
**Textile glass — Rovings — Manufacture
of test specimens and determination of
tensile strength of impregnated rovings**

*Verre textile — Stratifils — Fabrication d'éprouvettes et essai de traction
sur stratifil imprégné*

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Contents

	Page
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Sampling and conditioning	2
6 Production of test specimens — Reference method	3
7 Production of test specimens — Short method	13
8 Tensile testing	15
9 Expression of results	18
10 Test report	19
Annex A (informative) Precision statement	20
Bibliography	21

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 9163 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

This second edition cancels and replaces the first edition (ISO 9163:1996), which has been technically revised, as follows:

- The reference method remains essentially the same, with a few corrections, as in ISO 9163:1996.
- The so-called “fast method” described in ISO 9163:1996 has been deleted because it was no longer used. It has been replaced by a method which needs far less time and gives results consistent with the reference method.

Textile glass — Rovings — Manufacture of test specimens and determination of tensile strength of impregnated rovings

1 Scope

This International Standard specifies two methods for the determination of the tensile stress at break of an impregnated roving:

- a reference method using test specimens produced with moulded epoxy tabs;
- a short method using test specimens with no tabs or simple cardboard or composite tabs.

The methods are applicable to both assembled (multistrand) and direct (multifilament) rovings; nevertheless the reference method may be used for various linear densities, but the short method is described for 1 200 tex rovings only, which is the linear density that allows the fibres in the roving to spread out most easily during impregnation.

2 Normative references

The following referenced documents are indispensable for the application 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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

ISO 1172, *Textile-glass-reinforced plastics — Prepregs, moulding compounds and laminates — Determination of the textile-glass and mineral-filler content — Calcination methods*

ISO 1887, *Textile glass — Determination of combustible-matter content*

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

ISO 2078, *Textile glass — Yarns — Designation*

ISO 2859-1, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

ISO 3951-1, *Sampling procedures for inspection by variables — Part 1: Specification for single sampling plans indexed by acceptance quality limit (AQL) for lot-by-lot inspection for a single quality characteristic and a single AQL*

ISO 7822, *Textile glass reinforced plastics — Determination of void content — Loss on ignition, mechanical disintegration and statistical counting methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply.

- 3.1
breaking force**
force or load required to rupture a test specimen in a tensile test, usually expressed in newtons
- 3.2
gauge length**
nominal length between the spines of contact of an extensometer (expressed in millimetres), by reference to which the length increase due to a tensile force is determined
- 3.3
relative elongation**
ratio of the increase in length (expressed in millimetres) between the spines of contact of an extensometer, resulting from application of a tensile force, to the gauge length of the extensometer (also expressed in millimetres)
- 3.4
breaking stress**
ratio (expressed in megapascals) of the breaking force (expressed in newtons) to the cross-sectional area of a roving (expressed in square millimetres)
- 3.5
loading stress**
ratio (expressed in megapascals) of the tensile force applied to a roving during a tensile test (expressed in newtons) to the cross-sectional area of the roving (expressed in square millimetres)

NOTE The force experienced by the resin can be neglected.

**3.6
cross-sectional area**

S
area of the cross-section of a roving given, in square millimetres, by the formula:

$$\frac{\rho_l \times 10^{-3}}{\rho_g} \tag{1}$$

where

- ρ_l is the exact linear density of the unsized roving, in tex;
- ρ_g is the density of the glass constituting the roving, in grams per cubic centimetre.

**3.7
proportional limit**
greatest stress (expressed in megapascals) for which the relative elongation is proportional to the applied force

4 Principle

A specimen of impregnated roving is subjected to tensile loading to rupture using a suitable mechanical apparatus, and the breaking stress of the specimen determined.

5 Sampling and conditioning

Carry out sampling in accordance with ISO 2859-1, using the “inspection by attributes” method, or in accordance with ISO 3951-1, using the “inspection by variables” method in order to minimize the number of elementary units (packages) to be selected.

Condition the packages selected for at least 12 h in one of the standard atmospheres given in ISO 291.

6 Production of test specimens — Reference method

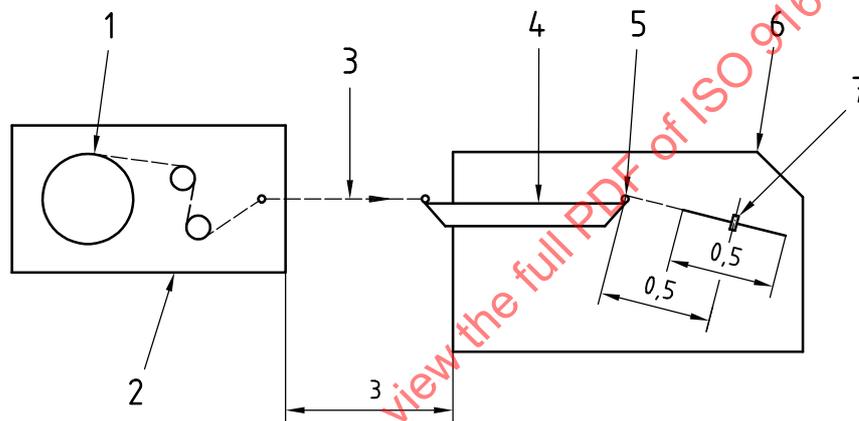
6.1 Apparatus

Ordinary laboratory apparatus, plus the following:

6.1.1 Impregnation apparatus (see Figure 1), including the following elements:

6.1.1.1 Reel, equipped with a tension-regulating system capable of maintaining the roving under a tension between 0,2 N and 20 N by positioning the tensioning bars (see Figure 2) to obtain a correct level of impregnation. The tension can be adjusted by measuring it with a tension-measuring instrument between the reel and the entrance guide.

