INTERNATIONAL STANDARD

ISO 9163

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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

Reference number
ISO 9163:1996(E)

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.

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.

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

Annex A forms an integral part of this International Standard.

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Introduction

There are several methods of measuring the tensile strength of rovings, using as a specimen either an unimpregnated roving or a roving impregnated with polyester or epoxy resin or with rosin/beeswax mixture.

The results obtained with these different methods are not equivalent.

The test carried out on rovings impregnated with cured resin is considered the reference method. It allows both the tensile stress at break and the modulus of elasticity to be measured. This method is the only one which gives results having a direct correlation with the tensile properties of reinforced plastics made from the continuous roving.

The test results obtained with rovings impregnated with rosin/beeswax are generally 10 % to 20 % lower than those obtained with the reference method. Moreover, the method using rovings impregnated with rosin/beeswax does not give the tensile modulus of elasticity. On the other hand, it is simpler than the reference method.

The measurement of the tensile strength using unimpregnated rovings is even simpler, but the results are lower than those obtained with rovings impregnated with rosin/beeswax mixture. They also show a greater variation. On the other hand, this method is a useful one for the user of the roving to check the quality of the product. This method is not covered by this International Standard but is described in ISO 3341.

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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:

— the reference method (roving impregnated with thermosetting resin);

— a rapid method (roving impregnated with rosin/beeswax).

The reference method is used to determine, in addition, the tensile modulus of elasticity of the glass.

The methods are applicable to both assembled (multistrand) and direct (multifilament) rovings.

2 Normative references

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

ISO 291:—[1], *Plastics — Standard atmospheres for conditioning and testing.*

ISO 1172:—[2], *Textile-glass-reinforced plastics — Prepegs, moulding compounds and laminates — Determination of the textile-glass and mineral-filter content — Calculation method.*

ISO 1886:1990, *Reinforcement fibres — Sampling plans applicable to received batches.*

ISO 1887:1995, *Textile glass — Determination of combustible-matter content.*

ISO 1889:—[3], *Reinforcement yarns — Determination of linear density.*

ISO 2078:1993, *Textile glass — Yarns — Designation.*

ISO 2602:1980, *Statistical interpretation of test results — Estimation of the mean — Confidence interval.*

ISO 3341:1984, *Textile glass — Yarns — Determination of breaking force and breaking elongation.*

ISO 7822:1990, *Textile glass reinforced plastics — Determination of void content — Loss on ignition, mechanical disintegration and statistical counting methods.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 breaking force: The maximum tensile force (expressed in newtons) required to break a test specimen in a tensile test.

1) To be published. (Revision of ISO 291:1977)

2) To be published. (Revision of ISO 1172:1975)

3) To be published. (Revision of ISO 1889:1987 and ISO 10120:1991)

3.2 gauge length: The nominal length (expressed in millimetres) between the spines of contact of an extensometer, by reference to which the length increase due to a tensile force is determined.

3.3 relative elongation: The 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: The ratio (expressed in megapascals) of the breaking force (expressed in newtons) to the cross-sectional area[4] of a roving (expressed in square millimetres).

The force experienced by the resin can be neglected.

3.5 loading stress: The ratio (expressed in megapascals) of the tensile force applied to a roving during a tensile test (expressed in newtons) to the cross-sectional area[4] of the roving (expressed in square millimetres).

The force experienced by the resin can be neglected.

3.6 tensile modulus of elasticity of glass: The ratio (expressed in megapascals) of the stress (expressed in megapascals) to the corresponding strain, when the stress is below the proportionality limit or can be determined by extrapolation beyond this limit.

3.7 proportionality limit: The 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, using suitable apparatus, to a tensile rupture test and the breaking stress determined. In the case of the reference method only, the tensile modulus of elasticity of the glass is also calculated.

5 Reference method

5.1 Apparatus

Ordinary laboratory apparatus, plus the following:

5.1.1 Impregnation apparatus (see figure 1), including the elements specified in 5.1.1.1 to 5.1.1.4.

5.1.1.1 Reel, equipped with a tension-regulating system capable of maintaining the roving under a tension between 0,2 N and 20 N.

5.1.1.2 Impregnation vat (see figure 2), equipped with yarn guides and capable of maintaining a temperature of up to 130 °C with a precision of ± 5 °C (the temperature is dependent 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.

