shear Modulus or Torsional Modulus) -- The ratio of stress to strain below the proportional limit for shear or torsional stress.

Moisture Content -- The amount of moisture in a material determined under prescribed condition and expressed as a percentage of the mass of the moist specimen, i.e., the mass of the dry substance plus the moisture present.

Moisture Equilibrium -- The condition reached by a sample when it no longer takes up moisture from, or gives up moisture to, the surrounding environment.

Mold Release Agent -- A lubricant applied to mold surfaces to facilitate release of the molded article.

Molded Edge -- An edge which is not physically altered after molding for use in final form and particularly one which does not have fiber ends along its length.

Molding -- The forming of a polymer or composite into a solid mass of prescribed shape and size by the application of pressure and heat.

Monolayer -- The basic laminate unit from which crossplied or other laminates are constructed.

Monomer -- A relatively simple compound which can react to form a polymer.

NDE -- Nondestructive evaluation. Broadly considered synonymous with NDI.

NDI -- Nondestructive Inspection. A process or procedure for determining the quality or characteristics of a material, part, or assembly without permanently altering the subject or its properties.

NDT -- Nondestructive Testing. Broadly considered synonymous with NDI.

Necking -- A localized reduction in cross-sectional area which may occur in a material under tensile stress.

Negatively Skewed -- A distribution is said to be negatively skewed if the distribution is not symmetric and the longest tail is on the left.

Nominal Specimen Thickness -- The nominal ply thickness multiplied by the number of plies.

Nominal Value -- A value assigned for the purpose of a convenient designation. A nominal value exists in name only.

Normal Distribution -- A two parameter (μ,σ) family of probability distributions for which the probability that an observation will fall between a and b is given by the area under the curve

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} e^{-(x-\mu)^2/2\sigma^2} \qquad 1.7(c)$$

between a and b.

Normalized Stress -- Stress calculated by multiplying the raw stress value by the ratio of measured fiber volume to the nominal fiber volume. This ratio is often approximated by the ratio of the measured specimen thickness to the nominal specimen thickness. Stresses for fiber-dominated failure modes are often normalized.

Observed Significance Level (OSL) -- The probability of observing a more extreme value of the test statistic when the null hypotheses is true.

Oligomer -- A polymer consisting of only a few monomer units such as a dimer, trimer, etc. or their mixtures.

One-Sided Tolerance Limit Factor -- See Tolerance Limit Factor.

Orthotropic -- A material having three mutually perpendicular planes of elastic symmetry.

Oven Dry -- The condition of a material that has been heated under prescribed conditions of temperature and humidity until there is no further significant change in its mass.

PAN Fibers -- Polyacrylonitrile spun and stabilized fibers.

Parallel Laminate -- A laminate of woven fabric in which the plies are aligned in the same position as originally aligned in the fabric roll.

Peel Ply -- A layer of resin free material used to protect a laminate for later secondary bonding.

pH -- A measure of acidity or alkalinity of a solution, with neutrality represented by a value of 7, with increasing acidity corresponding to progressively smaller values, and increasing alkalinity corresponding to progressively higher values.

Pick Count -- The number of filling yarns per inch of woven fabric.

Pitch Fibers -- Fibers derived from a special petroleum pitch.

Plastic -- A material that contains one or more organic polymers of large molecular weight, is solid in its finished state, and, at some state in its manufacture or processing into finished articles, can be shaped by flow.

Plasticizer -- A material of lower molecular weight added to a polymer to separate the molecular chains. This results in a depression of the glass-transition temperature, reduced stiffness and brittleness, and improved processability. (Note, many polymeric materials do not need a plasticizer.)

Plied Yarn -- A yarn formed by twisting together two or more single yarns in one operation.

Poisson's Ratio -- The ratio of transverse strain to the corresponding axial strain below the proportional limit caused by a uniformly distributed axial stress.

Polymer -- An organic material composed of molecules characterized by the repetition of one of more types of monomeric units.

Polymerization -- A chemical reaction in which the molecules of monomers are linked together to form polymers.

Population -- The set of measurements about which inferences are to be made or the totality of possible measurements which might be obtained in a given testing situation. For example, "all possible ultimate tensile strength measurements for Hexcel, conditioned at 95% relative humidity and room temperature". In order to make inferences about a population, it is often necessary to make assumptions about its distributional form. The assumed distributional form may also be referred to as the population.

Population Mean -- The average of all potential measurements in a given population weighted by their relative frequencies in the population.

Population Median -- That value in the population such that the probability of exceeding it is 0.5 and the probability of being less than it is 0.5.

Population Variance -- A measure of dispersion in the population.

Porosity -- A condition of trapped pockets of air, gas, or vacuum within a solid material, usually expressed as a percentage of the total nonsolid volume to the total volume (solid plus nonsolid) of a unit quantity of material.

Positively Skewed -- A distribution is said to be positively skewed if the distribution is not symmetric and the longest tail is on the right.

Postcure -- Additional elevated temperature cure, usually without pressure, to improve final properties or complete the cure or both.

Pot Life -- The period of time during which a reacting thermosetting composition remains suitable for its intended processing after mixing with a reaction initiating agent.

Precision -- The degree of agreement within a set of observations or test results obtained. Precision involves repeatability and reproducibility.

Precursor (for Carbon or Graphite Fiber) -- Either the PAN or pitch fibers from which carbon and graphite fibers are derived.

Preply -- A composite material lamina in the raw material stage ready to be fabricated into a finished laminate. The lamina is usually combined with other raw laminae prior to fabrication. A preply includes all of the fiber system placed in position relative to all or part of the required matrix material that together will comprise the finished lamina. An organic matrix preply is called a prepreg. (Metal matrix preplies include green tape, flame-sprayed tape, and consolidated monolayers).

Prepreg -- Ready to mold or cure material in sheet form which may be fiber, cloth, or mat impregnated with resin and stored for use. The resin is partially cured to a B-stage and supplied to the fabricator for lay-up and cure.

Pressure - The force or load per unit area.

Proportional Limit -- The maximum stress that a material is capable of sustaining without any deviation from the proportionality of stress to strain (also known as Hooke's law).

Quasi-Isotropic Laminate -- A laminate approximating isotropy by orientation of plies in several or more directions.

Reduction of Area -- The difference between the original cross sectional area of a tension test specimen and the area of its smallest cross section, usually expressed as a percentage of the original area.

Refractive Index - The ratio of the velocity of light (of specified wavelength) in air to its velocity in the substance under examination. Also defined as the sine of the angle of incidence divided by the sine of the angle of refraction as light passes from air into the substance.

Reinforced Plastic -- A plastic with relatively high stiffness or very high strength fibers imbedded in the composition. This improves some mechanical properties over that of the base resin.

Release Agent -- See Mold Release Agent.

Resilience -- A property of a material which is able to do work against restraining forces during return from a deformed condition.

Resin -- A solid or pseudo-solid organic material usually of high molecular weight which exhibits a tendency to flow when subjected to stress, usually has a softening or melting range, and fractures conchoidally.

Resin Content -- The amount of matrix present in a composite either by percent weight or percent volume.

Resin Starve Area -- Area of composite part where the resin has a non-continuous smooth coverage of the fiber.

Resin System -- A mixture of resin, with ingredients such as catalyst, initiator, diluents, etc. required for the intended processing and final product.

Roving -- A number of strands, tows, or ends collected into a parallel bundle with little or no twist.

S-Basis (or S-Value) -- The mechanical property value which is usually the specified minimum value of the appropriate government specification or SAE Aerospace Material Specification for this material.

Sample -- A small portion of a material or product intended to be representative of the whole. Statistically, a sample is the collection of measurements taken from a specified population.

Sample Mean -- The arithmetic average of the measurements in a sample. The sample mean is an estimator of the population mean.

Sample Median -- Order the observation from smallest to largest. Then the sample median is the value of the middle observation if the sample size is odd; the average of the two central observations if n is even. If the population is symmetric about its mean, the sample median is also an estimator of the population mean.

Sample Standard Deviation -- The square root of the sample variance.

Sample Variance -- The sum of the squared deviations from the sample mean, divided by n-1.

Sandwich Construction -- A structural panel concept consisting in its simplest form of two relatively thin, parallel sheets of structural material bonded to, and separated by, a relatively thick, light-weight core.

Scrim (also called Glass Cloth, Carrier) -- A low cost fabric woven into an open mesh construction, used in the processing of tape or other B-stage material to facilitate handling.

Secondary Bonding -- The joining together, by the process of adhesive bonding, of two or more already-cured composite parts, during which the only chemical or thermal reaction occurring is the curing of the adhesive itself.

Selvedge -- The woven edge portion of a fabric parallel to the warp.

Set -- The strain remaining after complete release of the force producing the deformation.

Shear Fracture (for crystalline type materials) -- A mode of fracture resulting from translation along slip planes which are preferentially oriented in the direction of the shearing stress.

Shelf Life -- The length of time a material, substance, product, or reagent can be stored under specified environmental conditions and continue to meet all applicable specification requirements and/or remain suitable for its intended function.

Significant -- Statistically, the value of a test statistic is significant if the probability of a value at least as extreme is less than or equal to a predetermined number called the significance level of the test.

Significant Digit -- Any digit that is necessary to define a value or quantity.

Size System -- See Finish.

Skewness -- See Positively Skewed, Negatively Skewed.

Slenderness Ratio -- The unsupported effective length of a uniform column divided by the least radius of gyration of the cross-sectional area.

Sliver -- A continuous strand of loosely assembled fiber that is approximately uniform in cross-sectional area and has no twist.

Solute -- The dissolved material.

Specimen -- A piece or portion of a sample or other material taken to be tested. Specimens normally are prepared to conform with the applicable test method.

Standard Deviation -- See Sample Standard Deviation.

Staple -- Either naturally occurring fibers or lengths cut from filaments.

Step-Growth Polymerization -- A chemical reaction in which polymers are formed by the stepwise intermolecular addition of molecules through reactive groups. Any two molecular species present can react. Monomers disappear early in the reaction and polymer molecular weight rises steadily throughout the reaction.

Strain -- The unit change, caused by a force, in the size or shape of a body. Strain is nondimensional but is often expressed in inches per inch of original size or shape.

Strain, Axial -- The linear strain in a plane parallel to the longitudinal axis of the specimen.

Strain, Bearing -- The ratio of the deformation of the bearing hole, in the direction of the applied force, to the pin diameter.

Strain, Initial -- The strain produced in a specimen by given loading conditions before creep occurs.

Strain, Residual -- The strain associated with residual stress.

Strain, Shear (also Angular) -- The tangent of the angular change, caused by a force, between two lines originally perpendicular to each other through a point in a body.

Strain, Transverse -- The linear strain in a plane perpendicular to the loading axis of a specimen.

Strain, True -- The natural logarithm of the ratio of gage length at the moment of observation to the original gage length for a body subjected to an axial force.

Strand -- Normally an untwisted bundle or assembly of continuous filaments used as a unit, including slivers, tow, ends, yarn, etc. Sometimes a single fiber or filament is called a strand.

Strength, Bearing -- The maximum bearing stress which can be sustained.

Strength, Compressive -- The maximum compressive stress which can be sustained. Compressive strength is calculated from the maximum load and the original cross section.

Strength, Shear -- The maximum shear stress which a material is capable of sustaining. Shear strength is calculated from the maximum load during a shear or torsion test and is based on the original cross-sectional area of the specimen.

Strength, Tensile -- The breaking load or force per unit cross-sectional area of the unstrained specimen.

Stress -- The intensity of the internal forces or components of force at a point in a body that act on a given plane through the point. Stress is expressed as force per unit area and is calculated on the basis of the original cross-sectional area for tension, compression, and shear testing.

Stress, Bearing -- The applied load divided by the bearing area.

Stress, Compressive -- The normal stress caused by forces directed toward the plane on which they act.

Stress, Fracture -- The true normal stress on the minimum cross-sectional area at the beginning of fracture.

Stress, Initial (Instantaneous) -- The stress produced by force in a specimen before stress relaxation occurs.

Stress, Nominal -- The stress at a point calculated on the net cross-section without taking into consideration the effect of geometric discontinuities such as holes, grooves, fillets, etc. on stress. (The calculation is made by simple elastic theory).

Stress, Normal -- The stress component which is perpendicular to the plane on which the forces act.

Stress, Principal (Normal) -- The maximum or minimum normal stress at a point in a plane.

Stress, Relaxed -- The initial stress minus the remaining stress at a given time during a stress relaxation test.

Stress, Remaining -- The stress remaining at a given time during a stress relaxation test.

Stress, Residual -- The stress existing in a body at rest, in equilibrium, at uniform temperature, and not subjected to external forces.

Stress, Shear -- The component of stress tangent to the plane on which the forces act.

Stress, Tensile -- The normal stress caused by forces directed away from the plane on which they act.

Stress, Torsional -- The shear stress on a transverse cross-section caused by a twisting action.

Stress, True -- The stress along the axis calculated on the actual cross-section at the time of observation instead of the original cross-sectional area. (Applicable to tension and compression testing).

Stress Relaxation -- The time dependent decrease in stress in a solid under given constraint conditions.

Stress-Strain Curve (Diagram) -- A graphical representation showing the relationship between the change in dimension of the specimen in the direction of the externally applied stress and the magnitude of the applied stress. Values of stress usually are plotted as ordinates (vertically) and strain values as abscissa (horizontally).

Surfacing Mat -- A thin mat of fine fibers used primarily to produce a smooth surface on an organic matrix composite.

Symmetrical Laminate -- A composite laminate in which the sequence of plies below the laminate midplane is a mirror image of the stacking sequence above the midplane.

Tack -- Stickiness of the prepreg.

Tape -- Prepreg fabricated in widths up to 12 inches wide for carbon and 3 inches for boron. Cross stitched carbon tapes up to 60 inches wide are available commercially in some cases.

Tenacity -- The tensile stress expressed as force per unit linear density of the unstrained specimen i.e., grams-force per denier or grams-force per tex.

Tex -- A unit for expressing linear density equal to the mass or weight in grams of 1000 meters of filament, fiber, yarn or other textile strand.

Thermoplastic -- A plastic that repeatedly can be softened by heating and hardened by cooling through a temperature range characteristic of the plastic, and when in the softened stage, can be shaped by flow into articles by molding or extrusion.

Thermoset -- A plastic that is substantially infusible and insoluble after having been cured by heat or other means.

Tolerance -- The total amount by which a quantity is allowed to vary.

Tolerance Limit -- A lower (upper) confidence limit on a specified percentile of a distribution. For example, the B-basis value is a 95% lower confidence limit on the tenth percentile of a distribution.

Tolerance Limit Factor -- The factor which is multiplied by the estimate of variability in computing the tolerance limit.

Toughness -- A measure of a material's ability to absorb work, or the actual work per unit volume or unit mass of material that is required to rupture it. Toughness is proportional to the area under the load-elongation curve from the origin to the breaking point.

Tow -- An untwisted bundle of continuous filaments. Commonly used in referring to man-made fibers, particularly carbon and graphite fibers, in the composites industry.

Transformation -- A transformation of data values is a change in the units of measurement accomplished by applying a mathematical function to all data values. For example, if the data is given by x, then $y = x + 1$, x^2, $1/x$, $\log x$, and $\cos x$ are transformations.

Transition, First Order -- A change of state associated with crystallization or melting in a polymer.

Transversely Isotropic -- Descriptive term for a material exhibiting a special case of orthotropy in which properties are identical in two orthotropic dimensions, but not the third; having identical properties in both transverse directions but not the longitudinal direction.

Twist -- The number of turns about its axis per unit of length in a yarn or other textile strand. It may be expressed as turns per inch (tpi).

Twist, Direction of -- The direction of twist in yarns and other textile strands is indicated by the capital letters S and Z. Yarn has S twist if when held in a vertical position the visible spirals or helices around its central axis are in the direction of slope of the central portion of the letter S and Z twist is in the other direction.

Typical Basis -- A typical property value is a sample mean. Note that the typical value is defined as the simple arithmetic mean which has a statistical connotation of 50% reliability with a 50% confidence.

Unbond -- An area within a bonded interface between two adherends in which the intended bonding action failed to take place. Also used to denote specific areas deliberately prevented from bonding in order to simulate a defective bond, such as in the generation of quality standards specimens. (See Disbond, Debond).

Unidirectional Laminate -- A laminate with nonwoven reinforcements and all layers layed up in the same direction.

Upper Confidence Limit -- See Confidence Interval.

Vacuum Bag Molding -- A process in which the lay-up is cured under pressure generated by drawing a vacuum in the space between the lay-up and a flexible sheet placed over it and sealed at the edges.

Variance -- See Sample Variance.

Viscosity -- The property of resistance to flow exhibited within the body of a material.

Void -- A physical and mechanical discontinuity occurring within a material or part which may be two-dimensional (e.g., disbonds, delaminations) or three-dimensional (e.g., vacuum-, air-, or gas-filled pockets). Porosity is an aggregation of micro-voids. Voids are essentially incapable of transmitting structural stresses or nonradiative energy fields. (See Inclusion).

Warp -- The longitudinally oriented yarn in a woven fabric (see Fill); a group of yarns in long lengths and approximately parallel.

Weibull Distribution -- A probability distribution for which the probability that a randomly selected observation from this population lies between a and b ($0 < a < b < \infty$) is given by

$$e^{-(a/\alpha)^{\beta}} - e^{-(b/\alpha)^{\beta}} \qquad 1.7(d)$$

where α is called the scale parameter and β is called the shape parameter.

Wet Lay-up -- A method of making a reinforced product by applying a liquid resin system while the reinforcement is put in place.

Wet Strength -- The strength of an organic matrix composite when the matrix resin is saturated with absorbed moisture. (Saturation is an equilibrium condition in which the net rate of absorption under prescribed conditions falls essentially to zero).

Wet Winding -- A method of filament winding in which the fiber reinforcement is coated with the resin system as a liquid just prior to wrapping on a mandrel.

Whisker -- A short single crystal fiber or filament. Whisker diameters range from 1 to 25 microns, with aspect ratios between 100 and 15,000.

Work Life -- The period during which a compound, after mixing with a catalyst, solvent, or other compounding ingredient, remains suitable for its intended use.

Woven Fabric Composite -- A major form of advanced composites in which the fiber constituent consists of woven fabric. A woven fabric composite normally is a laminate comprised of a number of laminae, each of which consists of one layer of fabric imbedded in the selected matrix material. Individual fabric laminae are directionally oriented and combined into specific multiaxial laminates for application to specific envelopes of strength and stiffness requirements.

Yarn -- A generic term for strands or bundles of continuous filaments or fibers, usually twisted and suitable for making textile fabric.

Yarn, Plied -- Yarns made by collecting two or more single yarns together. Normally, the yarns are twisted together though sometimes they are collected without twist.

Yield Point -- The first stress in a material at which an increase in strain occurs without an increase in stress. (The stress is less than the maximum attainable). It should be noted that only materials that exhibit the unique phenomenon of yielding have a "yield point".

Yield Strength -- The stress at which a material exhibits a specified limiting deviation from the proportionality of stress to strain. (The deviation is expressed in terms of strain such as 0.2 percent for the Offset Method or 0.5 percent for the Total Extension Under Load Method).

X-Axis -- In composite laminates, an axis in the plane of the laminate which is used as the 0 degree reference for designating the angle of a lamina.

X-Y Plane -- In composite laminates, the reference plane parallel to the plane of the laminate.

Y-Axis -- In composite laminates, the axis in the plane of the laminate which is perpendicular to the x-axis.

Z-Axis -- In composite laminates, the reference axis normal to the plane of the laminate.

REFERENCES

1.6(a) Military Standardization Handbook, "Metallic Materials and Elements for Aerospace Vehicle Structures", MIL-HDBK-5D, Change Notice 2, (May, 1985).

1.6(b) DOD/NASA Advanced Composites Design Guide, Air Force Wright Aeronautical Laboratories, Dayton, OH, prepared by Rockwell International Corporation (1983) (distribution limited).

1.6(c) ASTM E206, "Definitions of Terms Relating to Fatigue Testing and the Statistical Analysis of Fatigue Data," 1984 Annual Book of ASTM Standards, 3.01, ASTM, Philadelphia, PA (1984).

2. OBJECTIVES IN GENERATING PROPERTY DATA

2.1 Introduction.

2.2 Recommendations for the generation of physical and mechanical properties.

2.2.1 General guidelines. Generation of design allowables for advanced composite materials requires physical and chemical property characterization of the prepreg material as well as physical and mechanical property tests on cured lamina and laminates. These guidelines contain recommendations for the number and type of tests sufficient to establish B-basis mechanical properties for advanced composite unidirectional tape and woven fabric materials. Recommendations are also provided for vendor acceptance tests by the manufacturer of the prepreg materials.

These guidelines present the methods recommended by MIL-HDBK-17 for generating statistically-based material properties for lamina coupons. Other methods involving statistical analysis of mechanical property tests on oriented laminates are equally acceptable when agreed to by the contractor and certifying agency.

2.2.2 Environmental sensitivity.

2.2.2.1 Moisture effects. Organic matrix composites absorb small amounts of moisture. The absorbed water causes a dimensional change (swelling), lowers the glass transition temperature of the resin, and results in a reduction of the matrix and matrix/fiber interface dependent mechanical properties of the composite, particularly at elevated temperatures. Except for the aramid fiber composites, it is usually assumed that the moisture absorption is limited to the organic matrix.

The degradation of composite properties as a result of exposure to high temperatures is nonlinear. A drastic reduction in properties occurs at a characteristic temperature level, defined here as the material operational limit (MOL). For composites, this behavior is more complicated because the introduction of moisture will result in more than one critical temperature, as illustrated in Figure 2.2.2.1(a). The various moisture contents are represented schematically by saturation at different relative humidity levels. There are no standard criteria for the determination of the MOL. One method (References 2.2.2.1(b) - (d)), illustrated in Figure 2.2.2.1(b), utilizes the glass transition temperature (T_g) with some safety factor K as the basis for the MOL. For epoxy composites, 50°F (10°C) has been proposed for the value of the safety factor. If service usage at high temperatures is limited to occasional excursions, it can be argued that lower safety factors are acceptable.

The elevated temperature wet testing required by MIL-HDBK-17 should be at the MOL temperature, corresponding to the highest practical relative humidity. For aircraft, the worst-case relative humidity is 85%. As can be seen from Figure 2.2.2.1(a), the effect of moisture is small for matrix-dependent properties at temperatures below room temperature (RT). However, if Figure 2.2.2.1(a) were produced for the fiber dependent properties, degradation in properties would be observed below room temperature. Therefore, MIL-HDBK-17 requires testing at cold temperatures. Since the changes in properties at these temperatures are not drastic, the lowest service temperature will suffice and there is no need to determine a MOL.

The equilibrium level of weight gain due to moisture absorption is generally considered to be a function of relative humidity (RH) only. The relationship is not necessarily linear as shown by the test data fitted curves for epoxy composites in Figure 2.2.2.1(c). The rate of moisture absorption is very strongly dependent on temperature as shown in Figure 2.2.2.1(d); a decrease of 60°F (16°C) increases the time to absorb 1% moisture by 5 times. The relationship between the diffusion coefficient and absolute temperature is of the Arrhenius form

$$D = D_o \exp(-E_d/RT) \qquad 2.2.2.1(a)$$

where D_o and E_d are material constants and R is the universal gas constant. The moisture absorption and desorption is mathematically modeled using a one-dimensional Fickian equation

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2} \qquad 2.2.2.1(b)$$

where c is moisture concentration. The boundary conditions are:

$$c = c_i \text{ at } t = 0$$

$$c = c_a \text{ at } t >> 0$$

and x is the thickness parameter varying between 0 and h. This equation assumes that the moisture diffusion and temperature are constant inside the material. Because the rate of temperature diffusion is about 10^6 times faster than the moisture diffusion, and because diffusivity changes very little with moisture content, these assumptions are quite valid. If a further simplification of constant boundary conditions with time is made, solution to Equation 2.2.2.1(b) can be approximated (Reference 2.2.2.1(a)) as

$$M_{avg} = G(M_m - M_i) + M_i \qquad 2.2.2.1(c)$$

where

M_{avg} = moisture content of slab in percent by weight

M_m = moisture content at equilibrium for the boundary relative humidity

M_i = initial uniform moisture content (assumed to be zero)

G = a time-dependent parameter defined below

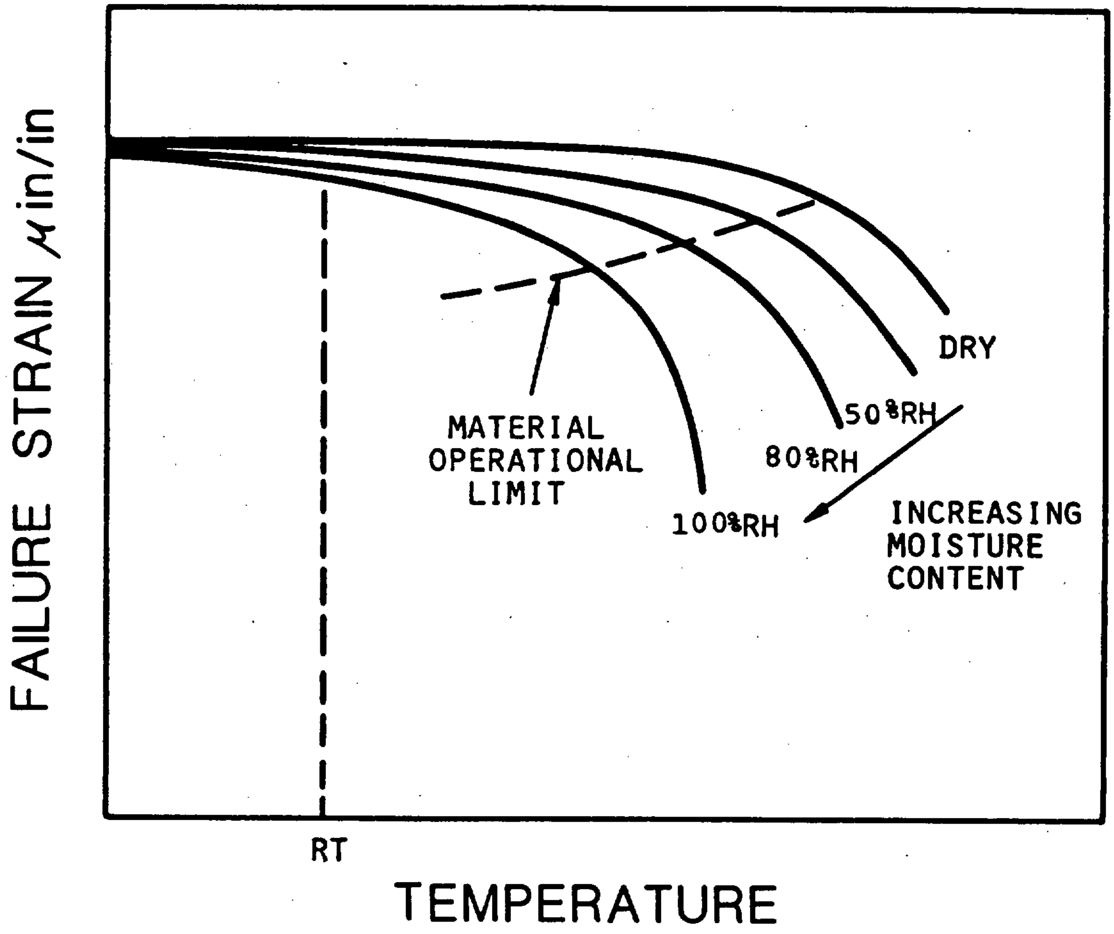
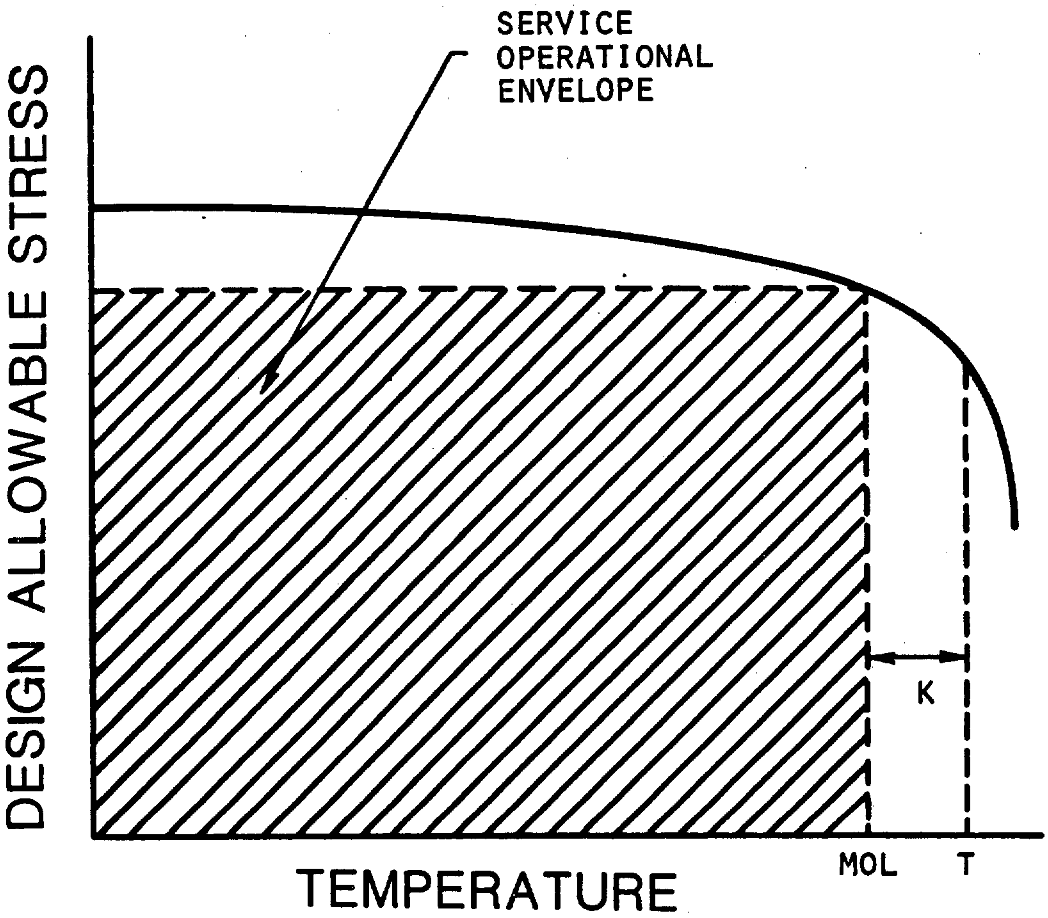


FIGURE 2.2.2.1(a) Influence of temperature and moisture on matrix dependent failure strain.



T = GLASS TRANSITION TEMPERATURE

K = MOL SAFETY FACTOR

FIGURE 2.2.2.1(b) Material selection criterion.

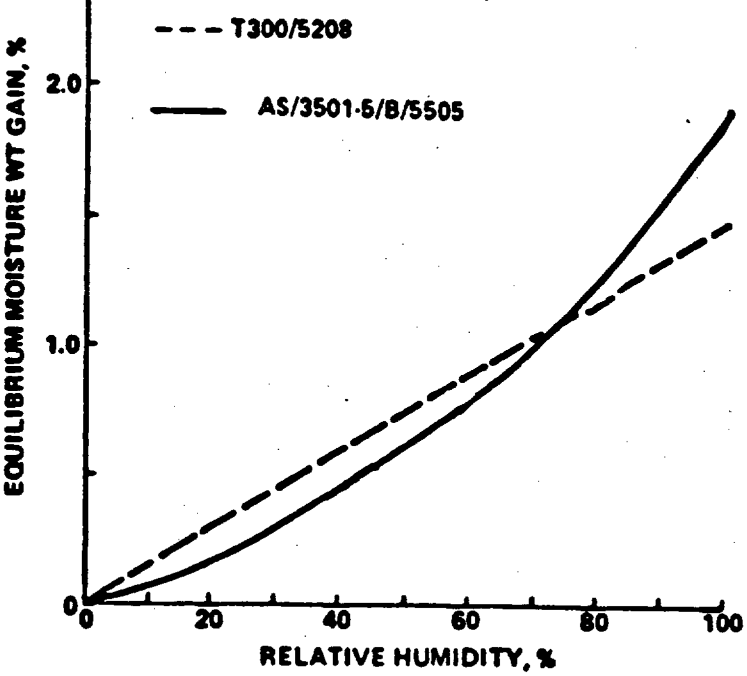


FIGURE 2.2.2.1(c) Maximum moisture content as a function of relative humidity (Reference 2.2.2.1(b)).

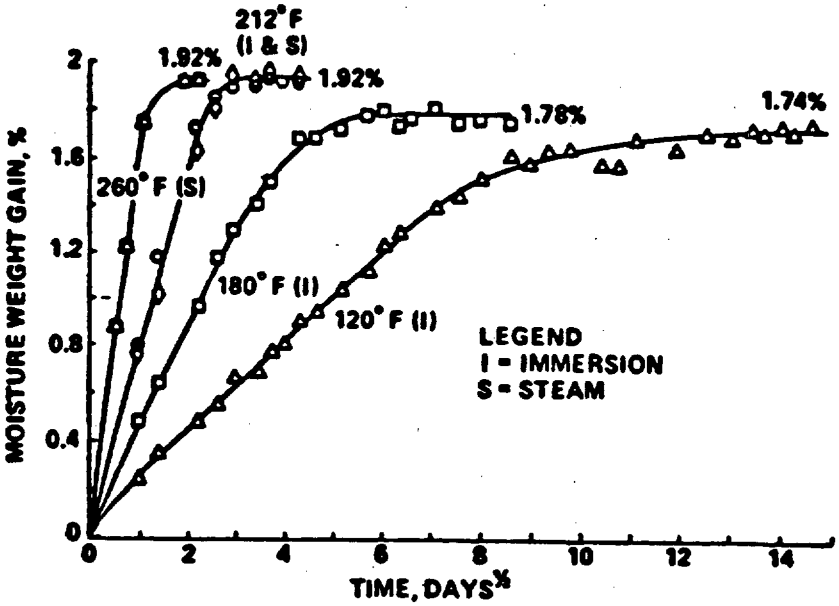


FIGURE 2.2.2.1(d) Effect of temperature on moisture absorption rate in hybrid boron-graphite/epoxy (5505/AS-3501) laminate (3.0 x 0.5 x 0.12 in. 76 x 13 x 3.0 mm) (Reference 2.2.2.1(b)).

The term G is a series solution parameter which can be adequately evaluated over the range of interest by the following approximation given in Reference 2.2.2.1(a).

$$G = 1 - \exp\left[-7.300\left(\frac{D_o t}{h^2}\right)^{0.75}\right] \qquad 2.2.2.1(d)$$

where

h = slab thickness exposed on both sides to the same environment

t = exposure time

M_m can be obtained from Figure 2.2.2.1(c).

For time variable boundary conditions, the finite difference method (Reference 2.2.2.1(c)) can be used to numerically integrate Equation 2.2.2.1(b) with respect to time.

In the design of a composite structure, the effects of moisture are accounted for in the design allowables. The end-of-life moisture content (usually defined as the design service moisture content) is either calculated using Equation 2.2.2.1(b) or established by previous experimental or flight data. After the end-of-life moisture content is determined, mechanical property tests are performed with specimens preconditioned to that moisture level. The conditioning procedure to be performed is described in the next section.

Before end-of-life moisture contents can be calculated, environmental definition of component usage must be determined. An example is illustrated in Reference 2.2.2.1(d), where worldwide weather data and USAF aircraft basing data were gathered to define runway storage environmental spectra for each of the three classes of Air Force vehicles: fighters, bombers, and cargo/tankers. A ranking procedure was developed and applied to select baseline and worst-case locations with respect to the absorption of moisture by carbon/epoxy composite structures. A guide to environmental exposure parameters is provided in MIL-STD-810D.

2.2.2.2 Conditioning of samples. To obtain test data for mechanical properties of wet material, specimens must be environmentally conditioned prior to mechanical testing. The goal for this procedure is a uniform moisture content throughout the thickness of the specimen. Uniformity in moisture content is achieved when the specimens reach an equilibrium moisture content. This moisture content is different at different humidity levels but is not, in general, a function of temperature, if the conditioning temperature is below the glass transition temperature of the material. The humidity level that is chosen depends on the application, the extreme being water immersion (100% RH). An 85% exposure would be more representative of worst usage conditions for aircraft based in the tropics. Because the moisture absorption rate is so strongly dependent on temperature, there is a tendency to increase conditioning temperatures to save time. However, long exposures to high temperatures combined with moisture are deleterious. For epoxy matrices, the conditioning temperature should be kept below 170°F (77°C) for a 350°F (177°C) cure material and below 155°F (68°C) for a 250°F (121°C) cure material.

In addition to specifying conditioning temperature and RH, time of exposure has to be specified. The best way to specify conditioning is to require specimens to reach equilibrium, i.e. the maximum moisture content (MC) by weight that can be achieved at the given humidity level. A shortcut prevalent in the industry is the specification of a time period, e.g. thirty days. The drawbacks of this approach are illustrated in Figure 2.2.2.2 where, using the absorption rate and equilibrium level for IM6/3501-6 carbon/epoxy, the maximum moisture content has been calculated for various composite laminate thicknesses exposed for 30 days at 95% relative humidity and 140°F. The required 1.2% MC (equilibrium at 78% RH) by weight can be obtained only if the laminate thickness is approximately 0.07 in. (1.8 mm). For thicknesses larger than 0.07 in. (1.8 mm), the MC will be lower than desired and, for thicknesses smaller than 0.07 in. (1.8 mm), the MC will be higher than required. Furthermore, the moisture distribution across the thickness will be non-uniform for all thicknesses greater than 0.035 in. (0.9 mm). Because a typical test matrix includes specimens with different thicknesses, different exposure times should be specified. Figure 2.2.2.2 was generated for a known material; such information may not be readily available for a new material system. Specifying final MC instead of time of exposure implies knowledge of the material properties at the resin and void content of the laminate.

Two-step conditioning schemes consisting of 95% RH exposure followed by a lower RH exposure can also be used. These save time and are perfectly acceptable if the last step is exposure at the required RH until equilibrium.

Moisture content measurements are taken by weighing either the actual specimens or travellers that have been cut from the same panel and conditioned at the same time as the specimens. To minimize ingress of moisture through the edges, the thickness of the travellers must be small in comparison to the length and width dimensions; a ratio of 1:10 is usually adequate. For greater thicknesses, the edges may have to be sealed. Because the weight gain is approximately 1%, measurements must be taken on a fine balance to an accuracy of 0.0001 g. If the specimens have fiberglass tabs, adhesive materials, etc., travellers must be used, unless the saturation moisture content is specified, and even in this case the specimen may be too large or heavy to be conveniently weighed.

Both specimens and travellers should be dried in an oven at temperatures up to 250°F (121°C) for 350°F (177°C) cure materials and 200°F (93°C) for 250°F (121°C) cure materials prior to any additional conditioning. Since the time from cure to completion of specimen fabrication may be long, a required time of drying cannot be specified. Periodic readings should be taken and, when three consecutive readings are within 0.01% weight change, the travellers and specimens are considered to be dry. The dry weight should be obtained at this time. The specimens and travellers should than be stored in plastic bags with a desiccator. From this time on, the travellers must accompany the specimens throughout the conditioning history. For travellers and specimens that are conditioned until equilibrium, equilibrium is defined as the point when three consecutive weighings give weight changes that average less than 1% of the total increase in weight. ASTM D570-81 "Standard Test Method for Water Absorption of Plastics" and ASTM D618-61 "Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing" provide guidance for test procedures (References 2.2.2.2(a) and (b)).

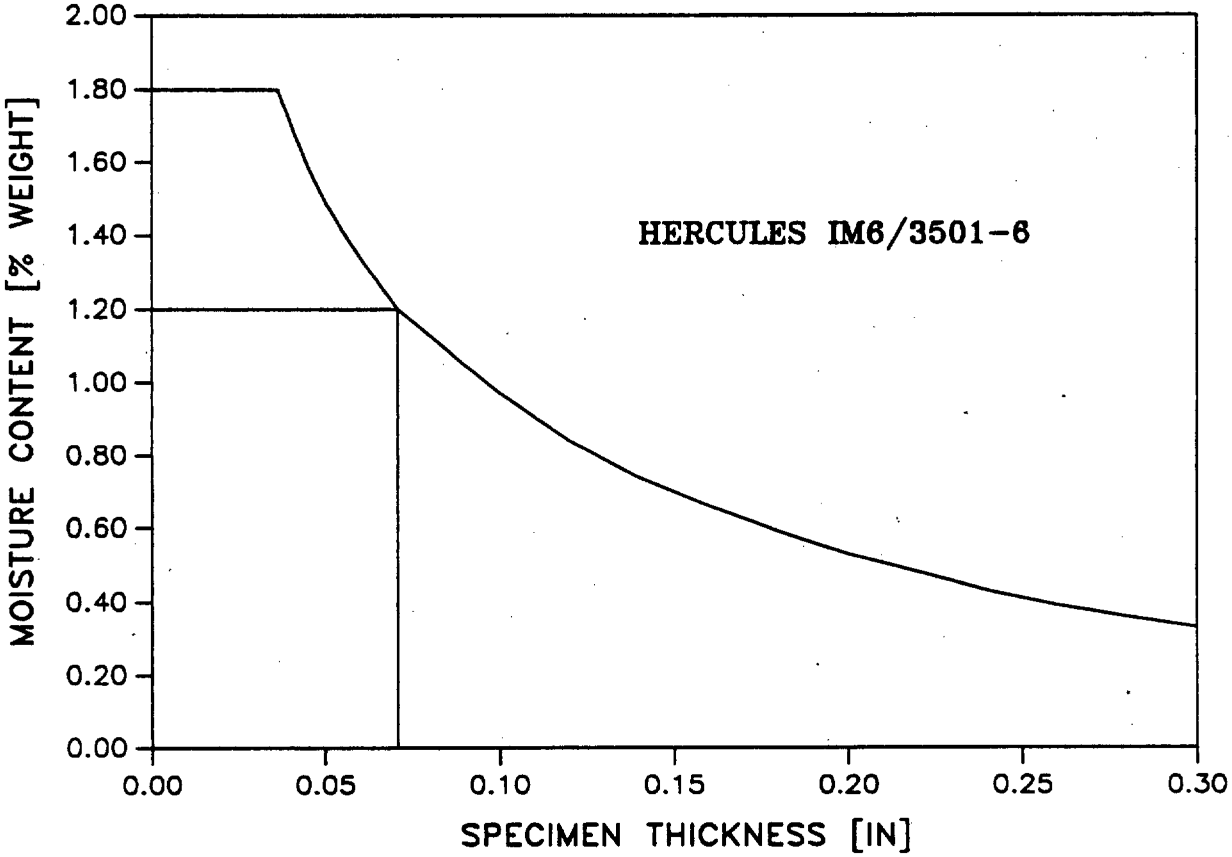


FIGURE 2.2.2.2 <u>Moisture absorption after 30 days at 95% RH and 140°F (60°C) for carbon/epoxy laminates with both sides exposed</u>.