Dimensions in metres



Key

- 1 reel
- 2 tension-regulating system
- 3 roving
- 4 impregnation vat
- 5 die
- 6 winding system
- 7 former

Figure 1 — General layout of impregnation apparatus

6.1.1.2 Impregnation vat, equipped with yarn guides (see Figure 2).

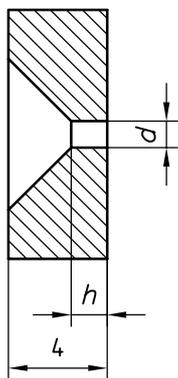
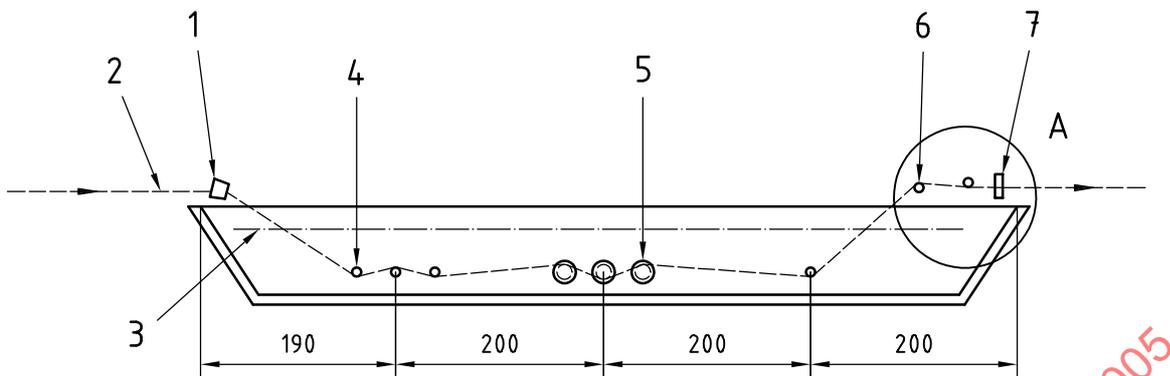
It shall be capable of maintaining a temperature of up to 130 °C with a precision of ± 5 °C (the actual temperature set will depend on the resin system).

The use of a double-walled vat, with heating fluid circulating between the walls, is recommended. If a vat of this kind is not available, a hotplate may be used.

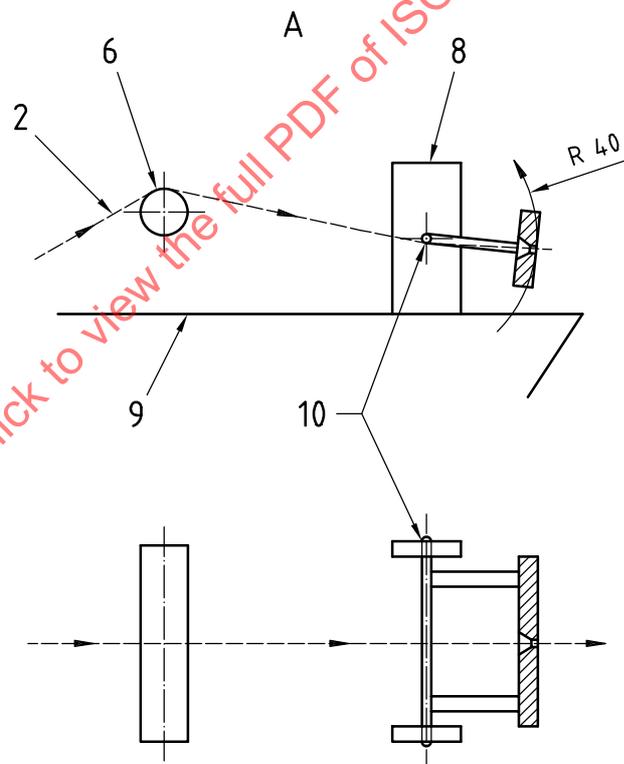
6.1.1.3 Stainless-steel die, designed to give the impregnated roving a defined circular cross-section.

The die shall be mounted on a spindle, permitting it to align itself automatically with the roving during reeling (see Figure 2).

Dimensions in millimetres



11



Key

- 1 entrance guide (eye type)
- 2 roving
- 3 resin level
- 4 tensioning bars (smooth)
- 5 tensioning bars (grooved)
- 6 exit tensioning bar
- 7 die
- 8 die support
- 9 impregnation vat
- 10 rotating spindle of die, \varnothing 3 mm

NOTE Any equipment which is equivalent to the one illustrated above may be used, provided satisfactory impregnation is obtained. Nevertheless, it is recommended that a die diameter given in Table 1 be used, in order to obtain a reproducible impregnation ratio.

Figure 2 — Vat and die

The diameter of the die will depend on the linear density of the roving. It is given by the equation:

$$d = \sqrt{\frac{4\rho_l[(1/G) - (1 + \rho_m)/\rho_g]}{1\,000 \rho_m \pi}} \quad (2)$$

where

ρ_m is the density of the matrix;

ρ_g is the density of the glass in the roving;

ρ_l is the linear density of the roving, in tex;

G is the proportion of glass, by mass, in the composite, expressed as a figure between 0 and 1.

The glass content by mass, determined as specified in ISO 1172, shall be approximately constant at (75 ± 4) %.

Table 1 indicates some typical parameters for dies for different tex counts.

