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Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods of tests for reinforcements — Determination of the tensile properties of resin-impregnated yarns

Céramiques techniques (céramique technique, céramique technique avancée) — Méthodes d'essais pour renforts — Détermination des propriétés en traction des fils imprégnés de résine

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Fine ceramics are widely used in various fields, such as machinery, electronics, chemistry, construction, energy, aerospace and the nuclear industry, and in environmental applications. Fibre-reinforced ceramic matrix composites (CMCs) have been the subject of extensive research and development. CMCs are lightweight, have suitable chemical and thermal stability, and exhibit high strength, elastic modulus and creep resistance. These composites have been applied in devices and components in the aerospace industry and in high-temperature applications.

CMCs have been put to practical use as components in the jet engines of passenger planes and are also being developed as components of gas turbines for electric power generation.

The reliability of CMCs is influenced by the properties of the reinforcing fibre. High reliability is particularly important for jet engine parts. The mechanical properties of these fibres also require fixed values and distribution.

This document establishes a method for the measurement of mechanical properties such as tensile strength, elastic modulus and strain at the maximum force of a resin-impregnated yarn specimen prepared from the reinforcing ceramic fibre in CMCs.

Fibre and CMC manufacturers will be able to use this method to perform quality control and relative comparison of ceramic fibres used as the reinforcement of CMCs.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods of tests for reinforcements — Determination of the tensile properties of resin-impregnated yarns

1 Scope

This document specifies a method for the determination of the tensile strength, tensile modulus of elasticity and strain at the maximum force of a resin-impregnated yarn specimen at ambient temperature. This method is applicable to yarns of ceramic fibres that are used as reinforcements in composite materials. The test results obtained by this method are applicable for quality control and comparison of the ceramic fibres.

The outputs of this method are not to be mixed up with the strength of filaments derived from tensile tests on dry tows specified in ISO 22459.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1889, *Reinforcement yarns — Determination of linear density*

ISO 7500-1, *Metallic materials — Calibration and verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Calibration and verification of the force-measuring system*

ISO 10119, *Carbon fibre — Determination of density*

ISO 10548, *Carbon fibre — Determination of size content*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
gauge length
L
distance between reference points on the test specimen during the tensile test

3.2
initial gauge length
L_0
distance between reference points on the test specimen at zero load

[SOURCE: ISO 22459:2020, 3.1, modified — term and definition revised and Note 1 to entry removed.]

3.3
longitudinal deformation
A
increase of the *initial gauge length* (3.2) during the tensile test

[SOURCE: ISO 19630:2017, 3.7, modified — definition revised.]

3.4
maximum tensile force
F_m
greatest tensile force applied to the test specimen when tested to failure

[SOURCE: ISO 19630:2017, 3.4, modified — definition revised.]

3.5
tensile strength
σ_f
maximum tensile force (3.4) divided by the *initial total cross-sectional area of the dry yarn* (3.9)

[SOURCE: ISO 19630:2017, 3.6, modified — definition revised.]

3.6
total compliance
C_t
ratio of the measured displacement to the corresponding force during the tensile test

[SOURCE: ISO 22459:2020, 3.4.1]

3.7
load train compliance
C_l
ratio of the load train elongation, excluding the specimen contribution, to the corresponding force during the tensile test

[SOURCE: ISO 22459:2020, 3.4.2]

3.8
tensile modulus of elasticity
E_f
slope of the force–deformation curve divided by the *initial total cross-sectional area of the dry yarn* (3.9)

3.9
initial total cross-sectional area of the dry yarn
S_f
linear density of the dry yarn (3.11) divided by the *density of the dry yarn* (3.13)

[SOURCE: ISO 22459:2020, 3.2, modified — term and definition revised.]

3.10
strain at maximum force
$\varepsilon_{t,m}$
ratio of the greatest *longitudinal deformation* (3.3) to the *initial gauge length* (3.2)

3.11
linear density of the dry yarn
T_{tf}
mass of the dry yarn without a sizing agent divided by its length

[SOURCE: ISO 1889:2009, 3.1, modified — term and definition revised and Note 1 to entry removed.]