The specimens or travellers should also be weighed just before and after mechanical testing, especially for elevated temperatures. At these temperatures, it also important not to soak the specimens longer than needed so as not to drive-out moisture. A typical soak time after the thermocouples on the specimens reach the desired temperature is half a minute for carbon/epoxy for temperatures up to 350°F (177°C).

2.2.3 Material acquisition and prepreg physical property characterization. For material property characterization suitable for inclusion in MIL-HDBK-17, five prepreg batches of each material type shall be prepared by the material supplier using prepreg production facilities. The five prepreg batches shall be made with five different fiber lots impregnated with five different production resin batches. The contractor shall prepare and coordinate a preliminary material specification with the supplier prior to manufacture of the prepreg batches. The preliminary material specification shall define agreed-to laminate and specimen fabrication procedures, test procedures, and physical and mechanical property requirements.

The five-batch requirement only applies to material properties that are to be incorporated in MIL-HDBK-17. An alternate number of tests and batches may be employed upon approval of the certifying agency. However, mechanical property test data should be examined by the recommended statistical methods in this document (MIL-HDBK-17) to ascertain that they are statistically acceptable.

Recommended tests to be performed on the prepreg materials are shown in Table 2.2.3. When applicable, the tests should be standard ASTM procedures.

2.2.4 Lamina physical and mechanical property tests. A minimum of two test panels for each prepreg batch shall be cured in two separate autoclave runs to be used in fabricating test coupons. The prepreg material shall be layed up, bagged, and cured in accordance with a process specification prepared by the contractor. Cured test panels shall be non-destructively evaluated (NDE) using ultrasonic inspection. Imperfections in the test panels shall be reported with questionable areas marked on the panels. Test coupons shall not be extracted from these areas.

Suggested physical and mechanical property tests are shown in Tables 2.2.4(a) and 2.2.4(b). A test procedures document should be prepared by the contractor prior to the start of testing. It is recommended that, whenever possible, the tests should be ASTM standard methods.

The test matrix shown in Table 2.2.4(b) is based on thirty tests per data point (six tests per batch) to provide for parametric/non-parametric analysis in determining B-basis properties. Fewer tests per data point may be acceptable for non-critical properties as agreed to by the prime contractor and the certifying agency. For generating B-basis properties, 0° and 90° tension, 0° and 90° compression, and in-plane shear tests are required. Short beam shear tests are required for determining process control test minimum values.

Extensometers or strain gages should be used to obtain strain measurements for each specimen. Two of each group of six compression specimens shall be strain gaged back-to-back with axial gages to assess possible premature buckling failure.

TABLE 2.2.3 - Recommended physical and chemical property tests to be performed by material supplier and prime contractor.

Test Property	Suggested Test Procedure*	Number of Tests per Batch**	Total Number of Tests
Resin Content, %	D3529	3	15
Volatile Content, %	D3530	3	15
Gel Time, min.	D3532	3	15
Resin Flow, %	D3531	3	15
Fiber Areal Wt., gm/M^2	†	3	15
Moisture Content, %	†	3	15
Tack	†	3	15
HPLC (High Performance Liquid Chromatography)	†	3	15
IR (Infrared Spectroscopy)	†	3	15
DMA (Dynamic Mechanical Analysis, neat resin only)	†	3	15
DSC (Differential Scanning Calorimetry)	†	3	15
RDS (Rheological Dynamic Spectroscopy)	†	3	15

* Test procedures shall be coordinated and agreed to prior to manufacture of prepreg material.

** Tests shall be performed on each of the five batches of prepreg material.

† Test procedures to be described at a later date.

TABLE 2.2.4(a) - Cured lamina physical property tests.

Physical Property	Suggested Test Procedure	Number of Tests Per Prepreg Batch*	Total Number of Tests
Fiber Volume,%	D3171	3	15
Resin Volume,%	D3171	3	15
Density, gm/cc	D792	3	15
Measurements of per ply thickness	-	10	50
Glass Transition Temperature, °F (dry)†	-	3	15
Glass Transition Temperature, °F (Wet)†	-	3	15

* Tests shall be performed on each of the five batches.

† Dry specimens are "as fabricated" specimens which have been maintained at ambient conditions in an environmentally-controlled test laboratory. Wet specimens are environmentally controlled by drying at 140°F (60°C) to constant weight, exposing them in a humidity chamber until they attain an equilibrium moisture content agreed to by the contractor and customer, then packaged in a heat-sealed aluminized polyethylene bag until required for test. Tests shall be performed in a manner which maintains the moisture content in specimens at the levels agreed to by the contractor and certifying agency.

TABLE 2.2.4(b) - Cured lamina mechanical property tests.

Mechanical Property	Suggested Test Procedure	Test Condition and Number of Tests Per Batch*			Total Number of Tests
		Min. Temp Dry**	RT Dry	Max. Temp Wet**	
0° Tension (warp)	D3039	6	6	6	90
90° Tension (fill)	D3039	6	6	6	90
0° Compression (warp)	D3410-Tape † Fabric	6	6	6	90
90° Compression (fill)	†	6	6	6	90
In-plane Shear	†	6	6	6	90
0° Short Beam Shear	D2344	-	6	-	30
					480

* Tests shall be performed on each of the five batches.

Dry specimens are "as fabricated" specimens which have been maintained at ambient conditions in an environmentally-controlled test laboratory. Wet specimens are environmentally-controlled by drying at 140 °F (60 °C) to constant weight, exposing them in a humidity chamber until they attain an equilibrium moisture content agreed to by the contractor and certifying agency, then packaged in a heat-sealed aluminized polyethylene bag until required for test. Tests shall be performed in a manner which maintains the moisture content in specimens at the levels agreed to by the contractor and certifying agency.

** Minimum and maximum temperature tests shall be performed within ±5 °F (±2.8 °C) of the nominal test temperature. Nominal test temperatures will be as agreed to by contractor and certifying agency.

† Test procedures for these properties are presently being reviewed by MIL-HDBK-17 committee.

2.2.5 Statistical development of mechanical properties. Data interpretation and statistical analysis procedures are defined in Chapter 8.

2.2.6 Data pooling requirements. The capability of pooling data may be desirable in order to obtain sufficient data to calculate B-basis values for material properties. Data to be pooled may be available for materials from different composite component manufacturers, different locations of one manufacturer, or slightly different processes from the same manufacturer. Data for the same material system and equivalent processing procedures may be pooled if the statistical batch-to-batch variation test criteria of Section 8.5.3.2 are satisfied. These criteria should be satisfied for all properties measured in order to pool data for any of the properties. For example, if tensile data meet the batch-to-batch variation criteria but shear data for the same material do not meet these criteria, shear data and tensile data should not be pooled.

2.3 Other useful test matrices.

2.3.1 Material system screening. The objective of this screening process is to reveal key mechanical property attributes and/or inadequacies in new material system candidates through use of a reduced test matrix. The screening process will identify the critical test and environmental conditions, and any special considerations for a particular composite material system. Proper test selection and environmental conditioning will enable comparisons with current productions systems.

Table 2.3.1 shows a typical mechanical property screening test matrix primarily intended for epoxy resin systems. The test matrix will vary depending on the purpose of the investigation. The 0° axial tensile tests examine fiber dominant properties, while 0° axial compression tests monitor matrix/fiber interactions. These tests will yield static strength and stiffness properties. To test matrix characteristics, ±45° tensile tests will be used. The ±45° tensile tests permit actual shear modulus and effective shear strength values to be obtained. Finally, damage tolerance will be evaluated using impact testing.

Testing will be conducted under three environmental conditions: cold temperature dry (CTD), room temperature ambient (RTA), and elevated temperature wet (ETW). These conditions are selected based on results for the epoxy resin systems currently in use which show that the CTD condition is a fiber critical environmental state and the ETW condition is the most severe test for the matrix. Moisture conditioning may be accomplished by placing the dried test specimens in an environmental chamber until an equilibrium moisture weight gain at the desired relative humidity is obtained.

The screening process presented allows key mechanical properties and environmental conditions to be evaluated. Dependent upon application, sensitivity to exposures to operational fluids and other special issues, additional tests may also be included in the screening evaluation. However, a reduced test matrix for mean property evaluations should permit preliminary efficient and economical assessment of new composite material systems.

The approach to screening is the performance of key static tests to provide sufficient data for mean property evaluations of stiffness and strength. The recommended specimens provide data at the lamina (ply) and laminate (application) levels. The lamina level tests provide key generic material stiffness and strength properties commonly used in classical lamination plate point stress analysis including tension, compression, and shear loadings. The laminate level tests provide screening strength data on application issues relating to effects of stress concentrations, manufactured stress risers (fastener holes), bolt bearing, or impact damage. Additional laminate level tests provide screening stiffness data to verify the use of lamina data with classical lamination plate theory for laminate stiffness predictions. The damage tolerance screening is patterned after NASA Reference Publication 1092 (Reference 2.3.1).

All laminate level tests suggested are performed at the RTD test condition. For screening purposes, it is assumed the lamina (ply) level unnotched specimens will conservatively provide mean data to assess environmental effects on strength and stiffness. The laminate level tests will provide mean data on the effects of stress concentrations.

2.3.2 Requirements for alternate material suppliers.

Table 2.3.1 Composite material static strength screening test matrix.

Test	Number of specimens			Evaluation Emphasis
	CTD	RTA	ETW	
Lamina				
0° Tension	3	3	3	fiber
0° Compression		3	3	fiber/matrix
±45° Tension		3		fiber/matrix (0°/90° shear - lamina) (±45° - laminate)
0° Compression Modulus		3	3	fiber/matrix
Laminate:				
Open Hole Compression*		3		stress riser
Open Hole Tension*		3		stress riser
Bolt-Bearing*		3		bearing
Compression after Impact†		3		impact damage

* Fastener hole effects
† per NASA Reference Publication 1092.

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3. EVALUATION OF REINFORCEMENT FIBERS.

4. Matrix Characterization

5. PREPREG MATERIAL CHARACTERIZATION

Page

5.1 Introduction. The processability and properties of high performance composites depend upon the composition of the fiber/resin preimpregnated materials (prepregs) from which they are manufactured. In general, prepregs consist of "modified" or surface-treated glass, graphite, or aramid fibers impregnated with 28-60 weight-percent of a reactive and chemically-complex thermoset resin formulation or a thermoplastic resin. A typical thermoset resin formulation may contain, for example, several different types of epoxy resins, curing agents, diluents, rubber modifiers, thermoplastic additives, accelerators or catalysts, residual solvents, and inorganic materials, plus various impurities and synthetic by-products. Furthermore, such resins are often "staged" or partially reacted during the prepregging process and may undergo compositional changes during transport, handling, and storage. Although less likely to undergo compositional changes, polymer molecular weight (MW), molecular weight distribution (MWD), and crystalline morphology have major effects on the processability and properties of thermoplastic prepregs and composites. Inadvertent or minor changes in resin composition may cause problems in processing and have deleterious effects on the performance and long-term properties of composites.

Modern analytical techniques and detailed knowledge relating to fibers, fiber surface treatments, and resin types and formulations are needed to characterize prepregs and composite materials. Characterization involves the identification and quantification of the fiber, fiber surface, and major resin components and should include information about the presence of impurities or contaminants. For thermoset resins and composites, characterization should include a description of the nature and extent of the prepreg resin reaction and the thermal/rheological and thermal/mechanical behavior. In the case of thermoplastics, the polymer molecular weight distribution, crystallinity, and time/temperature viscosity profile should also be analyzed. However, few laboratories are equipped or have the knowledgeable technical personnel to characterize prepregs and composites completely, and few studies have been published describing how variations in fiber type and resin chemistry/morphology affect the physical properties and long-term performance of composites. Also, until recently, prepreg compositions were considered proprietary, processing conditions were only recommended, and acceptance was based primarily upon mechanical testing of fabricated specimens. The purpose of this chapter is to provide an overview of characterization techniques and, more specifically, to address the application of state-of-the-art techniques for the chemical and physical characterization of resins and prepreg materials used in the manufacture of high performance organic matrix composites.

5.2 Characterization techniques - overview. According to a recent survey (Reference 5.2), the most widely utilized techniques for the characterization and quality assurance of composite material precursors are -

1. High Performance Liquid Chromatography (HPLC)
2. Infrared (IR) Spectroscopy
3. Thermal Analysis
4. Rheological Analysis

HPLC and IR spectroscopy provide the capability for rapid screening and quality control fingerprinting of individual resin constituents as well as of the prepreg resin and, therefore, may be used advantageously by both the prepregger and composite manufacturer. Thermal analytical techniques, such as thermal gravimetrical analysis (TGA), differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermal mechanical analysis (TMA), dynamic mechanical analysis (DMA), and torsional braid analysis (TBA) are not strictly chemical analysis techniques; however, they provide useful information relating to the composition and processability of resins. Similarly, rheological and dielectric techniques are used frequently to evaluate the chemoviscosity properties of thermoset resins during cure, and there is increasing interest in applying such techniques for process monitoring and process control of both thermoset and thermoplastic resins.

5.2.1 High performance liquid chromatography. High performance liquid chromatography (HPLC) is a versatile and economically viable quality assurance technique for soluble resin materials (References 5.2.1(a) - 5.2.1(g)). HPLC involves the liquid-phase separation and monitoring of separated resin components. Dilute solutions of resin samples are prepared and injected into a liquid mobile phase which is pumped through column(s) packed with a stationary phase to facilitate separation and then into a detector. The detector monitors concentrations of the separated components; its signal response, recorded as a function of time after injection, provides a "fingerprint" of the sample's chemical composition. Quantitative information may be obtained if the sample components are known and sufficiently well-resolved, and if standards for the components are available. Size exclusion chromatography (SEC), an HPLC technique, is particularly useful in determining the average molecular weights and molecular weight distributions of thermoplastic resins (Reference 5.2.1(g)). Recent advances have resulted in improved and automated HPLC instrumentation that is relatively low cost and simple to operate and maintain.

5.2.2 Infrared spectroscopy. Infrared (IR) spectroscopy is sensitive to changes in the dipole moments of vibrating groups in molecules and, accordingly, yields useful information for the identification of resin components. IR spectroscopy provides a fingerprint of the resin composition and is not limited by the solubility of resin components (References 5.2.2(a) - 5.2.2(c)). Indeed, gases, liquids and solids may be analyzed by IR spectroscopy. Advances in technology have led to the development of Fourier transform infrared spectroscopy (FTIR) - a computer-supported IR technique for rapidly scanning and storing infrared spectra. Multiple scans and Fourier transformation of the infrared spectra enhance the signal-to-noise ratio and provide improved spectra for interpretation. In addition, the FTIR attenuated total reflection (ATR) technique may be applied for quality assurance of thermoset composite materials to assess their state of cure; i.e., residual epoxide concentration.

Other chromatographic and spectroscopic techniques have also been considered (References 5.2.2(a), 5.2.2(d) - 5.2.2(h)). Gas chromatography (GC), GC headspace analysis, and GC-mass spectroscopy are useful for analyzing residual solvents and some of the more volatile resin components. Combined thermal analysis - GC-mass spectroscopy can be used to identify volatile reaction products during cure (References 5.2.2(i) and 5.2.2(j)). Elemental analysis, titration, and wet chemical analysis for specific functional groups are useful techniques for characterizing individual epoxy components but have limited application and may provide misleading results when complex resin formulations are analyzed. When necessary, ion chromatography, atomic absorption (AA), x-ray fluorescence, or emission spectroscopy are applied to analyze specific elements, such as boron or fluorine. X-ray diffraction may be used to identify crystalline components, such as fillers, and determine the relative percent crystallinity for certain resins.

5.2.3 Thermal analysis. Thermal gravimetric analysis (TGA) monitors the weight changes in a sample as a function of temperature. Although primarily used for studying the degradation processes, TGA can also be applied as a quality assurance technique to provide information about the volatiles, resin, fiber, and inorganic residue content of prepreg materials Reference (5.2.3(a)). Since dissimilar materials often degrade and volatilize at different temperature and rates, compositional differences may be reflected by differences in their TGA thermograms. Thermal oxidative degradation rates determined by TGA are useful for estimating the life cycles of resin materials (Reference 5.2.3(b)).

Differential scanning calorimetry (DSC) and differential thermal analysis (DTA) techniques are frequently employed for characterizing resins and composite materials (References 5.2.1(g), 5.2.2(a), 5.2.3(c), and 5.2.3(d)). Both DSC and DTA monitor enthalpy changes in materials as a function of temperature (DSC directly and DTA indirectly) and thereby provide similar information useful for quality assurance of prepreg materials. DTA measures the temperature difference (ΔT) between the epoxy resin specimen and a reference material; whereas DSC measures the rate of heat evolution (dH/dt) or enthalpy absorption of the specimen relative to a reference. DTA and DSC measure thermal changes (1) as a function of time with both the specimen and reference material held at the same temperature (isothermal), or (2) as a function of temperature with both the specimen and reference material heated at the same heating rate (dynamic). For quality assurance applications, DTA and DSC are usually run in the dynamic mode with the weighed specimen in an aluminum specimen holder and an empty holder used as the reference. Dynamic DTA and DSC measure the glass transition temperature T_g and heat of reaction ΔH of the prepreg resin but do not directly provide information about chemical composition. By monitoring the fraction of heat evolved as a function of temperature or time, information relating to the extent of cure and curing kinetics can be obtained. DSC and DTA may also be applied to evaluate the melting temperature T_m and to estimate the degree of crystallinity of thermoplastic resins and composites. Since the average specimen size used in DSC is only about 10 mg (0.00002 lb), special care must be taken in obtaining representative materials. Multiple specimens runs are advisable.

Thermal mechanical analysis (TMA) is used in conjunction with DTA and DSC to study the thermal transition behavior (e.g., T_g) of prepreg resins and cured laminates. TMA simulates a linear dilatometer to measure the thermal expansion and contraction of specimens under dynamic or isothermal heating conditions. Adjustable loads are applied via a specially designed probe resting upon the specimen surface. Sensitive displacement devices are employed to monitor the "nominal" thermal response of a material. Since thermal transition behavior is related to the chemical composition and extent of cure of a prepreg resin, TMA can be applied as a quality assurance technique.

5.2.4 Rheological analysis. Dynamic mechanical analysis (DMA), torsional braid analysis (TBA), and various mechanical spectrometers measure the rheological response of resins and composites as a function of frequency, temperature and/or state of cure. Both DMA and TBA provide information relating to the storage modulus (E') and the loss modulus (E") of the resin during cure and to the T_g of the cured resin (References 5.2.3(c), 5.2.4(a) - 5.2.4(C)). The dynamic complex viscosity (η*) and tan delta parameters can also be determined from rheological measurements of thermoset resins during cure. Rheological techniques are most often used to optimize processing parameters and evaluate the onset of gelation and vitrification during cure. However, since rheological properties are related to resin composition and morphology, rheological techniques may also be applied for the quality assurance of prepregs. Rheological data are often less reproducible when taken from fiber reinforced samples. Whenever possible, neat resin or extruded film samples of the resin lot should be procured along with the prepreg for rheological testing.

Dynamic dielectric analysis (DDA) techniques involve the use of electrical measurements to monitor changes in the dielectric constant, the dissipation factor, capacitance, and/or conductance of the prepreg resin during processing as a function of frequency, time, and temperature. Measured electrical parameters are highly responsive to changes in resin viscosity and, similar to the rheological techniques, are often employed to investigate and optimize prepreg processing parameters such as resin flow and gelation time/temperature. Since chemical composition affects the electrical properties and curing behavior of prepregs, DDA techniques may also be applied for their quality assurance (References 5.2.4(c) - 5.2.4(j)).

Whenever possible, complementary techniques should be used for the chemical quality assurance of resins and composites. Techniques, such as HPLC and IR spectroscopy, are fundamentally different from one another and provide direct, but different, information about a resin's composition. If appropriate test methods are applied, HPLC and IR spectroscopy are usually powerful enough to detect differences or changes in the chemical compositions of resins. DTA and DSC complement HPLC and IR spectroscopy by providing information relating to the handleability (i.e., the T_g and extent of reaction of the resin) and the processability of the prepreg. TGA and GC head-space analysis techniques for volatile components are secondary, but important, techniques. Special techniques for analyzing specific components or elements should be used if knowledge of the concentrations of the components is critical for processing the resin or if their presence could adversely effect the performance and durability of the cured composite. The information provided by mechanical, rheological, and dielectric analysis techniques is related to the chemical composition of the prepreg resin and thereby complements the more direct chemical techniques. However, caution is recommended in applying non-chemical techniques since the information obtained is complex and frequently ambiguous when attempts are made to relate measured parameters to chemical composition.

5.3 <u>Sampling, handling and storage</u>. Prepregs are specified by the manufacturer's trade name, resin type (e.g., 250°F) and lot number, fiber type and form (tape, fabric, roving, etc.), prepreg lot and roll numbers, and date of manufacture. The shipping date and expected shelf life are also usually designated along with recommended processing conditions. Generally, prepregs are shipped as rolls of impregnated woven fabric or unidirectional tape. (Standard widths are 38" (~97 cm) for woven aramid and glass, 42" (~107 cm) for woven graphite, and 12" (~30 cm) for tapes). The prepreg layers are separated by thin, removable plastic or coated paper films which are removed when the prepregs are analyzed.

To check uniformity, it is recommended that approximately 6 × 6 inch (15 x 15 cm) sections be cut from the center and each side of the front-end (first off) of each prepreg roll. The amount and number of samples required for a particular analysis or test depends upon the homogeneity of the resin and the uniformity of the prepreg fibers. For some techniques, such as HPLC, 0.5 to 2.0 grams of prepreg may be needed to provide a representative sample. Other techniques (e.g., DSC) which utilize relatively small specimens (10 to 20 milligrams) may demand multiple specimens to provide an "average" value or test result.

Care must be taken not to contaminate or in any way alter samples during handling and storage. The samples should be placed in clean, dry, sealable containers and be carefully labeled. The containers must not react with the samples and precautions must be taken not to expose the prepregs to moisture nor allow them to stand unrefrigerated for long periods of time. For reactive prepreg resins such as epoxies, it is recommended that the prepregs be stored in hermetically sealed containers at -10 to -15°F (-23 to -26 C). Upon removing the containers from cold storage, they should be allowed to achieve room temperature before opening to prevent condensation of moisture on the samples.

5.4 General characteristics of prepregs and composites.

5.4.1 Physical description of reinforcement. The physical description of the reinforcement used in a composite shall be described using the standard definitions of ASTM D3878 (Reference 5.4.1). The filaments in the prepreg should be uniformly wetted by the resin. No particulates should be observable upon visual examination.

5.4.1.1 Alignment. In unidirectional prepregs, the filament bundles must be parallel to the longitudinal direction of the prepreg within an angle of 0.5° when examined visually using appropriate aids to measure angular alignment.

5.4.1.2 Gaps. Any gap within or between filament bundles in unidirectional prepregs generally should comply with the following:

a. No gap shall exceed 0.060 inch (1.54 mm) in width.

b. The length of any portion of the gap with an average width of 0.040 inch (1.02) shall not exceed 24 inches.

c. Gaps in line with each other and no more than one inch (25 mm) apart shall be considered as one gap, regardless of number.

d. Gaps with excessive width or length shall be considered defective and will be the basis for flagging or replacing the prepreg.

5.4.1.3 Width. Width tolerance for unidirectional prepreg tape shall be ± 0.030 inch (0.76 mm) or as specified.

5.4.1.4 Length. The length of each roll of prepreg shall be provided together with sequence in production and batch identification as supporting data to prepreg certification. The maximum length of prepreg on any single roll shall be specified. Alternatively, suppliers and users may agree to specify the maximum prepreg weight (lbs, kg) or area (ft^2, m^2) per roll.

5.4.1.5 Edges. Maximum acceptable waviness of any 12-inch (30-cm) length of tape shall be 0.030 inch (0.76 mm) from the edge when measured with an appropriate straight edge.

5.4.1.6 Splices. Prepreg splices shall be permitted on any roll of tape where processing is continuous without change in fiber or resin batch. Such splices shall be appropriately marked as a nonconforming area.

5.4.1.7 Packaging. Prepregs shall be packaged in accordance with ASTM D3951 (Reference 5.4.1.7)

5.4.2 Resin content. The resin content of prepregs may be determined by extracting the resin from the prepreg fibers using a solvent in which the resin material is fully soluble and the fibers are not attacked. Soxhlet extraction procedures are described in ASTM C613 (Reference 5.4.2(a)). Procedures for determining the resin content of carbon fiber-epoxy prepregs are provided in ASTM D3529. Special procedures may be required to extract high molecular weight or thermoplastic resins. For example, polyether ether ketone (PEEK) can be extracted from carbon fiber prepregs using a 50/50 mixture of 1,2,4-trichlorobenzene (TCB) and phenol at 356°F (180°C). An alternate procedure for determining the resin content in epoxy resin prepregs is outlined in Section 5.6.1.

5.4.3 Fiber content. Procedures used to determine resin content often provide information about the fiber content of prepregs. Alternatively, acid digestion methods (ASTM D3171) may be applied to remove the matrix resin from the fibers as long as the fibers are not degraded (Reference 5.4.3). Digestion methods are not preferred for graphite and aramid fiber prepregs since such fibers are subject to oxidative degradation. Section 5.6.1 describes a procedure for determining the fiber and resin contents of glass, carbon, and aramid fiber/epoxy resin prepregs.

5.4.4 Volatiles content. The volatiles content of prepregs may be determined by ASTM D3530 (Reference 5.4.4). Thermogravimetric analysis (TGA) may also be applied to estimate weight percent volatiles in a prepreg.

5.4.5 Moisture content. The moisture content of prepregs may be determined by coulometry in accordance with ASTM D4019 (Reference 5.4.5) or by automated moisture meters based on the Karl Fischer titration method.

5.4.6 Inorganic fillers and additives content. The quantitative determination of inorganic fillers and additives in the prepreg resin requires considerable care. For example, the weight percent inorganic fillers and additives in a prepreg resin may be determined according to the procedure described in Section 5.6.1. Assuming that the organic resin material is fully soluble in tetrahydrofuran (THF) and that the inorganic fillers and additives are insoluble, the solution prepared in step 6 can be centrifuged to precipitate the insoluble components. The precipitate is washed at least three times with the solvent, dried, and then weighed.

5.4.7 Areal weight. The areal weight (mass per unit area) of a prepreg material may be determined using ASTM D3776 (Reference 5.4.7).

5.4.8 Tack and drape. Tack refers to the ability of a prepreg to adhere to itself or to other material surfaces and is a key factor in determining prepreg suitability for component/part fabrication. There is no quantitative method for measuring tack. Subjective terms such as high, medium, and, low are often used in describing tack. Although there is no generally accepted method for measuring tack, some composite manufacturer's use a Monsanto Tack Tester to obtain a relative index for prepeg tack. Drape is also a subjective term which relates to the ease of handling and conforming prepregs to complex surfaces.

5.4.9 Resin flow. Resin flow under specified test conditions relates to the composition, extent of reaction, and/or morphology of the prepreg resin, as well as the resin content. Prepreg processability and resin content in the processed laminate are affected by resin flow. Test conditions (temperature, pressure, layers of prepreg, number of bleeder plies) depend upon the type of resin. The resin flow of prepreg materials may be determined by ASTM D3531 (Reference 5.4.9).

5.4.10 Gel time. Gel time relates to the chemical composition and extent of reaction of thermosetting prepreg resins. Prepreg processability is affected by the resin gel time. The test temperature depends upon the type of resin. The gel times of prepregs may be determined by ASTM D3532 (Reference 5.4.10).

5.5 General scheme for resin and prepreg characterization. The following questions deserve careful consideration when developing procedures for preparing and characterizing polymer and polymer precursor (thermosetting resins and resin formulations) samples -

What are the inherent characteristics of the polymer or prepolymer?

Will certain operations cause irreversible changes in the sample?

What requirements does the characterization technique impose upon the sample?

Is it necessary to isolate the polymer or prepolymer from other sample components?

It should be recognized that the properties of polymer compounds and prepolymer formulations are often quite different from those of the pure polymers and polymer precursors. Polymer properties are greatly influenced by the presence of other components, e.g. fillers, additives, processing aids, dyes, residual catalysts, impurities, solvents and other polymers, low molecular weight oligomers and monomers.

One must decide whether the specimen needs to be modified or specially treated for a particular analysis. Chemical structure, thermal transition behavior and solubility determine what can be done with a specimen. Operations, such as heating or extraction, may alter morphology or change the chemical composition of a specimen and thereby affect its properties and compromise the validity of certain tests. Many characterization techniques require polymer specimen to be modified or have a particular shape or form. If a specimen does not conform precisely to test criteria, the test may be invalid. On the other hand, in order to apply certain techniques (e.g., light scattering and membrane osmometry for MW analysis), it is essential that the polymer be totally isolated from nonpolymeric components.

Foreknowledge of the type of polymer or prepolymer is important in developing characterization procedures. If the material is unidentified, a simple series of tests (Level I in Figure 5.5) may be applied, first to answer the question of

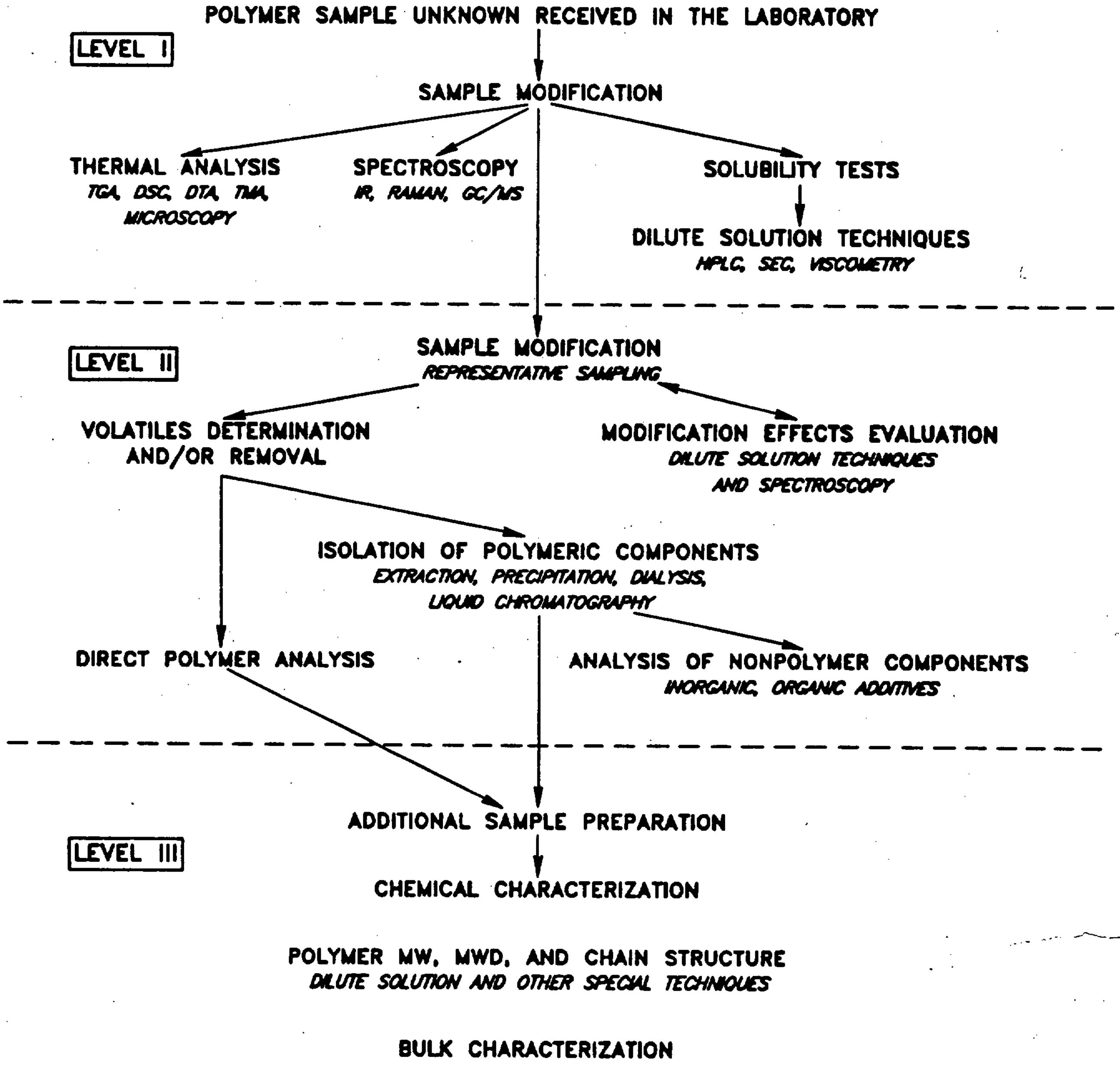

FIGURE 5.5 Polymer/prepolymer characterization scheme.

whether the sample actually contains polymer, and then to determine its characteristics and identify the polymer or prepolymer.

Specimen modification for Level I merely involves breaking or cutting a small section from the sample and, if possible, further reducing the specimen size by grinding. To facilitate thermal and spectroscopic analysis and solubility testing, the specimen should have a large surface area. Liquid and heterogeneous specimens should be thoroughly mixed before removing an aliquot for analysis. Each test can be run using as little as 10 mg sample.

5.5.1 Thermal analysis. As previously discussed, TGA provides an indication of a sample's thermal decomposition temperature T_d and is used to estimate the relative amounts of volatiles, polymer, nonpolymeric additives, and inorganic residues. DSC or DTA is applied to evaluate the extent of cure and curing characteristics of thermoset resins, to determine a polymer's T_g, and, if the polymer is semi-crystalline, to determine its crystalline melting temperature T_m. Suggested procedures for measuring T_g and T_m are given in ASTM Standards D3417 and D3418 (References 5.5.1(a) and 5.5.1(b)). TMA can also be used to determine the T_g and to obtain further information about a polymer's heat distortion temperature and thermal expansion coefficient. For pelletized or molded samples, a razor blade or microtome can be used to cut samples to approximately fit the dimensions (thickness and diameter) of the sample holder. If the sample has been cut or is already in film or sheet form with a thickness no greater than 0.015 in (0.04mm), a punch or cork borer may be used to cut disks of an appropriate size.

Alternatively, a hot state microscope may be used to observe the heat distortion temperature and onset of flow of powdered samples. Initially the powder particles have sharp, rough edges. As the sample is heated and the heat distortion temperature is approached, the edges first become blurred and then the particles start to agglomerate. Finally, at T_m, for semi-crystalline polymers, or T_g, in the case of glassy polymers, flow occurs and a clear melt or liquid forms. Microscopes equipped with cross polarizers are useful for defining crystal-crystal transitions and the onset of melting of semi-crystalline polymers.

5.5.2 Spectroscopy. Infrared spectroscopy (IR) provides more useful information for identifying polymers and polymer precursors that any other absorption or vibrational spectroscopy technique and is generally available in most laboratories. Advances like Fourier transform IR (FTIR) spectroscopy have broadened the areas of application while simplifying the requirements for specimen preparation and interpretation of spectra. IR yields both qualitative information concerning a polymer specimen chemical nature; e.g., structural repeat units, end groups and branch units, additives, and impurities. (Reference 5.5.2) Computerized libraries of spectra for common polymeric materials exist for direct comparison and identification of unknowns. Computer software allows the spectrum of a standard polymer to be subtracted from that of the unknown in order to estimate its concentration and perhaps to determine whether another type of polymer is also present in the sample.

The quality of a polymer's IR spectrum is directly related to the care taken in specimen preparation. Impurities, solvent residue, nonuniformity, interference fringes, or incorrect concentration or film thickness may cause poor results and lead to misinterpretation. For transmission IR, the concentration of the polymer solution should be the highest possible in order to minimize the contribution of solvent. Polymer films should be large enough to occupy the entire cross-section of the light beam and range from about 0.001 to 0.02 mm in thickness. The following procedures are recommended:

Soluble polymers - solvent casting to form thin films.

Thermoplastic polymers - molding or melt casting to form thin films.

Brittle and thermosetting polymers - pressed-disk or liquid dispersion technique.

Fibers and other polymers - direct analysis using IR microscope or pressing a grid of fibers into a thin film (for fibers with diameters between 0.015 and 0.03 mm) and microtoming (for thicker fibers > 0.03 mm).

Although not as popular as IR, laser Raman spectroscopy complements IR as an identification technique and is relatively simple to apply. (Reference 5.5.2) As long as the specimen is stable to the high intensity incident light and does not contain species that fluoresce, little or no sample preparation is necessary. Solid specimens need only be cut to fit into the sample holder. Transmission spectra are obtained directly with transparent specimens. For translucent specimens, a hole may be drilled into the specimen for passage of the incident light and a transmission spectra obtained by analyzing light scattered perpendicular to the incident beam. The spectrum of a turbid or highly scattering specimen is obtained by analyzing the light reflected from its front surface. Powdered samples are simply tamped into a transparent glass tube and fibers can be oriented in the path of the incident beam for direct analysis.

A powerful, but technically more demanding technique for directly analyzing polymers is pyrolysis GC/MS (gas chromatography/mass spectroscopy). In this case, the sample only needs to be rendered sufficiently small to fit onto the pyrolysis probe. Not only can the polymer type be identified by comparing the resulting spectrum with standards, but volatiles and additives can be identified rapidly and quantitatively, and polymer branching and crosslink density can sometimes be measured.

5.5.3 Dilute solution characterization. Once the solubility characteristics of a polymer are known, a suitable solvent can be selected for dilute solution characterization. THF is most often the solvent of choice for SEC. Toluene, chloroform, TCB, DMF (or DMP) and m-cresol are also used. If the polymer's Mark-Houwink constants, $\underline{k}$ and $\underline{a}$, in the solvent are known, size-exclusion chromatography (SEC) can be applied to determine the polymer's average molecular weights and molecular weight distribution (Reference 5.5.3(a)). If the constants are unknown or the polymer has a complex structure (e.g., branched, a copolymer or mixture of polymers), SEC still may be used to estimate the molecular weight distribution and other parameters relating to the structure and composition of the polymer. Although SEC indicates the presence of soluble non-polymeric components, high performance liquid chromatography (HPLC) is the better technique for characterizing residual monomers, oligomers, and other soluble, low molecular weight sample components.

Dilute solution viscometry is a simple technique for determining the limiting viscosity number or intrinsic viscosity [η] of soluble polymers (Reference 5.5.3(a)). The apparatus is inexpensive and simple to assemble and operate. The [η] of a polymer depends upon its hydrodynamic volume in the solvent and is related to the molecular weight of the polymer.

Structural and compositional information obtained by the tests in Level I is used to help develop more sophisticated specimen preparation schemes and support the application of more detailed or specialized characterization techniques. The major concern of Level II is representative sampling and insuring that specimen modification procedures (cutting, grinding, molding, etc.) do not compromise polymer characteristics to be evaluated. Level II also addresses the "quantitative" aspects of sample composition (percent polymer, additives, volatiles, and inorganic and other organic residues) and, if necessary, deals with the identification of nonpolymeric components.

A general scheme for polymer analysis is illustrated in Figure 5.5.3(a). The polymer sample should be uniform and have a large surface area. Once volatile components are removed, the polymer can be directly analyzed, or a variety of techniques (e.g., extraction, precipitation, filtration, liquid chromatography) may be applied to isolate the polymer. If required, special procedures are applied to prepare the polymer sample for chemical characterization - molecular weight, molecular weight distribution, and chain structure evaluation, and bulk characterization (Level III in Figure 5.5).

Chemical characterization techniques are listed in Table 5.5.3(a). Elemental analysis and functional group analysis provide basic and quantitative information relating to chemical composition. The analysis of reactive functional groups is particularly important in determining equivalent weights of prepolymers. Spectroscopic analysis provides detailed information about the molecular structure, conformation, morphology, and physical-chemical characteristics of polymers. Chromatographic techniques separate sample components from one another, thereby simplifying compositional characterization and make a more accurate analysis possible. Employing spectroscopic techniques to monitor components separated by gas or liquid chromatography greatly enhances characterization, providing a means to identify and quantitatively analyze even the most minor components.

Techniques for evaluating polymer molecular weights, molecular weight distribution, and chain structure are listed in Table 5.5.3(b). Size-exclusion chromatography (SEC) is the most versatile and widely used method for analyzing polymer molecular weights and molecular weight distribution. Light scattering, osmometry, and viscometry are also used to analyze polymer molecular weights. Although seldom applied to synthetic polymers, sedimentation is an excellent technique for characterizing the molecular weights of polymers having very large molecular weights. The "special" techniques tend to be somewhat empirical or have limited utility and therefore are used less often.

New techniques which show great promise for characterizing polymer chain structure also are listed in Table 5.5.3(b). One of the most promising new techniques is dynamic laser light scattering. Unlike SEC, dynamic light scattering can be applied to any soluble polymer, regardless of temperature or solvent, and does not require polymer standards for calibration. Figure 5.5.3(b) illustrates the molecular weight distribution of poly(1,4-phenyleneterephthalamide) (i.e., Kevlar™) measured by the laser light scattering (Reference 5.5.3(b)). As indicated, the polymer's molecular weight distribution can be fully characterized using very little sample and a single solution with concentrated sulfuric acid as the solvent.

Polymer Sample (fine powder or thin film)

Volatiles Removal and/or Determination

- Weight loss on drying
- TGA (Thermal Gravimetric Analysis)
- Head-Space analysis (GC/MS)
- Moisture analyzer

Isolation of Polymeric Component(s)

- Extraction
- Dissolution
- Filtration
- Precipitation
- Centrifugation

Chemical Characterization Techniques

- Elemental analysis
- Functional group analysis
- Spectroscopic analysis
- Chromatographic analysis

Polymer Molecular Weight, Molecular Weight Distribution, and Chain Structure

- Dilute solution techniques
- Other special techniques

Bulk Characterization Techniques

- Thermal analysis
- Microscopy
- Rheological analysis
- Mechanical testing
- Miscellaneous

FIGURE 5.5.3(a) General scheme for polymer analysis.