Table 1 — Typical parameters for dies for different tex counts

ρ_l (tex)	800	1 200	1 600	2 400	4 800
d^a (mm)	0,8	1,1	1,15	1,4	2
h^a (mm)	1	1,3	1,5	2	3
^a h et d are as indicated in Figure 2.					

6.1.1.4 Winding device (mechanical or manual) with former, for collecting the impregnated roving, ensuring that

- the roving is kept under tension;
- the impregnated strands are kept separate from each other.

The collecting speed shall be as constant as possible.

6.2 Preparation of test specimens

6.2.1 Type and dimensions

Each test specimen shall consist of an impregnated roving with moulded end tabs. Its dimensions shall be as given in Figure 3.

A method for the fabrication of tabs is given in 6.3.

6.2.2 Number

For each of the packages selected (see Clause 5), at least 20 test specimens shall be prepared to obtain at least 10 valid readings (five for the determination of the breaking stress and five for the tensile modulus). Testing of more specimens might be necessary for statistical purposes.

Dimensions in millimetres

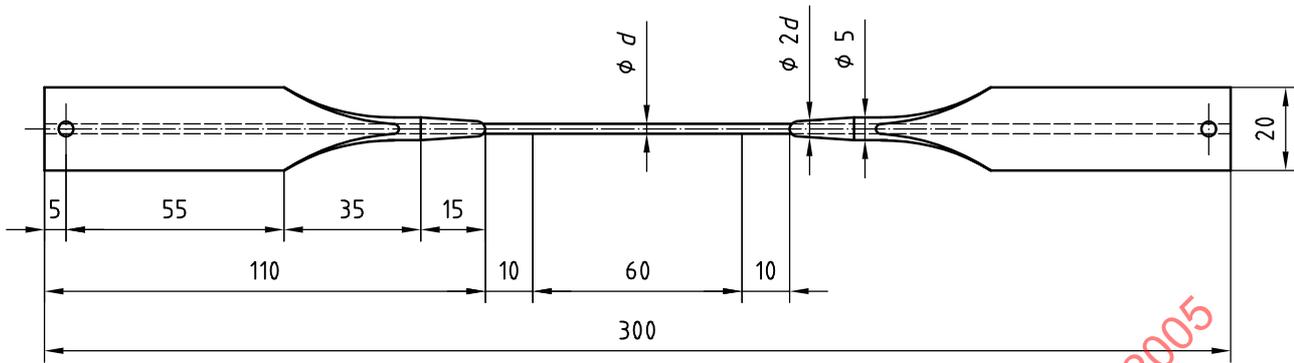


Figure 3 — Test specimen

6.2.3 Impregnation procedure

Set up the impregnating apparatus (6.1.1) as shown in Figure 1. Details of the apparatus may vary depending on the type of roving package (reel or ball) and the way in which it is unwound (unravelling or unrolled). Verify that:

- The tension-regulating system and the yarn guide in front of the vat do not cause any damage to the roving.
- The impregnating system and the conditions of passage through the vat are such that they provide an impregnated roving of good quality. The quality of the impregnated roving may be verified by measuring the void content of the material in accordance with ISO 7822. For the purposes of this International Standard, the void content shall be lower than 2 %. If the void content is higher, adjust the tension and/or the diameter of the die to obtain the correct level.

Carry out the following operations:

Place a package on the reel of the tension-regulating system and unwind and discard at least the first three layers of roving.

Install the die appropriate to the linear density of the roving to be impregnated (see Figure 2).

Prepare an impregnating system (polyester or epoxy resin) with the following characteristics:

- viscosity at impregnation temperature lower than $0,4 \text{ Pa}\cdot\text{s}^1$;
- pot life of the system longer than twice the time required for the winding operation;
- minimum elongation at break: 5 %.

Pour the impregnation resin into the vat (see Figure 2) and maintain a continuous check on its temperature. Start the impregnation of the roving when the resin has reached the vat temperature.

Adjust the unwinding tension to about 3 mN/tex .

Rotate the former (see Figure 4) at about 2 rpm and as regularly as possible.

Wind on to the former a number of turns equal to at least two times the number required for the test specimens to be produced; cut the roving and attach the free end to the former.

1) $1 \text{ Pa}\cdot\text{s} = 10 \text{ poises}$

Keep the frame turning for about 10 min to prevent the resin from flowing along the roving.

Place the former vertically in an oven at a temperature which will ensure partial curing of the resin (a method in which curing is complete is described in Clause 7).

Remove the former from the oven, and unwind and discard the first three turns and the last turn of the impregnated roving.