3.12
linear density of the impregnated yarn
T_{ti}
mass of the test specimen divided by its length

3.13
density of the dry yarn
ρ_f
mass of the dry yarn without a sizing agent divided by its volume

4 Principle

A fibre yarn impregnated with an epoxy resin is used as the specimen. A tensile force is applied to the specimen at a constant displacement rate by a suitable mechanical testing machine and grip system until failure. The longitudinal deformation of resin-impregnated yarn is measured directly by using an extensometer or is determined from the displacement between two grips using a compliance correction. The correction takes into account the contributions of the loading train, the grips and the tabbing materials.

The tensile strength, tensile modulus of elasticity and the strain at maximum force are calculated from the force–deformation relationship. For ceramic fibre yarns in epoxy matrix, the relation between force and longitudinal deformation is linear and, hence, the tensile modulus of elasticity is calculated from the slope of the force–deformation curve.

5 Apparatus and material

5.1 Resin

The impregnating resin shall be compatible with the sizing agent and the type of yarn. The viscosity of the resin or resin solution shall be such that sufficient resin pick-up and uniform impregnation are achieved. The failure strain of the cured resin should be at least twice as large, preferably more than three times as large, as that of the specimen. In this respect, thermosetting epoxy-resin systems are suitable (see Annex A, Table A.1 for an example) as is any formulation capable of giving test specimens that fulfil the requirements of this document.

5.2 Impregnation apparatus

Test specimens can be prepared by any method that produces a uniformly impregnated, smooth specimen.

An impregnation apparatus consists of a yarn bobbin holder, an impregnation bath, rollers for resin impregnation and removal of excess resin, and an impregnated yarn winder.

An impregnation apparatus may consist of:

5.2.1 Yarn bobbin holder for the sample yarn bobbin, with yarn-tensioning devices.

5.2.2 Impregnation bath with temperature-control devices and impregnation rollers or yarn-tensioning bars.

5.2.3 Roller for removal of excess resin from the impregnated yarn, that passes its rollers covered with fabric, paper or felt through a die.

5.2.4 Impregnated yarn winder to wind up the impregnated yarn, preferably made of wood or metal coated with rubber. Examples of impregnation apparatuses are given in Annex B, Figure B.1.

5.3 Curing oven with temperature control

A fan circulation oven is preferable to ensure uniform curing of the resin.

5.4 Tensile-testing machine and extensometer

5.4.1 Tensile-testing machine

Use a tensile-testing machine (with a constant cross-head speed) equipped with force- and extension-recording devices. The machine shall conform to grade 1 or better in ISO 7500-1. The accuracy of the force indicated shall be better than 1 % of the recorded value. The specimen-gripping system shall ensure that the test specimen is aligned with the axis of the test machine. A suitable example is shown in Annex C, Figure C.1.

5.4.2 Extensometer

Use an extensometer linked to a continuous-recording device that automatically records the extension within the gauge length of the extensometer as a function of force on the test specimen. The extensometer should be sufficiently light to induce only negligible stresses in the test specimen. For the specimens with long gauge section lengths, it is recommended that the weight of the extensometer is supported, for example by using a thread, in order to prevent bending of the specimen and contact forces.

The gauge length of the extensometer shall be at least 25 mm. The gauge length shall be determined with a tolerance of ±1 %. The use of an extensometer with a gauge length as long as possible is recommended to increase the accuracy of the measurement.

The extensometer shall have a tolerance on deviation from linearity of not more than 0,1 % over the required extension measurement range.

Examples of suitable extensometers are given in Annex C. Other strain-measuring instruments, such as optical or laser instruments, may be used if suitable.

5.5 Balance

Use a balance readable to 0,1 mg to weigh the test specimens to determine the linear density of the impregnated yarn.

5.6 Ruler

Use a graduated ruler or other measuring device with a precision of ±0,5 mm to determine the initial distance between two grips, i.e. the gauge length of the specimen.