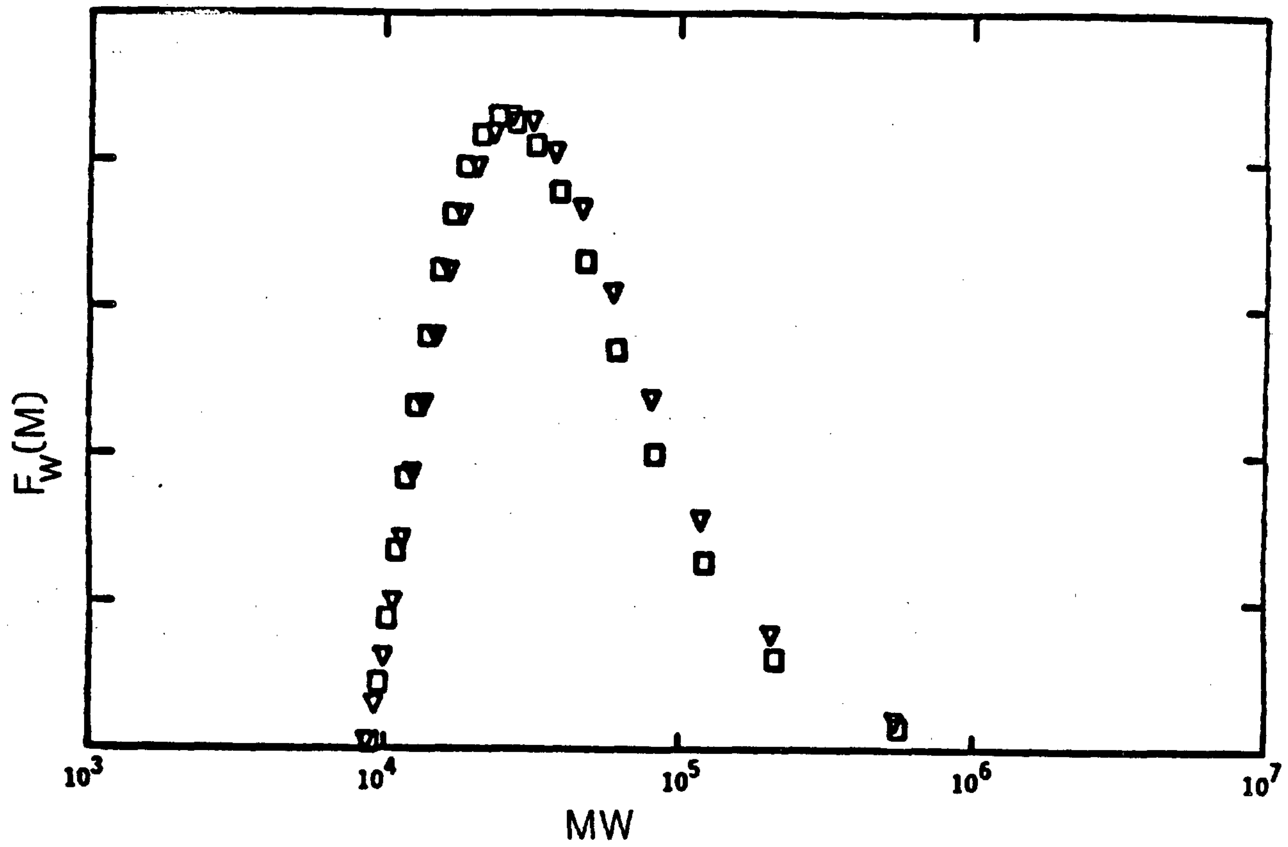

FIGURE 5.5.3(b) Molecular weight distribution (MWD) of Kevlar^{TM} in concentrated sulfuric acid, using dynamic laser light scattering.

A number of techniques and approaches used to characterize the bulk properties of polymers are listed in Table 5.5.3(c). A variety of physical properties can be measured by thermal and microscopic analysis. One of the most promising new techniques for characterizing polymers is thermo-microphotometry (TMP) - a combination of thermomicroscopy and thermo-photometry (Reference 5.5.3(c)). Like DSC and DTA, TMP determines glass transition temperature (T_g), crystalline melting temperature (T_m), thermal decomposition temperature, and polymorphic transformations. However TMP is unique in that it can also detect changes in residual stress and polymer orientation. TMP measurements employ unpolarized, linearly polarized, or circularly polarized light. Each state of polarization measures the change in a specific property of the polymer as a function of temperature. Very fast, as well as very slow, changes in state can be monitored, and the high sensitivity of TMP permits subtle changes to be detected. Although TMP shows great promise for characterizing polymer materials, an instrument which uses this technique has not been developed commercially.

TABLE 5.5.3(a) - Techniques for chemical characterization.

Elemental Analysis -	Conventional Analytical Techniques X-Ray Fluorescence Atomic Absorption (AA) ICAP EDAX Neutron Activation Analysis
Functional Group Analysis -	Conventional Wet Chemical Techniques Potentiometric Titration Coulometry Radiography
Spectroscopic Analysis -	Infrared (Pellet, Film, Dispersion, Reflectance) Fourier Transform IR (FTIR), Photoacoustic FTIR Internal Reflection IR, IR Microscopy, Dichroism Laser Raman Nuclear Magnetic Resonance (NMR) 13C, 1H, 15N Conventional (Soluble Sample) Solid State (Machined or Molded Sample) Fluorescence, Chemiluminescence, Phosphorescence Ultraviolet-Visible (UV-VIS) Mass Spectroscopy (MS), Election Impact MS, Field Desorption MS, Laser Desorption MS, Secondary Ion Mass Spectroscopy (SIMS), Chemical Ionization MS Electron Spin Resonance (ESR) ESCA (Electron Spectroscopy for Chemical Analysis) X-Ray Photoelectron X-Ray Emission X-Ray Scattering (Small Angle-Saxs) Small-Angle Neutron Scattering (SANS) Dynamic Light Scattering
Chromatographic Analysis -	Gas Chromatography (GC) or GC/MS (Low MW Compounds) Pyrolysis-GC and GC/MS (Pyrolysis Products) Headspace GC/MS (Volatiles) Inverse GC (Thermodynamic Interaction Parameters) Size-Exclusion Chromatography (SEC), SEC-IR Liquid Chromatography (LC or HPLC), HPLC-MS, Multi-Dimensional/Orthogonal LC, Microbore LC Supercritical Fluid Chromatography (SFC) Thin-Layer Chromatography (TLC), 2-D TLC

TABLE 5.5.3(b) Polymer molecular weights, molecular

Standard Techniques	Parameters Measured
Size-Exclusion Chromatography (SEC)	Mol. wgt. averages and MWD, also provides information relating to polymer chain branching, copolymer composition, and polymer shape.
Light Scattering (Rayleigh)	Weight-average mol. wgt., M_w (g/mol), virial coefficient A_2 (mol. cc/g^2), radius of gyration $<R_g^2>_z$ (A), polymer structure, anisotrophy, polydispersity.
Membrane Osmometry	Number-average mol. wgt. M_n (g/mol), virial coefficient A_2 (mol cc/g^2). Good for polymers with MW's in the range $5000 < MW < 10^6$, lower MW species must be removed.
Vapor Phase Osmometry	Same as membrane osmometry except that the technique is best suited for polymers with MW < 20,000 g/mol.
Viscometry (dilute solution)	Viscosity-average mol. wgt. M_η (g/mol) as determined by intrinsic viscosity $[\eta]$ (ml/g) relationship $[\eta] = KM_v^a$ where K and a are constants.
Ultracentrifugation or Sedimentation	Sedimentation-diffusion average mol. wgt. M_{sd} as defined by the relationship $M_{sd} = S_w/D_w$. Number- and z-average mol. wgt., M_n and M_z. MWD determined by the relation $S = kM^a$ where k and a are constants. Also provides information on the size and shape of polymer molecules.
Special Techniques	**Parameters Measured**
Ebulliometry	Number-average mol. wgt. M_n (g/mol) for $M_n < 20{,}000$ g/mol.
Cryoscopy	Number-average mol. wgt. M_n (g/mol) for $M_n < 20{,}000$ g/mol.
End Group Analysis	Number-average mol. wgt. M_n (g/mol generally for $M_n < 10{,}000$. Upper limit depends on the sensitivity of the analytical method used.
Turbidimetry	Weight-average mol. wgt. M_w (g/mol) and MWD based upon solubility considerations and fractional precipitation of polymers in very dilute solutions

weight distribution and chain structure.

Principle
Liquid chromatography technique. Separates molecules according to their size in solution and employs various detectors to monitor concentrations and identify sample components. Requires calibration with standard polymers.
Measurement of scattered light intensities from dilute polymer solutions dependent upon solute concentration and scattering angle. Requires solubility, isolation, and in some cases fractionation of polymer molecules.
Measurement of pressure differential between dilute polymer solution and solvent separated by a semi-permeable membrane. colligative property method based upon thermodynamic chemical potential for polymer mixing.
Involves isothermal transfer of solvent from a saturated vapor phase to a polymer solution and measurement of energy required to maintain thermal equilibrium. A colligative property.
Employs capillary or rotational viscometer to to measure increase in viscosity of solvent caused by the presence of polymer molecules. Not an absolute method, requires standards.
Strong centrifugal field is employed with optical detection to measure sedimentation velocity and diffusion equilibrium coefficients S_w and D_w. Sedimentation transport measurements of dilute polymer solutions corrected for pressure and diffusion provides the sedimentation coefficient S. Permits analysis of gel containing solutions.

Principle
Measures boiling point elevation by polymer in dilute solution. A colligative property.
Measures freezing point depression by polymer in dilute solution. A colligative property.
The number or concentration of polymer chain end groups per weight or concentration of polymer are determined by specific chemical or instrumental techniques.
Optical techniques are applied to measure the extent of precipitation as polymer solution is titrated with a non-solvent under isothermal conditions or as the solution prepared with a poor solvent is slowly cooled.

TABLE 5.5.3(b) Polymer molecular weights, molecular

Special Techniques	Parameters Measured
Chromatographic Fractionation	Molecular weight distribution An absolute MW technique is needed to analyze fractions.
Melt Rheometry	Weight-average mol. wgt. M_w (g/mol) and weight-fraction differential molecular weight distribution semi-empirical method.
Gel-Sol Analysis of Crosslinked Polymers	Gel fraction Cross-link density
Swelling Equilibrium	Network structure, crosslink density, number-average mol. wgt. of chains between crosslinks M_c.
Promising Techniques	Parameters Measured
Laser Light Scattering (quasi-elastic, line-broadening or dynamic)	Same as Rayleigh light scattering plus trans-diffusion coefficient, molecular weight distribution, and information relating to gel structure.
Field Flow Fractionation (FFF)	Mol. wgt. averages and MWD. Requires calibration.
Non-Aqueous Reverse-Phase High Performance Liquid Chromatography HPLC and Thin-Layer Chromatography TLC	Mol. wgt. averages and MWD. Requires calibration
Supercritical Fluid Chromatography (SFC)	Mol. wgt. averages and MWD. Requires calibration.
Neutron Scattering Small Angle (SANS)	Weight-average mol. wgt. M_w (g/mol) Virial coefficient A_2 (mol-cc/g^2) Radius of gyration $\langle R_g \rangle_z$ (A)

weight distribution, and chain structure - Continued.

Principle
Polymer is coated onto silica particles packed in thermostated column and separated according using solvent gradient elution. Polymer solubility decreases with increasing MW.
Dynamic melt rheological method involving measurement of spectrum of diffusional relaxation times for polymer during oscillatory deformation.
Extraction, filtration, and centrifugation are employed to isolate soluble polymer from gel. MW of soluble polymer is determined separately
Molar volume of crosslinked polymer immersed in swelling liquid and density of the swollen polymer are determined. Theory of partial molar free energy of mixing is applied.

Principle
Same as above but also involves measurement of the low-frequency line broadening of the central Rayleigh line of the scattered light. The structure of polymers in both dilute and concentrated solutions can be analyzed.
Separates polymers according to their size and shape in solution. An elution technique, like chromatography, except that a field/gradient (thermal, gravitational, flow, electrical, etc.) is applied perpendicular to the axis of solution flow through a capilliary or ribbon-shaped channel and a single phase is employed.
Liquid chromatography technique based upon equilibrium distribution of polymer molecules between a non-aqueous binary solvent mobile phase and a nonpolar stationary (packing) phase.
Liquid chromatography technique involving the use of a mobile phase under supercritical conditions (100 bars, 250 c).
Measurement of amplitude of neutron scattering momentum vector for polymer in dilute solution or blend with another polymer. Scattering angle and polymer concentration are varied. Deuterated solvents are used. Dilute solid solutions and polymer blends have been studied.

TABLE 5.5.3(c) - Techniques for bulk characterization.

Thermal Analysis

- TGA (Thermal Stability and Component Analysis)
- DSC (Differential Scanning Calorimetry- C_p, T_g, T_m)
- DTA (Differential Thermal Analysis- T_g, T_m)
- DMA (Differential Mechanical Analysis- T_g, T_m)
- TMA (Thermal Mechanical Analysis- T_g, Expansion)
- TBA (Torsional Braid Analysis- T_g, T_m)
- Dilatometry (Thermal Expansion Coefficient, T_g, T_m)

Microscopy

- Optical (Morphological Studies)
- Electron (Transmission and Scanning - Morphology)

Rheological

- Rotating Disc Rheometer
- Capillary Rheometer
- Melt Viscosity
- Relaxation (Viscoelastic Behavior)

Mechanical Testing

- Dielectric Spectroscopy
- Relaxation Times
- Moduli
- Compliances
- Impact Testing
- Hardness

Miscellaneous

- Density
- Birefringence Relaxation
- Refractive Index
- Transparency
- Particle Size Analysis
- Gas/Liquid Diffusion/Permeation Behavior
- Dipole Moment
- Solubility Studies
- Chemical Reactivity

5.6 Test methods.

5.6.1 Resin extraction procedure for epoxy resin prepregs. This procedure is applicable for determining the fiber and resin contents of glass, carbon, and aramid fiber/epoxy resin prepregs. Solutions prepared according to this procedure can be used directly for HPLC analysis. Recommended sampling, specimen handling procedures, and standard laboratory safety procedures should be followed.

1. Cut a rectangular specimen (approx. 1 g) from prepreg section and weigh on analytical balance (±0.001g or better). Record weight as W_o (grams).

2. Place specimen in 25 mL Erlenmeyer flask (fitted with a ground-glass stopper) and add about 20 mL THF (tetrahydrofuran, fresh, HPLC grade, distilled-in-glass, with no inhibitor added).

3. Stopper the flask and allow the specimen to soak in the THF for at least 4 hours.

4. Place flask on vortex mixer and agitate for 1 minute.

5. Carefully decant the THF solution into a 50 mL volumetric flask. The fibers should remain bunched together in the 25 mL flask.

6. Add about 10 mL THF to rinse the fibers in the 25 mL flask, mix on the vortex mixer, and decant the THF into the 50 mL volumetric flask containing the primary solution (step 5).

7. Repeat step 6.

8. Add THF to fill the volumetric flask to the 50 mL mark.

9. Carefully remove the graphite fibers from the 25 mL Erlenmeyer flask (using forceps), wrap fibers in Kimwipes^{TM} or equivalent, place in labeled paper envelop, place the envelope in fume hood air stream, and allow fibers to dry overnight. Alternatively, residual THF may be removed by placing the envelope with fibers in a vacuum oven (fitted an appropriate vapor trap) set at 40°C and maintaining a vacuum for at least 1 hour.

10. The fibers are removed from the Kimwipes^{TM} and weighed on an analytical balance. Record the fiber weight as W_f (grams).

11. Calculate the concentration of the resin solution (see step 8) and record concentration as C_o (μg/μL). This concentration will be useful in the analysis of HPLC data.

$$C_o = (W_o - W_f)/0.050 \ \mu g/\mu L \qquad 5.6.1(a)$$

12. Mix resin solution (from step 11) on vortex mixer and immediately filter about 4 mL of the resin sample solution through a 0.2 μm Teflon membrane filter into a dry, clean glass vial. Immediately cap the vial to prevent contamination and solvent loss. This solution will be used for HPLC analysis.

13. The extractable resin content and fiber content, not corrected for the presence of volatiles and insoluble components in the prepreg resin and remaining on the fibers, are calculated -

$$\text{wt-\% extractable resin} = 100\% \times (W_o - W_f)/W_o \qquad 5.6.1(b)$$
$$\text{wt-\% fiber} = 100\% - \text{wt\% extractable resin} \qquad 5.6.1(c)$$

14. Glass fibers may be placed in a muffle furnace and heated at 650 to 800°C to remove nonextractable surface material. After cooling to room temperature, the fibers are reweighed and their weights are recorded as $W_{f'}$.

15. The amount of nonextractable fiber surface material in glass fiber prepregs is calculated -

$$\text{wt-\% nonextractables} = 100 \times (W_f - W_{f'})/W_o \qquad 5.6.1(d)$$

16. The extractable or THF-soluble resin content may also be determined by filtering the solution prepared in step 8 through a 0.2 μm Teflon™ membrane filter. Using a volumetric pipet, an aliquot (e.g., 10 mL) of the filtered solution is transferred to a pre-weighed aluminum pan (weight W_A) which is then placed into a fume hood to evaporate the solvent. A stream of filtered air or nitrogen can be directed over the surface of the pan to accelerate evaporation. After 9 mL or more of the solvent is removed leaving an oily residue of resin, the pan can be placed in a vacuum oven and heated at about 50°C for several hours to remove residual solvent. After cooling to room temperature, the pan is reweighed ($W_{A'}$) and the resin content is calculated -

$$\text{wt-\% soluble resin} = 100\% \times (W_{A'} - W_A) \times 5/W_o \qquad 5.6.1(e)$$

Differences in the weight-percent resin determined using Equations 5.6.1(b) and 5.6.1(e) may be attributed to the presence of volatiles and insoluble (nonfibrous) components in the prepreg.

5.6.2 Procedure for HPLC/HPSEC analysis of glass, aramid, and graphite fiber prepregs. Mix resin solution (prepared in Section 5.6.1, step 12) on a vortex mixer and immediately filter about 4 mL of the resin sample solution through a 0.2 μm Teflon membrane filter into a dry, clean glass vial.

Immediately cap the vial to prevent contamination and solvent loss. The sample is now ready for HPLC analysis.

If the HPLC analysis is not run immediately, the sample solution should be kept in a cool, dark location. If care is taken during storage, the THF solution will remain stable and may be analyzed weeks after its preparation with no apparent effect on the HPLC analysis.

5.6.2.1 Reverse phase HPLC analysis. The epoxy resin prepreg analysis can be run using any of a number of commercially available HPLC instruments. An integrator/recorder or state-of-the-art HPLC data analysis system is recommended for data acquisition, plotting, and reporting. HPLC operating conditions were selected for simplicity and compatibility with most commercial HPLC equipment.

HPLC System: Waters Associates model-244 instrument with M6000A solvent delivery systems, M720 system controller, 710B WISP auto-injection system, M440 UV detector, and M730 data module. Similar systems available from other manufacturers may also be used.

Solvents: Acetonitrile (distilled-in-glass) and reagent grade water prepared from distilled water using a Millipore Milli-Q2 (Millipore Corp., Bedford, MA) or equivalent water purification system. Purging the solvents with helium is recommended.

Column: Waters Associates μBondapak C18. (Similar columns available from other manufacturers may also be used).

Flow Rate: 2.0 mL/min

Mobile Phase (solvent program):

Time	% Acetonitrile	% Water	Curve
0	45	55	*
12 min	100	0	7
16 min	100	0	*
20 min	45	55	6

Detector: UV 254nm   Run Time: 20 minutes

5.6.2.2 Size Exclusion Chromatography (SEC) analysis. The SEC analysis of the prepreg resins can be run using HPLC instrumentation as described above.

Solvent: THF (distilled-in-glass) A helium purge should be maintained on THF for optimum results.

Columns: IBM SEC type A and type C, 5 micron (columns from other manufacturers, such as the Waters μStyragel 1000, 500, 100, 100 A, are also acceptable).

Injection Volume: 10 μL   Flow Rate: 1 mL/min
Detector: UV 254nm   Run Time: 15 minutes

Calculations: Integrated peak areas are converted to area percentages (% area).

5.6.3 Procedure for Fourier transform infrared spectroscopy (FTIR). Several droplets of the resin/THF solution (prepared in Section 5.6.1, step 12) are placed on the surface of a polished salt plate (preferably KBr). The sample is analyzed as soon as the THF has evaporated. A Perkin-Elmer model 1550 or 1700 FTIR spectrometer with model 7500 computer or an equivalent instrument is used to scan and record the spectrum (500 to 4000 cm^{-1}) of the salt plate with and without the sample on its surface. The analysis should be conducted with the salt plate and sample in a purged, dry nitrogen atmosphere at room temperature. Depending upon the sample, 100 to 200 scans of the spectrum may be required to optimize spectral resolution. It also may be necessary to deposit more or less sample on the salt plate. The spectrum of the sample obtained by subtracting that of the salt plate is plotted, reported, and stored on a computer disk.

5.6.4 Procedure for differential scanning calorimetry (DSC). This test can be performed using a DuPont Instruments 9900 Thermal Analyzer/Controller and model 912 DSC accessory or an equivalent instrument.

Specimen:	Prepreg (10 to 30 mg) in an aluminum sample pan
Reference:	Empty sample pan
Heating Rate:	10°C/min
Temperature Range:	Room temperature to 350°C
Atmosphere:	Dry nitrogen gas purge
Data Handling:	Data is stored on a computer disk and a plot of heat flow dH/dt (mW/sec) vs temperature (°C) is produced.
Heat of Reaction:	The calibration routine and integration program provided with the thermal analyzer is used to calculate heats of reaction ΔH of thermoset prepreg resins.
Glass Transition:	A cooling device attached to the DSC cell may be needed to facilitate glass transition temperature T_g measurements of thermoset prepreg resins; i.e., it is often necessary to initiate temperature scans at -50°C or lower since such resins typically have T_g values below room temperature. The thermal analyzer may have a software routine to assist in determining T_g values.

5.6.5 Procedure for dynamic mechanical analysis (DMA). A single ply of prepreg is cut into a 1.1 cm x 1.7 cm strip and the strip is mounted in a DuPont model 982 or 983 DMA accessory. A DuPont 9900 or 1090 controller is used to run the test and plot the results. Equivalent instruments may also be used.

Heating Rate:	5°C/min
Temperature Range:	Room temperature to 350°C
Atmosphere:	Dry, nitrogen gas purge
Data Handling:	Data is stored on a computer disk and a plot of storage modulus and tan δ is plotted as a function of temperature.
Glass Transition:	The temperature of the damping peak maximum is assigned as the T_g value.
Gelation:	Gelation occurs when the Young's modulus starts increasing rapidly (several orders of magnitude) over a narrow temperature range. Gelation temperature depends upon heating rate and mechanical frequency. Therefore, both heating rate and frequency should be included when DMA gelation temperatures are reported.
Gelation Time:	In the isothermal mode, the time to gelation is determined by rapidly heating a sample to the desired temperature, holding the temperature constant and monitoring the change in Young's modulus with time. Gelation time is defined as

the time it takes for the modulus to start rapidly increasing (several orders of magnitude).

5.6.6 Procedure for rheological characterization. A Rheometrics Dynamic Spectrometer (RDS) or equivalent system is used for this test. Samples are prepared by cutting three 25-mm diameter circles from a single ply of prepreg. The three plies are stacked and placed between the rheometer's parallel plates.

Heating Rate:	2°C/min
Temperature Range:	Room temperature to the onset of gelation (for thermosets)
Atmosphere:	Air or a blanket of nitrogen gas
Geometry:	25-mm diameter parallel plate
Gap:	Typically 0.8 mm, but may be adjusted according to sample characteristics.
Data Reporting:	Shear moduli (storage and loss) and complex viscosity are plotted as a function of temperature.

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6. LAMINA AND LAMINATE CHARACTERIZATION

Page

6.1 Introduction. The use of composite materials continues to increase as new performance, reliability, and durability requirements drive hardware designs to higher levels of structural efficiency. Additionally, government requirements are becoming more stringent to assume proper levels of structural integrity are maintained. These design drivers, among others, have resulted in a growing recognition that certification or qualification of aerospace structure requires an extensive combination of analysis, testing, and documentation.

Further, because of the large number of design variables inherent to composite structure, analytic models are even more necessary than for metallic structure to assume completeness of the hardware qualification process. Inherent in all structural analysis models are material, physical, and mechanical property characterization data. Ideally, these analytic models would permit analysts to predict full-scale structural response (e.g. stability, deflections, strength, life) directly from a generic (lamina) material database. In truth, test data is required at design development (element, subcomponent, component) and full-scale article test levels as well as the generic (coupon) levels of evaluation.

The purpose of Chapter 6 is to provide guidelines on testing procedures for characterization of lamina (ply) physical and mechanical properties. While current procedures emphasize development of a lamina-level database, this does not preclude higher-level testing.

6.2 Chemical property tests.

6.3 Physical property tests.

6.3.1 Glass transition temperature.

6.3.2 Density.

6.3.3 Water absorption.

6.3.4 Thermal expansion.

6.3.5 Porosity.

6.4 Thermal stability.

6.5 Chemical stability.

6.6 Mechanical property tests.

6.6.1 General. Section 6.6 contains test methods for determining mechanical property data for composite materials. The purpose of this section is to provide a basis for uniformity in the use of standard test methods and, ultimately, to provide for combining the experimental data. The reader is referred to Chapter 8 for reporting of data to MIL-HDBK-17. The test methods are representative of procedures used in the composite materials industry and were selected after review of user material specifications. Specific standards are included to allow the user to perform tests consistent with industry practice; however, inclusion of these standards should not be considered an endorsement of any standard or organization by MIL-HDBK-17. Moisture effects and conditioning of specimens are described in Section 2.2.2.

6.6.2 Tensile tests.

6.6.2.1 General considerations. Tensile testing of laminates will generally be accomplished in accordance with ASTM D3039. This test employs a straight-sided specimen with bonded end tabs for gripping, and is applicable to 0° and 90° unidirectional constructions as well as bidirectional and fabric laminates. Details for specific orientations are discussed in Sections 6.6.2.2, 6.6.2.3, and 6.6.2.4. The test yields tensile ultimate strength, tensile modulus, and the major Poisson's ratio.

Experience within the aerospace industry has indicated that the critical factors in obtaining consistent tensile test results are tab design, tab application, and specimen machining quality. With respect to machining, it is important that specimen edges be wet ground, cut, or polished to a fine surface finish to preclude premature failure.

The purpose of tabbing is to introduce uniform loading into the specimen. The degree to which this is realized depends upon a number of factors including tab bevel angle, and the mechanical properties of the tab material relative to the laminate being tested. In general, the tab material should be less stiff than the test laminate.

Surface preparation prior to tab bonding may include light sanding or grit blasting to promote adhesion. Care must be taken not to abrade any part of the laminate gage section (any area not covered by the tab), as this damage could cause premature failure.

Thermal considerations are also important. If the coefficient of thermal expansion of the tab material is significantly different from that of the laminate, bond failure could occur either during bond cycle cool-down, or during elevated temperature testing. In addition, bond cycles that will advance the cure state of the laminate cannot be used, as this will invalidate the data. For this reason, many companies use 250°F (120°C) curing adhesives when tabbing 350°F (180°C) cured laminates. This is of less concern when postcured laminates are being tested.

6.6.2.2 0° Unidirectional laminae. When preparing 0° specimens, care must be taken to insure that the fibers are aligned parallel to the coupon load axis. A misalignment of only 0.5° has been shown to affect ultimate strength by as much as

5%. To accomodate capacities of standard composite test machines, the width of 0° unidirectional specimens as usually specified as 0.500 inches (12.7 mm).

6.6.2.3 90° Unidirectional laminae. As specified in ASTM D3039, the 90° tensile specimen is wider and shorter than the 0° coupon. Since this test is totally matrix dependent, the handling and precise alignment of the specimen are critical. Special alignment fixtures are often used in conjunction with hydraulic or pneumatic test grips to prevent introduction of out-of-plane forces.

As an alternate, a sandwich beam specimen in accordance with ASTM C393 may be used for 90° testing. This specimen is less susceptible to handling damage, but panel warpage is a problem.

6.6.2.4 Bidirectional and fabric laminates. The specimen for these laminates is wider than the 0° unidirectional coupon. For fabrics it is mandatory to orient the warp and fill for each ply in the same direction, and to report the direction tested. A modified ASTM D-3039 dog-bone shaped specimen is frequently utilized for testing fabrics. As larger tow, fewer ends per inch fabrics have become more common, it has become practical to increase the gage width to 1.000 inch (25.4 mm) to reduce scatter of test results.

6.6.3 Compression tests. Compression test methods for composite materials generally fall into three categories: unsupported coupon, supported coupon, and sandwich beam. ASTM D 3410, Standard Test Method for Compressive Properties of Unidirectional or Cross-ply Fiber-Resin Composites, describes the specimens and fixtures for the unsupported coupon and sandwich beam tests. The unsupported coupon has a recommended gage length of 0.500 inch (12.7 mm) and can be placed in one of the two compression fixtures given in ASTM D 3410. The two fixtures have split collet-type grips at both ends but they differ in the way load is introduced and in the gripping arrangement. The sandwich beam is a rectangular bonded beam with a 6 ply, all 0° composite test skin. The skin is bonded to a high density core. It should be noted that the sandwich beam specimen is generally expensive to test and unsuitable for environmental testing. There are also questions concerning the influence of honeycomb on the test results.

ASTM D 695, Standard Test Method for Compressive Properties of Rigid Plastics, was not designed for composite materials, but nonetheless it has been modified by the aerospace industry for use with composites. References will often be made to a "modified ASTM D 695 test", but each company has developed its own modification and there is no one accepted test fixture, gage length, or test procedure. In general, a modified ASTM D 695 test supports the face of the specimen and uses a gage section ranging from 0.1 to 0.2 inches (2.5 to 5.1 mm) in length. The supported specimen face of this test method, in addition to the smaller gage section, generally results in higher compressive strength and moduli values than ASTM D 3410. These higher values make it the method of choice for many designers.

There is considerable debate as to whether a modified ASTM D 695 test results in a true compressive failure. The constrained specimen may not be representative of a compressive failure in composite materials. ASTM D 3410 may produce a more accurate mechanistic compressive failure in composites, but the design values are generally lower and may not be representative of a compressive failure mode in an aircraft structure.

Regardless of choice, ASTM D-3410 or modified ASTM D-695, it has become more practical to incorporate 1.000 inch (25.4 mm) gage width laminate specimens for fabrics with low end count per inch weaves (such as 6000 filament graphite tow fabrics).

ASTM C 393, Flexure Test of Flat Sandwich Constructions, has been regarded as a dependable compression test for composites. However, the sandwich beam specimen is generally expensive to test, wasteful of material, and unsuitable for environmental testing. There are also questions concerning the influence of honeycomb on the test results.

6.6.4 <u>Flexure tests</u>. There is not a recommended test method for determining the flexural properties of composite laminates. Even though there are approved flexure test methods, there is some debate as to the validity of the results.

Within the aerospace industry, flexure testing is primarily used for quality control. ASTM D790, Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials, was originally written for plastics but has since been modified and approved for composites. In some cases, ASTM C393, Flexure Test of Flat Sandwich Constructions, has been adapted for use with composite laminates.

6.6.5 <u>Shear tests</u>. The use of continuous fiber reinforced composites as high performance structural materials in modern aerospace vehicles necessitates the accurate measurement of the material response to mechanical and thermal loads. Most of the current composite material shear test methods were originally developed for metals, wood, and adhesives. However, coupling effects, nonlinear behavior of the matrix or the fiber-matrix interface, and the presence of normal stresses combine to make the present shear test methods of questionable value for composite materials (References 6.6.5(a) - (d)).

The shear test methods most widely used in the aerospace industry to measure the interlaminar and in-plane shear moduli and shear strengths of composite materials are subject to the above-mentioned limitations. For instance, in the Short Beam Shear test (ASTM D2344), the shear stress distribution depends upon the beam length-to-thickness ratio and eccentricities of the rollers applying the loads (Reference 6.6.5(a)). Stinchcomb et al. (Reference 6.6.5(b)) found that a good quality specimen failed in microbuckling or a combination of shear and microbuckling and that a shear failure only occurred in poor quality specimens. The Short Beam Shear test should not be utilized for determining the interlaminar shear strength. Short Beam Shear is useful for quality control and perhaps for selecting candidate materials, but it does not provide an accurate measurement of interlaminar shear strength.

Similar problems exist for the Rail Shear test (ASTM D4255), which is one of the most analyzed methods for determining the in-plane shear modulus of composite materials. Stress concentrations at the corners and significant normal stresses are produced in the test section depending upon the method of load introduction, stiffness of rails, and properties of the composite (Reference 6.6.5(c)).

The torsional shear of a thin-walled circular tube (ASTM E143) is considered the most desirable method to obtain both the shear strength and the shear modulus of a material. Though from the applied mechanics viewpoint this is the ideal shear test, the torsional tube presents many problems to the experimentalist. Cost of fabricating tubular specimens can be prohibitive unless the manufacturer is involved in filament winding. Preparation of a tube is time-intensive and requires more material than a flat specimen. Composite tubes can also be extremely fragile and difficult to handle. Further, the specimen must be mounted concentrically in the test apparatus to prevent the introduction of bending moments, and the tube must be free to move axially to avoid introducing axial forces. Buckling of the tube must also be prevented. This requires special test equipment which can be expensive to manufacture. Yet, for all its limitations, when shear properties from this test are available, these values are the standard by which results from other shear tests are judged.

The ±45 Off-Axis Tensile Shear Test (ASTM D3518) consists of loading a ±45-degree symmetric laminate uniaxially in tension. This test has the advantage of being economical with material and time and it involves a simple test procedure (Reference 6.6.5(d)). Researchers have reported good correlation with unidirectional laminates between the results of the ±45-degree shear test and other shear test methods (References 6.6.5(d-g)), including the torsion tube. However, like all other shear test methods, this shear test is not without its problems. The stress/strain response tends toward ductile characteristics as a result of interply effects (Reference 6.6.5(f)). The stiffness is considered reliable up to 1.3% strain, but at higher stress levels the stiffness is underestimated due to edge effects (Reference 6.6.5(d)). Strength is also governed by these edge effects and is considered a conservative, lower-bound design value. Note that ±45 off-axis shear test results with fabric materials become more difficult to interpret due to the nature of the product form, mechanical testing, etc.

However, for determining the initial in-plane shear modulus, the ±45 Off-Axis Tensile Shear Test (ASTM D3518) may provide a value which reflects the actual stress state in a laminated structure. The specimen's ductile stress-strain response resulting from interply effects is indicative of the fact that a "pure" shear stress does not exist in the specimen, yet it may mimic the actual stress state that may occur in a laminate and may be representative of the interaction of one lamina upon adjoining laminae. The resulting value is an "effective" shear modulus, and may be a more realistic determination for the designer.

6.6.6 <u>Fatigue</u>. Static testing of unidirectional composite coupons is useful for material characterization, comparison of materials, and for prediction of application laminate properties through the use of lamination plate theory. In the area of fatigue, however, no generalized methodology has yet been devised to predict laminate behavior from unidirectional coupon data. Hence, the development of fatigue design values becomes a unique problem for each application lay-up.

Many studies have been undertaken, and much has been written concerning life prediction for specific laminates under cyclic loading spectra. Even at this level, empirical methods have been favored due to the inadequacy of results obtained from cumulative damage models, fracture mechanics analysis, and other theoretical approaches (References 6.6.6(a) and (b)).

ASTM D 3479, Tension - Tension Fatigue of Oriented Fiber, Resin Matrix Composites, is a generalized coupon testing method. However, because composite fatigue is so application dependent, it is important that the laminates represent the application and that the laminates be tested accounting for the usage load spectra and environmental conditions. Currently this is accomplished in composite hardware programs through a "building block" test approach involving coupon, element, and component specimens, all representative of full-scale structural details.

It is important to note that, for the majority of current aircraft composite structure, fatigue capability does not become a limiting factor if all static strength concerns have been thoroughly and successfully addressed. Exceptions to this are high-cycle components such as those found in helicopter dynamic systems.

6.6.7 Creep.

6.6.8 Damage tolerance and laminate testing. The resistance of composite materials to damage has gained broad interest in the aerospace community. To work toward characterizing the damage tolerance of these materials, several tests have been developed. These include laminate tests such as compression after impact and various tests for determining strain energy release rate.

For compression after impact testing, the most widely used configuration is that discussed in NASA Publications 1092 and 1141 (References 6.6.8(a) and (b)). Among the factors to be considered in using these methods are the actual energy of the falling tup in impact, the rigidity of the holding fixture, specimen preparation techniques, and the materials used and actual configuration of the compression test fixture. Other compression after impact configurations are also in use, and no industry wide standard has been accepted.

Strain energy release rate, G_c, is currently the subject of considerable research. The most common method for determining G_c, the mixed mode strain energy release rate, is the edge delamination test. This test involves measuring the load and strain at onset of the first delamination of a specified lay-up when tested in tension. For determination of G_c, basic laminate properties must be known. Detailed procedures outlining lay-up, test method, and calculations are given in NASA Publications 1092 and 1142 (References 6.6.8(a) and (b)). Further discussion may be found in Reference 6.6.8(c).

Mode I strain energy release rate, G_{Ic}, is usually determined by using a hinged double cantilever beam specimen. The two halves of a pre-cracked specimen are pulled apart in tension so that the crack propagates between laminate layers in the center of the specimen. Load and crack-opening displacement are monitored throughout the test. Details of the specimen configuration and test procedure are given in Reference 6.6.8(a).

Factors that may effect the test results include resin content and specimen geometry. There are indications that an optimal resin content range may exist outside of which G_{Ic} values will be lower. The crack length over which the data is reduced is another variable which requires further standardization.

Mode II strain energy release rate, G_{IIc}, may be determined by using an end-notched flexure specimen. This approach is discussed in Reference 6.6.8(d). The test is designed so that a precrack is allowed to propagate through the middle plane of the specimen during Mode II loading. Issues of test specimen geometry, loading rates, lay-up, and other factors have not yet been standardized.

ASTM Committee D 30 is currently working on test procedures for these properties. The approaches currently being considered are: Mode I - the double cantilever beam test; Mode II - the end-notch flexure test; and mixed mode - the edge delamination and the cracked-lap shear tests.

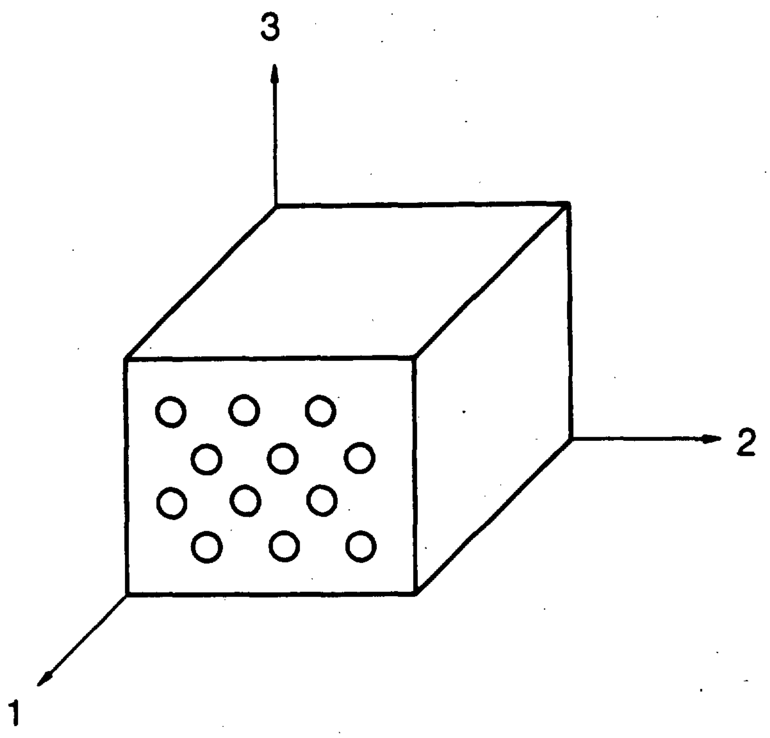
6.6.9 Filament winding. The mechanical behavior of filament wound structures is typically different from the behavior of flat laminated structures. Some noted differences result from the type of cure, resin void content, microcracking, and free edge construction. However, filament wound structures require the same mechanical property data for design and analysis as used for general laminated structures. The majority of filament would structures are used in the rocket motorcase community and, consequently, most of the test specimens are in the form of cylinders or bottles that more closely simulate the geometry of the structures to be designed and analyzed.

In November 1983, the Joint Army, Navy, NASA, and Air Force (JANNAF) Interagency Propulsion Committee charted by the Department of Defense (DoD) formed the Composite Motorcase Subcommittee (CMCS) (Reference 6.6.9(a)). The CMCS is concerned with the application of composites materials in the construction of rocket motorcases for strategic and tactical missiles, space propulsion systems, and cartridge cases for gun propulsion. The CMCS consists of four working panels: (1) Testing and Inspection (T&I), (2) Design and Analysis (D&A), (3) Processing, and (4) Materials. The T&I Panel surveyed industry on test methods which resulted in 17 tension, 17 compression, and 16 different shear tests that were being used to obtain mechanical property data. The T&I and D&A Panels joined to evaluate the test methods via a JANNAF Workshop (Reference 6.6.9(b)). A panel of experts in filament wound composites was selected and tasked to make recommendations for test methods. A joint T&I and D&A Member JANNAF Workshop was held in April 1986 to discuss the panel of experts' recommendations and to have an industry selection of JANNAF Interim Standard Test Methods to be used for the determination of uniaxial material properties for the design and analysis of filament wound structures. Table 6.6.9 contains the test methods selected and the output parameters obtained from each test. An element of orientation is included to identify directions of the output parameters.

The JANNAF CMCS is presently engaged in Round Robin Testing (RRT) of the Interim Standard Test Methods. The RRT effort includes industry and government laboratories. Until the results of the RRT are known, JANNAF cannot recommend detailed test methods for uniaxial material characteristics of filament wound structures. The composite motorcase community also has a vital interest in characterizing the mechanical behavior of filament wound structures subjected to biaxial and triaxial states of stress. The JANNAF efforts are being coordinated with MIL-HDBK-17, ASTM D-30 Committee, SACMA, IDA, and the DoD Standardization Program for Composites Technology (CMPS).

TABLE 6.6.9 JANNAF interim standard test methods.

0° Tension:	Pressurized NOL Ring, Pressurized Tube (90° Wind)	E_{11}, ν_{12}, σ_{11}, ϵ_{11}
90° Tension:	Tube (90° Wind)	E_{22}, ν_{21}, σ_{22}, ϵ_{22}
0° Compression:	Flat Laminate (0°)	E_{11}, ν_{12}, σ_{11}, ϵ_{11}
90° Compression:	Tube (90° Wind)	E_{22}, ν_{21}, σ_{22}, ϵ_{22}
In-Plane Shear:	Torsion Tube (90° Wind)	G_{12}, τ_{12}, γ_{12}
Transverse Shear	Iosipescu	G_{23}, τ_{23}, γ_{23}



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7. STRUCTURAL ELEMENT CHARACTERIZATION

7.1 Introduction.

7.1.1 Background. Testing and analysis of composite joints are essential for maintaining the structural integrity of composite structures and to ensure their reliability. Any joint in a composite structure is a potential failure site. Without proper design, a joint can act as a damage initiation point, which can lead to a loss in structural stability and eventual failure of a component. Two types of joints are in common use, namely (1) mechanically-fastened joints and (2) adhesively-bonded joints. Only mechanically-fastened joints are considered in MIL-HDBK-17 at this time. These guidelines define the recommended approach for testing bolted joints to determine bearing strength properties. A detailed analysis of the stress distribution around a fastener hole is not presented here. Discussion on both theoretical and empirical approaches to the stress analysis of bolted joints in composite materials can be found in Reference 7.1.1.