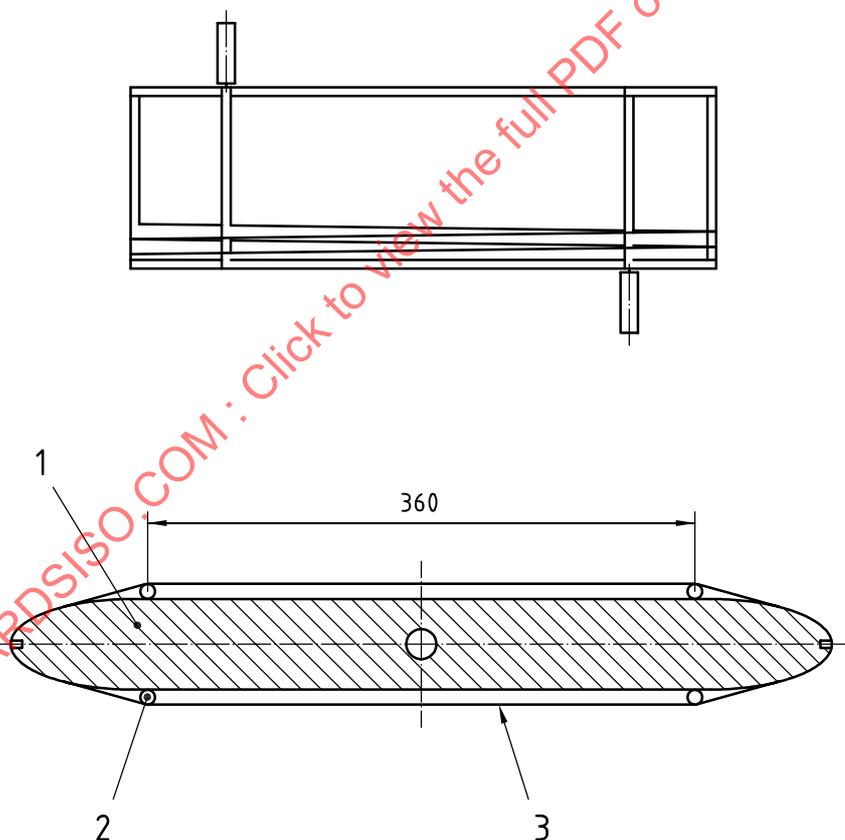
Take 10 test specimens from each side of the former. The impregnated roving remaining on the former shall be used to determine the percentage of glass by loss on ignition in accordance with ISO 1172.

Label the impregnated rovings and store them in a cardboard box to protect them against light, humidity and shocks. Store the box, as far as possible, in a conditioned environment in accordance with ISO 291.

Determine the exact linear density of the unimpregnated roving remaining in the package, in accordance with ISO 1889. If it is not possible to follow ISO 1889 exactly, determine the linear density by taking five test specimens of length 1 m and weighing them before and after ignition (in accordance with ISO 1887).

Provide each test specimen with tabs as described below.

Dimensions in millimetres



Key

- 1 former
- 2 silicone rubber bar, \varnothing 5 mm
- 3 impregnated roving

Figure 4 — Former on which impregnated roving is wound

6.3 Moulding of tabs

6.3.1 Fabrication of moulds

6.3.1.1 Apparatus (see Figure 5) consisting of:

- a mould production box;
- a glass plate measuring 400 mm × 400 mm × 5 mm;
- a rigid transparent plastic sheet;
- a flexible transparent plastic sheet;
- aluminium templates (see Figure 6).

6.3.1.2 Procedure (see Figure 5)

The moulds are made by casting a resin system which will cure at room temperature and gives low shrinkage (e.g. silicone rubber) in the mould production box in which the two templates have been positioned and fixed.

Carry out the following operations to produce the moulds:

Stir the resin thoroughly before and after the addition of the catalyst, in order to obtain a homogeneous mixture.

Pour the resin into the mould production box, taking care to distribute the resin evenly over the whole surface.

Wait for any bubbles in the resin to disappear, then lay the flexible transparent plastic sheet over the mould, beginning at the centre and taking care to push any pockets of air trapped under the sheet out to the sides.

Place the rigid transparent plastic sheet on the flexible one, followed by the glass plate, and finally five weights of 1 kg each, evenly distributed.

Allow the resin to cure at room temperature in accordance with the resin manufacturer's instructions.

Take the block of resin out of the mould production box and trim off excess resin with a sharp tool (e.g. a razor blade).

Take out the templates.

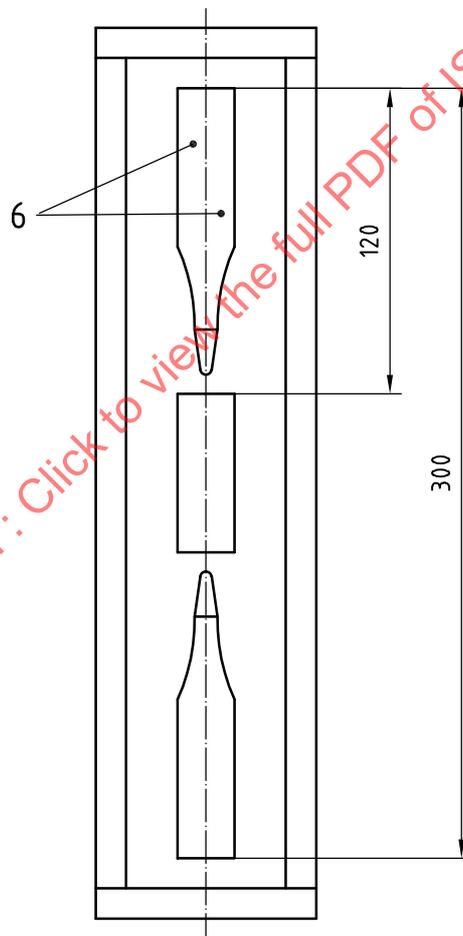
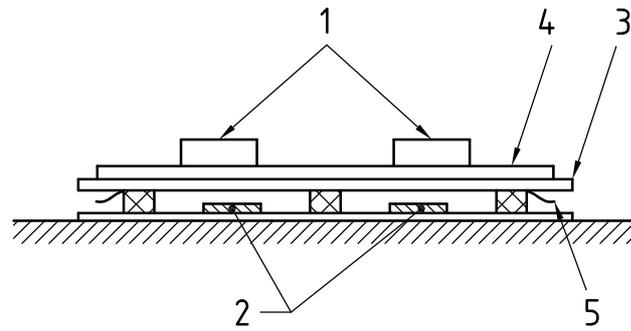
Cut a slit in the mould to allow the impregnated roving to be inserted

6.3.2 Casting the tabs

6.3.2.1 Apparatus (see Figure 7) consisting of:

- a flat-topped three-legged stand;
- two glass plates;
- a spirit level;
- a template for cutting the test specimens from the impregnated rovings (see Figure 8);
- an oven, the temperature of which can be controlled between 20 °C and 190 °C, with temperature programming and automatic switch-off facilities.