6 Test specimens

6.1 Number of test specimens

Prepare a sufficient number of test specimens to perform five determinations. If any of the specimens fails within the grips at the tabs, or because of damage caused by the extensometer, discard the result and carry out a repeat determination on a fresh test specimen.

6.2 Impregnation of test specimens

The procedure for using the impregnation apparatus described in 5.2 is as follows:

a) Place the yarn bobbin on the holder.

b) Pour the impregnating-resin mixture into the resin bath (see 5.2.2) and adjust the temperature and viscosity to the desired values.

c) Draw the yarn from the bobbin holder through the rollers, resin bath and system designed to remove the excess resin while ensuring adequate resin impregnation (see 5.2.3).

d) Adjust the unwinding tension by applying a force of 80 gram-force (gf) to 130 gf.

e) Wind the impregnated yarn onto the frame (see 5.2.4).

f) Place the frame in the oven (see 5.3).

g) Cure the resin in accordance with the resin manufacturer's instructions.

h) When the resin has been cured, remove the frame from the oven. After removing the impregnated yarn from the frame, cut off a sufficient number of test specimens.

i) Select the test specimens according to the criteria given in 6.4.1.

6.3 Determination of other fibre properties

6.3.1 General

In order to perform the calculations of tensile strength and tensile modulus given in Clause 9 the properties specified from 6.3.2 to 6.3.4 shall be determined.

6.3.2 Linear density of the yarn

Determine the linear density of the yarn using the method given in ISO 1889 for carbon fibre.

6.3.3 Size content of the yarn

Determine the size content of the yarn using the method given in ISO 10548, method C.

6.3.4 Density of the ceramic fibre

Determine the density of the ceramic fibre using one of the methods given in ISO 10119.

6.4 Criteria for selection of the test specimens

6.4.1 Specimens

Each test specimen shall be confirmed as straight by checking with a metallic measuring ruler. The specimen shall be uniform in appearance and without any of the following defects:

— broken filaments;

— resin droplets;

— fibre misalignment.

6.4.2 Resin content of the specimens

The resin content shall be at least 30 % by mass. The resin content of the specimens is calculated from the linear densities of the specimen and dry yarn using Formula (1):

$$m_r = 100 \times \frac{T_{ti} - T_{tf}}{T_{ti}} \quad (1)$$

where

m_r is the resin content of the test specimens, in per cent;

T_{ti} is the linear density of the test specimen, in mg/m;

T_{tf} is the linear density of the dry yarn, in mg/m.

6.4.3 Impregnated yarn

The yarn shall be uniformly impregnated. No excess resin beads shall form on the surface of the specimen.

7 Atmospheres for conditioning and testing

During the test, the apparatus and specimens shall be maintained at the same conditions as used for conditioning. The preferred conditions are 23 °C ± 5 °C and 50 % ± 20 % relative humidity.

8 Procedure for tensile testing

8.1 Procedure

a) Clamp a test specimen in the grips of the test machine. Equip two sheets of abrasive paper tabs between the specimen and flat faces of the grips to avoid specimen slippage.

   The use of air-pressure or hydraulic grips is recommended to avoid specimen damage during specimen mounting.

   The length of the gauge section of the specimen, i.e. the distance between two grip ends, should be at least 50 mm and the recommended length is 200 mm ± 5 mm.

b) Measure the gauge length of the specimen, i.e. the distance between two grips with an accuracy of ± 0,5 mm.

c) When the longitudinal deformation of the specimen is measured by an extensometer, mount the extensometer (see 5.4.2) carefully on the test specimen.

d) Set the cross-head speed of the tensile-testing machine (see 5.4.1). Set the tensile force at a constant displacement rate between 0,1 mm/min and 60 mm/min. Select a loading rate to produce final failure in 60 s. The recommended rate is 5 mm/min for any length of specimens.

e) Start the recording device and load the test specimen to failure. Record the applied force, cross-head displacement and elongation of the specimen.

8.2 Determination of load train compliance

When longitudinal deformation of resin-impregnated yarn is determined from the displacement between two grips, the load train compliance shall be determined. Repeat step 8.1 three times for each of the gauge lengths of 100 mm, 200 mm and 300 mm.