An important consideration in joint testing and analysis is the selection of the type of test method with due attention to the failure mode which is likely to result with a specific joint design in a particular composite system. A brief discussion on various failure modes is provided in the next section.

7.1.2 Failure modes. The occurrence of a particular failure mode is dependent primarily on joint geometry. Composite bolted joints may fail in various modes as shown in Figure 7.1.2. The likelihood of a particular failure mode is influenced by bolt diameter (D), laminate width (b), edge distance (e), and thickness (t).

Net section tension failures occur when the bolt diameter is a sufficiently large fraction of the strip width. This fraction is about one-quarter or more for near-isotropic lay-ups in graphite-epoxy systems. Bearing failures typically occur when the bolt diameter is a small fraction of the strip width. Shear-out failures are essentially a special case of bearing failure. Quite often a shear-out failure is the result of a bearing failure with short edge distance (e). For highly orthotropic laminates, shear-out failures may occur at very large edge distances.

Cleavage failures occur because of the proximity of the end of the specimen. A cleavage failure can be triggered from a net-section tension failure. This type of failure often initiates at the end of the specimen rather than adjacent to the fastener.

In some instances the bolt head may be pulled out through the laminate after the bolt is bent and deformed. This mode is frequently associated with countersunk fasteners.

Finally, it is important to note that for any given geometry, the failure mode may vary as a function of fiber pattern and lay-up sequence.

Within MIL-HDBK-17, only the bearing strength test method is discussed in detail to allow proper measurement and computation of joint strength. Strength measurements based on other failure modes may be included in MIL-HDBK-17 in the future.

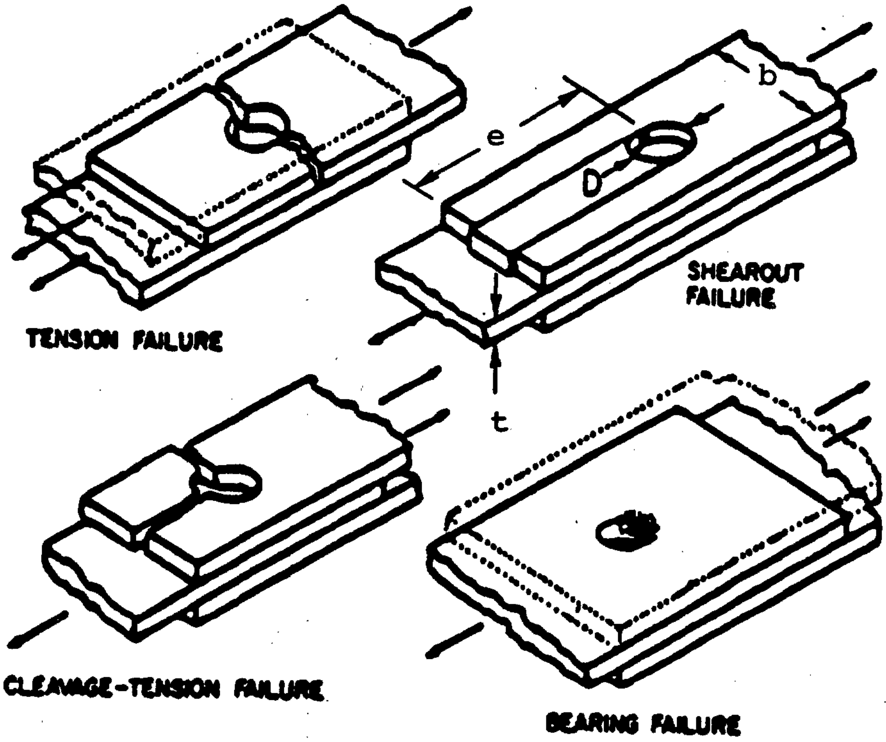


Figure 7.1.2 Typical failure modes for bolted joints in advanced composites.

7.2 Bearing strength characterization. Determination of bearing strength of continuous fiber-reinforced advanced composites is discussed in this section.

7.2.1 Significance. Bearing strength is considered a structural property for relative evaluation purposes, and should not be used improperly in the design of bolted composite structures. In an actual structural application, factors like laminate lay-up and load eccentricity will significantly influence the realizable fraction of the bearing strength measured in the proposed test.

The proposed test introduces the bearing load in a double shear configuration. In actual applications, load transfer in a single shear configuration is more commonplace, resulting in larger stress concentrations in the thickness direction, and lowering the realizable bearing strength.

The proposed test specimen suffers pure bearing failures because it contains a low percentage of 0° plies. If the structural application calls for over forty percent of 0° plies in the laminate, the failure mode will generally not be pure bearing, and a lowering of the bearing stress at failure will result. A high percentage of 0° plies will normally result in a shear-out mode of failure. In this case, the bearing stress at failure will be influenced by the shear-out area, which will be dependent on the edge distance.

Only a tensile loading condition is proposed for evaluating bearing failures; under compression, the larger edge distance (e >> 3D) should only influence the bearing stress at failure minimally unless a shear-out mode of failure is possible (e.g., a laminate with a large percent of 0° plies).

The bearing deformation effects on reinforced composites is vastly different from those on plastics and metals, making the definition of bearing strength ambiguous under the same situations. Therefore, it is recommended that the bearing stress variation as a function of hole deformation be documented, and that bearing stress values corresponding to the proportional limit, prescribed hole elongation values of four percent of the initial hole diameter, and ultimate failure be recorded.

7.2.2 Definitions. The following definitions are relevant to this chapter.

Edge Distance Ratio -- The distance from the center of the bearing hole to the edge of the specimen in the direction of the applied load, divided by the diameter of the hole.

Bearing Area -- The diameter of the hole multiplied by the thickness of the specimen.

Bearing Load -- A compressive load on an interface.

Bearing Strain -- The ratio of the deformation of the bearing hole in the direction of the applied force, to the pin diameter.

Bearing Strength -- The maximum bearing stress which can be sustained.

Bearing Stress -- The applied load divided by the bearing area.

Proportional Limit Bearing Stress -- The bearing stress value corresponding to the deviation from linearity of the bearing stress versus hole elongation curve.

Intermediate Bearing Stress -- The bearing stress at the point on the bearing load-deformation curve where the tangent is equal to the bearing stress divided by a designated percentage (usually 4%) of the original hole diameter.

Ultimate Bearing Stress -- The bearing stress corresponding to total failure of the test specimen.

7.2.3 Specimen design and testing. Using standard test equipment, the specimen shall be tested in the double shear arrangement shown in Figure 7.2.3. Test specimens shall conform to the dimensions and tolerances shown. Recommended b/D and e/D values are 6 and 3, respectively, and D = 1/4 inch (6.4 mm). A [±45/0/90]3s lay-up based on a cured ply nominal thickness of 0.0052 inch (0.13 mm) is recommended.

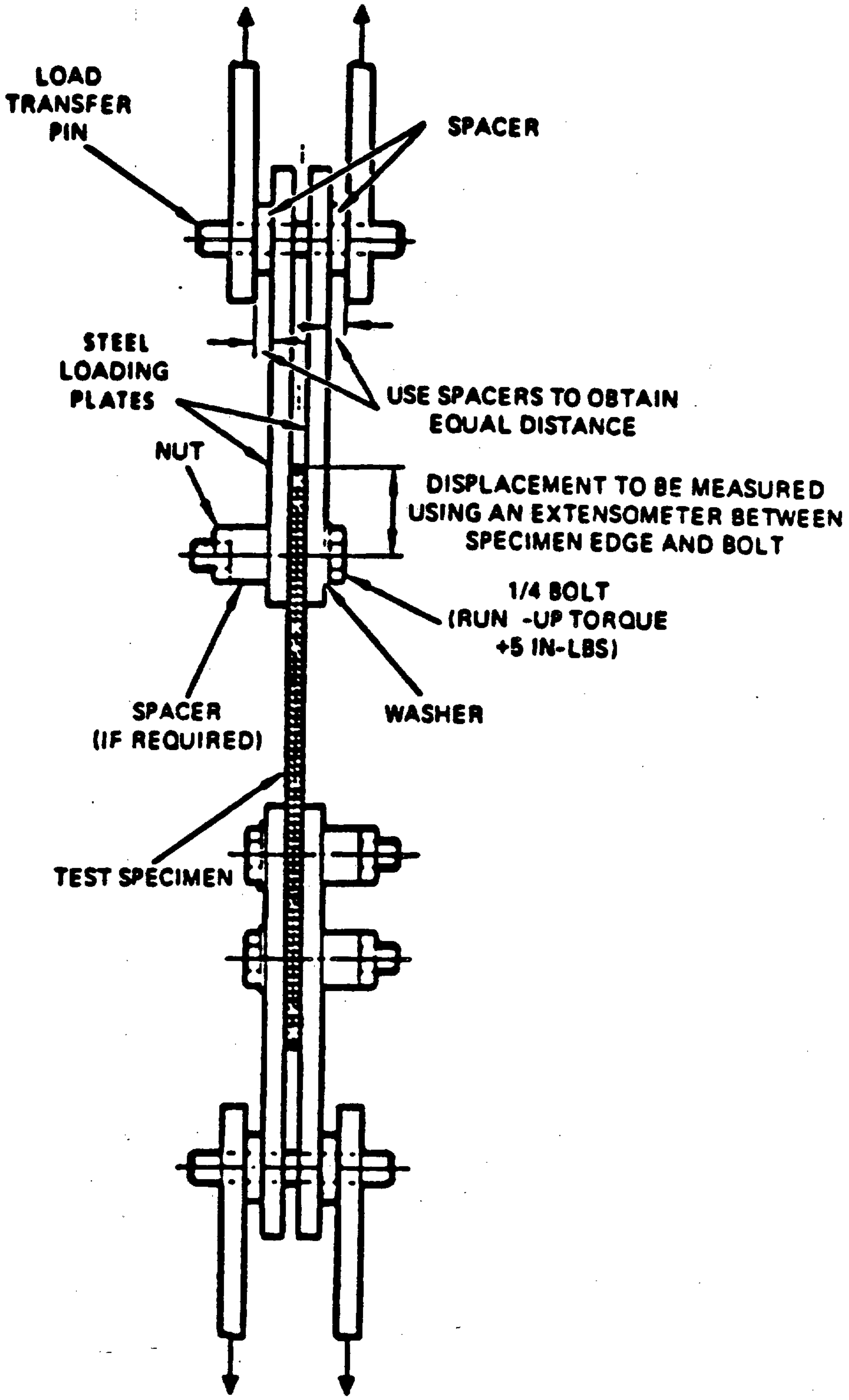
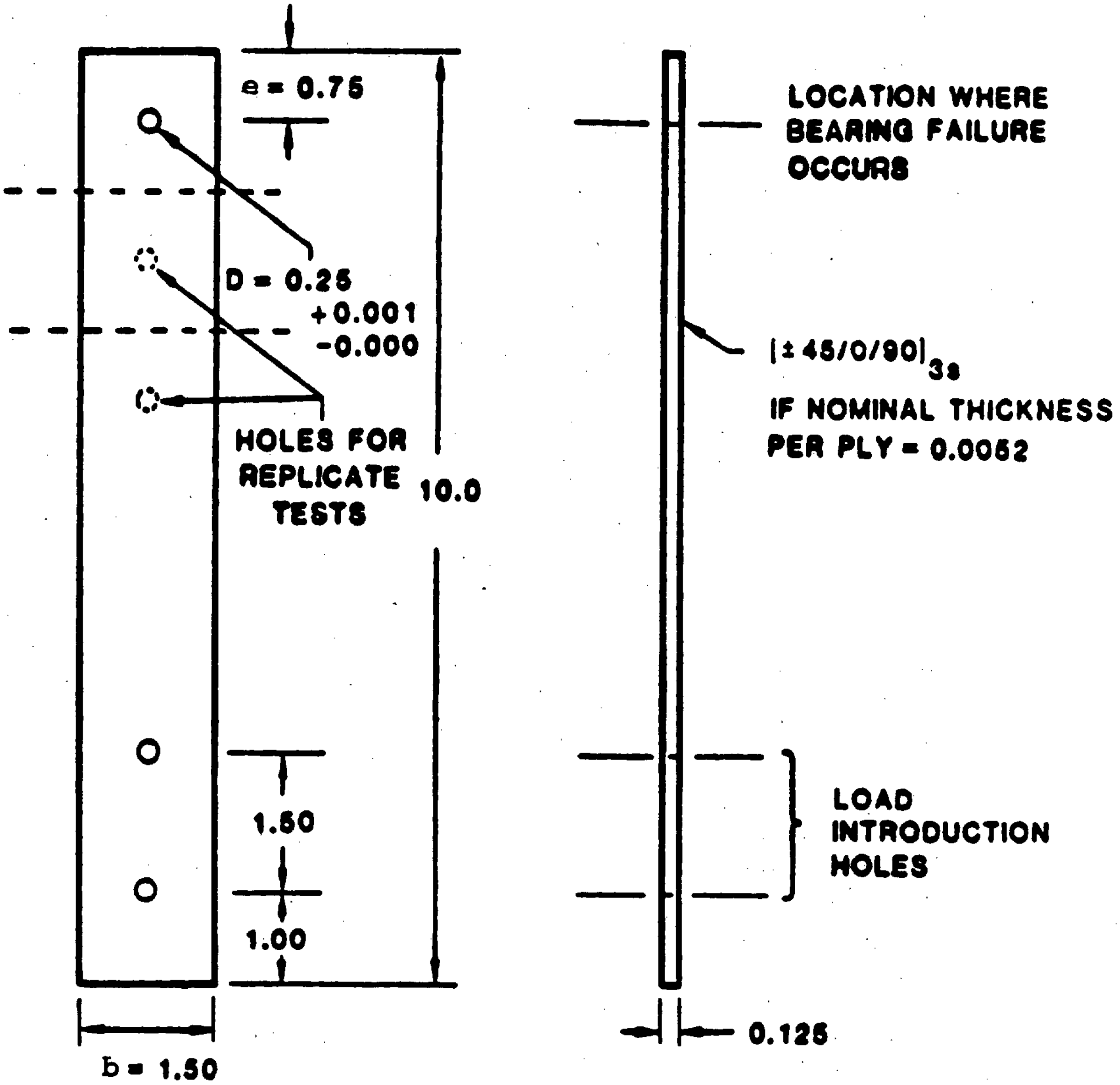


Figure 7.2.3. Test arrangement for bearing strength measurement.



ALL DIMENSIONS IN INCHES

Figure 7.2.4. Test specimen geometry.

7.2.4 Replication requirements. Five replicates shall be tested as a minimum. If three tests are conducted on one specimen, as shown in Figure 7.2.4, a total of six tests on two specimens will suffice.

7.2.5 Test conditions. Tests shall be conducted under as-fabricated or ambient dry or room temperature dry (RTD) conditions, and two hot, wet conditions. The hot, wet tests shall be conducted on specimens after they are preconditioned to near saturation level moisture contents, at temperatures that are dependent on the wet glass transition temperature for the material (wet T_g). Recommended test temperatures for hot, wet tests are T_g - 50°F (T_g - 28°C) and T_g - 20°F (T_g - 11°C).

Measurement of specimen dimensions, test procedures, etc., shall follow ASTM D953 recommendations (Reference 7.2.5). Four percent bearing stress values shall be obtained, as described in ASTM D953, with the help of a template (as described in sections 7.2.6 and 7.2.7).

7.2.6 Determination of the intermediate bearing stress. The intermediate bearing strength of a material is defined as the bearing stress at which the bearing hole is deformed some specified amount. This measurement is complicated by uncertainties in the zero displacement point on the load-displacement curve. The following graphical procedure for finding a representative bearing stress higher than the proportional limit bearing stress has been found to eliminate this ambiguity. It provides a bearing strength measurement which is clearly defined once a deformation measure is specified. This deformation measure is usually specified as a percentage of the hole diameter (commonly 4%).

The method is illustrated in Figure 7.2.6(a). First the load-deflection curve for each specimen must be plotted. Given the bearing load-deflection curve, ABC, the tangent is determined at a point, B, such that when the tangent is projected through the point, E, on the zero load axis, the distance between E and F is equal to four percent of the bearing hole diameter.

A template to facilitate the determination of point B on the curve is shown in Figure 7.2.6(b). It should be designed to fit the coordinate paper upon which the load-deformation curve is drawn in such a way that a "4% line" on the template is established at a distance from the reference point, E, equal to 4% of the bearing hole diameter. It should consist of a thin rigid sheet of transparent plastic upon which the rectangular coordinates are ruled. A strip of transparent plastic can be mounted on the sheet so that it can rotate about point E. The strip should be designed with a reference center line passing through the point of rotation.

In practice, the deformation axis of the template should be superimposed on the zero load axis of the load-deformation curve. This can best be done with the aid of a drawing board and a parallel straightedge. The template should then be slid to the right or left, while in superposition, until the rotating arm can be made tangent to the load-deformation curve at its intersection with the 4% line. The intersection is point B on the curve in Figure 7.2.6(a). B should be projected horizontally to the load axis to read the bearing load.

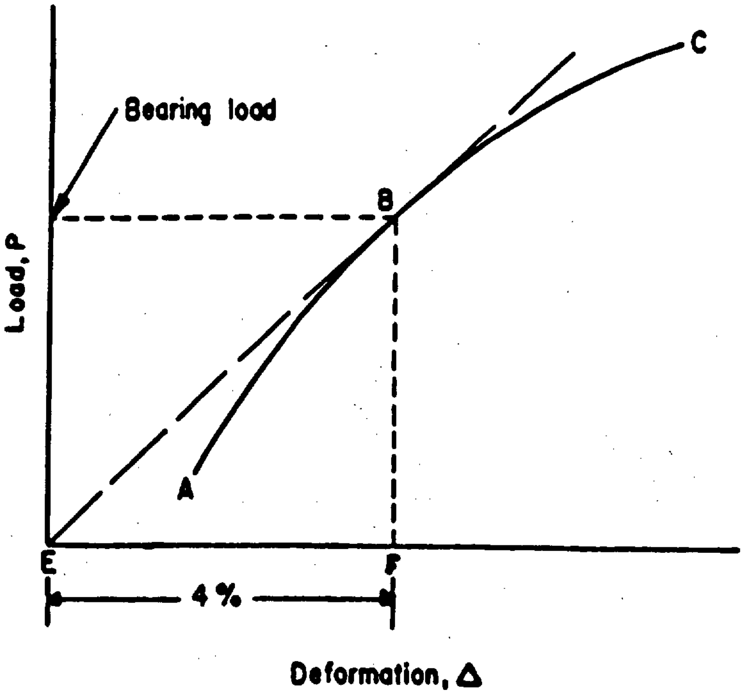


Figure 7.2.6(a) Illustration of method of determining bearing strength from bearing load-deformation curve.

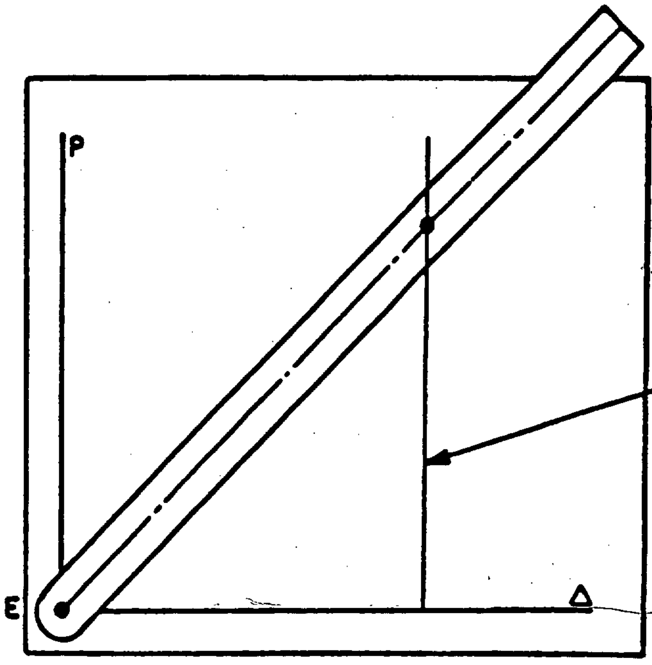


Figure 7.2.6(b). Template for determining point B on bearing load-deformation curve.

7.2.7 Bearing strength calculations. Bearing strength should be calculated using the following equation:

$$F^{br} = P/tD \qquad 7.2.7$$

where

F^{br} = bearing strength, psi (Pa)
P = bearing load at 4 percent hole deformation, lb_f (N)
D = bearing hole diameter, in. (m)
t = specimen thickness, in. (m)

Bearing strength values are reported in MIL-HDBK-17 as typical or average values. Therefore, bearing strength values which are available for each specific condition should be analyzed to produce typical property values as described in Chapter 7.

REFERENCES

7.1.1 DOD/NASA Advanced Composites Design Guide, Air Force Wright Aeronautical Laboratories, Dayton, OH, 1-A (1983).

7.2.5 ASTM D953, "Standard Method of Test for Bearing Strength of Plastics," 1984 Annual Book of ASTM Standards, 8.01, ASTM, Philadelphia, PA (1984).

8. ANALYSIS AND PRESENTATION OF DATA

Page

STATISTICAL TABLES

8.1 General. This section of Chapter 8 covers general information applicable to the sections that follow. Information specific to individual properties is to be found in the pertinent sections.

8.1.1 Introduction. The mechanical properties in MIL-HDBK-17 are used in the design of aerospace structures and elements. Thus it is exceedingly important that the values presented in MIL-HDBK-17 reflect as accurately as possible the actual properties of the materials covered.

The statistical procedures used in determining the materials properties values presented in this handbook are described in Sections 8.5 through 8.8. Section 8.2 presents the definitions for typical and B-basis values and defines the minimum number of batches and specimens to obtain a B-basis property value. Guidelines for the computation of individual mechanical properties are presented in Section 8.3. Section 8.4 describes the procedures and formats for presenting the material property data in Volume II.

8.1.2 Data documentation requirements.

8.1.3 Normalization. The values of fiber-dominated strength and stiffness properties (i.e., 0° tension, 0° compression) are dependent upon the volume fraction of fiber present in the laminate. In typical applications, the strength and stiffness have been found to vary linearly with percent fiber. Properties other than 0° tension and 0° compression strengths and stiffnesses may also be dependent on fiber volume fraction, but not in a well-defined way. For example, interlaminar shear properties are frequently found to remain fairly constant over a given fiber fraction range, but become lower when the fiber fraction falls above or below this range. Normalization is not considered appropriate for such properties.

In order to compare the properties of laminates of different fiber contents, it has become common practice to report values normalized to a given fiber volume fraction. This is accomplished by determining the fiber volume fraction in the cured test laminates by an appropriate method, and then multiplying the raw property values by the factor: (nominal fiber volume fraction)/(determined fiber volume fraction).

It may be shown that, for fiber/fabric of a given areal weight, the per ply thickness depends entirely on the fiber volume fraction. Hence, normalization may also be performed on a cured ply thickness basis. Although the true relationship is of the form, cured ply thickness = K/fiber fraction (where K is a constant), the departure from linearity in the 0.45 to 0.65 fiber volume fraction range is less than 0.1%. The normal thickness is chosen to correspond to a given fiber volume fraction or to the expected thickness under given process conditions.

The thickness method may be particularly useful for fiber reinforcements such as Kevlar(TM), where direct, repeatable methods for determining fiber volume of cured laminates are not well established or easily applied on a routine basis. The thickness method has the added advantage of allowing normalization of each individual test specimen, rather than applying a common fraction to all specimens from a given panel (based on panel fiber volume fraction). The latter approach might be less accurate since the thickness (and hence fiber fraction) will vary throughout the panel. The per ply thickness approach is not considered appropriate for test panels wound from rovings, however, since the per ply thickness is influenced greatly by the wind spacing and roving bandwidth, and may not consistently correspond to a fiber volume fraction.

The recommended data practices are as follows:

1. For 0° tension and 0° compression strengths and stiffnesses, normalized values shall be reported. Normalization may be by fiber volume fraction or thickness as appropriate for the material. The method of normalization shall be stated, and the normalizing factor shall be within the material process specification range. Raw data before normalization shall also be reported.

2. For properties other than those in (1.) above, raw data without normalization shall be reported. The fiber volume fraction and thickness for panels used shall be reported for information.

8.1.4 Symbols. The symbols which are used in Chapter 8, particularly those that are used in Sections 8.5 through 8.8, and not commonly used throughout this handbook are listed below, each with its definition and the section in which it is first used. These are primarily used to denote statistical variables.

SYMBOL	DEFINITION	SECTION
A_i	population	8.6.3.1
ADK	k-sample Anderson-Darling statistic	8.6.3.1
B	B-basis value	8.5.1.1
C.V.	critical value	8.6.2.1
e	error, residual	8.6.4.4
EV	equality of variances test statistic	8.6.3.2
F	F statistic	8.5.8
$F_{0.2}$	0.20 quantile of the F-distribution	8.5.4
IQ	informative quantile function	8.6.5.2
k	number of batches	8.5.4
k^*	total number of observations/root-mean-square number of observations per sample	8.5.4
k_B	one-sided tolerance limit factor	8.5.5.1
MNR	maximum normed residual test statistic	8.6.2.1

SYMBOL	DEFINITION	SECTION
N	total number of observations in several batches	8.5.4
n	number of observations in a sample	8.5.1.1
n'	effective sample size	8.5.4
n^*	see Equation 8.5.4(d)	8.5.4
n_i	number of observations in sample i	8.6.6.2
OSL	observed significance level	8.5.3.4
Q	quantile function	8.6.5.1
$\hat{Q}$	quantile function estimate	8.6.5.1
$q_{0.10}$	quantile of the underlying population distribution	8.5.7.1
R	ratio of between-batch to within-batch variances	8.5.4
$\hat{R}$	upper bound estimate of R	8.5.4
r	rank of observation	8.5.7.1
r_i	normed residual	8.6.2.1
RME	relative magnitude of error	8.8
S	sample standard deviation	8.5.1.1
S^2	sample variance	8.5.1.1
S_b^2	sample between-batch variance	8.5.4
S_e^2	sample within-batch variance	8.5.4
S_y	root-mean-square error of the regression	8.5.8
T	tolerance limit factor	8.5.4

SYMBOL	DEFINITION	SECTION
$t_{\gamma,0.95}(\delta)$	0.95 quantile of the non-central t-distribution with non-centrality parameter δ and degrees of freedom γ	8.5.4
$t_{\gamma,0.95}$	0.95 quantile of the central t-distribution with degrees of freedom γ	8.5.4
TIQ	truncated informative quantile function	8.6.5.2
V	one-sided tolerance limit factor for the Weibull distribution	8.5.6.2
$\bar{X}$	sample mean, overall mean	8.5.1.1
x_i	observation i in a sample	8.5.1.1
x_{ij}	jth observation in sample i	8.6.7
$x_{(r)}$	rth observation, sorted in ascending order, observation of rank r	8.5.7.1
α	(1) significance level	8.8.9
	(2) true intercept of regression equation	8.6.6.1
	(3) scale parameter of Weibull distribution	8.5.6.1
$\hat{\alpha}$	estimate of scale parameter	8.5.6.1
β	(1) true slope of regression equation	8.6.6.1
	(2) shape parameter of Weibull distribution	8.5.6.1
$\hat{\beta}$	estimate of shape parameter	8.5.6.1
γ	degrees of freedom	8.5.4
δ	noncentrality parameter	8.5.4
ϵ	error	8.6.6.1
μ	population mean	8.5.1.1

SYMBOL	DEFINITION	SECTION
σ	population standard deviation	8.6.6.1
σ^2	population variance	8.6.6.1
σ_b^2	population between-batch variance	8.5.4
σ_e^2	population within-batch variance	8.5.4

8.2 Material and specimen requirements.

8.2.1 Statistically-based material properties. The statistically-based properties included in MIL-HDBK-17 are B-basis values. A B-value, as defined in Section 1.7, is the value above which at least 90 percent of the population of values is expected to fall, with a confidence of 95 percent. In order to obtain a B-value by using a nonparametric method as described in Section 8.5.7.1, twenty-nine measurements are required. For the purposes of obtaining a reasonable evaluation of material variation, properties as presented in this handbook are based on a minimum of six measurements from at least five batches of a material. This criterion provides for a minimum of thirty measurements. Fewer measurements can be evaluated according to the statistical methods described in Section 8.5.7.2; these data will be included in the handbook but B-basis values will not be reported for them. B-values are included for all ultimate strength and strain-to-failure properties.

8.2.2 Typical properties. Typical, sample mean, values are presented for all properties in MIL-HDBK-17. Statistical assurance is associated only with the ultimate strength and strain-to-failure.

8.3 Determination of properties.

8.3.1 Introduction. This section outlines the procedures for calculating material property values from the experimental records of the individual specimens. The statistical methods described in Sections 8.5 through 8.8 and the statistical definitions presented in Section 1.7 are used to determine the necessary values.

8.3.2 Mechanical properties.

8.3.2.1 Ultimate stress and strain. The ultimate stress and strain-to-failure will receive the full statistical treatment described in Section 8.5.2 including outlier detection, data pooling testing, and determination of distribution.

8.3.2.2 Transverse tensile properties. A B-basis value will not be reported for transverse strength in tension for unidirectional tape materials but the remaining statistical parameters will be tabulated.

8.3.2.3 Elastic constants and Poisson's ratio. The elastic constants, Young's modulus or shear modulus, shall be collected from each of the experiments, with a minimum of six specimens from each of five batches, for each property. For each experiment, the modulus will be calculated from the slope of the load-deformation curve within the linear portion of the curve or, if the load-deformation curve is nonlinear, the modulus will be calculated from the slope of a secant line between load-deformation points at 0.001 in/in (m/m) and 0.003 in/in (m/m) strain. The results will be examined for batch-to-batch variation and pooled if possible. Minimum and maximum values will be reported in Volume II. If stress-strain data are provided for each experiment, the data can be fit by algebraic functions as described in Section 8.3.7. Poisson's ratio values will be determined for the same range of data as the modulus values. Typical values of Poisson's ratios determined from the thirty experiments for tensile and compressive loading conditions will be included in the data summary for a given material.

8.3.3 Chemical properties.

8.3.4 Physical properties. The following requirements have been established for determining physical properties from experimental results and reporting those properties in MIL-HDBK-17.

8.3.4.1 Density. The density will be reported for each batch used in the experiments to determine any of the mechanical properties. Typical density values will be part of the material description presented with each set of data.

8.3.4.2 Composition.

8.3.4.3 Water absorption. The equilibrium moisture content for the relative humidity values from Tables 2.2.4(a) and (b) will be determined from a minimum of three specimens. This value will be presented in the data summary for each material. If additional information is available for moisture content as a function of temperature and relative humidity, those values will be presented graphically.

Values for the moisture expansion coefficient will be treated in the same way as those for the thermal expansion coefficient.

8.3.4.4 Glass transition temperature. The glass transition temperature will be determined from a minimum of three measurements per dry and wet test condition. This information will be a basis for determining the maximum use temperature.

8.3.5 Thermal properties. The coefficient of linear thermal expansion will be determined from a minimum of five measurements at each condition. Room temperature values will be listed in the data summary and additional values of the thermal expansion coefficient will be presented graphically as a function of temperature.

The specific heat and the thermal conductivity will be presented in a similar fashion. When data are adequate to present curves showing specific heat, thermal conductivity, and mean coefficient of thermal expansion over a range of temperatures, graphical presentation is used in addition to tabular presentation. A smooth curve is drawn through the plotted points to depict the overall trend of the data. The smooth curves for specific heat, thermal conductivity, and thermal expansion are then shown in a single figure as shown in Figure 8.3.5. The reference temperature for thermal expansion should be shown on the figure. In Figure 8.3.5, the reference temperature of 70°F (21°C) indicates that the mean coefficient of expansion between 70°F (21°C) and the indicated temperature is plotted.

8.3.6 Electrical properties.

8.3.7 Typical stress-strain curves.

FIGURE 8.3.5. Thermal properties as a function of temperature.

8.4 Presentation of data. All data is presented in Volume II of MIL-HDBK-17B. This section describes how the data is presented and organized in that volume.

8.4.1 Properties and definitions. The properties and their definitions are found in the appropriate chapters of Volume I. Fiber properties and methods for obtaining them are discussed in Chapter 3. Resin properties are presented in Chapter 4. Methods for characterizing prepreg materials are discussed in Chapter 5 and properties and definitions for laminae and laminates are presented in Chapter 6. The statistical methods used in determining these properties are discussed in this chapter. Material system codes and laminate orientation codes are defined in Chapter I of Volume II.

8.4.2 Organization of data in handbook. The data in Volume II is divided into chapters of fiber properties, resin properties, and composite properties organized by fiber and then resin.

8.4.2.1 Fiber properties. Chapter 2 in Volume II provides data for fiber properties. Sections are included for different types of fiber, e.g. glass fibers and carbon fibers. In each section, the general characteristics of the type of fiber are given, as well as an index of suppliers, designations, and abbreviations. For each specific fiber, data are organized in the following manner. The X's in the subsection number are determined by the type of fiber and the specific fiber described.

2.X.X.1	Supplier and product data
2.X.X.2	Chemical and physical properties
2.X.X.2.1	Typical range of chemical constituents
2.X.X.2.2	Expected bound in physical properties
2.X.X.3	Thermal-mechanical properties
2.X.X.3.1	Stress-strain curves
2.X.X.3.2	Environmental effects

8.4.2.2 Matrix properties. Matrix or resin properties are included in Chapter 3 which is divided into sections according to the type of resin. For example, section 3.2 gives data for epoxies and section 3.3 provides data for polyester resins. The subsections for each specific resin are the same as those in Chapter 2 given above.

8.4.2.3 Composite properties. The remaining chapters of Volume II provide data for prepreg, lamina, laminate, and joint properties. There are individual chapters for each family of composites based on fiber type. For example, Chapter 4 describes glass fiber composites. Within each chapter, there is an index of suppliers, designations, and abbreviations. Sections are included based on the resin type used with the fiber described in the chapter, e.g. section 4.3 provides properties for epoxy-glass composites.

Properties are organized in the following manner for each specific composite:

X.X.X.1	Supplier and product data
.2	Prepreg chemical and physical properties
.2.1	Physical description
.2.2	Resin content
.2.3	Fiber content

X.X.X.2.4 Volatiles content
.2.5 Moisture content
.2.6 Inorganic fillers and additives content
.2.7 Areal weight
.2.8 Tack and drape
.2.9 Resin flow
.2.10 Gel time
.3 Lamina chemical properties
.4 Lamina physical properties
.5 Lamina mechanical properties
.5.1 Data summaries
.5.2 Typical stress-strain curves
.6 Thermal properties
.7 Electrical properties
.8 Laminate thermal-mechanical properties
.8.1 Index of properties by lay-up
.8.2 Strength properties
    a. Lay-up No. 1
    b. Lay-up No. 2
.8.3 Thermal properties
.8.4 Electrical properties
.6 Joint thermal-mechanical properties
.6.1 Index of properties by joint and composite system
.6.2 Bearing strength
    a. System No.1
    b. System No. 2

8.4.3 Sample summary tables.

8.4.4 Sample graphs.

8.5 Calculation of statistically-based material properties.

8.5.1 Introduction. Section 8.5 contains computational methods for computing B-basis values from composite material failure data. In Section 8.5.1.1, definitions are provided for a number of statistical terms which are used frequently in these guidelines. A discussion of the data requirements for publishing a B-basis value in MIL-HDBK-17 is presented in Section 8.5.1.2.

A step-by-step procedure for selecting the appropriate computational method is outlined in Section 8.5.2 with a flowchart showing the steps which must be taken. Section 8.5.3 contains procedures which evaluate several different statistical models and determine which, if any, of the models adequately describes the data. These procedures include methods for detecting outliers, for testing the compatibility of several batches of data, and for investigating the form of the underlying population from which a sample is drawn.

There are various methods for computing B-basis values. The assumption of a statistical model for the data determines which of these methods should be used. Sections 8.5.4 through 8.5.8 contain methods for computing B-basis values under the various models. Statistical tools used in these methods, as well as supplemental methods and tables, are presented in Section 8.6.

Finally, Section 8.7 contains examples illustrating the computational methods. Illustrative sets of data are used to demonstrate the steps that must be followed and the calculations which must be performed in calculating B-basis values.

8.5.1.1 Statistical terms. Proper use of the following statistical terms and equations will do much to alleviate misunderstanding in the presentation of data analyses. Definitions for other statistical terms are included in Section 1.7.

Population. - The set of measurements about which inferences are to be made or the totality of possible measurements which might be obtained in a given testing situation. For example, "all possible ultimate tensile strength measurements for Hexcel, conditioned at 95% relative humidity and room temperature". In order to make inferences about a population, it is often necessary to make assumptions about its distributional form. The assumed distributional form may also be referred to as the population.

Sample. - The collection of measurements or observations taken from a specified population.

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.

Location parameters and statistics:

Population mean. - The average of all potential measurements in a given population weighted by their relative frequencies in the population. Also the limit of the sample mean as the sample size increases.

Sample mean. - The average of all observations in a sample and an estimate of the population mean. If the notation $X_1, X_2, \ldots, X_n$ is used to denote the n observations in a sample, then the sample mean is expressed as:

$$\bar{X} = \frac{X_1 + X_2 + \ldots + X_n}{n} \qquad 8.5.1.1(a)$$

or

$$\bar{X} = \frac{\sum_{i=1}^{n} X_i}{n} \qquad 8.5.1.1(b)$$

Sample median. - After ordering the observations in a sample from least to greatest, the sample median is the value of the middle-most observation if the sample size is odd and the average of the two middle-most observations if the sample size is even. If the population is symmetric about its mean, the sample median is also a satisfactory estimator of the population mean.

Dispersion statistics:

Sample variance. - The sum of the squared deviations from the sample mean, divided by n - 1, where n denotes the sample size. The sample variance is expressed as:

$$S^2 = \frac{\sum_{i=1}^{n} (X_i - \bar{X})^2}{n - 1} \qquad 8.5.1.1(c)$$

or

$$S^2 = \frac{n \sum_{i=1}^{n} (X_i)^2 - (\sum_{i=1}^{n} X_i)^2}{n (n - 1)} \qquad 8.5.1.1(d)$$

Sample standard deviation. - The square root of the sample variance. The sample standard deviation is denoted by S.

Distribution terms:

Distribution. - A formula which gives the probability that a value will fall within prescribed limits.

Normal Distribution - A two parameter (μ,σ) family of probability distributions for which the probability that an observation will fall between a and b is given by the area under the curve

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} e^{-(x-\mu)^2/2\sigma^2} \qquad 8.5.1.1(e)$$

between a and b.

Lognormal Distribution - A probability distribution for which the probability that an observation selected at random from this population falls between a and b ($0 < a < b < \infty$) is given by the area under the normal distribution between ℓog a and ℓog b.

Weibull Distribution - A probability distribution for which the probability that a randomly selected observation from this population lies between a and b ($0 < a < b < \infty$) is given by

$$e^{-(a/\alpha)^{\beta}} - e^{-(b/\alpha)^{\beta}} \qquad 8.5.1.1(f)$$

where α is called the scale parameter and β is called the shape parameter.

Probability function terms:

Cumulative Distribution Function - A function, usually denoted by F(x), which gives the probability that a random variable lies between any prescribed pair of numbers, that is

$$P\{\, a < x \le b \,\} = F(b) - F(a) \qquad 8.5.1.1(g)$$

Such functions are non-decreasing and satisfy

$$\lim_{x \to -\infty} F(x) = 0 \quad \text{and} \quad \lim_{x \to \infty} F(x) = 1$$

The cumulative distribution function is related to the probability density function by

$$f(x) = \frac{d}{dx} F(x) \qquad 8.5.1.1(h)$$

provided that F(x) is differentiable.

F-distribution - A probability distribution which is employed in the analysis of variance, regression analysis, and tests for equality of variance. Tables of this distribution are readily available.

Failure Rate - see Hazard Rate.

Hazard Rate - If F(x) is the cumulative distribution function, and f(x) is the corresponding probability density function, then the hazard rate, h(x), is defined by

$$h(x) = \frac{f(x)}{1 - F(x)} \qquad 8.5.1.1(i)$$

The above formula has the following intuitive interpretation. h(x) is proportional to the probability of "instantaneous failure" at time x, given that a failure has not occurred by time x, where F(x) is the probability that a failure occurs on or before time x.

Probability Density Function - A function $f(x) \leq 0$ for all x and

$$\int_{-\infty}^{\infty} f(x)\, dx = 1 \qquad 8.5.1.1(j)$$

The probability density function determines the cumulative distribution function F(x) by

$$F(x) = \int_{-\infty}^{x} f(t)\, dt \qquad 8.5.1.1(k)$$

Note that the limits $(-\infty, \infty)$ may be conventional; that is, a random variable with a restricted range, for example, the exponential random variable which assumes only positive values satisfies the definition by defining its probability density function as

$$f(x) = \begin{cases} 0 & x \leq 0 \\ e^{x} & x > 0 \end{cases} \qquad 8.5.1.1(l)$$

The probability density function is used to calculate probabilities as follows:

$$P\ (\ a < x \leq b\ ) = \int_{a}^{b} f(x)\, dx \qquad 8.5.1.1(m)$$

8.5.1.2 Sample size requirements. The sample size requirements for publishing a B-basis value in MIL-HDBK-17 depend upon the status of material and process specifications for the subject composite material system. If no standard specifications have been developed for the material system, a minimum of five batches of material and six specimens per batch are required for each fabricator that wishes to publish a B-basis value in MIL-HDBK-17.

In the event that standard material and process specifications have been developed for a composite material system, several fabricators may jointly develop data to publish a single B-basis value in MIL-HDBK-17. In this case, a minimum of three fabricators, three batches of material per fabricator, and six specimens per batch are required.

The sample specimens should be obtained from randomly selected areas of randomly selected sheets of material fabricated to the material specification for which the B-basis value is desired. The number of specimens from each batch should be as nearly equal as possible with the largest batch size being no more than one and a half times the smallest batch size.

It should be noted that the magnitude of a B-basis value is a function of the amount of data obtained, the number of batches represented, and the uniformity of the batches produced. In general, B-basis values will tend to increase if the number of batches is increased or the number of specimens per batch is increased. B-basis values will also tend to increase if the batch-to-batch variability or the within-batch variability is reduced.

If the minimum data requirements specified above cannot be met, these guidelines may be used to calculate "preliminary" B-basis values. These data will be included as interim data in MIL-HDBK-17 but the B-basis values will not be reported in the handbook until the minimum data requirements are met.

8.5.2 Definition of computational procedures. The procedure used to determine a B-basis value depends on the characteristics of the data. The step-by-step procedure for selecting the appropriate computational method is illustrated by the flowchart in Figure 8.5.2. Details for the specific computational methods are provided in later sections.