Dimensions in metres

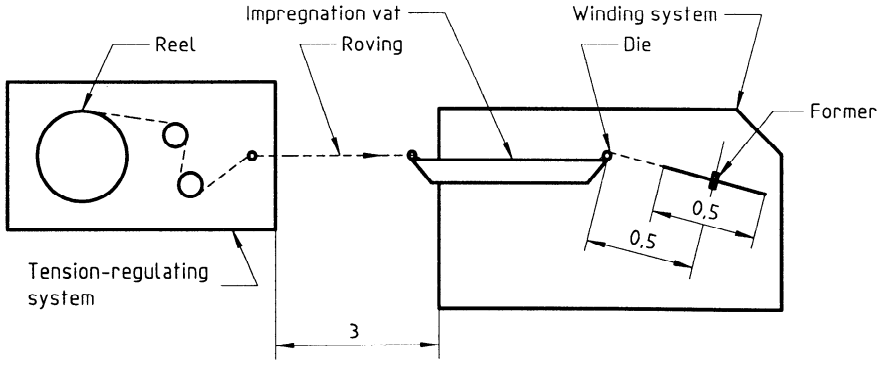


Figure 1 — General layout of impregnation apparatus

4) The cross-sectional area S of the roving, expressed in square millimetres, is given by the formula