8.3 Test validity

The following circumstances invalidate a test:

— failure to specify and record test conditions;

— the test specimen fails within the grips or because of the extensometer;

— any slippage in the force–displacement curve.

9 Calculation of results

9.1 Tensile strength

For each test specimen, calculate the tensile strength of the resin-impregnated yarn using Formulae (2) to (4).

$$\sigma_f = \frac{F_f}{S_f} \tag{2}$$

where

σ_f is the tensile strength, in megapascals;

F_f is the maximum tensile force, in newtons;

S_f is the initial total cross-sectional area of the dry yarn, in square millimetres, given by Formula (3).

$$S_f = \frac{T_{tf}}{\rho_f} \times 10^{-3} \tag{3}$$

where

T_{tf} is the linear density, in mg/m, of the dry yarn without a sizing agent that is calculated from the linear density determined in accordance with ISO 1889 and the size content determined in accordance with ISO 10548;

ρ_f is the density of the dry yarn without a sizing agent, in grams per cubic centimetre, determined in accordance with ISO 10119.

If using a sized specimen, calculate the linear density, in grams per kilometre, using Formula (4).

$$T_{tf} = \frac{m \times 10^3}{l} \times \frac{(100 - S_c)}{100} \tag{4}$$

where

m is the mass, in grams, of the specimen;

l is the length, in metres, of the specimen;

S_c is the sizing content, in per cent, of the specimen;

If the size content is sufficiently low for no error to be introduced, then the linear density and density of the yarn with a sizing agent may be used.

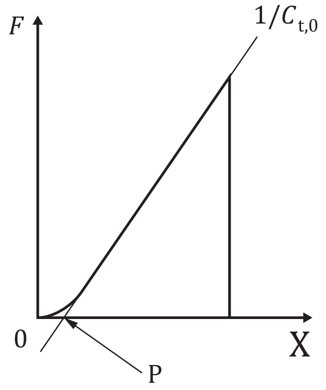
9.2 Calculation of the load train compliance

The load train compliance shall be determined when longitudinal deformation of the specimen is obtained from the grip displacement. The longitudinal deformation shall be calculated according to 9.3.

Calculate the initial total compliance $C_{t,0}$ (mm/N) for the tests at each of the gauge lengths from the slope $1/C_{t,0}$ of the linear rising part of the force (N)-displacement (mm) curve (see Figure 1). Calculate the average value $\bar{C}_{t,0}$ at each of the three gauge lengths.

Plot $\bar{C}_{t,0}$ against L_0 (see Figure 2).

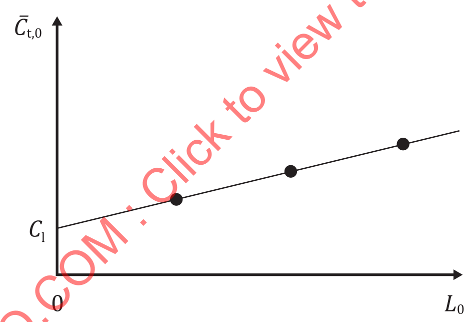
Perform a linear regression analysis of $C_{t,0}$ versus L_0 and determine the load train compliance C_l from the intercept at $L_0 = 0$.



Key

F force
X displacement
P true origin
$C_{t,0}$ initial total compliance

Figure 1 — Force-displacement curve and determination of true origin



Key

$\bar{C}_{t,0}$ average of initial total compliance
C_l load train compliance
L_0 initial gauge length

Figure 2 — Determination of load train compliance

9.3 Calculation of instantaneous total compliance and longitudinal deformation of resin-impregnated yarn

Calculate the instantaneous total compliance $C_{t,i}$ from the slope $1/C_{t,i}$ of the line through the points on the force–grip displacement curve and the origin of the curve.