If the sample is made up of data from several different batches, each of the batches should be screened for outliers by the method described in Section 8.5.3.1. One should then determine whether or not the data from the different batches should be analyzed as one sample by employing the k-sample Anderson-Darling test as specified in Section 8.5.3.2. If the k-sample test does not

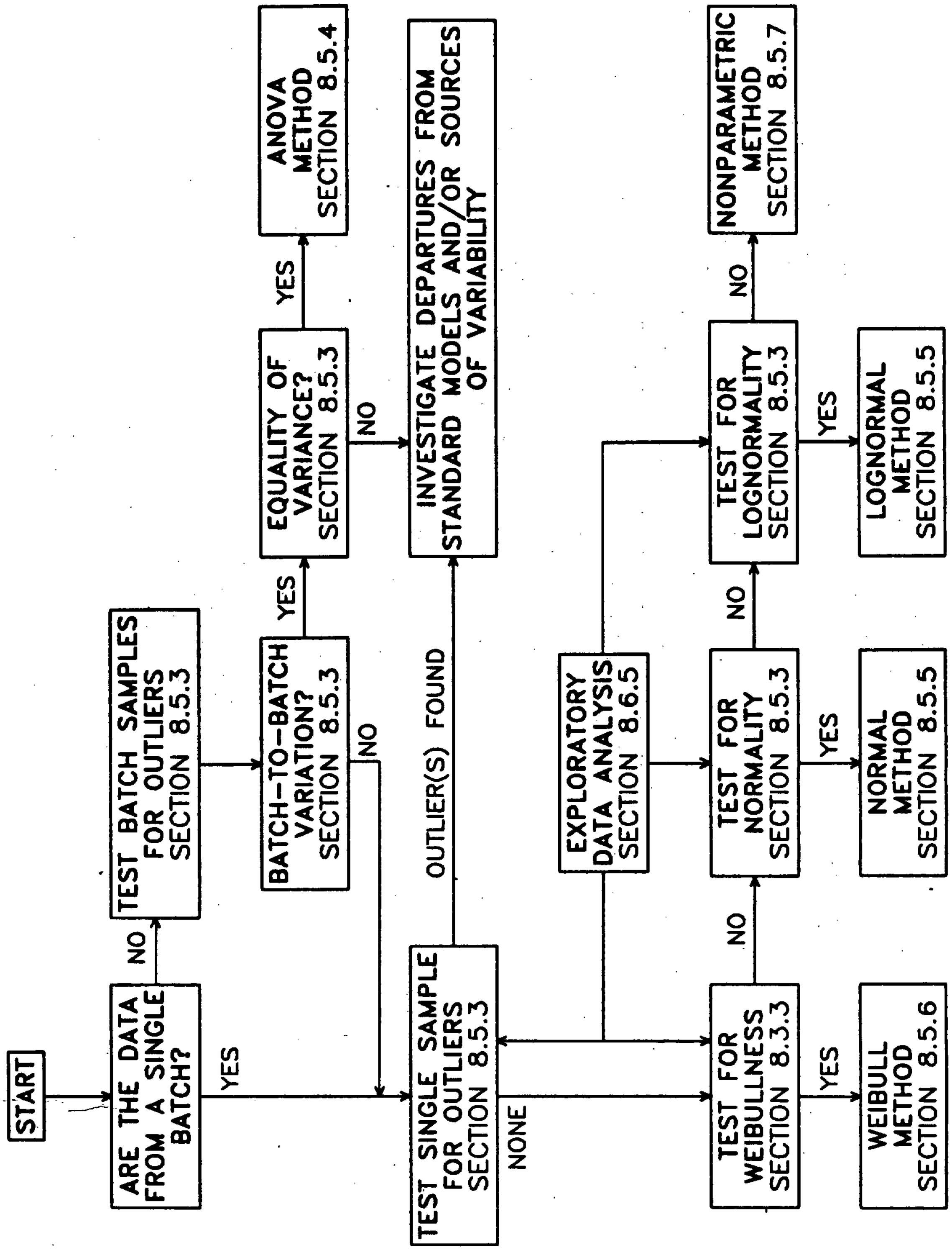


Figure 8.5.2 Flow chart illustrating computational procedures for B-basis material property values.

reject the hypothesis that the batches are from the same population, the batches should be combined and the data should be analyzed as a single sample.

If the hypothesis that the batches are from the same population is rejected, indicating significant batch-to-batch variability, then the B-basis value should be computed by the ANOVA Method of Section 8.5.4, provided that it can be assumed that the data in each batch are normally distributed and that the within-batch variances are equal. A test for the latter assumption is discussed in Section 8.5.3.3. If either of these assumptions appears to be invalid, then there is currently no approved method for computing a B-basis value.

If the sample represents a single batch of material, or several batches which are to be analyzed as a single sample, the single sample of data should be screened for outliers as discussed in Section 8.5.3.1. Note that if only a single batch is represented, then the B-basis value should only be considered a "preliminary" value. One should then determine which, if any, distributional form will be assumed in calculating the B-basis value. The use of goodness-of-fit tests for the two-parameter Weibull, normal, and lognormal distributions is described in Section 8.5.3.4. These tests establish the degree to which the underlying population may be fitted by one of these distributions. The two-parameter Weibull distribution is tested first. If it does not adequately fit the data, then the normal distribution is tested. If neither of these provide an adequate fit, then the lognormal distribution is tested. Exploratory data analysis (EDA) techniques, described in Section 8.6.5, can provide graphical illustrations of the distribution of the sample in support of the goodness-of-fit tests.

If one of the distributions mentioned above provides a good fit to the data, the B-basis value is computed by the corresponding two-parameter Weibull, normal, or lognormal method as described in Sections 8.5.5 or 8.5.6. If none of the distributions adequately fit the data, and the sample contains 29 or more observations, the nonparametric method described in Section 8.5.7.1 is used to compute the B-basis value. If none of the distributions fit the data adequately, and the sample size is less than 29, the Hanson-Koopmans method, described in

Section 8.5.7.2, may be employed. This method, however, does not produce an approved B-basis value due to the small sample size.

For strength measurements measured in thousands of pounds per square inch (ksi), data are generally measured to the nearest tenth of a ksi. B-basis values are generally rounded to ksi units by the following rule. Values with a fractional part less than 0.75 are rounded down to the next lowest integer. B-basis values with a fractional part greater than or equal to 0.75 are rounded up to the next highest integer.

8.5.3 <u>Detecting outliers and characterizing the population</u>. Before a B-basis value is computed, the data must be screened for erroneous values and the structure of the population from which the data were sampled must be investigated in order to determine the appropriate method. The methods discussed in this section address these two issues. These methods include the maximum normed residual outlier test (Section 8.5.3.1), the k-sample Anderson-Darling test (Section 8.5.3.2), an equality of variance test (Section 8.5.3.3), and goodness-of-fit tests for the two-parameter Weibull, normal, and lognormal distributions (Section 8.5.3.4).

8.5.3.1 <u>Detecting outliers</u>. An observation is said to be an outlier if it is an observation that has been recorded in error. For example, an erroneous observation could be the result of clerical error or the incorrect setting of environmental conditions during testing. Since outliers may have a substantial influence on the statistical analysis of the data, it is mandatory that the data be screened for outliers prior to the calculation of a B-basis value.

Initially, the data is subjected to the statistical outlier procedure discussed in Section 8.6.2, using a significance level of 0.05, to identify potential outliers. The test screens for outliers in a single sample. When different batches are represented, the data from each batch should be tested separately. Since the outlier detection procedure may miss erroneous observations, the sample should also be visually inspected for observations suspected of being outliers.

If the statistical outlier procedure or the visual inspection identifies an observation as a potential outlier, the observation will be investigated to ascertain if a cause for the outlying value can be ascribed. If it can be corrected, as is often the case for clerical errors, this should be done. If it is determined to be an erroneous observation and it cannot be corrected, as might be the case with improper setting of environmental conditions during testing, then it is discarded. If no cause can be determined for the outlying value, it must be retained in the data set.

When errors in data collection and recording are discovered, it should be determined whether similar errors occurred for other data points that were not identified as potential outliers and these values should also be corrected or discarded. If any observations are corrected or discarded, both the statistical outlier procedure and the visual inspection should be repeated.

8.5.3.2 The k-sample Anderson-Darling test. The k-sample Anderson-Darling test is used to test the hypothesis that the mechanical property data from different batches are independent random samples from the same population. The procedure for performing this test is presented in Section 8.6.3.1. If the test statistic is less than the critical value from Equation 8.6.3.1(j), then the batches should be treated as a single sample and one should proceed to the goodness-of-fit tests for investigating the form of the distribution discussed in Section 8.5.3.4.

If the test statistic is greater than or equal to the critical value, it is concluded that the batches are not identically distributed and the test for equality of variance should be performed. This test is discussed in the next section.

8.5.3.3 Equality of variance test. The ANOVA method (Section 8.5.4) is derived under the assumption that the variances within each batch are equal. The recommended procedure for testing this assumption is presented in Section 8.6.3.2. If the test does not reject the hypothesis that the variances are equal, then the ANOVA method should be used to compute the B-basis value.

If the test rejects the equality of variance assumption, then there is currently no approved method for computing a B-basis value. In this case, it is recommended that an investigation of the reason for the non-equal variances be carried out. This may reveal problems in the generation of the data or in the fabrication of the material.

8.5.3.4 Investigating the form of the distribution. The method employed in calculating the B-basis value for a single sample depends on the distributional form which is assumed for the data. The most frequently used parametric procedures involve the two-parameter Weibull, normal, or lognormal distributions. Section 8.6.4 contains procedures for performing a goodness-of-fit test for each of these distributions. These goodness-of-fit tests yield an observed significance level (OSL) for each of the distributions listed above. The OSL from each of the tests measures the probability of observing an Anderson-Darling statistic as extreme as the value calculated assuming that the given distribution is the correct one.

To determine which method should be used in calculating the B-basis value, the goodness-of-fit tests should be employed in the following manner. The two-parameter Weibull distribution is tested first. If the OSL is larger than 0.05, the Weibull distribution method of Section 8.5.6 should be used to compute the B-basis value. If the OSL is less than or equal to 0.05, the hypothesis that the data is a sample from a normal distribution is tested next. If this test for normality gives an OSL larger than 0.05, the normal distribution method of Section 8.5.5 should be used to compute the B-basis value. If the normal OSL is less than or equal to 0.05, the lognormal distribution is tested next. If this results in an OSL greater than 0.05, the lognormal distribution method of Section 8.5.5 should be used to compute the B-basis value. If this OSL is less than or equal to 0.05, it is concluded that the data is not a sample from any of these families of parametric distributions, and the nonparametric procedure of Section 8.5.7.1 should be used to compute the B-basis value, provided that the sample size is greater than or equal to 29. If the sample size is less than 29, then the Hanson-Koopmans method, described in Section 8.5.7.2, may be employed.

The exploratory data analysis (EDA) techniques of Section 8.6.5 may be used to graphically display the distribution of measurements in the sample and to provide graphical evidence for the conclusions obtained through the goodness-of-fit tests.

8.5.4 Normal analysis procedure in the presence of batch-to-batch variation. When failure data from different batches are to be analyzed, it is possible that the variation from batch to batch will be significant. This will prevent pooling the data and using the methods of Section 8.5.5, 8.5.6 or 8.5.7 to compute B-basis values from a single sample. This section contains a method, referred to as the ANOVA method, for computing B-basis values when there is significant batch-to-batch variation. It is a modification of a method suggested in References 8.5.4(a) and 8.5.4(b).

The method is based on the one-way analysis of variance (ANOVA) random effects model discussed in Section 8.6.7. The assumptions are that

(1) the data from each batch are normally distributed,
(2) the within-batch variance is the same from batch to batch, and
(3) the batch means are normally distributed.

The second assumption should be validated by performing the test described in Section 8.6.3.2. There is generally insufficient data to validate the third assumption and therefore no test of this assumption is required. If the between-batch variance is larger than the within-batch variance, i.e., if S_b^2 is larger than S_e^2, then there may be differences in fabrication process that warrant investigation.

In the analysis, all batches are treated the same, and no distinction is made between batches from different fabricators. For this reason, the sample size requirements in Section 8.5.2 require that data from at least three fabricators be included in the sample, with at least three batches from each fabricator. This attempts to insure that any variability among fabricators, while not explicitly estimated, will be included in the analysis via its contribution to the batch-to-batch variability.

In what follows, X_{ij} denotes the jth data value in the ith batch, n_i denotes the sample size of batch i, k is the number of batches, and N is the total number of data values ($N = n_1 + n_2 + \ldots + n_k$). If the entire set of data values is not available, means, standard deviations, and sample sizes for the batches are sufficient to perform the calculations.

Computational Procedure. The first step in computing the B-basis value is to compute estimates of the between-batch and within-batch variances (S_b^2 and S_e^2, respectively). Computation of the intermediate values MSB and MSE, is discussed in Section 8.6.7.2. The within-batch variance (σ_e^2) is estimated by

$$S_e^2 = MSE \qquad 8.5.4(a)$$

The between-batch variance (σ_b^2) is estimated by

$$S_b^2 = (MSB - MSE)/n' \qquad 8.5.4(b)$$

where

$$n' = (N-n^*)/(k-1) \qquad 8.5.4(c)$$

and

$$n^* = \sum_{i=1}^{k} \frac{n_i^2}{N} \qquad 8.5.4(d)$$

If (MSB-MSE)/n' is negative, then set S_b^2 equal to 0. For more information on estimation procedures for the one-way random effects model, see Reference 8.5.4(c).

The quantity n' may be thought of as the "effective sample size" when the number of specimens in each batch is not the same. When the number of specimens per batch is the same, n' is equal to the common sample size, n.

The ratio of the between-batch to within-batch variances ($R = \sigma_b^2/\sigma_e^2$) must also be estimated. An upper bound for this ratio is estimated by the quantity $\hat{R}$, which is computed as

$$\hat{R} = [(MSB/MSE)/F_{0.2} - 1]/n' \qquad 8.5.4(e)$$

where $F_{0.2}$ is the 0.20 quantile of an F distribution with k - 1 numerator degrees of freedom and N - k denominator degrees of freedom. $\hat{R}$ is an upper 80 percent confidence bound on the true ratio. If $\hat{R}$ is negative, then set $\hat{R}$ equal to zero. Table 8.8.4 contains the $F_{0.2}$ values required in Equation 8.5.4(e).

The formula for the B-basis value is then

$$B = \bar{X} - T(S_b^2 + S_e^2)^{0.5} \qquad 8.5.4(f)$$

where $\bar{X}$ is the overall mean (average of all N measurements) defined in Equations 8.6.7.1.1(a) and 8.6.7.1.2(a), and T is a tolerance limit factor defined below and tabulated in Tables 8.8.6.

The tolerance limit factor T is defined in statistical terms as

$$T = \{(n^*\hat{R}+1)/[N(\hat{R}+1)]\}^{0.5}\ t_{\gamma,0.95}(\delta) \qquad 8.5.4(g)$$

where $t_{\gamma,0.95}(\delta)$ is the 0.95 quantile of the non-central t distribution with noncentrality parameter

$$\delta = 1.282\ [N(\hat{R} + 1)/(n^*\hat{R} + 1)]^{0.5} \qquad 8.5.4(h)$$

and degrees of freedom approximated by

$$\gamma = \frac{(\hat{R} + 1)^2}{\dfrac{(\hat{R} + \frac{1}{n*})^2}{k* - 1} + \dfrac{(\frac{n*-1}{n*})^2}{k*(n* - 1)}} \qquad 8.5.4(i)$$

where $k^* = N/n^*$. T may be computed from the following approximate formula:

$$T = 1.282 + 1.282\,(t_{\gamma,0.95}/\delta) + 1.80\,(1/\gamma) - 1.85\,(1/\gamma^2) + 0.567\,(\delta/\gamma) + 5.24\,(\delta/\gamma^2) - 1.08\,(\delta^2/\gamma^2) + 0.0166\,(\delta^3/\gamma^2) + 7.79\,(1/\gamma^4) \qquad 8.5.4(j)$$

(Note: $t_{\gamma,0.95}$ is the 0.95 quantile of the central t-distribution.) Tolerance limit factors are tabulated in Table 8.8.6 for various values of n^*, k^* and $\hat{R}$.

Equations 8.5.4(e) through 8.5.4(i) were developed under the assumption of equal sample sizes. The performance of the method is not affected substantially by unequal sample sizes as long as the largest batch size is no more than one and one half times the smallest batch size. If sample sizes are equal, then $k^* = k$ and $n^* = n$.

8.5.5 Normal analysis procedure for a single population. This procedure should be used when a B-basis value is to be computed from a single sample of failure data that is assumed to be normally distributed. See Section 8.5.1.1 for a definition of the normal distribution. Section 8.5.5.1 contains the computational procedure and Section 8.5.5.2 describes how this procedure is applied to lognormal data. Further information on this procedure may be found in Reference 8.5.5.

8.5.5.1 Computational procedure. In order to compute B-basis values for a normal population, it is first necessary to obtain estimates of the population mean and standard deviation. The sample mean and standard deviation, $\bar{X}$ and S, are computed from the sample of available measurements using the equations presented in Section 8.5.1.1.

The B-basis value is then calculated by use of the following formula:

$$B = \bar{X} - k_B S \qquad 8.5.5.1$$

where

$\bar{X}$ = the sample mean based on n observations
S = the sample standard deviation
k_B = the one-sided tolerance-limit factor from Table 8.8.1.

A numerical approximation to the k_B values is given in Equation 8.8.1.

8.5.5.2 Lognormal procedure. If the sample is assumed to follow a lognormal distribution, then the computational procedure presented in Section 8.5.5.1 is used to calculate the B-basis value. However, the calculations are performed using the logarithms of the data rather than the original observations. Either the natural or the common logarithm may be used. The computed B-basis value must then be transformed back to the original units by applying the inverse of the log transformation which was used. See Section 8.5.1.1 for definition of the lognormal distribution.

If, for example, the natural logarithm is used to transform the data, the B-basis value would be calculated as follows:

$$B = \exp(\bar{X}_L - k_B S_L) \qquad 8.5.5.2(a)$$

where

$$\bar{X}_L = \frac{\sum_{i=1}^{n} \ell n(X_i)}{n} \qquad 8.5.5.2(b)$$

$$S_L = \left[\frac{\sum_{i=1}^{n} [\ell n(X_i) - \bar{X}_L]^2}{n-1}\right]^{0.5} \qquad 8.5.5.2(c)$$

and k_B is the one-sided tolerance-limit factor from Table 8.8.1.

8.5.6 Two-parameter Weibull analysis procedure for a single population. This procedure should be used when a B-basis value is to be computed from a single sample of failure data which is assumed to follow a two-parameter Weibull distribution. See Section 8.5.1.1 for a definition of the two-parameter Weibull distribution.

In order to compute a B-basis value for a two-parameter Weibull population, it is first necessary to obtain estimates of the population shape and scale parameters. Section 8.5.6.1 contains a step-by-step procedure for calculating maximum likelihood estimates of these parameters. The computational procedure for calculating B-basis values using these estimates and the tolerance limit factors in Table 8.8.2 is outlined in Section 8.5.6.2. For further information on these procedures, see Reference 8.5.6.

8.5.6.1 Estimating the shape and scale parameters of a Weibull distribution. In order to calculate the B-basis value for a random sample from the two-parameter Weibull distribution, estimates of the shape and scale parameters are needed. The procedure described below is the maximum likelihood method, and the estimates obtained are known as the maximum likelihood estimates. The shape parameter estimate is denoted by $\hat{\beta}$, and the scale parameter by $\hat{\alpha}$. The estimates are obtained by solving the pair of likelihood equations given below for $\hat{\beta}$ and $\hat{\alpha}$.

$$\alpha \beta n - \frac{\hat{\beta}}{\hat{\alpha}^{\hat{\beta}-1}} \sum_{i=1}^{n} X_i^{\hat{\beta}} = 0 \qquad 8.5.6.1(a)$$

$$\frac{n}{\hat{\beta}} - n \ln \hat{\alpha} + \sum_{i=1}^{n} \ln X_i - \sum_{i=1}^{n} \left(\frac{X_i}{\hat{\alpha}} \right)^{\hat{\beta}} (\ln X_i - \ln \hat{\alpha}) = 0 \qquad 8.5.6.1(b)$$

Equation 8.5.6.1(a) can be rewritten as

$$\hat{\alpha} = \left(\frac{\sum_{i=1}^{n} X_i^{\hat{\beta}}}{n} \right)^{1/\hat{\beta}} \qquad 8.5.6.1(c)$$

By substituting Equation 8.5.6.1(c) into Equation 8.5.6.1(b), the following equation is obtained.

$$\frac{n}{\hat{\beta}} + \sum_{i=1}^{n} \ln X_i - \frac{n}{\sum_{i=1}^{n} X_i^{\hat{\beta}}} \sum_{i=1}^{n} X_i^{\hat{\beta}} \ln X_i = 0 \qquad 8.5.6.1(d)$$

Since Equation 8.5.6.1(d) depends only on the data $X_1, X_2, \ldots, X_n$, it can be solved numerically, and the solution of $\hat{\beta}$ that is obtained is substituted into Equation 8.5.6.1(c) to obtain $\hat{\alpha}$, the estimate of α.

Figure 8.5.6 shows FORTRAN source code for three routines which compute the estimates of $\hat{\alpha}$ and $\hat{\beta}$ by the method described above. WBLEST is a subroutine which returns the estimates of the parameters, $\hat{\beta}$ and $\hat{\alpha}$. FNALPH is a function which calculates the estimate of the scale parameter, $\hat{\alpha}$. GFUNCT is a function which evaluates Equation 8.5.6.1(d). Arguments to WBLEST are

X = a vector of length NOBS containing the data (input),
NOBS = the number of data values, N (input),
BETA = estimate of the shape parameter (output),
ALPHA = estimate of the scale parameter (output).

The algorithm by which the FORTRAN code computes the estimates is described in the following paragraph.

Equation 8.5.6.1(d) is a monotonically increasing continuous function of $\hat{\beta}$. Designate the left-hand side of Equation 8.5.6.1(d) divided by n as $G(\hat{\beta})$ and obtain a solution for $\hat{\beta}$ by the following iterative procedure. Let S_y denote the standard deviation of $Y_1, \ldots, Y_n$ where $Y_i = \ell n(X_i)$ for $i = 1, \ldots, n$. Calculate $I = 1.28/S_y$ as an initial guess at the solution and calculate G(I). If $G(I) > 0$, then find the smallest positive integer k such that $G(I/2^k) < 0$ and let $L = I/2^k$ and $H = I/2^{k-1}$. If $G(I) < 0$, then find the smallest positive integer k such that $G(2^k I) > 0$ and let $L = 2^{k-1} I$ and $H = 2^k I$. In either case, the interval (L,H) contains the solution to $G(\hat{\beta}) = 0$. Now calculate G(M) where $M = (L + H)/2$. If $G(M) = 0$, then the solution is $\hat{\beta} = M$. If $G(M) > 0$, then let $H = M$. If $G(M) < 0$ then let $L = M$. The new interval (L,H) still contains the solution to $G(\hat{\beta}) = 0$ but is only half as long as the old interval. Calculate a new M-value and begin the process of interval halving again. The process is repeated until $H-L < 2I/10^6$ The solution to $G(\hat{\beta}) = 0$ is then taken to be $M = (L + H)/2$. The solution is in error by at most $I/10^6$.

8.5.6.2 Computational procedure. With a suitable sample of size n and the population parameter estimates discussed above at hand, the computation of a B-basis value is carried out by use of the formula

$$B = \hat{Q} \exp\{-V/(\hat{\beta}\sqrt{n})\} \qquad 8.5.6.2(a)$$

```
C ----------------------------------------------------------
      SUBROUTINE WBLEST(X,NOBS,ALPHA,BETA)
C
C COMPUTE MLES FOR SHAPE PARAMETER (BETA) AND SCALE
C  PARAMETER (ALPHA) BY SOLVING THE EQUATION  G(BETA)=0, WHERE G IS
C  A MONOTONICALLY INCREASING FUNCTION OF BETA.
C  THE INITIAL ESTIMATE IS:  RI=(1.28)/(STD. DEV. OF LOG(X)'S)
C  AND THE TOLERANCE IS    :   2*RI/(10**6).
      DIMENSION X(NOBS)
C
      RN=FLOAT(NOBS)
      SUMY=0.0
      SUMYSQ=0.0
      DO  2 I=1,NOBS
           Y=ALOG(X(I))
           SUMY=SUMY+Y
         SUMYSQ=SUMYSQ+(Y**2)
    2 CONTINUE
      YSTD=SQRT((SUMYSQ-(SUMY**2)/RN)/(RN-1.0))
      XGM=EXP(SUMY/RN)
      RI=1.28/YSTD
      TOL=2.0*.000001*RI
      BETAM=RI
      GFM=GFUNCT(X,NOBS,BETAM,XGM)
C
C  IF G(BETAM) .GE. 0, DIVIDE THE INITIAL ESTIMATE BY 2 UNTIL
C  THE ROOT IS BRACKETED BY BETAL AND BETAH.
      IF(GFM.GE.0.0)THEN
           DO 3 J=1,20
                BETAH=BETAM
                BETAM=BETAM/2.0
                GFM=GFUNCT(X,NOBS,BETAM,XGM)
                IF(GFM.LE.0.0)GO TO 4
    3      CONTINUE
           STOP 'GFM NEVER LE 0'
    4      CONTINUE
           BETAL=BETAM
      ENDIF
C
C  IF G(BETAM) .LT. 0, MULTIPLY THE INITIAL ESTIMATE BY 2 UNTIL
C  THE ROOT IS BRACKETED BY BETAL AND BETAH.
      IF(GFM.LT.0.0)THEN
           DO 7 J=1,20
                BETAL=BETAM
                BETAM=BETAM*2.0
                GFM=GFUNCT(X,NOBS,BETAM,XGM)
                IF(GFM.GE.0.0)GO TO 8
    7      CONTINUE
           STOP 'GFM NEVER GE 0'
    8      CONTINUE
           BETAH=BETAM
      ENDIF
C
C SOLVE THE EQUATION G(BETA)=0 FOR BETA BY BISECTING THE
C   INTERVAL (BETAL,BETAH) UNTIL THE TOLERANCE IS MET
   10 CONTINUE
      BETAM=(BETAL+BETAH)/2.0
      GFM=GFUNCT(X,NOBS,BETAM,XGM)
      IF(GFM.GE.0.0)THEN
           BETAH=BETAM
      ENDIF
      IF(GFM.LT.0.0)THEN
           BETAL=BETAM
      ENDIF
      IF(BETAH-BETAL.GT.TOL)GO TO 10
C
      BETA=(BETAL+BETAH)/2.0
      ALPHA=FNALPH(X,NOBS,BETA,XGM)
      RETURN
      END
C ----------------------------------------------------------
```

Figure 8.5.6 FORTRAN routines for calculating two-parameter Weibull shape and scale parameter estimates.

```
C ---------------------------------------------------------
      FUNCTION FNALPH(X,NOBS,BETA,XGM)
C
C COMPUTE MLE FOR TWO-PARAMETER WEIBULL SCALE PARAMETER (ALPHA)
C    XGM IS THE GEOMETRIC MEAN OF THE X'S
C
      DIMENSION X(NOBS)
      RN=FLOAT(NOBS)
C
      SUMX=0.0
      DO 20 I=1,NOBS
        SUMX=SUMX+(X(I)/XGM)**BETA
   20 CONTINUE
C
      FNALPH=XGM*(SUMX/RN)**(1./BETA)
C
      RETURN
      END
C ---------------------------------------------------------


C ---------------------------------------------------------
      FUNCTION GFUNCT(X,NOBS,BETA,XGM)
C
C  COMPUTE G FUNCTION USED IN ESTIMATING THE TWO-PARAMETER WEIBULL
C  SHAPE PARAMETER (BETA).
C  XGM IS THE GEOMETRIC MEAN OF THE X'S USED IN ESTIMATING ALPHA.
C
      DIMENSION X(NOBS)
      RN=FLOAT(NOBS)
C
      ALPHA=FNALPH(X,NOBS,BETA,XGM)
      SUMYX=0.0
      DO 10 I=1,NOBS
           SUMYX=SUMYX+ALOG(X(I))*((X(I)/ALPHA)**BETA-1.)
   10 CONTINUE
C
      GFUNCT=(SUMYX/RN)-1.0/BETA
C
      RETURN
      END
C ---------------------------------------------------------
```

Figure 8.5.6 FORTRAN routines for calculating two-parameter Weibull shape and scale parameter estimates - Continued.

where

$$\hat{Q} = \hat{\alpha}\,(0.10536)^{1/\hat{\beta}} \qquad 8.5.6.2(b)$$

and V is the value in Table 8.8.2 corresponding to a sample of size n. A numerical approximation to the V values is given in Equation 8.8.2(b).

8.5.7 Nonparametric analysis procedure for a single population. This procedure should be used when a material property value is to be computed from failure data and the form of the distribution of the population is unknown. The distribution should be considered unknown if tests indicate significant departure from the two-parameter Weibull, normal, and lognormal distributions.

8.5.7.1 Sample size greater than 28. First, order the sample so that the observations are in increasing order. Then, using Table 8.8.3, determine the r value corresponding to the sample size n. For sample sizes lying between tabulated values, use the r value associated with the largest tabulated sample size which is smaller than the actual n. The B-basis value is the rth lowest observation in the ordered sample. For example, in a sample of size $n = 30$, the lowest ($r = 1$) observation is the B-basis value. As another example, in a sample of size $n = 600$, the 48th ordered observation ($r = 48$) is the B-basis value. For sample sizes less than 29, a B-basis value acceptable for MIL-HDBK-17 cannot be calculated with this procedure; the Hanson-Koopmans procedure can provide a B-basis value for these small sample sizes.

The value of r for any sample size n can be computed directly by using the following relationships. If $q_{0.10}$ is the quantile of the underlying population distribution, then the number of observations in the sample less than $q_{0.10}$ is a binomial random variable with n trials and probability of success $p = 0.10$. Letting $X_{(r)}$ denote the rth ordered sample value, the statement $X_{(r)} = q_{0.10}$ is true if and only if there are r or more failure values in the sample which are less than or equal to $q_{0.10}$. Using this relationship, the B-basis value may be found as the rth ordered sample failure value, $X_{(r)}$, where $r \geq 1$ is the largest integer solution to

$$\sum_{w=r}^{n} \binom{n}{w} (0.10)^{w} (0.90)^{n-w} \geq 0.95 \qquad 8.5.7.1(a)$$

where

$$\binom{n}{w} = n!/w!(n-w)! \qquad 8.5.7.1(b)$$

Table 8.8.3 was generated from this equation. A numerical approximation to the tabulated r values as a function of n is also given in Section 8.8. Further information on this procedure may be found in Reference 8.5.5.

8.5.7.2 <u>The Hanson-Koopmans method</u>. Although it can be demonstrated that for $n < 29$ there is no useful nonparametric method, the following procedure, due to D. L. Hanson and L. H. Koopmans (Reference 8.5.7.2), may be a useful method for obtaining a B-basis value for sample sizes not exceeding 28. This procedure requires the assumption that the observations are a random sample from a distribution with a non-decreasing hazard rate. That is, if the probability function is f(x), and the cumulative distribution function is F(x), then

$$h(x) = \frac{f(x)}{1 - F(x)} \qquad 8.5.7.2(a)$$

is a non-decreasing function of x, for $x \leq 0$. This assumption is satisfied by the Weibull distribution with shape parameter $\beta \leq 1$ and by the normal distribution. It is not satisfied by the Weibull distribution with shape parameter $\beta < 1$ or by the lognormal distribution.

There is substantial empirical evidence that suggests the the composite strength data that will be encountered by users of this handbook will have increasing hazard rates and, consequently, this procedure can be recommended for use when n is less than 29. However, in view of the required assumption, this is not an unconditional recommendation.

The B-basis value is calculated as follows. Order the observations from smallest to largest. Let $X_{(1)}$ be the smallest observation, $X_{(2)}$ the second smallest, and so on. In general, we denote the t-th smallest value by $X_{(t)}$. Then the B-basis value is given by

$$B = X_{(t)} - k(X_{(t)} - X_{(1)}) \qquad 8.5.7.2(b)$$

where t and k depend on n and are determined from Table 8.8.11. This equation for the B-basis value should not be employed if $X_{(t)} = X_{(1)}$.

Example. Assume that 25 observations have been obtained. Order these in increasing value. From the table, for n = 25, t = 11; therefore, we record the first and eleventh values. For purposes of illustration, let the smallest value be 36.64 and the eleventh value be 47.50. Then the B-basis value is

$$B = 47.50 - 1.087(47.50 - 36.64) = 35.70$$

8.5.8 Linear regression analysis procedure. In order to determine that the average of a property for which a B-basis value is being calculated varies linearly with some specimen property such as thickness, number of plies, or percent fiber volume, the linear regression analysis procedure may be appropriate. Linear regression analysis assumes that the property to be regressed is normally distributed about the true regression line. The steps for obtaining B-basis values using linear regression analysis are as follows.

(1) Fit a regression equation of the form

$$y = a + bx \qquad 8.5.8$$

to the data as described in Section 8.6.6 where y is the material property to be regressed and x is the specimen property such as thickness, number of plies, or percent fiber volume. Obtain the estimates a and b and the root mean square error of the regression (s_y). Also obtain the F statistic for testing the significance of the regression (F).

(2) If F indicates that the regression is insignificant, one of the other appropriate analysis techniques, as described in Sections 8.5.5, 8.5.6, or 8.5.7, should be used. Otherwise,

proceed to step 3.

(3) Evaluate the validity of the normality assumption for y by performing the Anderson-Darling test presented in Section 8.6.4.4. If the hypothesis of normality is rejected, there is currently no approved method for computing B-basis values using linear regression analysis. Otherwise, proceed to step 4.

(4) At selected values of x, determine the B-basis value for the property according to the following procedure.

8.5.8.1 Computational procedure. To compute a B-basis value at $x = x_o$, use Equation 8.5.8.1(a).

$$B = (a+bx_o) - k'_B s_y \qquad 8.5.8.1(a)$$

where s_y is the root mean square error for the regression. The tolerance limit factor is

$$k'_B = [(1+\Delta)/n]^{0.5}\ t_{\gamma,0.95}(\delta) \qquad 8.5.8.1(b)$$

with noncentrality parameter

$$\delta = 1.282/[(1+\Delta)/n]^{0.5} \qquad 8.5.8.1(c)$$

where $t_{\gamma,0.95}(\delta)$ is the 0.95 percentile of the noncentral t-distribution with $\gamma = n - 2$ degrees of freedom,

$$\Delta = \frac{(x_o - \bar{x})^2}{\sum_{i=1}^{n} (x_i - \bar{x})^2/n} \qquad 8.5.8.1(d)$$

and

$$\bar{x} = \sum_{i=1}^{k} \frac{x_i}{n} \qquad 8.5.8.1(e)$$

is the average of the x values in the sample. The quantity represents the relative distance of x_o from the average x value.

The following approximation to k_B may be used when n is greater than or equal to 10 and $0 \leq \Delta \leq 10$.

$$k'_B \approx 1.282 + \exp(0.595 - 0.508\,\ell n(n) + 4.62/n + (0.486 - 0.986/n)\,\ell n(1.82 + \Delta)) \qquad 8.5.8.1(f)$$

Under the conditions stated above, the approximation is accurate to within ±1.0% (relative magnitude of error, see Section 8.8).

8.6 Statistical properties.

8.6.1 Introduction. This section includes a number of statistical tools for use in the analysis of data for MIL-HDBK-17. These tools are intended to supplement the basic procedures described in Section 8.5.

Various statistical techniques are employed in the analysis of material-property data. This section presents brief descriptions of procedures that are used most frequently in this application. More detailed descriptions of these and other statistical techniques and tables can be found in a number of workbooks and texts, some of which are given as references.

When procedures other than those described below are employed in the preparation of data proposals, they should be described adequately in the proposal to allow a proper evaluation of their validity and pertinence.

8.6.2 Outlier detection procedure. An observation is said to be an outlier if it is an observation that has been recorded in error. Since outliers may have a substantial influence on the statistical analysis of a set of data, it is often desirable that a data set be screened for outliers prior to data analysis. The maximum normed residual method for detecting potential outliers is described below. For more information on this test procedure, see References 8.6.2(a) and 8.6.2(b).

8.6.2.1 The maximum normed residual method. The maximum normed residual (MNR) test is a screening procedure for identifying outliers in a single set of data. It involves an examination of the residuals (signed distances from the mean divided by the standard deviation) to determine whether or not they are unusually large. The test assumes that the non-erroneous data values are independently and identically normally distributed. Also, the outlier procedure searches for one outlier at a time and, therefore, the significance level pertains to a single decision.

The first step is to compute the normed residuals and the MNR statistic. If X_1, X_2, ... X_n denote the data values in the sample of size n, the normed residuals are defined as:

$$r_i = \frac{X_i - \bar{X}}{S}, \qquad i = 1, 2, \ldots, n \qquad 8.6.2.1(a)$$

where $\bar{X}$ and S are the sample mean and sample standard deviation, respectively, defined in Section 8.5.1.1. The MNR statistic is the maximum of the absolute values of the normed residuals:

$$MNR = \max_i \left(| r_i | \right) \qquad 8.6.2.1(b)$$

Next, the MNR statistic is compared to the critical value corresponding to the sample size n from either Table 8.8.9 or 8.8.10. The critical values for this test are computed from the following formula:

$$C.V. = \frac{n - 1}{\sqrt{n}} \left(\frac{t^2}{n - 2 + t^2} \right)^{0.5} \qquad 8.6.2.1(c)$$

where t is the $[1 - \alpha/(2n)]$ quantile of the t distribution with n - 2 degrees of freedom and α represents the significance level of the test. Critical values for sample sizes 3 through 200 and a significance level of $\alpha = 0.01$ are tabulated in Table 8.8.9. Critical values for the same sample sizes and a significance level of $\alpha = 0.05$ are presented in Table 8.8.10. Values for other sample sizes or other significance levels may be computed using the above formula.

If MNR is smaller than the critical value, then no outliers are detected in the sample. If MNR is larger than the critical value, then the data value associated with the largest absolute normed residual is declared to be a potential outlier.

If an outlier is detected, the procedure is repeated on the reduced set of data (eliminating the potential outlier). This process is repeated until no potential outliers are detected. Note that in the second iteration, the mean, standard deviation, and critical value are computed using a sample size of n - 1. In the third iteration, the sample size will be n - 2, and so on.

8.6.3 Subpopulation compatibility tests. This section contains tests for determining the compatibility of two or more populations. The k-sample Anderson-Darling test in Section 8.6.3.1 is used to test the hypothesis that the populations from which two or more independent samples were taken are identically distributed. The equality of variance test in Section 8.6.3.2 is used to test the hypothesis that the variances of the populations from which two or more independent random samples were taken are equal.

8.6.3.1 The k-sample Anderson-Darling test. The k-sample Anderson-Darling statistic tests the hypothesis that the populations from which two or more independent random samples were drawn are identical. The test can be applied to determine whether two or more products differ with regard to their strength distributions. The test is a nonparametric statistical procedure and only requires the assumption that the samples are independent random samples from their respective populations.

Consider the populations A_1, $A_2, \ldots,$ A_k. Let X_{11}, X_{12}, ..., X_{1n_1}, denote a sample of n_1 data points from population A_1, let X_{21} X_{22}, ..., X_{2n_2}, denote a sample of the n_2 data points from population A_2, and so forth. Furthermore, let $N = n_1 + n_2 + \ldots + n_k$ represent the total number of data points in the combined samples.

Let L denote the total number of distinct data points in the combined samples and $Z_{(1)}$, $Z_{(2)}, \ldots,$ $Z_{(L)}$ denote the distinct values in the combined data set ordered from least to greatest. The k-sample Anderson-Darling statistic is defined by

$$ADK = \frac{1}{N(k-1)} \sum_{i=1}^{k} \left[\frac{1}{n_i} \sum_{j=1}^{L} h_j \frac{(NF_{ij} - n_i H_j)^2}{H_j (N-H_j) - Nh_j/4} \right] \quad 8.6.3.1(a)$$

where h_j = the number of values in the combined samples equal to $Z_{(j)}$

H_j = the number of values in the combined samples less than $Z_{(j)}$ plus one half the number of values in the combined samples equal to $Z_{(j)}$, and

F_{ij} = the number of values in sample corresponding to population A_i which are less than $Z_{(j)}$ plus one half the number of values in the sample corresponding to population A_i which are equal to $Z_{(j)}$.

Under the hypothesis of no difference in the sampled populations, the mean and variance of ADK are approximately k-1 and

$$\mathrm{Var(ADK)} = \frac{aN^3 + bN^2 + cN + d}{(N-1)(N-2)(N-3)} \tag{8.6.3.1(b)}$$

with

$$a = (4g-6)(k-1) + (10-6g)S \tag{8.6.3.1(c)}$$

$$b = (2g-4)k^2 + 8Tk + (2g-14T-4)S - 8T + 4g - 6 \tag{8.6.3.1(d)}$$

$$c = (6T+2g-2)k^2 + (4T-4g+6)k + (2T-6)S + 4T \tag{8.6.3.1(e)}$$

$$d = (2T+6)k^2 - 4Tk \tag{8.6.3.1(f)}$$

where

$$S = \sum_{i=1}^{k} \frac{1}{n_i} \tag{8.6.3.1(g)}$$

$$T = \sum_{i=1}^{N-1} \frac{1}{i} \tag{8.6.3.1(h)}$$

and

$$g = \sum_{i=1}^{N-2} \sum_{j=i+1}^{N-1} \frac{1}{(N-1)j} \tag{8.6.3.1(i)}$$

If

$$\mathrm{ADK} \geq 1 + \frac{\mathrm{Var(ADK)}}{k-1} \left[1.645 + \frac{0.678}{(k-1)^{0.5}} - \frac{0.362}{k-1} \right] \tag{8.6.3.1(j)}$$

one may conclude (with a five percent risk of being in error) that the samples were drawn from different populations. Otherwise, the hypothesis that the samples were selected from identical populations is not rejected. Table 8.8.7 contains the above critical values for the case of equal sample sizes.

For more information on the k-sample Anderson-Darling test, see Reference 8.6.3.1.

8.6.3.2 Test for equality of variances. This section describes a test suggested by Lehmann (Reference 8.6.3.2) for determining whether two or more estimates of variance differ significantly. With k independent random samples, the sample variance for the ith sample is denoted by S_i^2 and has $\gamma_i = n_i - 1$ degrees of freedom, where n_i is the size of the ith sample.

Letting $Z_i = \ell n\, (S_i^2)$, the test statistic is computed as

$$EV = \frac{1}{2} \sum_{i=1}^{k} \gamma_i Z_i^2 - \frac{1}{2} (N-k)\, \bar{Z}^2 \qquad 8.6.3.2(a)$$

where

$$\bar{Z} = \sum_{i=1}^{k} \gamma_i\, Z_i/(N-k) \qquad 8.6.3.2(b)$$

is the weighted average of the Z_i values, and N is the total sample size. This statistic is compared to the 0.95th quantile of a chi-square distribution with k - 1 degrees of freedom, values of which are tabulated in Table 8.8.8. (A numerical approximation to the tabulated values is given in Section 8.8.8.) If the test statistic is greater than or equal to the tabulated value, then the variances are declared to be significantly different. If the statistic is less than the tabulated value, then the hypothesis of equality of variance is not rejected.