Dimensions in millimetres



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Key

- 1 weights of 1 kg
- 2 templates
- 3 rigid, transparent plastic sheet
- 4 glass plate
- 5 flexible, transparent plastic sheet
- 6 locating pins

Figure 5 — Fabrication of moulds

Dimensions in millimetres

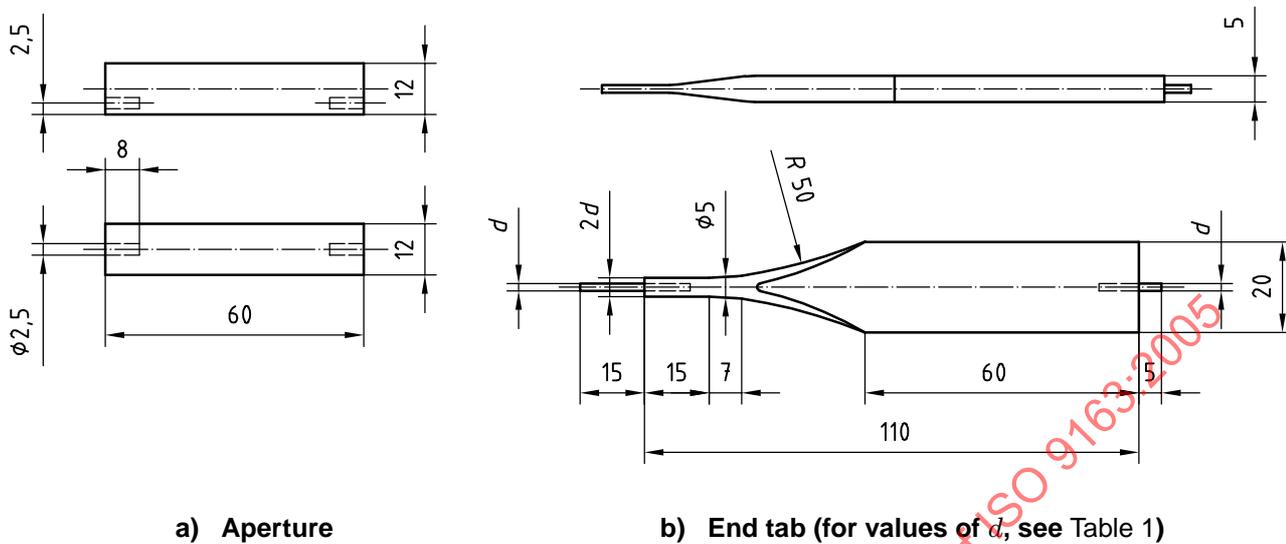


Figure 6 — Templates for fabrication of moulds

6.3.2.2 Procedure

Carry out the following operations:

Select from the impregnated roving test specimens which are free from defects, and cut them to the appropriate length on the template.

Place the test specimens in the moulds (see 6.3.1).

Place the three-legged stand in the oven and adjust it so that it is horizontal when checked by the spirit level.

Place the moulds on a glass plate, which serves as a support, and place the glass plate on the three-legged stand.

Turn on the oven, set at the curing temperature, at least 15 min before pouring the resin into the moulds.

Prepare a resin system based on an epoxy resin having the following properties:

- a) high bonding power;
- b) high breaking strength, i.e.
 - an elongation at break higher than 15 %;
 - a breaking stress higher than 30 MPa;
 - a tangent modulus of elasticity higher than 2 000 MPa;
 - the ability to reach an adequate degree of curing when subjected to the curing cycle used for the impregnated roving.

Pour the resin into the moulds so that the moulds are slightly more than full, i.e. the resin meniscus is clearly visible above the rim of the mould (see Figure 9).

Cure the resin until it is hard, but do not cure it completely in order to make the following operation easier, and also to reduce the utilization time of the moulds.