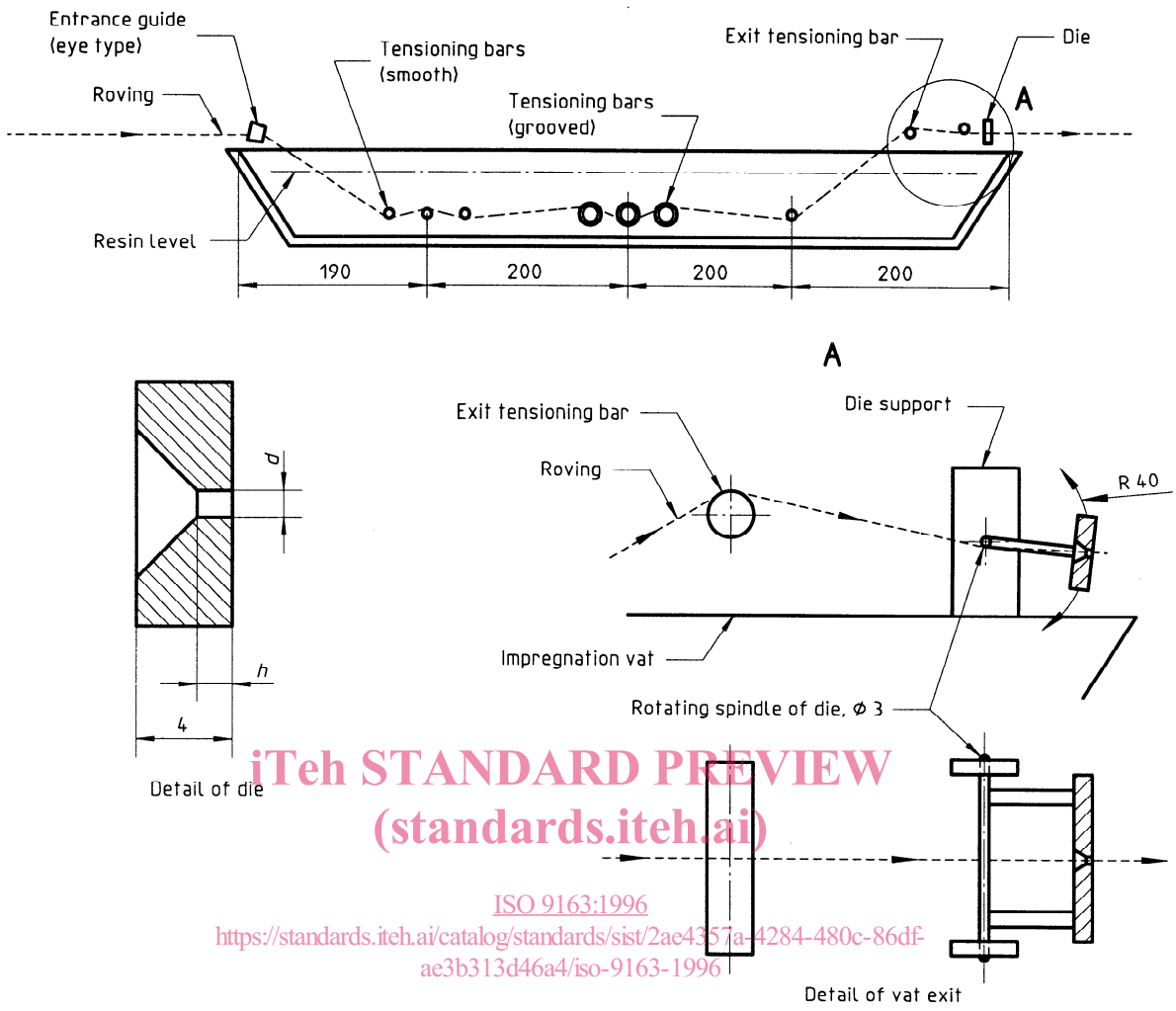
$$\frac{\rho_l \times 10^{-3}}{\rho}$$

where

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

ρ is the density, in grams per cubic centimetre, of the glass constituting the roving.

Dimensions in millimetres



ρ_l (tex)	800	1 600	2 400	4 800
d (mm)	0,8	1,15	1,4	2
h (mm)	1	1, 5	2	3

$d = 0{,}028\,3\sqrt{\rho_l}$ (round up to the nearest 0,05 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 the table be used, in order to obtain a reproducible impregnation ratio.

Figure 2 — Vat and die

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

The die shall be mounted on a spindle, so that it will align itself automatically with the roving during reeling (see figure 2).

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

5.1.1.4 Device (mechanical or manual) **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.

5.1.2 Test machine.

5.1.2.1 A tensioning machine with clamps fitted with a pin designed to hold the test specimen centrally between the jaws of the clamps shall be used. The machine shall also be equipped with a load-indicating or recording device. It shall be of the constant-rate-of-extension (CRE) type and shall be used between 20 % and 80 % of the selected load scale.

The machine shall be accurate to within ± 1 % of the indicated load over the selected load scale.

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

The jaws 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.

5.1.2.2 An extensometer shall be used with associated equipment for the continuous recording of elongation as a function of the applied force.

The extensometer shall be attached to the test specimen in such a way that no slipping occurs and the test specimen is not damaged in any way. It shall be sufficiently light to induce negligible stresses in the test specimen. The inertia of the moving parts shall be low enough to have no effect on the force/elongation curve.

The use of an extensometer with a gauge length of 50 mm is recommended.

5.1.3 Conditioning apparatus (see 5.2.2).

5.2 Sampling and conditioning

5.2.1 Sampling

Carry out sampling in accordance with ISO 1886. Use the "inspection by variables" method in order to minimize the number of elementary units (packages) to be selected.

5.2.2 Conditioning

Condition the packages selected as specified in 5.2.1 for 12 h to 24 h in one of the standard atmospheres specified in ISO 291.

5.2.3 Test atmosphere

Conduct the test in the same atmospheric conditions as those under which the rovings were conditioned (see 5.2.2).

5.3 Test specimens

5.3.1 Type and dimensions

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

A method for the fabrication of end tabs is given in annex A.

5.3.2 Number

For each of the packages selected as specified in 5.2.1, at least 10 test specimens shall be prepared to obtain at least 10 valid readings (five for the determination of the breaking stress and five for the determination of the tensile modulus of elasticity).

5.3.3 Impregnation procedure

Set up the impregnating apparatus (5.1.1) as shown in figure 1.

NOTE 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 (unravelled or unrolled).

The tension-regulating system and the yarn guide in front of the vat shall not cause any damage to the roving.

Dimensions in millimetres

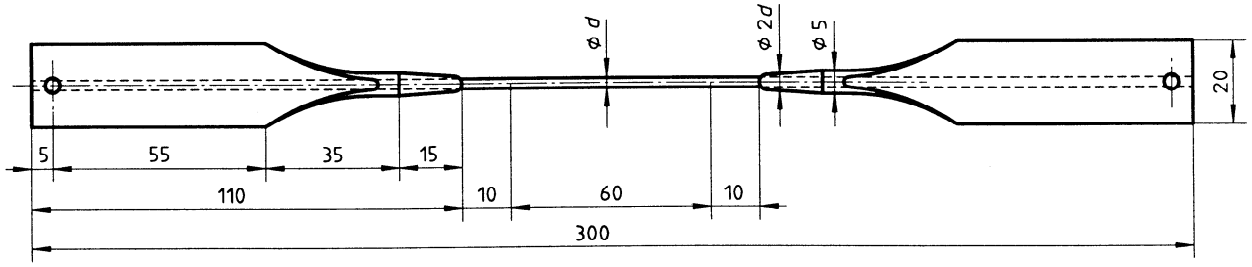


Figure 3 — Test specimen

The impregnating system and the conditions of passage of the roving through the vat shall be 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 %.