8.6.4 Goodness-of-fit tests. The computational procedure selected to establish B-basis values by statistical techniques is dependent upon the underlying distribution of strength measurements. The most frequently used parametric procedures involve the use of the normal, lognormal, or two-parameter Weibull distributions. This section contains methods that are used to establish the degree to which a population may be fitted by one of these distributions.

Four goodness-of-fit test procedures are described below. The purpose of each is to indicate whether an initial distributional assumption should be rejected. The methods presented are based on the Anderson-Darling goodness-of-fit test statistic, which is particularly sensitive to discrepancies in the tail regions. The tests of fit to the normal, lognormal, and Weibull distributions for a single sample are presented in Sections 8.6.4.1, 8.6.4.2 and 8.6.4.3, respectively. A test for normality in a regression setting is discussed in Section 8.6.4.4.

An observed significance level (OSL) may be computed for each test. The OSL is the probability of obtaining a value of the test statistic as large as that obtained if the hypothesis that the data are actually from the distribution being tested is true. If the OSL is less than or equal to 0.05, the hypothesis is rejected (with at most a five percent risk of being in error) and one proceeds as if the data are not from the distribution being tested.

In what follows, unless otherwise noted, the sample size is denoted by n, the sample observations by $X_1, \ldots, X_n$, and the sample observations ordered from least to greatest by $X_{(1)}, \ldots, X_{(n)}$.

8.6.4.1 <u>Anderson-Darling test for normality</u>. The Anderson-Darling test for normality is used to test the hypothesis that the population from which a given sample of data was drawn is normally distributed. See Section 8.5.1.1 for a definition of the normal distribution.

The test compares the cumulative normal distribution function that fits the observed data best with the cumulative distribution function of the observed data. Let

$$Z_{(i)} = (X_{(i)} - \bar{X}) / S \qquad i = 1, \ldots, n \qquad 8.6.4.1(a)$$

where $X_{(i)}$ is the ith smallest sample observation, $\bar{X}$ is the sample average, and S is the sample standard deviation. Equations for computing these sample statistics are presented in Section 8.5.1.1.

The Anderson-Darling test statistic is

$$AD = \sum_{i=1}^{n} \frac{1-2i}{n} \{\ell n[F_o(Z_{(i)})] + \ell n\,[1-F_o(Z_{(n+1-i)})]\} - n \qquad 8.6.4.1(b)$$

where F_o is the standard normal distribution function. The standard normal distribution function F_o is that function such that $F_o(x)$ is equal to the area under the standard normal curve to the left of the value x.

The observed significance level (OSL) is calculated as

$$OSL = 1/\{1+\exp[-0.48 + 0.78\,\ell n(AD^*) + 4.58(AD^*)]\} \qquad 8.6.4.1(c)$$

where

$$AD^* = (1 + 4/n - 25/n^2)AD \qquad 8.6.4.1(d)$$

The OSL is a measure of the goodness-of-fit of a normal distribution to the data. Specifically, the OSL measures the probability of observing an Anderson-Darling statistic as extreme as the value calculated if a normal distribution is in fact the underlying distribution.

If OSL ≤ 0.05, one may conclude (at five percent risk of being in error) that the population from which the sample was drawn is not normally distributed. Otherwise, the hypothesis that the population is normally distributed is not rejected. For further information on this test procedure, see Reference 8.5.6.

8.6.4.2 Anderson-Darling test for lognormality. The lognormal distribution is a positively skewed parametric distribution which is related to the normal distribution. If a variable is lognormally distributed, then the logarithm of that variable is normally distributed. Thus, if a sample of data has a lognormal distribution, taking the logarithm of the observations will enable the analyst to use normal analysis procedures. The natural (base e) logarithm is used in MIL-HDBK-17.

In order to test the goodness-of-fit of the lognormal distribution, take the log of the data and perform the Anderson-Darling test for normality from Section 8.6.4.1. Using the natural logarithm, let

$$Z_{(i)} = \frac{\ell n\ (X_{(i)}) - \bar{X}_L}{S_L} \qquad 8.6.4.2(a)$$

where $X_{(i)}$ is the ith smallest sample observation and X_L and S_L are the mean and standard deviation of the $\ell n\ (X_i)$ values defined by

$$\bar{X}_L = \frac{\sum_{i=1}^{n} \ell n\ (X_i)}{n} \qquad 8.6.4.2(b)$$

$$S_L = \left[\frac{\sum_{i=1}^{n} (\ell n\ (X_i) - \bar{X}_L)^2}{n-1} \right]^{0.5} \qquad 8.6.4.2(c)$$

The Anderson-Darling statistic is computed using equation 8.6.4.1(b) and the observed significance level (OSL) is computed as in Equation 8.6.4.1(c).

8.6.4.3 Anderson-Darling test for Weibullness. The Anderson-Darling test for two-parameter Weibullness is used to test the hypothesis that the population from which a given sample of data was drawn is a two-parameter Weibull population. See Section 8.5.1.1 for a definition of the two-parameter Weibull distribution.

The test compares the cumulative Weibull distribution function that fits the observed data best with the cumulative distribution function of the observed data. The first step is to compute the parameter estimates $\hat{\alpha}$ and $\hat{\beta}$. A procedure for computing these estimates is given in Section 8.5.6.1. Then let

$$Z_{(i)} = [X_{(i)}/\hat{\alpha}]^{\hat{\beta}} \qquad i = 1, \ldots, n \qquad 8.6.4.3(a)$$

The Anderson-Darling test statistic is then

$$AD = \sum_{i=1}^{n} \frac{1-2i}{n} \{\ln[1 - \exp(-Z_{(i)})] - Z_{(n+1-i)}\} - n \qquad 8.6.4.3(b)$$

and the observed significance level is calculated as

$$OSL = 1/\{1+\exp[-0.10 + 1.24 \ln(AD^*) + 4.48 (AD^*)]\} \qquad 8.6.4.3(c)$$

where

$$AD^* = (1 + 0.2/\sqrt{n})AD \qquad 8.6.4.3(d)$$

The OSL is a measure of the goodness-of-fit of the two-parameter Weibull distribution to the data. Specifically, the OSL measures the probability of observing an Anderson-Darling statistic as extreme as the value calculated if the two-parameter Weibull distribution is in fact the underlying distribution.

8.6.4.4 Testing for normality in a regression setting. This section contains a test for normality of the dependent variable in a regression setting. The test is an Anderson-Darling test for normality performed on the residuals

$$e_i = y_i - (a + b\,x_i) \qquad i = 1,\ldots,n \qquad 8.6.4.4(a)$$

from the regression (see Section 8.6.6) assuming equality of variance of the residuals over the range of the independent variable. Letting

$$Z_{(i)} = e_{(i)} / S_y \qquad i = 1,\ldots,n \qquad 8.6.4.4(b)$$

where $e_{(1)},\ldots,e_{(n)}$ are the ordered residuals from smallest to largest and S_y is the root-mean-square error of the regression defined in Section 8.6.6. The Anderson-Darling test statistic is computed using Equation 8.6.4.1(b), and the observed significance level (OSL) is computed as in Equation 8.6.4.1(c).

If OSL ≤ 0.05, conclude that the dependent variable is not normally distributed. Otherwise, the assumption that the dependent variable is normally distributed is not rejected. The justification for this procedure may be found in Reference 8.6.4.5.

8.6.5 Exploratory Data Analysis. Exploratory Data Analysis (EDA) techniques can be used to help familiarize the analyst with a set of data. The advantage of such techniques is that they are simple and visual and often point out the problems in the data which can be corrected, or at least noted before more involved techniques are undertaken. The disadvantage of these techniques is that they are only exploratory in nature and yield no quantitative measure upon which conclusions can be based. Due to the relative unfamiliarity with the materials being analyzed, the use of EDA as both an educational tool and a data screening technique can be invaluable; however, due to the subjectivity involved with the use of these techniques, it is essential to confirm any and all conclusions based on EDA with other statistical procedures. Two EDA techniques are described below; the Quantile Box Plot and the Informative Quantile Functions. Those readers desiring a more complete treatment of the subject are referred to Reference 8.6.5.

8.6.5.1 The quantile box plot. The quantile box plot provides a graphic summary of the sample values. This procedure depicts the symmetry, tail sizes, and median failure value of the sample as well as indicating the possible existence of outliers and multiple populations.

Let $F(x)$ be the underlying distribution function. The uth quantile of $F(x)$, q_u, is given as the solution to the equation $F(q_u) = u$. The quantile function, $Q(u)$, is defined by

$$Q(u) = F^{-1}(u) \qquad 0 < u < 1 \qquad 8.6.5.1(a)$$

(see Figure 8.6.5.1(a)). Letting $X_{(1)} \leq X_{(2)} \leq \ldots \leq X_{(n)}$ denote the ordered measurements for a sample of size n, $Q(u)$ is estimated by the piecewise linear function

$$\hat{Q}(u) = (nu - j + \tfrac{1}{2})\, X_{(j+1)} + (j + \tfrac{1}{2} - nu)\, X_{(j)} \qquad 8.6.5.1(b)$$

for

$$\frac{2j-1}{2n} \leq u < \frac{2j+1}{2n} \qquad 8.6.5.1(c)$$

The quantile box plot is a graph of the above function, with three rectangles (boxes) drawn to represent various quantiles and a line segment illustrating the median value of the distribution. The innermost box has boundaries of 0.25 and 0.75 on the u axis, and boundaries of Q(0.25) and Q(0.75) on the Q(u) axis. The other two boxes are drawn in a similar manner with boundaries of 0.125 to 0.875 and 0.0625 to 0.9375, respectively, on the u axis. The line segment illustrating the median is drawn from coordinates (0.25, Q(0.5)) to (0.75, Q(0.5)).

Figure 8.6.5.1(b) is an example of a quantile box plot. The boxes enable the analyst to get a feeling for the symmetry and tail sizes of the underlying distribution. Flat spots in Q(u) indicate modal values. Sharp rises in Q(u) for u in the vicinity of 0 or 1 indicate the possible presence of outliers in the data. Sharp rises in Q(u) within the boxes indicate the possible existence of two (or more) populations or gaps in the data. A thorough treatment of the use of the Quantile Box plot can be found in Reference 8.6.5.1.

8.6.5.2 <u>The Informative Quantile function</u>. Techniques for obtaining B-basis values for unimodal data can be divided into two main categories: techniques for specific parametric families, and nonparametric techniques. The Informative Quantile (IQ) function may be used as an aid in identifying a parametric model which provides a satisfactory fit to the data. Parametric techniques have been most thoroughly discussed for the normal, lognormal, and two-parameter Weibull parametric families; thus only these techniques will be considered here. Henceforth in this section, any reference to the Weibull parametric family should be interpreted as a reference to the two-parameter Weibull parametric family.

The IQ function was developed to identify which univariate location-scale parametric distribution best describes an ordered group of data. A univariate location-scale parametric distribution is one whose distribution function F(x) can be expressed as

$$F(x) = F_o[(x - a)/b] \qquad 8.6.5.2(a)$$

where a and b are the location and scale parameters respectively, and $F_o(x)$ is the "standard" distribution with a = 0 and b = 1. The IQ function identifies the

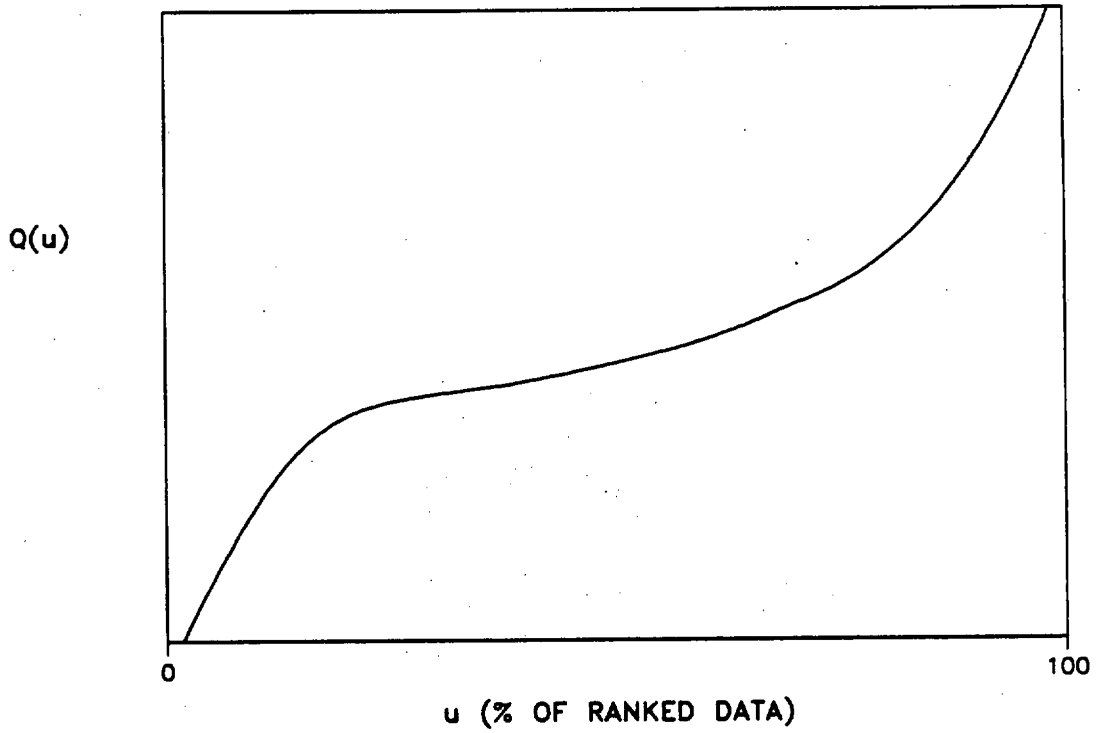


FIGURE 8.6.5.1(a) The quantile function.

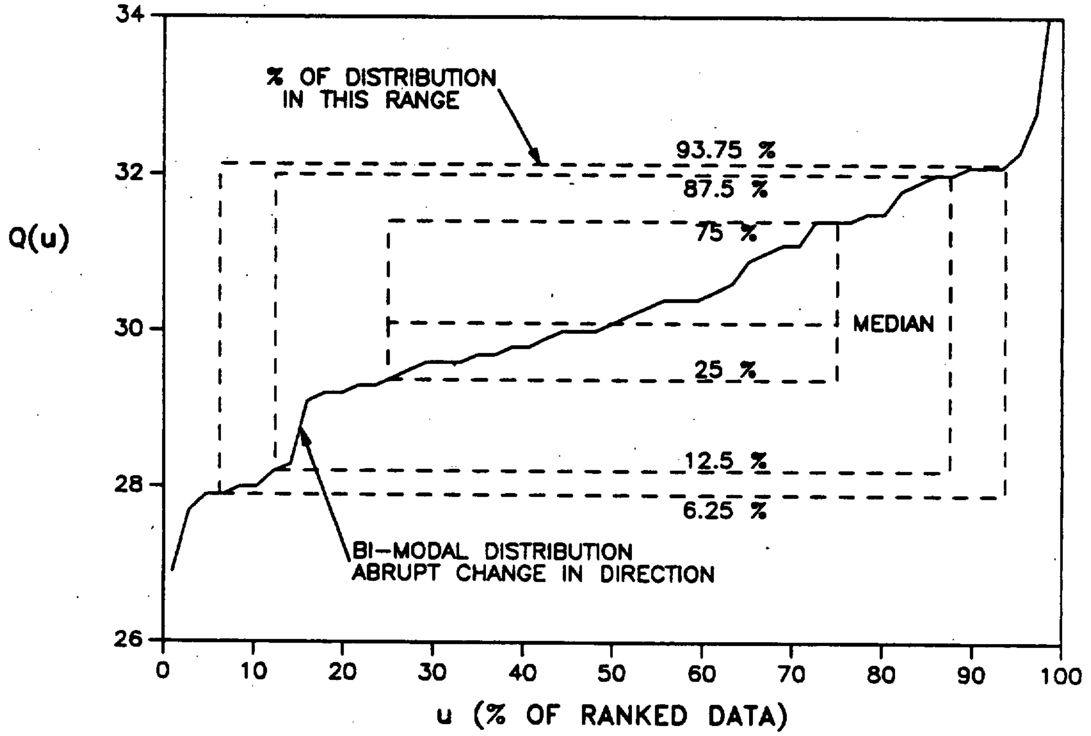


FIGURE 8.6.5.1(b) Example of a quantile box plot.

standard distributional form and is thus independent of the values of the location and scale parameters.

The Weibull and lognormal parametric families are not location-scale parametric families. However, the appropriateness of these families may be observed due to their relationship to two known location-scale families: the normal and the extreme value families.

The IQ function is defined as

$$IQ(u) = \frac{Q(u) - Q(0.5)}{2[Q(0.75) - Q(0.25)]} \qquad 8.6.5.2(a)$$

where Q(u) is the quantile function defined in Equation 8.6.5.1(a). In order to determine whether the data can be adequately modeled by either the normal or extreme value distribution, a plot of the truncated IQ function, defined by

$$TIQ(u) = \begin{cases} -1 & \text{if } IQ(u) < -1, \\ IQ(u) & \text{if } -1 < IQ(u) < 1, \\ 1 & \text{if } IQ(u) > 1, \end{cases} \qquad 8.6.5.2(b)$$

is compared to the graph of the exact uniform TIQ plots for these distributional forms (see Figures 8.6.5.2(a) and (b)). Though the TIQ plots for the data will be considerably less smooth than the exact TIQ plots, they may be compared for general shape and tail behavior.

In order to determine the adequacy of either the lognormal or the Weibull distribution, use the natural logarithms of the data to define the quantile function. Thus, Equation 8.6.5.1(b) becomes

$$\hat{Q}(u) = (nu - j + \frac{1}{2})\, \ell n(X_{(j+1)}) + (j + \frac{1}{2} - nu)\, \ell n(X_{(j)}) \qquad 8.6.5.2(c)$$

for

$$\frac{2j-1}{2n} \le u < \frac{2j+1}{2n} \qquad 8.6.5.2(d)$$

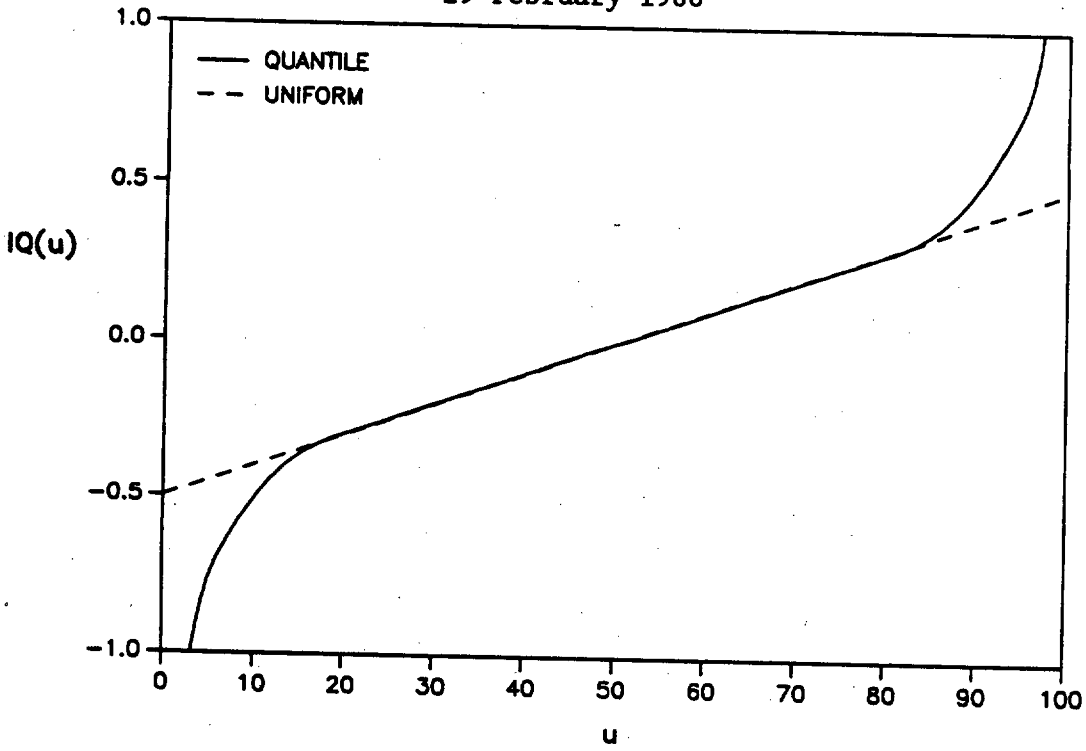


FIGURE 8.6.5.2(a) TIQ plot of the normal distribution parametric family.

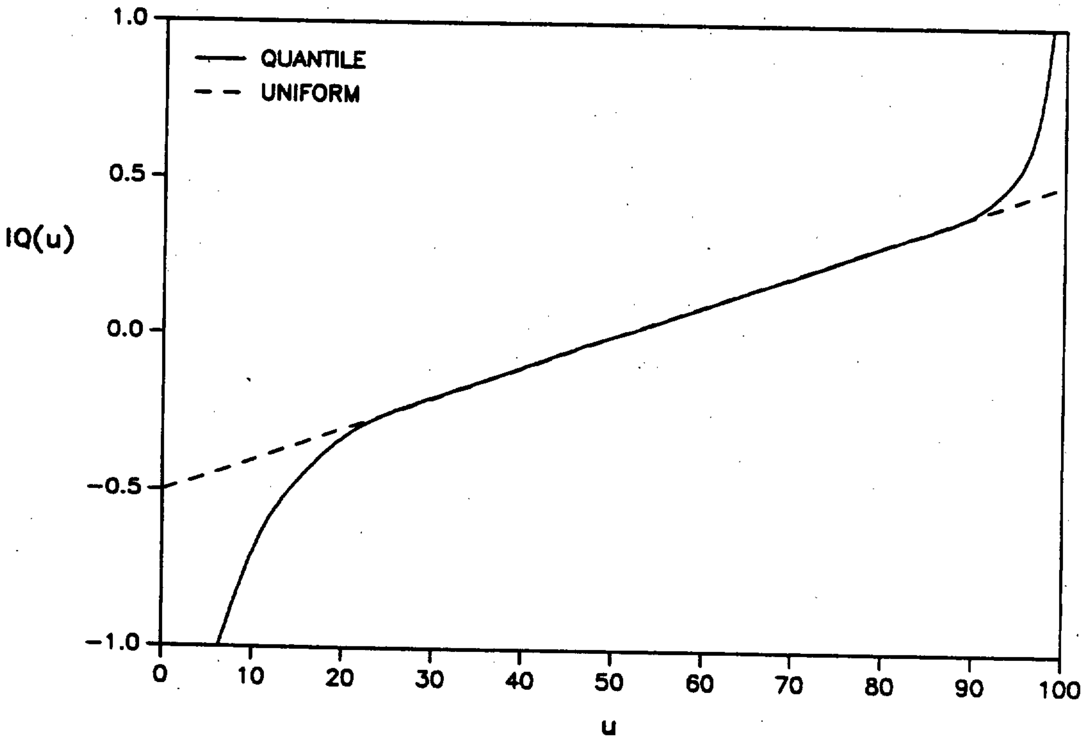


FIGURE 8.6.5.2(b) TIQ plot of the extreme value distribution parametric family.

The IQ and TIQ functions in Equations 8.6.5.2(a) and 8.6.5.2(b) are defined using this quantile function.

Thus, to determine whether the data can be adequately modeled by the normal distribution, compare the TIQ plot for the original data to the TIQ plot for the normal distribution in Figure 8.6.5.2(a). To determine whether the data can be adequately modeled by the lognormal distribution, compare the TIQ plot for the log data to the TIQ plot for the normal distribution in Figure 8.6.5.2(a). The adequacy of the two-parameter Weibull distribution is determined by comparing the TIQ plot for the log data to the exact TIQ plot for the extreme value distribution in Figure 8.6.5.2(b). For further information concerning the quantile function and the informative quantile function, the reader is referred to References 8.6.5.2(a) and 8.6.5.2(b).

8.6.6 Simple linear regression analysis. In order to determine if the average of one measured value varies linearly with another measured value, a simple linear regression analysis may be employed. The mathematical techniques for performing a simple linear regression analysis are presented in Section 8.6.6.1. Statistical tests to determine whether a straight line adequately fits a set of data and whether a regression relationship is significant are described in Section 8.6.6.2. Section 8.7 contains an example (Problem 8) illustrating the necessary computations for these statistical procedures. See Reference 8.6.6 for a thorough treatment of regression analysis.

8.6.6.1 Least-squares simple linear regression. Simple linear regression analysis is a statistical procedure often used to investigate and describe the relationship between two measured quantities, a dependent and an independent variable. The assumed model for the relationship between the two quantities is expressed algebraically by an equation of the form

$$Y = \alpha + \beta x + \epsilon \qquad 8.6.6.1(a)$$

where Y = the dependent variable
x = the independent variable
α = the true intercept of the regression equation

β - the true slope of the regression equation

ϵ - the measurement or experimental error by which Y differs from the ideal linear relationship; assumed to be normally distributed with mean zero and variance σ^2.

Aside from the error term, ϵ, this is the equation of a straight line. The parameter α is the point where the line intersects the Y-axis, and the parameter β represents its slope. Assuming such an approximate linear relationship between x and Y, a simple linear regression analysis can be performed to obtain estimates of the parameters α, β, and σ^2.

In order to use regression analysis, n x-values, not necessarily distinct are selected by the experimenter. For each of these x values, a corresponding y value is observed. Thus the data consists of n pairs, (x_1,y_1), (x_2,y_2), ..., (x_n,y_n) where the x's are fixed constants (frequently chosen in advance by the experimenter, in order to obtain specific experimental objectives) and each y_i is a random variable whose distribution may depend on the corresponding x_i. Such a sample is often represented graphically by plotting the n data points on a coordinate system, in which x is plotted horizontally and y vertically. A subjective method for obtaining estimates of α and β is drawing a line that, by visual inspection, appears to fit the points satisfactorily. An objective solution is afforded by the method of least squares.

The method of least squares is a procedure for obtaining a line having the property that the sum of squares of the vertical deviations of the sample points from this line is less than that for any other line. Denote the least-squares line by the equation

$$y^* = a + bx \qquad 8.6.6.1(b)$$

in which y^* is the predicted value of y for any value of x and a and b are the estimates of the parameters α and β in the true-regression equation obtained by the least squares method presented below. The values of a and b that minimize the sum of squares of the vertical deviations are given by the formulas:

$$a = \frac{\Sigma y - b\,\Sigma x}{n} \qquad 8.6.6.1(c)$$

and

$$b = \frac{S_{xy}}{S_{xx}} \qquad 8.6.6.1(d)$$

where

$$S_{xy} = \Sigma xy - \frac{\Sigma x\,\Sigma y}{n} \qquad 8.6.6.1(e)$$

and

$$S_{xx} = \Sigma x^2 - \frac{(\Sigma x)^2}{n} \qquad 8.6.6.1(f)$$

The root mean square error of y is expressed as

$$S_y = \left[\frac{\Sigma(y - y*)^2}{n-2}\right]^{0.5} \qquad 8.6.6.1(g)$$

where $y*$ is the predicted value of y defined in Equation 8.6.6.1(b). S_y^2 is an estimate of σ^2, the variance of the distribution of Y about the regression line. A convenient computational formula for S_y is

$$S_y = \left[\frac{S_{yy} - b^2 S_{xx}}{n-2}\right]^{0.5} \qquad 8.6.6.1(h)$$

where

$$S_{yy} = \Sigma y^2 - \frac{(\Sigma y)^2}{n} \qquad 8.6.6.1(i)$$

The quantity $R^2 = (b^2 S_{xx})/S_{yy}$ measures the proportion of the total variation in the y data that is explained by the regression. An R^2 equal to 1 indicates that the regression model describes the data perfectly. R^2 provides an idea of how well the data are described by a linear regression. A more precise determination of the adequacy of a linear regression is discussed in the next section.

8.6.6.2 <u>Tests for significance of the slope of a simple linear regression equation</u>. In order to determine if there is a statistically significant linear relationship between x and y, one tests the hypothesis that the slope β is zero. If this hypothesis is rejected, then one concludes that the variable x provides information about y by means of a linear relationship. If the hypothesis that β is zero is not rejected, this should not be regarded as conclusive evidence that x and y are independent. The possibilities include the presence of a relationship, which in addition to being nonlinear, does not have an adequately locally-linear approximation over the range of data values.

The logic of the test may be described as follows. One compares s_y^2 with S_{yy}. If s_y^2 is so much less than S_{yy} that the reduction cannot be ascribed to chance, then the regression model (i.e., the variable x) does provide an explanation for some of the variability in y.

Let

$$SSR = b^2 S_{xx} \qquad 8.6.6.2(a)$$

$$SST = S_{yy} \qquad 8.6.6.2(b)$$

$$SSE = SST - SSR \qquad 8.6.6.2(c)$$

Then, the test procedure may be summarized in the following ANOVA (analysis of variance) table.

Source of Variation	Degrees of Freedom, γ	Sum of Squares, SS	Mean Squares, MS	F
Regression	1	SSR	MSR	MSR/MSE
Error	n-2	SSE	MSE	
Total	n-1	SST		

If F is greater than or equal to the 95th percentile of the F distribution with 1 and n - 2 degrees of freedom (see Table 8.8.5), then the hypothesis that β is zero is rejected and x provides information about y by means of the calculated linear relationship.

8.6.7 Analysis of variance procedures. This section contains a discussion of the standard one-way analysis of variance (ANOVA) procedures for analyzing multiple samples of data. Two different models are referred to: the fixed effects model and the random effects model. The fixed effects model is appropriate when inferences are to be made only to the populations from which the actual samples were drawn. The random effects model is appropriate when the samples are viewed as merely representing a random selection from many possible populations. In the random effects model, inferences are made to the whole collection of populations, not just those from which samples were actually drawn. Although the two models involve different assumptions and lead to different interpretations of the data, the computational formulas for computing sums of squares are the same.

The basic model can be written as

$$X_{ij} = \mu + b_i + e_{ij}, \qquad \begin{array}{l} i = 1,\ldots,k \\ j = 1,\ldots,n_i \end{array} \qquad 8.6.7(a)$$

where k is the number of different samples, n_i is the number of observations in the ith sample, and X_{ij} represents the jth observation in the ith sample.

The ANOVA models represent each observation as the sum of three components; μ is the overall average of the population, b_i is the effect attributed to the ith sample, and e_{ij} is a random error term which represents unexplainable sources of variation. The error terms, e_{ij}, are assumed to be independently distributed normal random variables with a mean of zero and a variance of σ_e^2 (the within-sample variance). In the fixed effects model, the b_i values are assumed to be population parameters, taking on a specific value for each sample. In the random effects model, the b_i are assumed to be independent random variables following a normal distribution with a mean of zero and a variance of σ_b^2 (the between-sample variance). See Reference 8.6.7 for more information on analysis of variance procedures.

8.6.7.1 Computational procedures. The analysis of data based on either of the above models involves computing sums of squares which measure variability between samples and within samples. Two procedures for computing these quantities are presented. The first assumes that all of the original observations are available. The second assumes that only the batch means, standard deviations and sample sizes are available. Both of these procedures yield the same results for the computed sums of squares.

8.6.7.1.1 Computations based on individual measurements. When all of the observations in a sample are available, the first step is to compute averages. The overall average is computed as

$$\bar{X} = \sum_{i=1}^{k} \sum_{j=1}^{n_i} X_{ij}/N \tag{8.6.7.1.1(a)}$$

where

$$N = \sum_{i=1}^{k} n_i \tag{8.6.7.1.1(b)}$$

is the total sample size. The sample averages are computed as

$$\bar{X}_i = \sum_{j=1}^{n_i} X_{ij}/n_i \qquad \text{for } i = 1, \ldots, k \tag{8.6.7.1.1(c)}$$

From these quantities, the required sums of squares can be computed. The between-sample sum of squares is computed as

$$SSB = \sum_{i=1}^{k} n_i \bar{X}_i^2 - N\bar{X}^2 \tag{8.6.7.1.1(d)}$$

and the total sum of squares is computed as

$$SST = \sum_{i=1}^{k} \sum_{j=1}^{n_i} X_{ij}^2 - N\bar{X}^2 \tag{8.6.7.1.1(e)}$$

The within-sample, or error, sum of squares is computed by subtraction

$$SSE = SST - SSB \qquad 8.6.7.1.1(f)$$

8.6.7.1.2 <u>Computations based on summary statistics</u>. It is often the case that only summary statistics are available for each sample. If these summary statistics contain the sample averages $\bar{X}_i$, the standard deviations of the data from each sample (S_i) and the sample sizes (n_i), the sums of squares can be computed as follows. First, compute the overall mean,

$$\bar{X} = \sum_{i=1}^{k} n_i \bar{X}_i / N \qquad 8.6.7.1.2(a)$$

The between-sample sum of squares is computed as in the previous section

$$SSB = \sum_{i=1}^{k} n_i \bar{X}_i^2 - N\bar{X}^2 \qquad 8.6.7.1.2(b)$$

where

$$N = \sum_{i=1}^{k} n_i \qquad 8.6.7.1.2(c)$$

Each of the sample standard deviations has n_i-1 degrees of freedom. The sum of squares within samples can be computed by taking a sum of the squared standard deviations weighted by their degrees of freedom:

$$SSE = \sum_{i=1}^{k} (n_i - 1)\, S_i^2 \qquad 8.6.7.1.2(d)$$

The total sum of squares, SST, is the sum of SSB and SSE.

8.6.7.2 <u>ANOVA Table.</u> Once the sum of squares computations have been completed, an ANOVA table is constructed to display the information about sources of variation in the data. A typical ANOVA table which is used for both the fixed effects and random effects models is shown below. The first column identifies the source of variation. The degrees of freedom and the computed sums of squares are listed in the second and third columns, respectively. The fourth column contains mean squares which are defined as the sum of squares divided by its degrees of freedom. The final column contains an F statistic which is equal to the ratio of the mean squares. This statistic is used to test the hypothesis that there is significant sample-to-sample variation. The statistic is compared to the upper 0.95th quantile of an F distribution with k - 1 numerator degrees of freedom and N - k denominator degrees of freedom. Table 8.8.5 contains these critical F values. If the computed statistic is greater than the tabulated F value, this indicates that there is statistically significant sample-to-sample variation. If the computed statistic is less than the tabulated value, then the variation between samples is not statistically significant.

Source	Degrees of Freedom	Sum of Squares	Mean Squares	F Test
Samples	k - 1	SSB	MSB = SSB/(k-1)	F = MSB/MSE
Error	N - k	SSE	MSE = SSE/(N-k)	
Total	N - 1	SST		

8.7 Samples of computational procedures. It is appropriate at this point to review the computational procedures described in Sections 8.5.3 through 8.5.8. To do so, hypothetical sets of data have been created which represent mechanical property measurements from both tensile and compression tests. The data sets for Problems 1 through 8, which follow, are reported in Table 8.7. In progressing through the sample problems, the flowchart in Figure 8.5.2 has been followed, and appropriate references to specific sections of these guidelines have been made.

8.7.1 Problem 1 - Outlier detection and multiple sample tests. The data set for this problem consists of tensile strength measurements from nine batches of material. This problem illustrates the outlier detection procedure, the k-sample Anderson-Darling test, the test for normality of multiple samples, and the homogeneity of variance test.

Problem 1 - Step 1. The first step is to screen the data for outliers using the MNR procedure as described in Sections 8.5.3.1 and 8.6.2. The screening procedure is performed separately on each batch. The relevant calculations for the seventh batch, with a sample mean of 72.52 and a sample standard deviation of 8.08, are shown in the table below.

X_i	$\lvert r_i \rvert = \left\lvert \frac{X_i - \bar{X}}{s} \right\rvert = \left\lvert \frac{X_i - 72.52}{8.08} \right\rvert$
65.4	0.88
69.4	0.39
69.9	0.32
71.5	0.13
86.4	1.72

The MNR statistic is the largest absolute residual, or 1.72. Since this is greater than the n = 5 critical value of 1.715 from Table 8.8.10, the observation 86.4 is identified as a potential outlier in this batch.

TABLE 8.7 Sample data sets for Section 8.7.

Problem 1		Problem 2		Problem 3	
Batch	Data	Batch	Data	Batch	Data
1	65.4	1	61.3	1	67.4
1	63.6	1	68.5	1	61.8
1	63.4	1	62.5	1	72.6
1	61.3	1	66.0	1	56.0
1	61.0	1	66.6	1	69.0
1	61.6	1	64.8	1	75.0
1	65.4	1	69.5	1	65.4
2	64.0	2	66.5	2	59.4
2	64.8	2	64.7	2	66.2
2	66.4	2	64.9	2	74.8
2	61.2	2	65.2	2	75.7
2	64.7	2	70.3	2	63.9
3	68.4	3	66.0	3	70.5
3	68.8	3	72.7	3	61.7
3	66.8	3	67.1	3	66.6
3	63.6	3	67.7	3	66.1
3	69.3	3	65.7	3	72.7
4	66.8	4	61.9	4	65.5
4	67.9	4	68.0	4	61.7
4	68.9	4	63.3	4	60.6
4	65.6	4	74.6	4	75.0
4	64.6	4	66.2	4	64.4
4	71.1	4	68.2	4	75.3
4	64.1	4	69.1	4	71.7
5	74.6	5	68.9	5	51.6
5	74.2	5	65.0	5	64.3
5	62.7	5	70.9	5	74.2
5	73.3	5	65.4	5	75.4
5	68.6	5	66.5	5	60.5
5	74.0	5	64.9	5	47.1
6	67.4	6	75.8	6	73.5
6	68.0	6	75.2	6	77.0
6	74.6	6	71.5	6	66.7
6	74.9	6	69.6	6	66.4
6	65.5	6	66.1	6	65.0
7	69.4	7	72.8	7	64.0
7	71.5	7	75.0	7	66.2
7	69.9	7	66.3	7	64.3
7	86.4	7	69.5	7	60.4
7	65.4	7	71.9	7	72.4
8	64.7	8	71.9	8	71.0
8	70.0	8	71.0	8	66.9
8	70.2	8	69.5	8	76.8
8	79.2	8	69.5	8	64.4
8	76.8	8	72.6	8	70.0
8	78.5	8	74.6	8	72.1
9	70.7	9	68.7	9	73.9
9	80.6	9	76.3	9	78.5
9	68.3	9	76.6	9	68.0
9	87.0	9	109.9	9	75.0
9	81.1	9	66.2	9	79.5
9	79.7	9	72.4	9	60.6
9	82.3	9	72.8	9	71.4

TABLE 8.7 Sample data sets for Section 8.7 - Continued.

Problem 4		Problem 5		Problem 6	
Batch	Data	Batch	Data	Batch	Data
1	30.4	1	30.4	1	72.3
1	29.6	1	29.0	1	65.3
1	29.3	1	34.8	1	74.3
1	27.9	1	30.5	1	68.5
1	29.8	1	27.3	1	65.7
1	30.5	1	27.9	1	64.3
1	29.4	1	29.4	1	72.8
2	31.5	2	29.8	2	74.5
2	31.1	2	32.4	2	66.3
2	27.9	2	33.2	2	74.3
2	34.5	2	29.9	2	69.3
2	29.5	2	30.5	2	64.9
3	29.6	3	31.1	3	75.3
3	29.8	3	32.0	3	68.5
3	29.1	3	31.0	3	75.2
3	30.0	3	29.3	3	74.5
3	30.4	3	29.9	3	69.6
4	32.1	4	32.2	4	65.6
4	29.7	4	29.9	4	74.6
4	26.9	4	29.1	4	76.5
4	30.0	4	29.9	4	72.0
4	31.4	4	32.7	4	69.2
4	28.3	4	31.6	4	74.3
4	31.9	4	31.6	4	78.7
5	30.0	5	27.5	5	68.1
5	31.5	5	29.3	5	72.1
5	32.0	5	29.1	5	66.6
5	30.2	5	29.5	5	75.0
5	30.6	5	28.7	5	67.2
5	31.1	5	29.5	5	73.4
6	29.7	6	29.9	6	66.4
6	29.3	6	28.9	6	72.6
6	30.4	6	29.2	6	67.5
6	32.1	6	29.1	6	76.3
6	32.8	6	31.1	6	65.1
7	29.9	7	31.1	7	73.3
7	30.9	7	30.4	7	72.6
7	32.1	7	33.4	7	67.8
7	31.4	7	30.0	7	69.4
7	32.0	7	27.3	7	67.3
8	29.2	8	31.3	8	75.2
8	31.0	8	30.2	8	72.8
8	31.8	8	30.2	8	67.8
8	30.3	8	29.1	8	76.2
8	28.2	8	33.0	8	72.8
8	28.0	8	34.4	8	68.7
9	30.1	9	28.4	9	72.3
9	29.2	9	30.1	9	76.0
9	27.7	9	30.5	9	65.8
9	28.0	9	29.4	9	69.9
9	31.4	9	28.7	9	67.8
9	29.6	9	32.3	9	74.6
9	32.3	9	29.6	9	71.3

Problem 7	
Batch	Data
1	63.9
1	76.2
1	76.9
1	67.9
1	80.4
2	62.2
2	79.6
2	62.4
2	76.6
2	80.5
3	72.2
3	61.3
3	74.9
3	81.6
3	55.9
4	58.2
4	77.0
4	87.5
4	63.0
4	77.4
5	66.2
5	75.8
5	62.6
5	78.0
5	78.4

Problem 8	
x	y
0.112	32.0
0.112	32.0
0.116	30.0
0.116	32.0
0.119	28.0
0.119	30.4
0.121	26.8
0.122	28.0
0.124	29.2
0.124	28.4
0.126	27.3
0.126	28.0
0.127	29.2
0.128	26.0
0.129	27.6
0.133	26.8
0.135	26.8
0.135	24.0
0.135	25.2
0.137	24.8

Similar calculations for the remaining batches identify no additional potential outliers in this set of data. Visual inspection of the data also does not identify any additional potential outliers.

No cause could be determined for the potential outlier, 86.4, and it is, therefore, retained in the data set. Repeating the outlier tests at the 0.01 significance level (Table 8.8.9) indicates that there are no extreme outliers which should be removed from the data set. .