The longitudinal deformation during the tensile loading of the specimen is calculated using Formula (5):

$$A = \left(C_{t,i} - C_l\right) F \tag{5}$$

where

A is the longitudinal deformation of the specimen, in millimetres (mm);

$C_{t,i}$ is this the instantaneous total compliance, in millimetres per newton (mm/N);

C_l is the load train compliance, in millimetres per newton (mm/N);

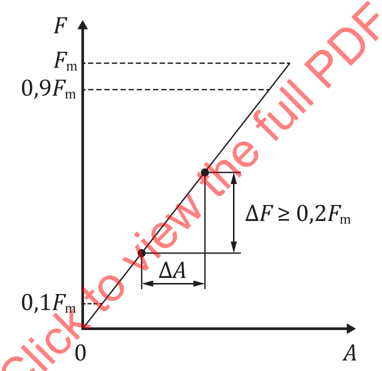
F is the force applied on the test specimen, in newtons (N).

9.4 Tensile modulus of elasticity

For each test, plot the force against the longitudinal deformation measured directly by the extensometer or determined from the grip displacement with the compliance correction.

The force–deformation curves of resin-impregnated yarns are usually linear until failure. A schematic diagram of the methods for determining the tensile modulus of elasticity is reported in Figure 3.

The tensile modulus is the slope calculated between the two points in the force–deformation curve. These two points shall lie between 10 % and 90 % of the maximum tensile force. The variation of the force between the two points shall be more than 20 % of the maximum tensile force.



Key

F force

A longitudinal deformation

F_m maximum tensile force

ΔF variation in force corresponding to the variation in the longitudinal deformation

ΔA variation in the longitudinal deformation corresponding to the variation in force

Figure 3 — Calculation of the tensile modulus of elasticity from the force–deformation curve

For each test specimen, calculate the tensile modulus of the yarn using Formula (6):

$$E_f = \left(\frac{\Delta F}{\Delta A}\right) \times \frac{L_0}{S_f} \times 10^{-3} \qquad (6)$$

where

E_f is the tensile modulus of elasticity, in gigapascals;

$\left(\frac{\Delta F}{\Delta A}\right)$ is the slope calculated between two points in the force–deformation curve;

ΔF is the variation in force, in newtons, corresponding to the variation in the longitudinal deformation;

ΔA is the variation in the longitudinal deformation, in millimetres, corresponding to the variation in force;

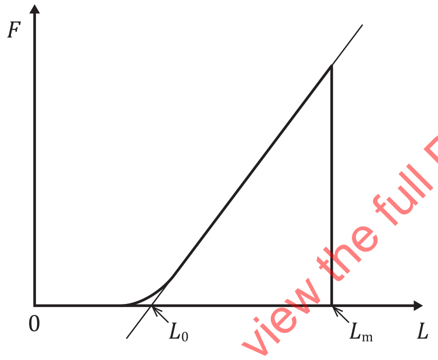
S_f is the initial total cross-sectional area of the dry yarn, in square millimetres;

L_0 is the initial gauge length at zero force, determined from the intersection of the linear rising part of the force–gauge length curve and abscissa, in millimetres (see Figure 4).

To determine the initial gauge length L_0, plot the force against the gauge length L during the tensile test (see Figure 4).

When an extensometer is used to measure the longitudinal deformation of the specimen, the gauge length L is defined as the gauge length of the extensometer during the test. When an extensometer is not used, the L is defined as the sum of the initial distance between two grips and the longitudinal deformation of the specimen.

The L_0 shall be determined by the intersection of the linear rising part of the force–gauge length curve with the abscissa, as shown in Figure 4.



Key

F force

L gauge length

L_0 initial gauge length at zero force

L_m gauge length at maximum force

Figure 4 — Determination of L_0 from the force–gauge length curve

9.5 Tensile strain at maximum force (percentage elongation at failure)

9.5.1 Calculation method (1)

The strain at maximum force is calculated using Formula (7):

$$\varepsilon_{t,m} = \frac{L_m - L_0}{L_0} \times 100 \quad (7)$$

where

$\varepsilon_{t,m}$ is the strain at maximum force;

L_m is the gauge length at the maximum force, in millimetres;

L_0 is the initial gauge length at zero force, determined from the intersection of the linear rising part of the force–gauge length curve and abscissa, in millimetres (see Figure 4).