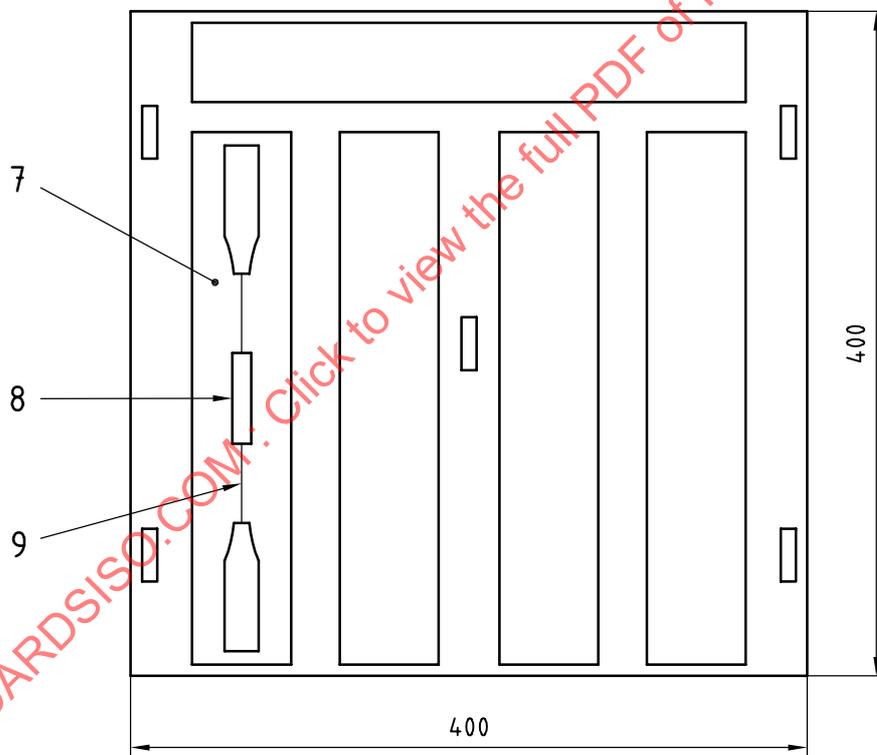
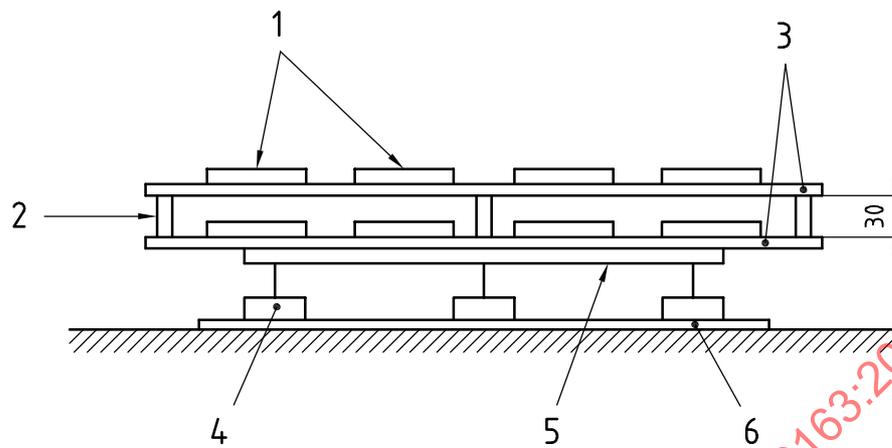
Remove the test specimens from the moulds and cut off, with a razor blade, all flashes on the tabs, as well as the flash of resin which forms on the impregnated roving along the spew groove (see Figure 10). Take care not to damage the impregnated roving during this operation.

Place the test specimens on another glass plate, protected with a flexible plastic sheet.

Complete the curing of the resin.

Pierce locating holes in the tabs (see Figure 3).

Dimensions in millimetres



Key

- 1 moulds
- 2 spacer blocks (all same height)
- 3 glass plates
- 4 metal blocks
- 5 flat-topped three-legged stand
- 6 rigid baseplate
- 7 mould without impregnated roving
- 8 aperture
- 9 slit for impregnated roving

Figure 7 — Production of end tabs

Dimensions in millimetres

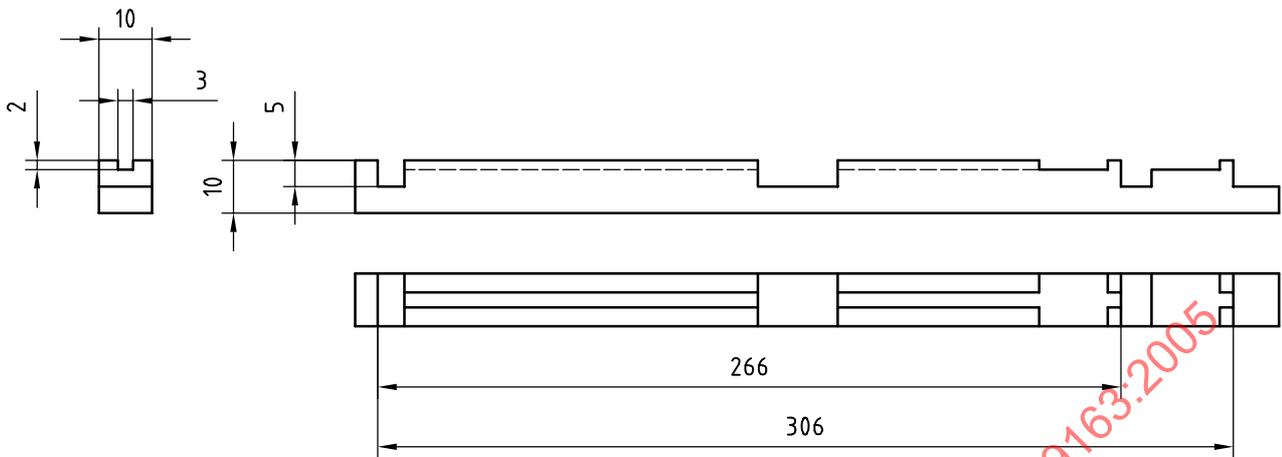
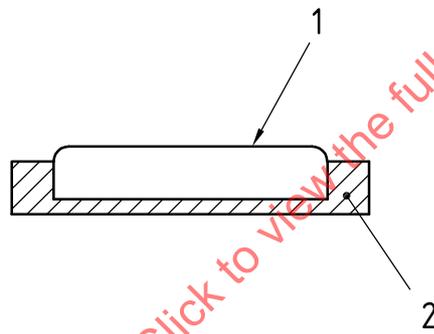


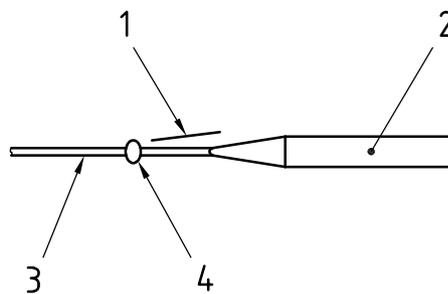
Figure 8 — Template for cutting test specimens from impregnated roving