Problem 1 - Step 2. The k-sample Anderson-Darling test described in Section 8.6.3.1 will be employed next to determine whether or not the data from the nine batches should be combined. The first step is to order the pooled sample. Table 8.7.1 lists the 47 sorted, distinct values in the column labeled $Z_{(j)}$. The remaining columns show the h_j, H_j, and F_{1j} values used in calculating the terms in the statistic arising from the first batch ($i=1$). The column labeled f_{1j} shows the number of times that $Z_{(j)}$ is represented in the first batch and is used in calculating F_{1j}. From these numbers, it follows that

$$\frac{1}{n_i}\sum_{j=1}^{L} h_j \frac{(NF_{ij} - n_iH_j)^2}{H_j(N - H_j) - Nh_j/4} = \frac{1}{7}\sum_{j=1}^{47} h_j \frac{(53F_{1j} - 7H_j)^2}{H_j(53 - H_j) - 53h_j/4} = 363.33$$

When these calculations are repeated for the remaining eight batches, the k-sample Anderson-Darling statistic is computed as

$$\begin{aligned} \text{ADK} &= \frac{1}{N(k-1)}\sum_{i=1}^{k}\left[\frac{1}{n_i}\sum_{j=1}^{L} h_j \frac{(NF_{ij} - n_iH_j)^2}{H_j(N - H_j) - Nh_j/4}\right] \\ &= \frac{1}{53(9-1)}(363.33 + \ldots + 324.11) \\ &= 2.72 \end{aligned}$$

The computed value of the statistic is compared to the critical value from Equation 8.6.3.1(j), which is 1.44. Since the computed value of 2.72 is greater than the critical value of 1.44, the hypothesis that the populations from which these groups were drawn are identical is rejected.

TABLE 8.7.1 Illustration of k-sample Anderson-Darling statistic calculations for the first batch ($i=1$).

j	$Z_{(j)}$	h_j	H_j	f_{1j}	F_{1j}
1	61.0	1	0.5	1	0.5
2	61.2	1	1.5	0	1.0
3	61.3	1	2.5	1	1.5
4	61.6	1	3.5	1	2.5
5	62.7	1	4.5	0	3.0
6	63.4	1	5.5	1	3.5
7	63.6	2	7.0	1	4.5
8	64.0	1	8.5	0	5.0
9	64.1	1	9.5	0	5.0
10	64.6	1	10.5	0	5.0
11	64.7	2	12.0	0	5.0
12	64.8	1	13.5	0	5.0
13	65.4	3	15.5	2	6.0
14	65.5	1	17.5	0	7.0
15	65.6	1	18.5	0	7.0
16	66.4	1	19.5	0	7.0
17	66.8	2	21.0	0	7.0
18	67.4	1	22.5	0	7.0
19	67.9	1	23.5	0	7.0
20	68.0	1	24.5	0	7.0
21	68.5	1	25.5	0	7.0
22	68.4	1	26.5	0	7.0
23	68.6	1	27.5	0	7.0
24	68.8	1	28.5	0	7.0
25	68.9	1	29.5	0	7.0
26	69.3	1	30.5	0	7.0
27	69.4	1	31.5	0	7.0
28	69.9	1	32.5	0	7.0
29	70.0	1	33.5	0	7.0
30	70.2	1	34.5	0	7.0
31	70.7	1	35.5	0	7.0
32	71.1	1	36.5	0	7.0
33	71.5	1	37.5	0	7.0
34	73.3	1	38.5	0	7.0
35	74.0	1	39.5	0	7.0
36	74.2	1	40.5	0	7.0
37	74.6	2	42.0	0	7.0
38	74.9	1	43.5	0	7.0
39	76.8	1	44.5	0	7.0
40	78.5	1	45.5	0	7.0
41	79.2	1	46.5	0	7.0
42	79.7	1	47.5	0	7.0
43	80.6	1	48.5	0	7.0
44	81.1	1	49.5	0	7.0
45	82.3	1	50.5	0	7.0
46	86.4	1	51.5	0	7.0
47	87.0	1	52.5	0	7.0

Problem 1 - Step 3. The equality of variance test described in Section 8.6.3.2 is used to determine if the within-batch variances are significantly different. Relevant sample statistics for the nine batches are given in the table below.

Batch	n_i	s_i^2	Y_i	$Z_i = \ln(s_i^2)$
1	7	3.46	6	1.24
2	5	3.61	4	1.28
3	5	5.34	4	1.68
4	7	6.25	6	1.83
5	6	22.37	5	3.11
6	5	19.01	4	2.95
7	5	65.29	4	4.18
8	6	33.76	5	3.52
9	7	44.09	6	3.79

The test statistic is then calculated as follows.

$$EV = \frac{1}{2} \sum_{i=1}^{k} \gamma_i Z_i^2 - \frac{1}{2} (n-k) \bar{Z}^2$$

$$= \frac{1}{2} \sum_{i=1}^{9} \gamma_i Z_i - \frac{1}{2} (53-9)(2.61)$$

$$= 24.63$$

The critical chi-square value from Table 8.8.8 corresponding to k - 1 = 8 degrees of freedom is 15.51. Since the test statistic is greater than the critical value, the hypothesis that the variances are equal is rejected. Since the different batches in this sample display significant batch-to-batch variability and unequal variances, a B-basis value cannot be computed. Potential problems with consistency in fabrication or processing of the different batches should be examined.

8.7.2 Problem 2 - Outlier detection and ANOVA method. The data set for this problem consists of tensile strength measurements from nine batches of material. This problem illustrates the outlier detection procedure and the calculation of B-basis values by the ANOVA method.

Problem 2 - Step 1. The first step is to screen the data for outliers using the MNR procedure described in Sections 8.5.3.1 and 8.6.2. The screening procedure is performed separately on each batch. (See Problem 1 for details of the outlier detection computations.)

A single observation, the value 109.9 in the ninth batch, is identified as a potential outlier. Assume that the potential outlier was the result of a clerical error, and its value should have been 69.6. The remaining calculations assume that this correction has been made. Upon repeating the entire outlier screening procedure, no potential outliers or extreme outliers are identified.

Problem 2 - Step 2. The k-sample Anderson-Darling test statistic for the data is ADK = 1.84. (See Problem 1 for a detailed computation of the k-sample statistic.) Since 1.84 is greater than the critical value of 1.44, conclude that the batches are not from the same population.

Problem 2 - Step 3. The test for equality of variances between the batches gives a calculated statistic of EV = 5.60, which is less than the critical value of 15.51 from Table 8.8.8. (See Problem 1 for a detailed computation of this test statistic.) Thus, one should proceed as if the data are normally distributed and the variances are equal, and use the ANOVA method of Section 8.5.4 to calculate a B-basis value.

Problem 2 - Step 4. Summary statistics for the data are given in the table below. Preliminary ANOVA calculations covered in Section 8.6.7.1.2 are:

$$\bar{\bar{X}} = \sum_{i=1}^{k} n_i \bar{X}_i / N = [7(65.60) + \ldots + 7(71.80)]/53 = 68.84$$

Batch	n_i	$\bar{X}_i$	S_i
1	7	65.60	2.99
2	5	66.32	2.33
3	5	67.84	2.84
4	7	67.33	4.17
5	6	66.93	2.45
6	5	71.64	4.03
7	5	71.10	3.33
8	6	71.52	1.96
9	7	71.80	3.88

$$SSB = \sum_{i=1}^{k} n_i\bar{X}_i^2 - N\bar{X}^2 = [7(65.60)^2 + \ldots + 7(71.80)^2] - 53(68.84)^2$$

$$= 317.255$$

$$SSE = \sum_{i=1}^{k} (n_i-1)S_i^2 = [6(2.99)^2 + \ldots + 6(3.88)^2] = 460.680$$

$$MSB = SSB/(k-1) = 317.255/(9-1) = 39.66$$

$$MSE = SSE/(N-k) = 460.680/(53-9) = 10.47$$

Preliminary calculations covered in Section 8.5.4 are:

$$n^* = \sum_{i=1}^{k} n_i^2/N = (7^2 + \ldots + 7^2)/53 = 6.0189$$

$$n' = (N-n^*)/(k-1) = (53-6.0189)/(9-1) = 5.8726$$

$$k^* = N/n^* = 53/6.0189 = 8.8056$$

$$S_e^2 = MSE = 10.47$$

$$S_b^2 = (MSB - MSE)/n' = (39.66-10.47)/5.8726 = 4.97$$

and

$$F_{0.2,k-1,N-k} = F_{0.2,8,44} = 0.56 \quad \text{(from Table 8.8.4)}$$

The upper bound for the ratio of between-batch to within-batch variability is

$$\begin{aligned} \hat{R} &= [(MSB/MSE)/\ F_{0.2} - 1]/n' \\ &= [(39.66/10.47)/0.56 - 1]/5.8726 \\ &= 0.98 \end{aligned}$$

The tolerance limit factor T should be obtained from the approximation in Section 8.5.4 rather than from Table 8.8.4 because $\hat{R}$, k^* and n^* are not integers. The noncentrality parameter is calculated as

$$\delta = 1.282 \left[\frac{N(\hat{R} + 1)}{n^*\hat{R} + 1} \right]^{0.5} = 1.282 \left[\frac{53\ (0.98 + 1)}{(6.0189)\ (0.98) + 1} \right]^{0.5} = 5.15$$

The degrees of freedom parameter is calculated as

$$\gamma = \frac{(\hat{R} + 1\ )^2}{\dfrac{(\hat{R} + \frac{1}{n^*})^2}{k^* - 1} + \dfrac{(\frac{n^*-1}{n^*})^2}{k^*(n^* - 1)}}$$

$$= \frac{(0.98 + 1)^2}{\dfrac{\left[0.98 + \frac{1}{6.0189} \right]^2}{8.8056 - 1} + \dfrac{\left[\frac{6.0189 - 1}{6.0189} \right]^2}{8.8056(6.0189-1)}}$$

$$= 21.43$$

and

$$t_{\gamma,0.95} = t_{21.43,0.95} = 1.72$$

The tolerance limit factor is calculated as follows:

$$T = 1.282 + 1.282\,(t_{\gamma,0.95}/\delta) + 1.80\,(1/\gamma) - 1.85\,(1/\gamma^2) + 0.567\,(\delta/\gamma) + 5.24\,(\delta/\gamma^2) - 1.08\,(\delta^2/\gamma^2) + 0.0166\,(\delta^3/\gamma^2) + 7.79\,(1/\gamma^4)$$

$$= 1.282 + 1.282\,(1.72/5.15) + 1.80\,(1/21.43) - 1.85\,(1/21.43^2) + 0.567\,(5.15/21.43) + 5.24\,(5.15/21.43^2) - 1.08\,(5.15^2/21.43^2) + 0.0166\,(5.15^3/21.43^2) + 7.79\,(1/21.43^4)$$

$$= 1.94$$

Thus, a B-basis value is calculated as

$$B = \overline{X} - T\,(S_b^2 + S_e^2)^{0.5} = 68.84 - 1.94\,(4.97 + 10.47)^{0.5} = 61.22$$

For presentation in MIL-HDBK-17, this B-basis value would be rounded to 61.

8.7.3 Problem 3 - Weibull distribution. The data set for this problem consists of tensile strength measurements from nine batches of material. This problem illustrates the two-parameter Weibull goodness-of-fit test and the calculation of B-basis values by the Weibull method.

Problem 3 - Step 1. There are no detected outliers in this set of data. (See Problem 1 for details of the outlier detection computations.)

Problem 3 - Step 2. The k-sample Anderson-Darling test statistic is ADK = 1.01. (See Problem 1 for details of the computation of the k-sample statistic.) Since this is less than the critical value of 1.44, conclude that the data from the batches may be combined into a single sample.

Problem 3 - Step 3. In order to perform the two-parameter Weibull goodness-of-fit test described in Section 8.6.4.3, it is necessary to compute estimates of the scale and shape parameters, $\hat{\alpha}$ and $\hat{\beta}$. A procedure for doing this is described in Section 8.5.6.1. The geometric mean of the data is computed as

$$\bar{X}_G = \exp\left[\frac{1}{n}\sum_{i=1}^{n} \ell n(X_i)\right]$$

$$= \exp\left[\frac{1}{53}\sum_{i=1}^{53} \ell n(X_i)\right]$$

$$= 67.501$$

For a given value of $\hat{\beta}$, $\hat{\alpha}$ is calculated as

$$\hat{\alpha} = \bar{X}_G \left[\frac{1}{n}\sum_{i=1}^{n}\left(\frac{X_i}{\bar{X}_G}\right)^{\hat{\beta}}\right]^{\frac{1}{\hat{\beta}}}$$

$$= (67.501)\left[\frac{1}{53}\sum_{i=1}^{53}\left(\frac{X_i}{67.501}\right)^{\hat{\beta}}\right]^{\frac{1}{\hat{\beta}}}$$

In order to calculate $\hat{\beta}$, define the function $G(\hat{\beta})$ by

$$G(\hat{\beta}) = \frac{1}{n} \sum_{i=1}^{n} \ell n(X_i) \left(\left[\frac{X_i}{\hat{\alpha}} \right]^{\hat{\beta}} - 1 \right) - \frac{1}{\hat{\beta}}$$

$$= \frac{1}{53} \sum_{i=1}^{53} \ell n(X_i) \left(\left[\frac{X_i}{\hat{\alpha}} \right]^{\hat{\beta}} - 1 \right) - \frac{1}{\hat{\beta}}$$

where $\hat{\alpha}$ is calculated as above. The estimate, $\hat{\beta}$, is the solution to the Equation $G(\hat{\beta}) = 0$. An iterative technique for solving this equation is given in Section 8.5.6.2, and begins by setting

$$\hat{\beta} = \frac{1.28}{S_y} = \frac{1.28}{0.1044} = 12.2605$$

The solution is $\hat{\beta} = 12.27$, which in turn gives $\hat{\alpha} = 70.77$.

The first five ordered observations are listed below with the transformations necessary to compute the goodness-of-fit test statistic.

$X_{(i)}$	$Z_{(i)} = \left(\frac{X_{(i)}}{\hat{\alpha}} \right)^{\hat{\beta}} = \left(\frac{X_{(i)}}{70.77} \right)^{12.27}$
47.1	0.0068
51.6	0.0207
56.0	0.0566
59.4	0.1166
60.4	0.1431
.	.
.	.
.	.

The goodness-of-fit statistic and observed significance level are calculated as follows.

$$AD = \sum_{i=1}^{n} \frac{1-2i}{n} \left[\ln [1 - \exp(-Z_{(i)})] \; - \; Z_{(n+1-i)} \right] - n$$

$$= \sum_{i=1}^{53} \frac{1-2i}{53} \left[\ln(1 - \exp(-Z_{(i)})) - Z_{(54-i)} \right] - 53$$

$$= 0.535$$

$$AD^* = (1 + 0.2/\sqrt{n})AD$$

$$= (1 + 0.2/\sqrt{53})\; 0.535$$

$$= 0.5497$$

$$OSL = 1/[1 + \exp(-0.10 + 1.24\; \ln(AD^*) + 4.48(AD^*))]$$

$$= 1/[1 + \exp(-0.10 + 1.24\; \ln(0.5497) + 4.48(0.5497))]$$

$$= 0.165$$

Since the Weibull goodness-of-fit test yields an OSL value greater than 0.05, there is insufficient evidence to contradict the assumption that the data follow a two-parameter Weibull distribution. Hence, the two parameter Weibull method in Section 8.5.6.2 should be used to compute the B-basis value.

<u>Problem 3 - Step 4</u>. The parameter estimates $\hat{\alpha}$ and $\hat{\beta}$ calculated in the previous step are used to compute the B-basis value for the sample as described in Section 8.5.6. The quantities necessary to compute the B-basis value are:

$$V = 4.670 \text{ (from Table 8.8.2)}$$

$$\hat{\alpha} = 70.77$$

$$\hat{\beta} = 12.27$$

$$\hat{Q} = \hat{\alpha}(0.10536)^{1/\hat{\beta}} = (70.77)(0.10536)^{1/12.27} = 58.91$$

The B-basis value is calculated as

$$B = \hat{Q} \exp \{-V/(\hat{\beta}\sqrt{n})\}$$

$$= 58.91 \exp\{-4.670/[(12.27)\sqrt{53}]\}$$

$$= 55.91$$

For presentation in MIL-HDBK-17, this B-basis value would be rounded to 56.

8.7.4 Problem 4 - Normal distribution. The data set for this problem consists of compression test measurements from nine batches of material. This problem illustrates the normal goodness-of-fit test and the calculation of B-basis values by the normal method.

Problem 4 - Step 1. There are no detected outliers in this set of data. (See Problem 1 for details of the outlier detection calculations.)

Problem 4 - Step 2. The k-sample Anderson-Darling test statistic is ADK=1.07 (see Problem 1 for a detailed computation of the k-sample statistic). Since this is less than the critical value of 1.44, conclude that the data from the batches may be combined and treated as a single sample. The next step is to investigate the form of the distribution.

Problem 4 - Step 3. The Weibull goodness-of-fit test yields an observed significance level of 0.025. (See Problem 3 for details of the computation for the Weibull goodness-of-fit test.) Since this is less than 0.05, the normal goodness-of-fit test described in Section 8.6.4.1 is performed.

Problem 4 - Step 4. The average and standard deviation of the sample are 30.25 and 1.49, respectively. The first five ordered observations are listed below with the Z-values and the values of the standard normal distribution necessary for calculation of the normal Anderson-Darling statistic.

$X_{(i)}$	$Z_{(i)} = \frac{X_{(i)} - \bar{X}}{S} = \frac{X_{(i)} - 30.25}{1.49}$	$F_o\ Z_{(i)}$
26.9	-2.25	0.0122
27.7	-1.71	0.0436
27.9	-1.58	0.0571
27.9	-1.58	0.0571
28.0	-1.51	0.0655
.	.	.
.	.	.
.	.	.

$$AD = \sum_{i=1}^{n} \frac{1 - 2i}{n} \left[\ell n[F_o(Z_{(i)})] + \ell n[1 - F_o(Z_{(n+1-i)})] \right] - n$$

$$= \sum_{i=1}^{53} \frac{1 - 2i}{53} \left[\ell n[F_o(Z_{(i)})] + \ell n[1 - F_o(Z_{(54-i)})] \right] - 53$$

$$= 0.363$$

$$AD^* = [1 + 4/n - 25/n^2]AD = [1 + 4/53 - 25/(53)^2](0.363) = 0.3872$$

$$OSL = 1/\{1 + \exp[-0.48 + 0.78\ \ell n(AD^*) + 4.58\ (AD^*)]\}$$

$$= 1/\{1 + \exp[-0.48 + 0.78\ \ell n(0.3872) + 4.58\ (0.3872)]\}$$

$$= 0.365$$

Since the normal goodness-of-fit test yields an OSL value (0.365) greater than 0.05, there is insufficient evidence to contradict the assumption that the data are normally distributed. Hence, the normal method in Section 8.5.5 is used to compute a B-basis value.

Problem 4 - Step 5. From Table 8.8.1, the one-sided tolerance limit factor, k_B, is 1.634. The B-basis value for a normally distributed sample is computed as

$$B = \bar{X} - k_B\ S = 30.25 - (1.634)(1.49) = 27.81$$

For presentation in MIL-HDBK-17, this B-basis value would be rounded to 28.

8.7.5 Problem 5 - Lognormal distribution. The data set for this problem consists of compression test measurements from nine batches of material. This problem illustrates the lognormal goodness-of-fit test and the calculation of B-basis values by the lognormal method.

Problem 5 - Step 1. There are no detected outliers in this set of data. (See Problem 1 for details of the outlier detection calculations.)

Problem 5 - Step 2. The k-sample Anderson-Darling test statistic is ADK = 1.26. (See Problem 1 for details of the computation of the k-sample statistic.) Since this is less than the critical value of 1.44, conclude that the data from the batches may be combined into a single sample.

Problem 5 - Step 3. The observed significance levels (OSL) for the two-parameter Weibull and the normal goodness-of-fit tests are given below:

Distribution	OSL
Two-parameter Weibull	0.000
Normal	0.034

(See Problems 3 and 4 for details of the computations for these tests.) Since the OSLs are both less than 0.05, neither of the distributions adequately describe the data. Thus, the lognormal goodness-of-fit test is performed.

Problem 5 - Step 4. In order to perform the lognormal goodness-of-fit test described in Section 8.6.4.2, the natural logarithms of the data are used. The average and standard deviation of the transformed data are

$$\bar{X}_L = 3.41$$

$$S_L = 0.055$$

The first five ordered observations are listed below with the transformations necessary to compute the goodness-of-fit statistic.

$X_{(i)}$	$\ell n(X_{(i)})$	$Z_{(i)} = \frac{\ell n(X_{(i)})-\bar{X}_L}{s_L} = \frac{\ell n(X_{(i)})-3.41}{0.055}$	$F_o\ Z_{(i)}$
27.3	3.31	-1.82	0.0344
27.3	3.31	-1.82	0.0344
27.5	3.31	-1.82	0.0344
27.9	3.33	-1.45	0.0735
28.4	3.35	-1.09	0.1379
.	.	.	.
.	.	.	.
.	.	.	.

The goodness-of-fit statistic and observed significance level are calculated as:

$$AD = \sum_{i=1}^{n} \frac{1-2i}{n} \left[\ell n[F_o(Z_{(i)})] + \ell n[1 - F_o(Z_{(n+1-i)})] \right] - n$$

$$= \sum_{i=1}^{53} \frac{1-2i}{53} \left[\ell n[F_o(Z_{(i)})] + \ell n[1 - F_o(Z_{(54-i)})] \right] - 53$$

$$= 0.650$$

$$AD^* = [1 + 4/n - 25/n^2]AE = [1 + 4/53 - 25/(53)^2](0.650) = 0.6933$$

$$OSL = 1/\{1 + \exp[-0.48 + 0.78\ \ell n(AD^*) + 4.58\ (AD^*)]\}$$

$$= 1/\{1 + \exp[-0.48 + 0.78\ \ell n(0.6933) + 4.58\ (0.6933)]\}$$

$$= 0.083$$

Since the lognormal goodness-of-fit test results in an OSL value greater than 0.05, there is insufficient evidence to contradict the assumption that the data are lognormally distributed. Hence, the lognormal method in Section 8.5.5.2 is used to compute a B-basis value.

Problem 5 - Step 5. The B-basis value for lognormally distributed data is computed as

$$B = \exp(\bar{X}_L - k_B S_L) = \exp\{3.41 - 1.634(0.055)\} = 27.66$$

For presentation in MIL-HDBK-17, this B-basis value would be rounded to 27.

8.7.6 Problem 6 - Nonparametric method. The data set for this problem consists of tensile strength measurements for nine batches of material. This problem illustrates the calculation of B-basis values by the nonparametric method.

Problem 6 - Step 1. There are no detected outliers in this set of data. (See Problem 1 for details of the outlier detection calculations.)

Problem 6 - Step 2. The k-sample Anderson-Darling test statistic is ADK = 0.90. (See Problem 1 for details of the computation of the k-sample statistic.) Since this is less than the critical value of 1.44, conclude that the data from the batches may be combined into a single sample.

Problem 6 - Step 3. The results of the goodness-of-fit tests for the three distributions are:

Distribution	OSL
Two-parameter Weibull	0.013
Normal	0.008
Lognormal	0.007

(See problems 3, 4, and 5 for details of the computations for each of these tests.)

Since all of the observed significance levels are less than 0.05, it is concluded that the data do not follow any of the three distributions. Thus, the nonparametric method described in Section 8.5.7 must be used to calculate the B-basis value.

Problem 6 - Step 4. The first step in computing a B-basis value by the nonparametric method is to order the data values from smallest to largest. The five smallest values are 64.3, 64.9, 65.1, 65.3, and 65.6. The next step is to obtain the appropriate rank from Table 8.8.3 corresponding to the sample of size n. With an n of 53, the rank of the observation to be used as a B-basis value is r = 2.

Thus, the second observation, or 64.9, is the B-basis value for this sample. For presentation in MIL-HDBK-17, this B-basis value would be rounded to 65.

8.7.7 Problem 7 - Insufficient data. The data set for this problem consists of tensile strength measurements for five batches of material. This problem illustrates the situation where none of the standard distributions adequately fit the data, but there is insufficient data to perform the nonparametric method.

Problem 7 - Step 1. There are no detected outliers in this set of data. (See Problem 2 for details of the outlier detection calculations.)

Problem 7 - Step 2. The k-sample Anderson-Darling test statistic is ADK = 0.61. (See Problem 1 for details of the computation of the k-sample statistic.) Since this is less than the critical value of 1.62, conclude that the data from the batches may be combined into a single sample.

Problem 7 - Step 3. The results of the goodness-of-fit tests for the three distributions are:

Distribution	OSL
Two-parameter Weibull	0.031
Normal	0.011
Lognormal	0.006

(See problems 3, 4, and 5 for details of the computations for each of these tests.)

Since all of the observed significance levels are less than 0.05, it is concluded that the data do not follow any of the three distributions. The nonparametric method cannot be used, however, because there are only 25 data values in the sample. The Hanson-Koopmans method should be used to calculate a B-basis value for these data.

Problem 7 - Step 4. Following the procedure described in Section 8.5.7.2, a B-basis value can be estimated. For n = 25, from Table 8.8.11 it is determined that r = 11 and k = 1.087. After ranking the data in ascending order, the first and eleventh values are found.

$X_{(1)} = 55.9$ $X_{(11)} = 72.2$

$$B = X_{(r)} - k\,[X_{(r)} - X_{(1)}]$$

$$= 72.2 - 1.087\ (72.2 - 55.9)$$

$$= 54.5$$

These data can be included in MIL-HDBK-17 as interim data, but the B-value would not be reported in the handbook.

8.7.8 Problem 8 - Regression analysis. The data set for this problem consists of compression test measurements and corresponding specimen thickness measurements. This problem illustrates the regression analysis procedures presented in Sections 8.5.8 and 8.6.6.

Problem 8 - Step 1. In this example, x represents the thickness and y the compressive yield strength determined from a group of compression tests. From the data in Table 8.7, the following quantities may be calculated:

$n = 20$

$\Sigma x = 2.496$

$\Sigma y = 562.5$

$\Sigma x^2 = 0.312638$

$\Sigma y^2 = 15923.85$

$(\Sigma x)^2 = 6.23$

$(\Sigma y)^2 = 316406.25$

$(\Sigma x)(\Sigma y) = 1404$

$\Sigma xy = 69.8974$

$$S_{xx} = \Sigma x^2 - (\Sigma x)^2/n = 0.312638 - (6.23)/20 = 0.001137$$

$$S_{xy} = \Sigma xy - (\Sigma x)(\Sigma y)/n = 69.8974 - (2.946)(562.5)/20 = -0.3026$$

$$S_{yy} = \Sigma y^2 - (\Sigma y)^2/n = 15923.85 - (316406.25)/20 = 103.5$$

The slope of regression line is:

$$b = \frac{S_{xy}}{S_{xy}} = \frac{-0.3026}{0.001137} = 266$$

The y-intercept of the regression line is:

$$a = \frac{\Sigma y - b\Sigma x}{n} = \frac{562.5}{20} - \frac{(-266)(2.496)}{20} = 28.1 - (-33.2) = 61.3$$

Thus, the final equation of the least squares regression line is:

$$y^* = a + bx = 61.3 - 266x$$

Using this equation, the following values of y^* may be computed for the values of x in the data set:

x	y^*	$e = y - y^*$
0.112	31.5	0.5
0.112	31.5	0.5
0.116	30.5	-0.4
0.116	30.5	1.6
0.119	29.7	-1.6
0.119	29.7	0.8
0.121	29.1	-2.3
0.122	28.9	-0.8
0.124	28.5	0.9
0.124	28.5	0.1
0.126	27.8	-0.5
0.126	27.8	0.2
0.127	27.5	1.7
0.128	27.3	-1.3
0.129	27.0	0.6
0.133	25.9	0.9
0.135	25.4	1.4
0.135	25.4	-1.4
0.135	25.4	-0.2
0.137	24.9	-0.1

The root mean square error is computed as follows:

$$S_y = \left[\frac{\Sigma(y - y^*)^2}{n - 2} \right]^{0.5} = \left[\frac{23.18}{18} \right]^{0.5}$$

or

$$S_y = \left[\frac{S_{yy} - b^2 S_{xx}}{n - 2} \right]^{0.5} = \left[\frac{103.5 - (-266)^2(0.001137)}{18} \right]^{0.5} = 1.13$$

R^2 is computed as follows:

$$R^2 = \frac{b^2 S_{xx}}{S_{yy}} = \frac{(-266)^2(0.001137)}{103.5} = 0.78$$

Thus, 78% of the variability in the y data about its average is explained by the linear relationship between y and x.

Problem 8 - Step 2. One of the assumptions made in linear regression analysis is that the residuals are normally distributed about the regression line. The validity of this assumption may be checked by performing a normal goodness-of-fit test on the residuals as discussed in Section 8.6.4.5. Note that the $Z_{(i)}$ values used in the Anderson-Darling statistic are defined as $Z_{(i)} = e_{(i)}/S_y$, where $e_{(i)}$ is the ith ordered residual and S_y is the root-mean-square error from the regression. The first five ordered residuals and the preliminary goodness-of-fit calculations are shown in the following table:

$e_{(i)}$	$Z_{(i)} = \frac{e_{(i)}}{S_y} = \frac{e_{(i)}}{1.13}$
-2.3	-2.04
-1.6	-1.42
-1.4	-1.24
-1.3	-1.15
-0.8	-0.71
.	.
.	.
.	.

The normal goodness-of-fit test statistic is 0.2391 with an OSL of 0.562. (See Problem 4 for details of the computation for the normal goodness-of-fit test.) Since the OSL is greater than 0.05, there is insufficient evidence to contradict the assumption that the residuals are normally distributed.

Problem 8 - Step 3. There are multiple y observations for several of the x values. Thus, it is possible to construct an analysis of variance table to test the adequacy of the regression as discussed in Section 8.6.6.2. The sums of squares for the three primary lines of the analysis of variance table are calculated as follows:

$$SSR = b^2 S_{xx} = (-266)^2 (0.001137) = 80.45$$

$$SST = S_{yy} = 103.5$$

$$SSE = SST - SSR = 103.5 - 80.45 = 23.05$$

The mean squares are calculated as shown below.

$$MSR = SSR = 80.45$$

$$MSE = SSE/(n - 2) = 23.05/18 = 1.28$$

$$F = MSR/MSE = 80.45/1.28 = 62.8$$

The analysis of variance table is shown below.

Source of Variation	Degrees of Freedom	Sum of Squares SS	Mean Squares, MS	F_{calc}
Regression	1	80.45	80.45	F = 62.8
Error	18	23.05	1.28	
Total	19	103.50		

The F value of 62.8 with 1 and n - 2 = 18 degrees of freedom is greater than the value of 4.41 from Table 8.8.5 corresponding to 1 and 18 degrees of freedom, so the regression is declared to be significant.

<u>Problem 8 - Step 4</u>. With the linear regression equation from step 1, lower tolerance limits may be calculated at any thickness (x value) by the procedure in Section 8.5.8. Details for computing a B-basis value at x = 0.120 are given below.

The average thickness value in the data set is:

$$\bar{x} = \Sigma x/n = 2.496/20 = 0.125$$

The Δ factor required to compute the tolerance limit factor, k'_B, is:

$$\Delta = \frac{(x_o - \bar{x})^2}{\sum_{i=1}^{n} (x_i - \bar{x})^2/n} = \frac{(0.120 - 0.125)^2}{(0.01137)/20} = 0.44$$

The approximation for the k'_B factor is:

$$\begin{aligned} k'_B &= 1.282 + \exp\{0.595 - 0.508\ \ell n(n) + 4.62/n \\ &\quad + (0.486 - 0.986/n)\ \ell n(1.82 + \Delta)\} \\ &= 1.282 + \exp\{0.595 - 0.508\ \ell n(20) + 4.62/20 \\ &\quad + (0.486 - 0.986/20)\ \ell n(1.82 + 0.44)\} \\ &= 1.99 \end{aligned}$$

Thus, a B-basis value at $x = 0.120$ is computed as

$$\begin{aligned} B &= (a + b\ x_o) - k'_B\ S_y \\ &= [61.3 - 266\ (0.120)] - 1.99\ (1.13) \\ &= 27.13 \end{aligned}$$

For presentation in MIL-HDBK-17, this value would be rounded to 27.

8.8 Statistical tables. This section contains a number of tables which are required for the analyses described in Sections 8.5 and 8.6. Tables 8.8.4 through 8.8.7 and Tables 8.8.9 and 8.8.10 were generated specifically for MIL-HDBK-17. The remaining tables were adapted from MIL-HDBK-5 (see Reference 8.5.5).

For some of the tabulated values, theoretical derivations and numerical approximations are provided below. The approximations are useful in computer applications when the software required to generate the tabulated values is not available. The accuracy of the approximations is measured by the relative magnitude of error (RME). The RME is defined as

$$\text{RME} = \left| \frac{\begin{matrix}\text{approximate}\\ \text{value}\end{matrix} - \begin{matrix}\text{actual}\\ \text{value}\end{matrix}}{\begin{matrix}\text{actual}\\ \text{value}\end{matrix}} \right| \qquad 8.8$$

and measures the percentage error in the approximate value with respect to the actual value.

8.8.1 One-sided tolerance factors, k_B, for the normal distribution, 0.95 confidence. The k_B values in Table 8.8.1 are calculated as $1/\sqrt{n}$ times the 0.95th quantile of the noncentral t-distribution with noncentrality parameter $1.282\sqrt{n}$ and n - 1 degrees of freedom. An approximation to the k_B values in Table 8.8.1 is:

$$k_B \approx 1.282 + \exp\{0.958 - 0.520\,\ell n(n) + 3.19/n\} \qquad 8.8.1$$

This approximation is accurate to within 0.2% of the tabulated values for n greater than or equal to 16.

8.8.2 One-sided tolerance factors, V, for the Weibull distribution, 0.95 confidence. The V values in Table 8.8.2 are calculated using the following statistical results. First, define the random variables

$$A_i = \frac{\ell n\,(X_i) - \ell n(\hat{\alpha})}{1/\hat{\beta}} \qquad i = 1, \ldots, n \qquad 8.8.2(a)$$

where X_i is a Weibull random variable with unknown shape and scale parameters β and α and $\hat{\alpha}$ and $\hat{\beta}$ are the maximum likelihood estimators (MLE's) of β and α given by Equations 8.5.6.1(a) and 8.5.6.1(c). For a particular n, the V value is the 0.95th quantile of the conditional distribution of the random variable

$$V = \frac{\sqrt{n}[\ln(\hat{Q}) - \ln(Q)]}{1/\hat{\beta}} \qquad 8.8.2(b)$$

given that

$$A_i = \frac{\ln(x_i') - \ln(\hat{\alpha}')}{1/\hat{\beta}'} \qquad 8.8.2(c)$$

where

$$x_i' = -\ln\left(1 - \frac{i - 0.5}{n + 0.25}\right) \quad i = 1,\ldots,n \qquad 8.8.2(d)$$

$$\hat{Q} = \hat{\alpha}(0.10536)^{1/\beta} \qquad 8.8.2(e)$$

$$Q = \alpha(0.10536)^{1/\beta} \qquad 8.8.2(f)$$

and $\hat{\alpha}'$ and $\hat{\beta}'$ are the MLE's of the two-parameter Weibull scale and shape parameters for the sample $x_1', \ldots, x_n'$. The conditional distribution of V is determined by the relationship

$$V = \sqrt{n}\,[Z + \ln(0.10536)] \qquad 8.8.2(g)$$

where the distribution of Z is given in Theorem 4.1.3 on page 150 of reference 8.6.4.1. Numerical integration was used to determine the V values in Table 8.8.2 based on these results.

An approximation to the V values in Table 8.8.2 is:

$$V \approx 3.803 + \exp\{1.79 - 0.516\ln(n) + 5.1/n\} \qquad 8.8.2(h)$$

This approximation is accurate to within 0.5% of the tabulated values for n greater than or equal to 16.

8.8.3 Ranks, r, of observations for determining B-values for an unknown distribution. For n > 29, an approximation to the ranks for B-basis values in Table 8.8.3 is

$$r = n/10 - 1.645\sqrt{9n/100} + 0.23 \qquad 8.8.3$$

rounded to the nearest integer. This approximation is exact for all but 12 values of n in the range of the table ($29 \leq n \leq 10499$). For this small percentage of n values (0.1%), the approximation errs by one rank on the conservative side.

8.8.4 0.20 Quantiles of the F distribution associated with γ_1 and γ_2 degrees of freedom. An approximation to the $F_{0.20}$ values in Table 8.8.4 is

$$F_{0.20} = \exp\left\{ 2\delta\left[1 + \frac{z^2-1}{3} - \frac{4\sigma^2}{3}\right] + 2\sigma z\left[1 + \frac{\sigma^2(z^2-3)}{6}\right]^{0.5} \right\} \qquad 8.8.4(a)$$

where

$$z = -0.84$$

$$\delta = 0.5\{1/(\gamma_2-1) - 1/(\gamma_1-1)\} \qquad 8.8.4(b)$$

$$\sigma^2 = 0.5\{1/(\gamma_2-1) + 1/(\gamma_1-1)\} \qquad 8.8.4(c)$$

γ_1 = numerator degrees of freedom

γ_2 = denominator degrees of freedom.

(See Reference 8.8.4.)

8.8.5 0.95 Quantiles of the F distribution associated with γ_1 and γ_2 degrees of freedom. An approximation to the $F_{0.95}$ values in Table 8.8.5 is

$$F_{0.20} = \exp\left\{ 2\delta\left[1 + \frac{z^2-1}{3} - \frac{4\sigma^2}{3}\right] + 2\sigma z\left[1 + \frac{\sigma^2(z^2-3)}{6}\right]^{0.5} \right\} \qquad 8.8.5(a)$$

where

$$z = 1.645$$

$$\delta = 0.5\{1/(\gamma_2-1) - 1/(\gamma_1-1)\} \qquad 8.8.5(b)$$

$$\sigma^2 = 0.5\{1/(\gamma_2-1) + 1/(\gamma_1-1)\} \qquad 8.8.5(c)$$

γ_1 = numerator degrees of freedom

γ_2 = denominator degrees of freedom.

(See Reference 8.8.4.)

8.8.6 <u>One-sided tolerance factors, T, for the one-way random effects ANOVA, 0.95 confidence</u>. The tolerance limit factor T is defined in as

$$T = \{(n^*\hat{R}+1)/[N(\hat{R}+1)]\}^{0.5}\ t_{\gamma,0.95}(\delta) \qquad 8.8.6(a)$$

where $t_{\gamma;0.95}(\delta)$ is the 0.95 quantile of the non-central t-distribution with noncentrality parameter

$$\delta = 1.282\ \{\ N(\hat{R}+1)/(n^*\hat{R}+1)\}^{0.5} \qquad 8.8.6(b)$$

degrees of freedom approximated by

$$\gamma = \frac{(\hat{R} + 1\ )^2}{\dfrac{(\hat{R} + \frac{1}{n*})^2}{k* - 1} + \dfrac{(\frac{n*-1}{n*})^2}{k*(n* - 1)}} \qquad 8.8.6(c)$$

Tolerance limit factors may also be computed from the following approximate formula:

$$
\begin{aligned}
T = 1.282 &+ 1.282\,(t_{\gamma,0.95}/\delta) + 1.80\,(1/\gamma) \\
&- 1.85\,(1/\gamma^2) + 0.567\,(\delta/\gamma) + 5.24\,(\delta/\gamma^2) \\
&- 1.08\,(\delta^2/\gamma^2) + 0.0166\,(\delta^3/\gamma^2) + 7.79\,(1/\gamma^4)
\end{aligned}
\qquad 8.8.6(d)
$$

This approximation is accurate to within 0.45% of the tabulated values. This approximation may be used for values of n^*, k^* and $\hat{R}$ both within and beyond the range of the table.

8.8.7 Critical values for the k-sample Anderson-Darling test at the 0.05 significance level. The k-sample Anderson-Darling test critical values in Table 8.8.7 were calculated using Equation 8.6.3.1(j) for the case of equal sample sizes.

8.8.8 0.95 Quantiles of the chi-squared distribution associated with γ degrees of freedom. An approximation to the chi-squared quantiles ($\chi^2_{0.95}$) in Table 8.8.8 is:

$$
\chi^2_{0.95} = \gamma \left[1 - \frac{2}{9\gamma} + 1.645\left(\frac{2}{9\gamma}\right)^{0.5}\right]^3 + \frac{9}{100\gamma} \qquad 8.8.8
$$

where γ is the degrees of freedom. This approximation is accurate to within 0.2% of the tabulated values. (See Reference 8.8.8.)

8.8.9 Critical values for the MNR outlier detection procedure ($\alpha = 0.05$). The critical values in Table 8.8.9 are computed by the following formula:

$$
C.V. = \frac{n-1}{\sqrt{n}} \left[\frac{t^2}{n - 2 + t^2} \right]^{0.5} \qquad 8.8.9
$$

where t is the $[1 - \alpha/(2n)]$ quantile of the t distribution with $n - 2$ degrees of freedom, α is the significance level of the test, and n is the sample size. Numbers in Table 8.8.9 are computed with a significance level of $\alpha = 0.05$. (See Reference 8.6.2(b))

8.8.10 Critical values for the MNR outlier detection procedure ($\alpha = 0.01$). The critical values in Table 8.8.10 are computed according to Equation 8.8.9 with significance level $\alpha = 0.01$.

8.8.11 Nonparametric B-basis material properties for small samples. The values in Table 8.8.11 are based on Reference 8.5.7.2.