9.5.2 Calculation method (2)

For the specimens with liner force–deformation behaviour, the strain at the maximum force can be calculated using Formula (8):

$$\varepsilon_{\mathrm{t,m}} = \frac{\sigma_{\mathrm{f}}}{E_{\mathrm{f}}} \times 0{,}1 \tag{8}$$

where

$\varepsilon_{\mathrm{t,m}}$ is the strain at maximum force;

σ_{f} is the tensile strength, in megapascals;

E_{f} is the tensile modulus of elasticity, in gigapascals.

10 Statistics

For each series test, calculate the mean, standard deviation and coefficient of variation for each measured value according to Formulae (9) to (11):

— mean
$$\bar{X} = \frac{\sum_{i=1}^{n} X_i}{n} \tag{9}$$

— standard deviation
$$S = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \bar{X})^2}{n-1}} \tag{10}$$

— coefficient of variation
$$C_v = \frac{100 \cdot S}{\bar{X}} \tag{11}$$

where

X represents the measured values;

n is the number of valid tests;

S is the standard deviation;

C_v is the coefficient of variation, in per cent.

11 Test report

The test report shall include the following:

a) a reference to this document, i.e. ISO 24046:2022;

b) all details necessary for complete identification of the yarn tested;

c) the linear density of the yarn;

d) the density of the yarn;

e) whether the longitudinal deformation of the specimen was measured by the extensometer or was determined from the grip displacement with compliance correction;

f) whether the strain at maximum force was determined from the gauge length at maximum force and initial gauge length or calculated using the tensile strength and modulus;

g) the number of tests carried out and the number of valid tests;

h) the results obtained from the determination tensile strength, tensile modulus of elasticity and, if required, the individual results for each test specimen and strain at maximum force;

i) the method of calculation of the tensile modulus of elasticity;

j) any additional details that have possibly had a bearing on the results obtained;

k) the date of the test.

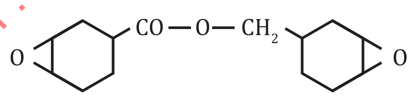
Annex A
(informative)

Examples of heat-curable epoxy-resin systems

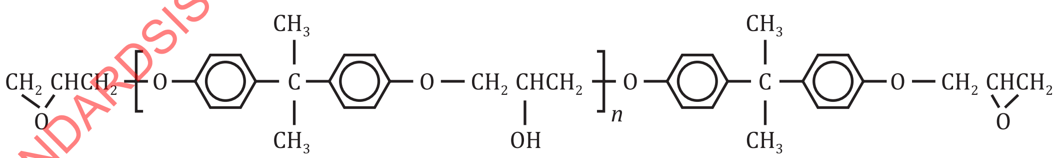
Table A.1 — Examples of heat-curable epoxy-resin systems

	Resin system	Amount	Cure conditions
1)	3,4-Epoxycyclohexyl 3,4-epoxycyclohexanecarboxylate[a] Boron trifluoride monoethylamine (BF_3MEA) 2-Propanone or ethanol	100 g 3 g 4 g	125 °C for 1 h or 130 °C for 30 min
2)	Diglycidyl ether bisphenol A (DGEBA)[b] with A viscosity of 11 000 mP·s to 14 000 mP·s at 25 °C and an epoxy equivalent of 184 g/equiv to 194 g/equiv Methyl nadic anhydrique (MNA)[c] Benzyldimetylamine 2-Propanone	100 g 90 g 1 g to 2 g 5 g	Room temperature for 10 h followed by 90 °C for 42 h and then 150 °C for 4 h or Room temperature for 10 h followed by 100 °C for 2 h and then 150 °C for 2 h
3)	Epoxy resin: same as in Example 2) Bis(4-aminophenyl)sulfone Boron trifluoride monoethylamine (BF_3MEA) Solvent	100 g 20 g 1,5 g Arbitrary	80 °C for 1 h followed by 200 °C for 2 h

[a] The structure is as follows:



[b] The structure is as follows:



[c] Methyl 5-norbomene-2,3-dicarboxylic. anhydride.