Key

- 1 resin
- 2 mould

Figure 9 — Pouring the resin



Key

- 1 razor blade
- 2 tab
- 3 impregnated roving
- 4 flash to be cut off

Figure 10 — Deflashing a tab

7 Production of test specimens — Short method

7.1 Apparatus

Ordinary laboratory apparatus, plus the following:

7.1.1 Impregnation apparatus (see Figure 11), including the following elements:

7.1.1.1 Tension-regulating system, capable of maintaining the roving at a tension between 0,2 N and 20 N.

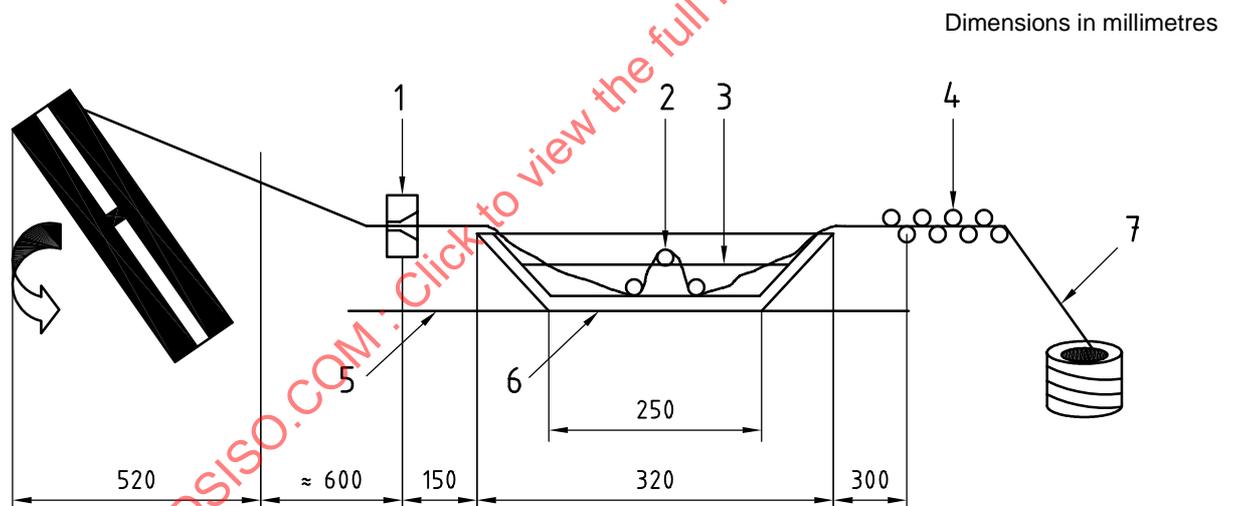
7.1.1.2 Impregnation vat, equipped with yarn guides.

It shall be capable of maintaining the temperature required for the resin system used with a precision of ± 5 °C.

The use of a double-walled vat, with heating fluid circulating between the walls, is recommended. If such a vat is not available, a hotplate may be used.

7.1.1.3 Stainless-steel die, diameter 1,1 mm (for 1 200 tex rovings), defined to give the impregnated roving a defined circular cross-section. It shall be fitted on an adjustable support.

The glass content by mass, determined as specified in ISO 1172, shall be approximately constant at (70 ± 5) %.



Key

- 1 die of $\varnothing 1,1$ mm
- 2 tensioning bars
- 3 resin level
- 4 tension-regulating device (21 teeth)
- 5 adjustable support
- 6 impregnation vat (temperature regulated)
- 7 unravellled 1 200 tex roving

Figure 11 — Impregnation apparatus

7.1.1.4 Winding device (mechanical or manual) with former, for collecting the impregnated roving, ensuring that

- the roving is kept under tension;
- the impregnated strands are kept separate from each other.

The collecting speed shall be as constant as possible.

7.2 Preparation of test specimens

7.2.1 Type and dimensions

Each test specimen shall consist of an impregnated roving without tabs or fitted with simple tabs.

The free length of the test specimen shall be 150 mm.

The total length shall 370 mm when no tabs are fitted, 300 mm when using composite tabs and 260 mm when using cardboard tabs.

A method for fitting simple tabs is given in 7.3.

7.2.2 Number

For each of the packages selected (see Clause 5), at least 20 test specimens shall be prepared to obtain at least 10 valid readings (five for the determination of the breaking stress and five for the tensile modulus). Testing of more specimens might be necessary for statistical purposes.

7.2.3 Impregnation procedure

Set up the impregnating apparatus (7.1.1) as shown in Figure 11. Check that:

- a) The tension-regulating system and the yarn guide in front of the vat do not cause any damage to the roving.
- b) The impregnating system and the conditions of passage through the vat are such that they provide an impregnated roving of good quality. The quality of the impregnated roving may be verified by measuring the void content of the material in accordance with ISO 7822. For the purposes of this International Standard, the void content shall be lower than 2 %. If the void content is higher, adjust the tension and/or the diameter of the die to obtain the correct level.

Carry out the following operations:

Place a package on the floor and unwind and discard at least the first three layers of roving. Pass the end of the roving from the package through the vat and attach it to the winding frame.

Taking account of the instructions of the resin manufacturer, prepare an impregnating system (polyester or epoxy resin) so that its viscosity at the impregnation temperature is lower than $0,4 \text{ Pa}\cdot\text{s}^2$.