TABLE 8.8.1 One-sided tolerance limit factors, k_B, for the normal distribution, 0.95 confidence.

n = 2 - 136

n	k_B	n	k_B	n	k_B
2	20.581	47	1.660	92	1.539
3	6.157	48	1.655	93	1.537
4	4.163	49	1.650	94	1.536
5	3.408	50	1.646	95	1.534
6	3.007	51	1.642	96	1.533
7	2.756	52	1.638	97	1.531
8	2.583	53	1.634	98	1.530
9	2.454	54	1.630	99	1.529
10	2.355	55	1.626	100	1.527
11	2.276	56	1.623	101	1.526
12	2.211	57	1.619	102	1.525
13	2.156	58	1.616	103	1.523
14	2.109	59	1.613	104	1.522
15	2.069	60	1.609	105	1.521
16	2.034	61	1.606	106	1.519
17	2.002	62	1.603	107	1.518
18	1.974	63	1.600	108	1.517
19	1.949	64	1.597	109	1.516
20	1.927	65	1.595	110	1.515
21	1.906	66	1.592	111	1.513
22	1.887	67	1.589	112	1.512
23	1.870	68	1.587	113	1.511
24	1.854	69	1.584	114	1.510
25	1.839	70	1.582	115	1.509
26	1.825	71	1.579	116	1.508
27	1.812	72	1.577	117	1.507
28	1.800	73	1.575	118	1.506
29	1.789	74	1.572	119	1.505
30	1.778	75	1.570	120	1.504
31	1.768	76	1.568	121	1.503
32	1.758	77	1.566	122	1.502
33	1.749	78	1.564	123	1.501
34	1.741	79	1.562	124	1.500
35	1.733	80	1.560	125	1.499
36	1.725	81	1.558	126	1.498
37	1.718	82	1.556	127	1.497
38	1.711	83	1.554	128	1.496
39	1.704	84	1.552	129	1.495
40	1.698	85	1.551	130	1.494
41	1.692	86	1.549	131	1.493
42	1.686	87	1.547	132	1.492
43	1.680	88	1.545	133	1.492
44	1.675	89	1.544	134	1.491
45	1.669	90	1.542	135	1.490
46	1.664	91	1.540	136	1.489

TABLE 8.8.1 <u>One-sided tolerance limit factors, k_B, for the normal distribution, 0.95 confidence</u> - Continued.

n = 137 - ∞

n	k_B	n	k_B	n	k_B
137	1.488	183	1.458	345	1.408
138	1.487	184	1.458	350	1.407
139	1.487	185	1.457	355	1.406
140	1.486	186	1.457	360	1.405
141	1.485	187	1.456	365	1.404
142	1.484	188	1.456	370	1.403
143	1.483	189	1.455	375	1.402
144	1.483	190	1.455	380	1.402
145	1.482	191	1.454	385	1.401
146	1.481	192	1.454	390	1.400
147	1.480	193	1.453	395	1.399
148	1.480	194	1.453	400	1.398
149	1.479	195	1.452	425	1.395
150	1.478	196	1.452	450	1.391
151	1.478	197	1.451	475	1.388
152	1.477	198	1.451	500	1.386
153	1.476	199	1.450	525	1.383
154	1.475	200	1.450	550	1.381
155	1.475	205	1.448	575	1.378
156	1.474	210	1.446	600	1.376
157	1.473	215	1.444	625	1.374
158	1.473	220	1.442	650	1.372
159	1.472	225	1.440	675	1.371
160	1.472	230	1.438	700	1.369
161	1.471	235	1.436	725	1.367
162	1.470	240	1.434	750	1.366
163	1.470	345	1.433	775	1.364
164	1.469	250	1.431	800	1.363
165	1.468	255	1.430	825	1.362
166	1.468	260	1.428	850	1.361
167	1.467	265	1.427	875	1.359
168	1.467	270	1.425	900	1.358
169	1.466	275	1.424	925	1.357
170	1.465	280	1.422	950	1.356
171	1.465	285	1.421	975	1.355
172	1.464	290	1.420	1000	1.354
173	1.464	295	1.419	1500	1.340
174	1.463	300	1.417	2000	1.332
175	1.463	305	1.416	3000	1.323
176	1.462	310	1.415	5000	1.313
177	1.461	315	1.414	10000	1.304
178	1.461	320	1.413	∞	1.282
179	1.460	325	1.412		
180	1.460	330	1.411		
181	1.459	335	1.410		
182	1.459	340	1.409		

TABLE 8.8.2 One-sided tolerance limit factors, V, for the Weibull distribution, 0.95 confidence.

n = 10 - 188

n	V	n	V	n	V
10	6.711	55	4.650	100	4.393
11	6.477	56	4.640	102	4.386
12	6.286	57	4.631	104	4.380
13	6.127	58	4.622	106	4.373
14	5.992	59	4.614	108	4.367
15	5.875	60	4.605	110	4.361
16	5.774	61	4.597	112	4.355
17	5.684	62	4.589	114	4.349
18	5.605	63	4.582	116	4.344
19	5.533	64	4.574	118	4.339
20	5.469	65	4.567	120	4.334
21	5.412	66	4.560	122	4.328
22	5.359	67	4.553	124	4.323
23	5.310	68	4.546	126	4.317
24	5.265	69	4.539	128	4.314
25	5.224	70	4.533	130	4.309
26	5.186	71	4.527	132	4.305
27	5.150	72	4.521	134	4.301
28	5.117	73	4.515	136	4.296
29	5.086	74	4.509	138	4.292
30	5.057	75	4.503	140	4.288
31	5.030	76	4.498	142	4.284
32	5.003	77	4.492	144	4.280
33	4.979	78	4.487	146	4.277
34	4.956	79	4.482	148	4.273
35	4.934	80	4.477	150	4.269
36	4.913	81	4.471	152	4.266
37	4.893	82	4.466	154	4.262
38	4.875	83	4.462	156	4.259
39	4.857	84	4.457	158	4.256
40	4.840	85	4.452	160	4.253
41	4.823	86	4.448	162	4.249
42	4.807	87	4.443	164	4.246
43	4.792	88	4.439	166	4.243
44	4.778	89	4.435	168	4.240
45	4.764	90	4.431	170	4.237
46	4.751	91	4.427	172	4.234
47	4.738	92	4.423	174	4.232
48	4.725	93	4.419	176	4.229
49	4.713	94	4.415	178	4.226
50	4.702	95	4.411	180	4.224
51	4.691	96	4.407	182	4.221
52	4.680	97	4.404	184	4.218
53	4.670	98	4.400	186	4.216
54	4.659	99	4.396	188	4.213

TABLE 8.8.2 One-sided tolerance limit factors, V, for the Weibull distribution, 0.95 confidence - Continued.

n = 190 - ∞

n	V	n	V
190	4.211	525	4.047
192	4.208	550	4.041
194	4.206	575	4.035
196	4.204	600	4.030
198	4.201	625	4.025
200	4.199	650	4.020
204	4.195	675	4.016
208	4.191	700	4.012
212	4.186	725	4.008
216	4.182	750	4.005
220	4.179	775	4.001
224	4.175	800	3.998
228	4.171	825	3.995
232	4.168	850	3.992
236	4.164	875	3.989
240	4.161	900	3.986
244	4.157	925	3.983
248	4.154	950	3.981
252	4.151	975	3.979
256	4.148	1000	3.976
260	4.145	1100	3.968
264	4.142	1200	3.960
268	4.140	1300	3.954
272	4.137	1400	3.948
276	4.134	1500	3.943
280	4.131	1600	3.939
284	4.129	1700	3.934
288	4.126	1800	3.931
292	4.124	1900	3.927
296	4.121	2000	3.924
300	4.119	3000	3.901
310	4.113	4000	3.887
320	4.108	5000	3.878
330	4.103	6000	3.872
340	4.098	7000	3.866
350	4.093	8000	3.862
360	4.089	9000	3.859
370	4.085	10000	3.856
380	4.081	15000	3.846
390	4.077	20000	3.840
400	4.073	∞	3.803
425	4.076		
450	4.067		
475	4.060		
500	4.053		

TABLE 8.8.3 Ranks, r, of observations for determining B-values for an unknown distribution.

n	r	n	r	n	r
28	†	781	65	4733	440
29	1	836	70	4836	450
46	2	890	75	4940	460
61	3	945	80	5044	470
76	4	999	85	5147	480
89	5	1053	90	5251	490
103	6	1107	95	5354	500
116	7	1161	100	5613	525
129	8	1269	110	5871	550
142	9	1376	120	6130	575
154	10	1483	130	6388	600
167	11	1590	140	6645	625
179	12	1696	150	6903	650
191	13	1803	160	7161	675
203	14	1909	170	7418	700
215	15	2015	180	7727	730
227	16	2120	190	8036	760
239	17	2226	200	8344	790
251	18	2331	210	8652	820
263	19	2437	220	8960	850
275	20	2542	230	9268	880
298	22	2647	240	9576	910
321	24	2752	250	9884	940
345	26	2857	260	10191	970
368	28	2962	270	10499	1000 *
391	30	3066	280		
413	32	3171	290		
436	34	3276	300		
459	36	3380	310		
481	38	3484	320		
504	40	3589	330		
526	42	3693	340		
549	44	3797	350		
571	46	3901	360		
593	48	4005	370		
615	50	4109	380		
638	52	4213	390		
660	54	4317	400		
682	56	4421	410		
704	58	4525	420		
726	60	4629	430		

† This procedure is unsatisfactory for $n < 28$.

* For $n > 10499$, $r \approx n/10 - 1.645 \sqrt{9n/100} + 0.23$

TABLE 8.8.4 0.20 Quantiles of the F distribution associated with γ_1 and γ_2 degrees of freedom.

	γ_1 numerator degrees of freedom								
γ_2 denominator degrees of freedom	1	2	3	4	5	6	7	8	9
1	0.11	0.28	0.37	0.43	0.46	0.48	0.50	0.51	0.52
2	0.08	0.25	0.35	0.40	0.44	0.47	0.49	0.50	0.52
3	0.08	0.24	0.34	0.40	0.44	0.47	0.50	0.51	0.53
4	0.07	0.24	0.34	0.40	0.45	0.48	0.50	0.52	0.53
5	0.07	0.23	0.34	0.40	0.45	0.48	0.51	0.53	0.54
6	0.07	0.23	0.34	0.40	0.45	0.48	0.51	0.53	0.55
7	0.07	0.23	0.34	0.41	0.45	0.49	0.51	0.54	0.55
8	0.07	0.23	0.34	0.41	0.45	0.49	0.52	0.54	0.56
9	0.07	0.23	0.34	0.41	0.46	0.49	0.52	0.54	0.56
10	0.07	0.23	0.34	0.41	0.46	0.49	0.52	0.54	0.56
11	0.07	0.23	0.34	0.41	0.46	0.49	0.52	0.55	0.56
12	0.07	0.23	0.34	0.41	0.46	0.50	0.52	0.55	0.57
13	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.55	0.57
14	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.55	0.57
15	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.55	0.57
16	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.55	0.57
17	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.55	0.57
18	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.56	0.58
19	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.56	0.58
20	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.56	0.58
21	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.56	0.58
22	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.56	0.58
23	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.56	0.58
24	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.56	0.58
25	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.56	0.58
26	0.07	0.23	0.34	0.41	0.46	0.50	0.53	0.56	0.58
27	0.07	0.22	0.34	0.41	0.46	0.50	0.54	0.56	0.58
28	0.07	0.22	0.34	0.41	0.46	0.50	0.54	0.56	0.58
29	0.07	0.22	0.34	0.41	0.46	0.50	0.54	0.56	0.58
30	0.07	0.22	0.34	0.41	0.46	0.50	0.54	0.56	0.58
40	0.07	0.22	0.34	0.41	0.46	0.51	0.54	0.56	0.59
60	0.06	0.22	0.34	0.41	0.47	0.51	0.54	0.57	0.59
120	0.06	0.22	0.34	0.41	0.47	0.51	0.54	0.57	0.59
∞	0.06	0.22	0.34	0.41	0.47	0.51	0.55	0.57	0.60

TABLE 8.8.4 0.20 Quantiles of the F distribution associated with r_1 and r_2 degrees of freedom - Continued.

r_2 denominator degrees of freedom	r_1 numerator degrees of freedom 10	12	15	20	24	30	40	60	120	∞
1	0.53	0.54	0.56	0.57	0.58	0.58	0.59	0.60	0.60	0.61
2	0.53	0.54	0.56	0.57	0.58	0.59	0.60	0.60	0.61	0.62
3	0.54	0.55	0.57	0.59	0.60	0.61	0.62	0.63	0.64	0.65
4	0.55	0.57	0.58	0.60	0.61	0.62	0.64	0.65	0.66	0.67
5	0.55	0.57	0.60	0.62	0.63	0.64	0.65	0.66	0.67	0.69
6	0.56	0.58	0.60	0.63	0.64	0.65	0.66	0.68	0.69	0.70
7	0.57	0.59	0.61	0.63	0.65	0.66	0.67	0.69	0.70	0.71
8	0.57	0.59	0.62	0.64	0.65	0.67	0.68	0.70	0.71	0.73
9	0.57	0.60	0.62	0.65	0.66	0.68	0.69	0.70	0.72	0.74
10	0.58	0.60	0.63	0.65	0.67	0.68	0.70	0.71	0.73	0.74
11	0.58	0.60	0.63	0.66	0.67	0.69	0.70	0.72	0.73	0.75
12	0.58	0.61	0.63	0.66	0.68	0.69	0.71	0.72	0.74	0.76
13	0.58	0.61	0.64	0.67	0.68	0.70	0.71	0.73	0.75	0.77
14	0.59	0.61	0.64	0.67	0.68	0.70	0.72	0.73	0.75	0.77
15	0.59	0.61	0.64	0.67	0.69	0.70	0.72	0.74	0.76	0.78
16	0.59	0.62	0.64	0.67	0.69	0.71	0.72	0.74	0.76	0.78
17	0.59	0.62	0.65	0.68	0.69	0.71	0.73	0.75	0.77	0.79
18	0.59	0.62	0.65	0.68	0.69	0.71	0.73	0.75	0.77	0.79
19	0.59	0.62	0.65	0.68	0.70	0.71	0.73	0.75	0.77	0.79
20	0.59	0.62	0.65	0.68	0.70	0.72	0.74	0.76	0.78	0.80
21	0.60	0.62	0.65	0.68	0.70	0.72	0.74	0.76	0.78	0.80
22	0.60	0.62	0.65	0.69	0.70	0.72	0.74	0.76	0.78	0.81
23	0.60	0.62	0.65	0.69	0.70	0.72	0.74	0.76	0.79	0.81
24	0.60	0.63	0.66	0.69	0.71	0.72	0.74	0.77	0.79	0.81
25	0.60	0.63	0.66	0.69	0.71	0.73	0.75	0.77	0.79	0.81
26	0.60	0.63	0.66	0.69	0.71	0.73	0.75	0.77	0.79	0.82
27	0.60	0.63	0.66	0.69	0.71	0.73	0.75	0.77	0.79	0.82
28	0.60	0.63	0.66	0.69	0.71	0.73	0.75	0.77	0.80	0.82
29	0.60	0.63	0.66	0.69	0.71	0.73	0.75	0.77	0.80	0.83
30	0.60	0.63	0.66	0.70	0.71	0.73	0.75	0.78	0.80	0.83
40	0.61	0.63	0.67	0.70	0.72	0.74	0.76	0.79	0.82	0.85
60	0.61	0.64	0.67	0.71	0.73	0.75	0.78	0.80	0.83	0.87
120	0.61	0.64	0.68	0.72	0.74	0.76	0.79	0.82	0.86	0.90
∞	0.62	0.65	0.69	0.73	0.75	0.78	0.81	0.84	0.89	1.00

TABLE 8.8.5 0.95 Quantiles of the F distribution associated with γ_1 and γ_2 degrees of freedom.

γ_2 denominator degrees of freedom	γ_1 numerator degrees of freedom 1	2	3	4	5	6	7	8	9
1	161.45	199.50	215.71	224.58	230.16	233.99	236.77	238.88	240.54
2	18.51	19.00	19.16	19.25	19.30	19.33	19.35	19.37	19.38
3	10.13	9.55	9.28	9.12	9.01	8.94	8.89	8.85	8.81
4	7.71	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00
5	6.61	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.77
6	5.99	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10
7	5.59	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68
8	5.32	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39
9	5.12	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18
10	4.96	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02
11	4.84	3.98	3.59	3.36	3.20	3.09	3.01	2.95	2.90
12	4.75	3.89	3.49	3.26	3.11	3.00	2.91	2.85	2.80
13	4.67	3.81	3.41	3.18	3.03	2.92	2.83	2.77	2.71
14	4.60	3.74	3.34	3.11	2.96	2.85	2.76	2.70	2.65
15	4.54	3.68	3.29	3.06	2.90	2.79	2.71	2.64	2.59
16	4.49	3.63	3.24	3.01	2.85	2.74	2.66	2.59	2.54
17	4.45	3.59	3.20	2.96	2.81	2.70	2.61	2.55	2.49
18	4.41	3.55	3.16	2.93	2.77	2.66	2.58	2.51	2.46
19	4.38	3.52	3.13	2.90	2.74	2.63	2.54	2.48	2.42
20	4.35	3.49	3.10	2.87	2.71	2.60	2.51	2.45	2.39
21	4.32	3.47	3.07	2.84	2.68	2.57	2.49	2.42	2.37
22	4.30	3.44	3.05	2.82	2.66	2.55	2.46	2.40	2.34
23	4.28	3.42	3.03	2.80	2.64	2.53	2.44	2.37	2.32
24	4.26	3.40	3.01	2.78	2.62	2.51	2.42	2.36	2.30
25	4.24	3.39	2.99	2.76	2.60	2.49	2.40	2.34	2.28
26	4.23	3.37	2.98	2.74	2.59	2.47	2.39	2.32	2.27
27	4.21	3.35	2.96	2.73	2.57	2.46	2.37	2.31	2.25
28	4.20	3.34	2.95	2.71	2.56	2.45	2.36	2.29	2.24
29	4.18	3.33	2.93	2.70	2.55	2.43	2.35	2.28	2.22
30	4.17	3.32	2.92	2.69	2.53	2.42	2.33	2.27	2.21
40	4.08	3.23	2.84	2.61	2.45	2.34	2.25	2.18	2.12
60	4.00	3.15	2.76	2.53	2.37	2.25	2.17	2.10	2.04
120	3.92	3.07	2.68	2.45	2.29	2.18	2.09	2.02	1.96
∞	3.84	3.00	2.61	2.37	2.21	2.10	2.01	1.94	1.88

TABLE 8.8.5 0.95 Quantiles of the F distribution associated with γ_1 and γ_2 degrees of freedom - Continued.

γ_2 denominator degrees of freedom	γ_1 numerator degrees of freedom									
	10	12	15	20	24	30	40	60	120	∞
1	241.88	243.91	245.95	248.01	249.05	250.10	251.14	252.20	253.25	254.31
2	19.40	19.41	19.43	19.45	19.45	19.46	19.47	19.48	19.49	19.51
3	8.79	8.74	8.70	8.66	8.64	8.62	8.59	8.57	8.55	8.53
4	5.96	5.91	5.86	5.80	5.77	5.75	5.72	5.69	5.66	5.63
5	4.74	4.68	4.62	4.56	4.53	4.50	4.46	4.43	4.40	4.37
6	4.06	4.00	3.94	3.87	3.84	3.81	3.77	3.74	3.70	3.67
7	3.64	3.57	3.51	3.44	3.41	3.38	3.34	3.30	3.27	3.23
8	3.35	3.28	3.22	3.15	3.12	3.08	3.04	3.01	2.97	2.93
9	3.14	3.07	3.01	2.94	2.90	2.86	2.83	2.79	2.75	2.71
10	2.98	2.91	2.85	2.77	2.74	2.70	2.66	2.62	2.58	2.54
11	2.85	2.79	2.72	2.65	2.61	2.57	2.53	2.49	2.45	2.40
12	2.75	2.69	2.62	2.54	2.51	2.47	2.43	2.38	2.34	2.30
13	2.67	2.60	2.53	2.46	2.42	2.38	2.34	2.30	2.25	2.21
14	2.60	2.53	2.46	2.39	2.35	2.31	2.27	2.22	2.18	2.13
15	2.54	2.48	2.40	2.33	2.29	2.25	2.20	2.16	2.11	2.07
16	2.49	2.42	2.35	2.28	2.24	2.19	2.15	2.11	2.06	2.01
17	2.45	2.38	2.31	2.23	2.19	2.15	2.10	2.06	2.01	1.96
18	2.41	2.34	2.27	2.19	2.15	2.11	2.06	2.02	1.97	1.92
19	2.38	2.31	2.23	2.16	2.11	2.07	2.03	1.98	1.93	1.88
20	2.35	2.28	2.20	2.12	2.08	2.04	1.99	1.95	1.90	1.84
21	2.32	2.25	2.18	2.10	2.05	2.01	1.96	1.92	1.87	1.81
22	2.30	2.23	2.15	2.07	2.03	1.98	1.94	1.89	1.84	1.78
23	2.27	2.20	2.13	2.05	2.01	1.96	1.91	1.86	1.81	1.76
24	2.25	2.18	2.11	2.03	1.98	1.94	1.89	1.84	1.79	1.73
25	2.24	2.16	2.09	2.01	1.96	1.92	1.87	1.82	1.77	1.71
26	2.22	2.15	2.07	1.99	1.95	1.90	1.85	1.80	1.75	1.69
27	2.20	2.13	2.06	1.97	1.93	1.88	1.84	1.79	1.73	1.67
28	2.19	2.12	2.04	1.96	1.91	1.87	1.82	1.77	1.71	1.65
29	2.18	2.10	2.03	1.94	1.90	1.85	1.81	1.75	1.70	1.64
30	2.16	2.09	2.01	1.93	1.89	1.84	1.79	1.74	1.68	1.62
40	2.08	2.00	1.92	1.84	1.79	1.74	1.69	1.64	1.58	1.51
60	1.99	1.92	1.84	1.75	1.70	1.65	1.59	1.53	1.47	1.39
120	1.91	1.83	1.75	1.66	1.61	1.55	1.50	1.43	1.35	1.25
∞	1.83	1.75	1.67	1.57	1.52	1.46	1.39	1.32	1.22	1.00

TABLE 8.8.6. One-sided tolerance factors, T, for the one-way random effects ANOVA, 0.95 confidence.

$\bar{R} = 0.00$

n^* \ k^*	2	3	4	5	6	7	8	9	10	15	20	∞
1	20.588	6.157	4.163	3.408	3.007	2.756	2.583	2.454	2.355	2.069	1.927	1.282
2	4.559	3.062	2.600	2.363	2.215	2.112	2.035	1.975	1.927	1.778	1.698	1.282
3	3.156	2.480	2.220	2.073	1.977	1.907	1.854	1.813	1.778	1.670	1.609	1.282
4	2.661	2.226	2.039	1.929	1.855	1.801	1.759	1.726	1.698	1.610	1.560	1.282
5	2.404	2.079	1.930	1.840	1.779	1.733	1.698	1.670	1.646	1.570	1.527	1.282
6	2.244	1.981	1.856	1.779	1.726	1.686	1.655	1.630	1.610	1.542	1.504	1.282
7	2.134	1.911	1.802	1.734	1.686	1.651	1.623	1.601	1.582	1.521	1.486	1.282
8	2.053	1.858	1.760	1.699	1.655	1.623	1.598	1.577	1.560	1.504	1.472	1.282
9	1.990	1.815	1.726	1.670	1.630	1.601	1.577	1.558	1.542	1.490	1.460	1.282
10	1.939	1.781	1.699	1.647	1.610	1.582	1.560	1.542	1.527	1.478	1.450	1.282
11	1.897	1.752	1.676	1.627	1.592	1.566	1.545	1.529	1.515	1.468	1.442	1.282
12	1.862	1.727	1.656	1.610	1.577	1.552	1.533	1.517	1.504	1.460	1.434	1.282
13	1.832	1.706	1.639	1.595	1.564	1.541	1.522	1.507	1.494	1.452	1.428	1.282
14	1.807	1.687	1.623	1.582	1.553	1.530	1.512	1.498	1.486	1.446	1.422	1.282
15	1.784	1.671	1.610	1.570	1.542	1.521	1.504	1.490	1.478	1.440	1.417	1.282
∞	1.282	1.282	1.282	1.282	1.282	1.282	1.282	1.282	1.282	1.282	1.282	1.282

$\bar{R} = 0.25$

n^* \ k^*	2	3	4	5	6	7	8	9	10	15	20	∞
1	20.588	6.157	4.163	3.408	3.007	2.756	2.583	2.454	2.355	2.069	1.927	1.282
2	5.341	3.288	2.729	2.455	2.288	2.174	2.089	2.024	1.972	1.810	1.724	1.282
3	3.759	2.717	2.373	2.191	2.075	1.992	1.930	1.882	1.842	1.718	1.650	1.282
4	3.196	2.470	2.208	2.063	1.969	1.901	1.849	1.808	1.775	1.668	1.609	1.282
5	2.908	2.331	2.111	1.987	1.905	1.845	1.799	1.763	1.733	1.638	1.584	1.282
6	2.732	2.241	2.047	1.936	1.861	1.807	1.766	1.732	1.705	1.616	1.566	1.282
7	2.614	2.178	2.002	1.899	1.830	1.780	1.741	1.710	1.684	1.601	1.553	1.282
8	2.529	2.131	1.967	1.872	1.807	1.759	1.722	1.692	1.668	1.588	1.543	1.282
9	2.464	2.095	1.941	1.850	1.788	1.742	1.707	1.679	1.655	1.579	1.536	1.282
10	2.414	2.067	1.919	1.832	1.773	1.729	1.695	1.668	1.645	1.571	1.529	1.282
11	2.373	2.043	1.902	1.818	1.761	1.718	1.685	1.659	1.637	1.565	1.524	1.282
12	2.340	2.023	1.887	1.806	1.750	1.709	1.677	1.651	1.629	1.559	1.519	1.282
13	2.312	2.007	1.875	1.796	1.741	1.701	1.670	1.644	1.623	1.555	1.515	1.282
14	2.288	1.993	1.864	1.787	1.734	1.694	1.664	1.639	1.618	1.551	1.512	1.282
15	2.268	1.981	1.855	1.779	1.727	1.688	1.658	1.634	1.614	1.547	1.509	1.282
∞	1.992	1.807	1.721	1.667	1.630	1.601	1.579	1.560	1.545	1.494	1.465	1.282

TABLE 8.8.6 One-sided tolerance limit factors, T, for the one-way random effects ANOVA, 0.95 confidence - Continued.

$\bar{R}$ = 0.50

n^* \ k^*	2	3	4	5	6	7	8	9	10	15	20	∞
1	20.588	6.157	4.163	3.408	3.007	2.756	2.583	2.454	2.355	2.069	1.927	1.282
2	6.157	3.511	2.855	2.544	2.358	2.232	2.140	2.069	2.012	1.839	1.747	1.282
3	4.434	2.957	2.522	2.302	2.165	2.070	1.999	1.943	1.898	1.759	1.683	1.282
4	3.807	2.716	2.369	2.187	2.071	1.989	1.928	1.879	1.840	1.717	1.649	1.282
5	3.485	2.581	2.280	2.118	2.014	1.940	1.885	1.841	1.805	1.691	1.628	1.282
6	3.289	2.494	2.221	2.073	1.977	1.908	1.856	1.814	1.781	1.673	1.614	1.282
7	3.157	2.434	2.180	2.041	1.950	1.885	1.835	1.796	1.763	1.661	1.603	1.282
8	3.063	2.390	2.149	2.016	1.930	1.867	1.819	1.781	1.750	1.651	1.595	1.282
9	2.992	2.355	2.125	1.998	1.914	1.853	1.807	1.770	1.740	1.643	1.589	1.282
10	2.936	2.328	2.106	1.983	1.901	1.842	1.797	1.761	1.732	1.637	1.584	1.282
11	2.891	2.306	2.091	1.970	1.891	1.833	1.789	1.754	1.725	1.632	1.580	1.282
12	2.855	2.288	2.078	1.960	1.882	1.826	1.782	1.748	1.719	1.628	1.577	1.282
13	2.824	2.273	2.067	1.951	1.875	1.819	1.776	1.742	1.714	1.624	1.574	1.282
14	2.798	2.260	2.058	1.944	1.868	1.814	1.772	1.738	1.710	1.621	1.571	1.282
15	2.776	2.248	2.050	1.937	1.863	1.809	1.767	1.734	1.707	1.618	1.569	1.282
∞	2.484	2.092	1.937	1.846	1.785	1.740	1.706	1.678	1.655	1.579	1.536	1.282

$\bar{R}$ = 1.00

n^* \ k^*	2	3	4	5	6	7	8	9	10	15	20	∞
1	20.588	6.157	4.163	3.408	3.007	2.756	2.583	2.454	2.355	2.069	1.927	1.282
2	7.682	3.892	3.063	2.688	2.470	2.325	2.220	2.139	2.076	1.883	1.782	1.282
3	5.787	3.376	2.771	2.482	2.309	2.191	2.104	2.037	1.984	1.820	1.732	1.282
4	5.063	3.148	2.635	2.384	2.230	2.125	2.047	1.986	1.938	1.787	1.706	1.282
5	4.683	3.020	2.557	2.326	2.184	2.086	2.013	1.956	1.910	1.767	1.690	1.282
6	4.451	2.937	2.505	2.288	2.153	2.059	1.990	1.935	1.891	1.754	1.680	1.282
7	4.294	2.880	2.469	2.261	2.131	2.041	1.973	1.921	1.878	1.745	1.672	1.282
8	4.181	2.838	2.442	2.241	2.115	2.027	1.961	1.910	1.868	1.737	1.666	1.282
9	4.095	2.805	2.422	2.225	2.102	2.016	1.951	1.901	1.860	1.732	1.662	1.282
10	4.028	2.780	2.405	2.213	2.092	2.007	1.944	1.894	1.854	1.727	1.658	1.282
11	3.975	2.759	2.392	2.203	2.084	2.000	1.938	1.888	1.849	1.724	1.655	1.282
12	3.931	2.742	2.381	2.194	2.077	1.994	1.932	1.884	1.844	1.720	1.653	1.282
13	3.894	2.727	2.371	2.187	2.071	1.989	1.928	1.880	1.841	1.718	1.650	1.282
14	3.863	2.715	2.363	2.181	2.066	1.985	1.924	1.876	1.838	1.716	1.649	1.282
15	3.836	2.704	2.356	2.176	2.062	1.981	1.921	1.873	1.835	1.714	1.647	1.282
∞	3.486	2.559	2.260	2.102	2.001	1.929	1.875	1.832	1.797	1.686	1.624	1.282

TABLE 8.8.6 One-sided tolerance limit factors, T, for the one-way random effects ANOVA, 0.95 confidence - Continued.

$\hat{R}$ = 2.00

n^* \ k^*	2	3	4	5	6	7	8	9	10	15	20	∞
1	20.588	6.157	4.163	3.408	3.007	2.756	2.583	2.454	2.355	2.069	1.927	1.282
2	10.092	4.419	3.337	2.875	2.613	2.441	2.319	2.226	2.153	1.936	1.824	1.282
3	8.129	3.979	3.105	2.716	2.491	2.342	2.234	2.152	2.087	1.891	1.789	1.282
4	7.325	3.779	2.995	2.640	2.432	2.293	2.192	2.115	2.054	1.869	1.771	1.282
5	6.890	3.664	2.931	2.595	2.397	2.264	2.167	2.093	2.034	1.855	1.760	1.282
6	6.617	3.591	2.890	2.566	2.374	2.245	2.151	2.079	2.021	1.846	1.753	1.282
7	6.431	3.539	2.860	2.545	2.358	2.231	2.139	2.068	2.012	1.839	1.748	1.282
8	6.296	3.501	2.838	2.529	2.345	2.221	2.130	2.061	2.005	1.835	1.744	1.282
9	6.193	3.471	2.821	2.517	2.336	2.213	2.123	2.054	1.999	1.831	1.741	1.282
10	6.112	3.448	2.808	2.508	2.328	2.207	2.118	2.050	1.995	1.828	1.739	1.282
11	6.047	3.429	2.797	2.500	2.322	2.202	2.113	2.046	1.991	1.825	1.737	1.282
12	5.994	3.413	2.788	2.493	2.317	2.197	2.110	2.042	1.988	1.823	1.735	1.282
13	5.949	3.400	2.780	2.488	2.312	2.194	2.107	2.039	1.986	1.821	1.734	1.282
14	5.911	3.389	2.773	2.483	2.309	2.190	2.104	2.037	1.984	1.820	1.733	1.282
15	5.878	3.379	2.768	2.479	2.305	2.188	2.101	2.035	1.982	1.818	1.731	1.282
∞	5.442	3.246	2.689	2.422	2.260	2.150	2.069	2.006	1.955	1.800	1.717	1.282

$\hat{R}$ = 10.00

n^* \ k^*	2	3	4	5	6	7	8	9	10	15	20	∞
1	20.588	6.157	4.163	3.408	3.007	2.756	2.583	2.454	2.355	2.069	1.927	1.282
2	16.565	5.564	3.893	3.237	2.883	2.658	2.501	2.384	2.294	2.029	1.896	1.282
3	15.433	5.382	3.808	3.183	2.843	2.626	2.474	2.361	2.273	2.016	1.886	1.282
4	14.901	5.294	3.766	3.156	2.823	2.610	2.461	2.350	2.263	2.009	1.881	1.282
5	14.592	5.242	3.741	3.139	2.811	2.600	2.453	2.343	2.257	2.005	1.878	1.282
6	14.390	5.207	3.724	3.129	2.803	2.594	2.448	2.338	2.253	2.002	1.875	1.282
7	14.248	5.183	3.713	3.121	2.797	2.590	2.444	2.335	2.250	2.000	1.874	1.282
8	14.142	5.165	3.704	3.116	2.793	2.586	2.441	2.332	2.248	1.999	1.873	1.282
9	14.061	5.151	3.697	3.111	2.790	2.584	2.439	2.330	2.246	1.998	1.872	1.282
10	13.996	5.139	3.692	3.108	2.787	2.581	2.437	2.329	2.245	1.997	1.871	1.282
11	13.943	5.130	3.687	3.105	2.785	2.580	2.436	2.328	2.244	1.996	1.871	1.282
12	13.899	5.123	3.684	3.102	2.783	2.578	2.434	2.327	2.243	1.996	1.870	1.282
13	13.863	5.116	3.680	3.100	2.782	2.577	2.433	2.326	2.242	1.995	1.870	1.282
14	13.831	5.111	3.678	3.099	2.780	2.576	2.432	2.325	2.241	1.995	1.870	1.282
15	13.804	5.106	3.675	3.097	2.779	2.575	2.432	2.324	2.241	1.994	1.869	1.282
∞	13.429	5.040	3.643	3.076	2.763	2.563	2.421	2.315	2.233	1.989	1.865	1.282

$\hat{R}$ = ∞

All n^*	20.588	6.157	4.163	3.408	3.007	2.756	2.583	2.454	2.355	2.069	1.927	1.282

TABLE 8.8.7 Critical values for the k-sample Anderson-Darling test at the 0.05 significance level, (equal sample sizes).

	k													
n	2	3	4	5	6	7	8	9	10	11	12	13	14	15
3	2.11	1.80	1.65	1.56	1.50	1.46	1.42	1.39	1.37	1.35	1.33	1.32	1.31	1.29
4	2.20	1.86	1.70	1.60	1.54	1.49	1.45	1.42	1.39	1.37	1.36	1.34	1.33	1.31
5	2.25	1.89	1.73	1.63	1.56	1.51	1.47	1.43	1.41	1.39	1.37	1.35	1.34	1.32
6	2.29	1.92	1.74	1.64	1.57	1.52	1.48	1.45	1.42	1.40	1.38	1.36	1.34	1.33
7	2.32	1.94	1.76	1.65	1.58	1.53	1.49	1.45	1.43	1.40	1.38	1.36	1.35	1.34
8	2.34	1.95	1.77	1.66	1.59	1.53	1.49	1.46	1.43	1.41	1.39	1.37	1.35	1.34
9	2.35	1.96	1.78	1.67	1.59	1.54	1.50	1.46	1.43	1.41	1.39	1.37	1.36	1.34
10	2.37	1.97	1.78	1.67	1.60	1.54	1.50	1.47	1.44	1.41	1.39	1.37	1.36	1.35
11	2.38	1.97	1.79	1.68	1.60	1.55	1.50	1.47	1.44	1.42	1.39	1.38	1.36	1.35
12	2.39	1.98	1.79	1.68	1.60	1.55	1.51	1.47	1.44	1.42	1.40	1.38	1.36	1.35
13	2.39	1.98	1.80	1.68	1.61	1.55	1.51	1.47	1.44	1.42	1.40	1.38	1.36	1.35
14	2.40	1.99	1.80	1.69	1.61	1.55	1.51	1.47	1.44	1.42	1.40	1.38	1.37	1.35
15	2.41	1.99	1.80	1.69	1.61	1.55	1.51	1.48	1.45	1.42	1.40	1.38	1.37	1.35
16	2.41	2.00	1.80	1.69	1.61	1.56	1.51	1.48	1.45	1.42	1.40	1.38	1.37	1.35
17	2.42	2.00	1.81	1.69	1.61	1.56	1.51	1.48	1.45	1.42	1.40	1.38	1.37	1.35
18	2.42	2.00	1.81	1.69	1.62	1.56	1.51	1.48	1.45	1.42	1.40	1.39	1.37	1.35
19	2.42	2.00	1.81	1.70	1.62	1.56	1.52	1.48	1.45	1.43	1.40	1.39	1.37	1.36
20	2.43	2.01	1.81	1.70	1.62	1.56	1.52	1.48	1.45	1.43	1.40	1.39	1.37	1.36
∞	2.49	2.05	1.84	1.72	1.64	1.58	1.53	1.50	1.46	1.44	1.42	1.40	1.38	1.37

TABLE 8.8.8. 0.95 Quantiles of the chi-squared distribution associated with γ degrees of freedom.

γ	$\chi^2_{0.95}$
1	3.84
2	5.99
3	7.82
4	9.49
5	11.07
6	12.60
7	14.07
8	15.51
9	16.93
10	18.31
11	19.68
12	21.03
13	22.37
14	23.69
15	25.00
16	26.30
17	27.59
18	28.88
19	30.15
20	31.42
21	32.68
22	33.93
23	35.18
24	36.42
25	37.66
26	38.89
27	40.12
28	41.34
29	42.56
30	43.78

TABLE 8.8.9 Critical values of the MNR outlier detection procedure, significance level, $\alpha = 0.01$.

n	CV	n	CV	n	CV
3	1.155	69	3.616	135	3.856
4	1.496	70	3.622	136	3.858
5	1.764	71	3.627	137	3.860
6	1.973	72	3.633	138	3.863
7	2.139	73	3.638	139	3.865
8	2.274	74	3.643	140	3.867
9	2.387	75	3.648	141	3.870
10	2.482	76	3.653	142	3.872
11	2.564	77	3.658	143	3.874
12	2.636	78	3.663	144	3.876
13	2.699	79	3.668	145	3.879
14	2.755	80	3.673	146	3.881
15	2.806	81	3.678	147	3.883
16	2.852	82	3.682	148	3.885
17	2.894	83	3.687	149	3.887
18	2.932	84	3.691	150	3.889
19	2.968	85	3.695	151	3.891
20	3.001	86	3.700	152	3.894
21	3.031	87	3.704	153	3.896
22	3.060	88	3.708	154	3.898
23	3.087	89	3.712	155	3.900
24	3.112	90	3.716	156	3.902
25	3.135	91	3.720	157	3.904
26	3.158	92	3.724	158	3.906
27	3.179	93	3.728	159	3.908
28	3.199	94	3.732	160	3.910
29	3.218	95	3.736	161	3.912
30	3.236	96	3.740	162	3.914
31	3.253	97	3.743	163	3.915
32	3.270	98	3.747	164	3.917
33	3.286	99	3.750	165	3.919
34	3.301	100	3.754	166	3.921
35	3.316	101	3.758	167	3.923
36	3.330	102	3.761	168	3.925
37	3.343	103	3.764	169	3.927
38	3.356	104	3.768	170	3.928
39	3.369	105	3.771	171	3.930
40	3.381	106	3.774	172	3.932
41	3.392	107	3.778	173	3.934
42	3.404	108	3.781	174	3.936
43	3.415	109	3.784	175	3.937
44	3.425	110	3.787	176	3.939
45	3.435	111	3.790	177	3.941
46	3.445	112	3.793	178	3.943
47	3.455	113	3.796	179	3.944
48	3.464	114	3.799	180	3.946
49	3.474	115	3.802	181	3.948
50	3.482	116	3.805	182	3.949
51	3.491	117	3.808	183	3.951
52	3.500	118	3.811	184	3.953
53	3.508	119	3.814	185	3.954
54	3.516	120	3.817	186	3.956
55	3.524	121	3.819	187	3.958
56	3.531	122	3.822	188	3.959
57	3.539	123	3.825	189	3.961
58	3.546	124	3.828	190	3.962
59	3.553	125	3.830	191	3.964
60	3.560	126	3.833	192	3.965
61	3.567	127	3.835	193	3.967
62	3.573	128	3.838	194	3.969
63	3.580	129	3.841	195	3.970
64	3.586	130	3.843	196	3.972
65	3.592	131	3.846	197	3.973
66	3.598	132	3.848	198	3.975
67	3.604	133	3.851	199	3.976
68	3.610	134	3.853	200	3.978

TABLE 8.8.10 Critical values for the MNR outlier detection procedure, significance level, $\alpha = 0.05$.

n	CV	n	CV	n	CV
3	1.154	69	3.252	135	3.483
4	1.481	70	3.258	136	3.486
5	1.715	71	3.263	137	3.488
6	1.887	72	3.268	138	3.491
7	2.020	73	3.273	139	3.493
8	2.127	74	3.278	140	3.495
9	2.215	75	3.283	141	3.497
10	2.290	76	3.288	142	3.500
11	2.355	77	3.292	143	3.502
12	2.412	78	3.297	144	3.504
13	2.462	79	3.302	145	3.506
14	2.507	80	3.306	146	3.508
15	2.548	81	3.311	147	3.511
16	2.586	82	3.315	148	3.513
17	2.620	83	3.319	149	3.515
18	2.652	84	3.323	150	3.517
19	2.681	85	3.328	151	3.519
20	2.708	86	3.332	152	3.521
21	2.734	87	3.336	153	3.523
22	2.758	88	3.340	154	3.525
23	2.780	89	3.344	155	3.527
24	2.802	90	3.348	156	3.529
25	2.822	91	3.352	157	3.531
26	2.841	92	3.355	158	3.533
27	2.859	93	3.359	159	3.535
28	2.876	94	3.363	160	3.537
29	2.893	95	3.366	161	3.539
30	2.908	96	3.370	162	3.541
31	2.924	97	3.374	163	3.543
32	2.938	98	3.377	164	3.545
33	2.952	99	3.381	165	3.547
34	2.965	100	3.384	166	3.549
35	2.978	101	3.387	167	3.551
36	2.991	102	3.391	168	3.552
37	3.003	103	3.394	169	3.554
38	3.014	104	3.397	170	3.556
39	3.025	105	3.401	171	3.558
40	3.036	106	3.404	172	3.560
41	3.047	107	3.407	173	3.561
42	3.057	108	3.410	174	3.563
43	3.067	109	3.413	175	3.565
44	3.076	110	3.416	176	3.567
45	3.085	111	3.419	177	3.568
46	3.094	112	3.422	178	3.570
47	3.103	113	3.425	179	3.572
48	3.112	114	3.428	180	3.574
49	3.120	115	3.431	181	3.575
50	3.128	116	3.434	182	3.577
51	3.136	117	3.437	183	3.579
52	3.144	118	3.440	184	3.580
53	3.151	119	3.442	185	3.582
54	3.159	120	3.445	186	3.584
55	3.166	121	3.448	187	3.585
56	3.173	122	3.451	188	3.587
57	3.180	123	3.453	189	3.588
58	3.187	124	3.456	190	3.590
59	3.193	125	3.459	191	3.592
60	3.200	126	3.461	192	3.593
61	3.206	127	3.464	193	3.595
62	3.212	128	3.466	194	3.596
63	3.218	129	3.469	195	3.598
64	3.224	130	3.471	196	3.599
65	3.230	131	3.474	197	3.601
66	3.236	132	3.476	198	3.603
67	3.241	133	3.479	199	3.604
68	3.247	134	3.481	200	3.606

TABLE 8.8.11 Nonparametric B-basis material properties for small samples (Reference 8.5.7.2).

n	r	k
2	2	35.177
3	3	7.859
4	4	4.505
5	4	4.101
6	5	3.064
7	5	2.858
8	6	2.382
9	6	2.253
10	6	2.137
11	7	1.897
12	7	1.814
13	7	1.738
14	8	1.599
15	8	1.540
16	8	1.485
17	8	1.434
18	9	1.354
19	9	1.311
20	10	1.253
21	10	1.218
22	10	1.184
23	11	1.143
24	11	1.114
25	11	1.087
26	11	1.060
27	11	1.035
28	12	1.010

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