Pour the impregnation resin into the vat and maintain a continuous check on its temperature. Start the impregnation of the roving when the resin has reached the vat temperature.

Adjust the unwinding tension to about 1,6 N by adjusting the tension-regulating device.

Rotate the former at about 4 rpm and as regularly as possible.

Wind on to the former a number of turns equal to the number required for the test specimens to be produced plus six turns for quality-control purposes and two turns for glass content determinations; cut the roving and attach the free end to the former.

Keep the former turning for about 10 min to prevent the resin from flowing along the roving.

2) $1 \text{ Pa}\cdot\text{s} = 10 \text{ poises}$

Place the former vertically in an oven at a temperature which will ensure the curing of the resin, for example:

- 2 h at 80 °C + 4 h at 120 °C for a polyester system;
- 2 h at 100 °C + 4 h at 140 °C for an epoxy system.

Remove the former from the oven and cut out at least 20 lengths of impregnated roving, discarding the first three and the last three turns of impregnated roving.

The impregnated roving remaining shall be used to determine the percentage of glass by loss on ignition in accordance with ISO 1172.

Label the impregnated rovings and store them in a cardboard box to protect them against light, humidity and shocks. Store the box, as far as possible, in a conditioned environment in accordance with ISO 291.

Determine the exact linear density of the unimpregnated roving remaining in the package, in accordance with ISO 1889.

If using tabs, attach them as described in 7.3.

7.3 Fitting of tabs

7.3.1 Production of template

Use a steel plate with four stops and notched end walls (see Figure 12).

7.3.2 Procedure

Prepare a suitable quantity of adhesive (epoxy system for cardboard tabs, polyester system for composite tabs).

Place a strip of tab material (55 mm wide in the case of cardboard, 75 mm wide in the case of composite) across each end of the template (along the notched end walls) and coat the strips with adhesive.

Then put the test specimens prepared in 7.2.3 on the strips. In order to improve adhesion, when necessary, the impregnated roving can be abraded lightly with abrasive paper.

Cover each of the strips with a second strip and compress with a metal plate.

Place the template + tabs + specimens + plate in an oven for curing (temperature and duration as recommended by the manufacturer).

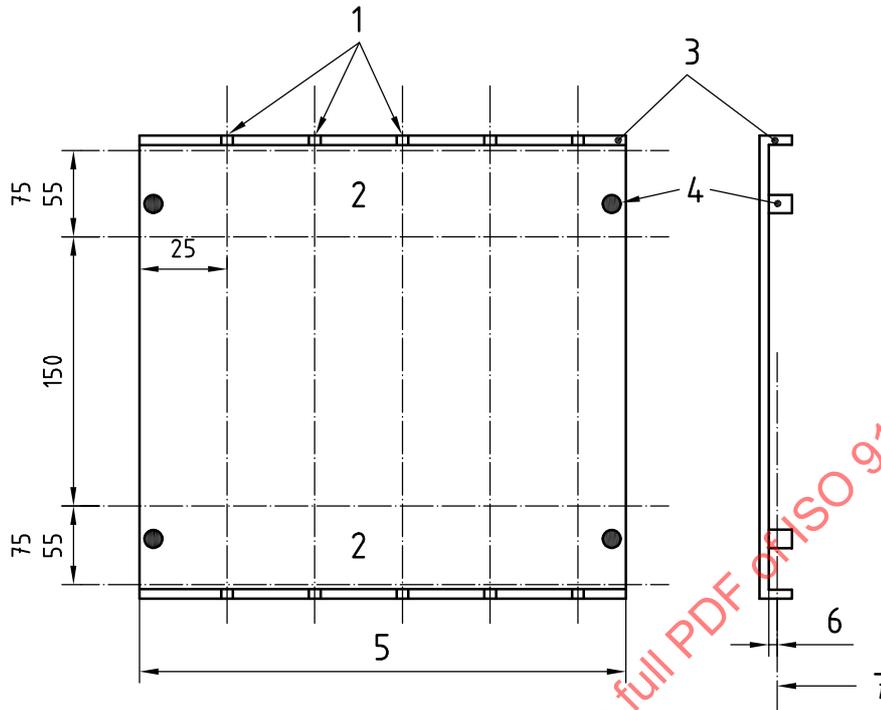
Cut the strips so as to obtain test specimens with tabs, taking care not to damage the impregnated roving. For easier preparation of tabs made of chopped-strand mat, the tabs can be cut with a sharp pair of scissors after the gelation period.

8 Tensile testing

8.1 Apparatus

8.1.1 Tensile-testing machine, of the constant-rate-of-extension (CRE) type, which shall be

- equipped with a load-indicating or recording device;
- used between 20 % and 80 % of the selected load scale;
- accurate to within ± 1 % of the indicated load over the selected load scale.



Key

- 1 notches for positioning impregnated rovings
- 2 position of strip of cardboard or composite
- 3 end wall
- 4 spacers
- 5 dimension will depend on number of test specimens
- 6 thickness of cardboard or composite
- 7 level of bottoms of notches

Figure 12 — Tab template

8.1.2 Specimen-clamping devices.

For tabbed test specimens:

- use clamps fitted with a pin designed to hold the test specimen centrally between the jaws;
- the jaws of the clamps shall be wider than the test specimen, and shall have a gripping length of at least 50 mm; their faces shall be plane and parallel, shall ensure uniform pressure over the whole width of the test specimen, and shall hold it firmly without slippage.

For test specimens without tabs, use grips of the type shown in Figure 13.

The use of a guiding device is recommended in order to avoid the necessity to lock manually the upper jaws or grips, which could result in damage to the test specimen.