Carry out the following operations:

Place a package on the reel of the tension-regulating system and unwind and discard at least the first layer 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 0,3 Pa·s[5];

— 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 3 mN/tex, i.e.

2,4 N for 800 tex;

4,8 N for 1 600 tex;

7,2 N for 2 400 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 three times the number required for the test specimens to be produced, cut the roving and attach the free end to the former.

Place the former vertically in an oven at a temperature which will ensure partial polymerization of the resin.

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

Take five 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.

Mark the impregnated rovings and store them in a cardboard box to protect them against light, humidity and knocks.

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

Dimensions in millimetres

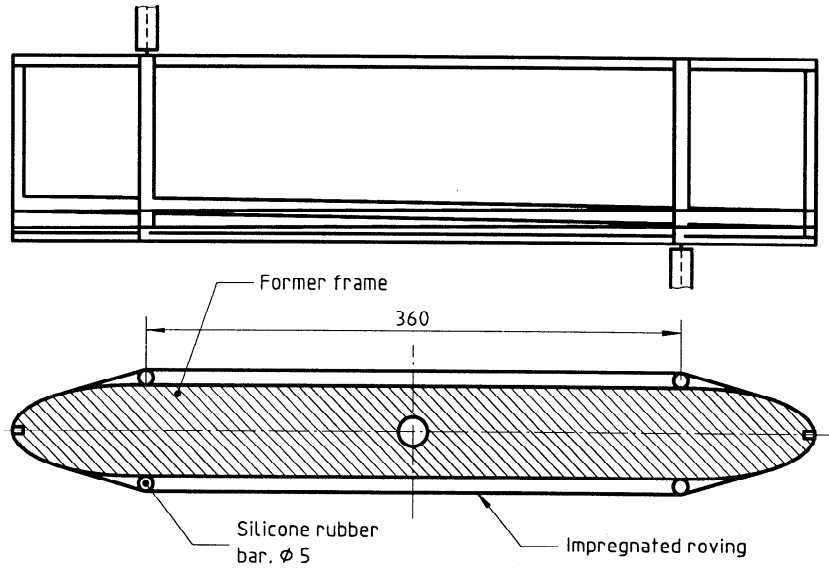


Figure 4 — Former on which impregnated roving is wound

5) 1 Pa·s = 10 poises

accordance with ISO 1887) in order to obtain the percentage of size and the exact linear density of the roving.

Provide each test specimen with end tabs as described in annex A.

5.4 Test procedure

5.4.1 Condition the test specimens for 16 h in the atmosphere specified in 5.2.3. Then carry out the test on the test machine described in 5.1.2, proceeding as follows:

Set the crosshead speed at 5 mm/min.

Fix a test specimen in the clamps, ensuring that a length of 50 mm of each end tab is held in the jaws of the clamps.

Apply a pre-tension, equal to less than 10 % of the expected breaking force, before tightening the jaws, in order to ensure correct alignment of the test specimen.

Test five specimens, without the extensometer, until break (stress determination).

Test the five remaining specimens with the extensometer (modulus determination).

As the extensometer could be damaged when the test specimen breaks, remove it when the applied force has reached approximately two-thirds of the expected breaking force.

5.4.2 After the test, note the type of rupture as follows:

— correct break (C.B.): simultaneous break of all fibres at centre of specimen;

— fibre rupture (F.R.): break not simultaneous (break of a fibre before final rupture);

— debonding (D): shear separation of roving and resin in tab, before breaking, causing a discontinuity in the force/elongation curve;

— incorrect break (I.B.): break outside central section.

Discard the results corresponding to D and I.B. ruptures and carry out the test with supplementary test specimens so that five valid readings are obtained for each property (stress and modulus).

5.5 Expression of results

In the following calculations, it is assumed that only the glass is involved in the rupturing process. This introduces a very slight error, but simplifies the calculation.

5.5.1 Breaking stress

The breaking stress σ_r, in megapascals, is given by the formula

$$\frac{F_r \times \rho}{10^{-3} \rho_l}$$

where

F_r is the breaking force, in newtons;

ρ is the density, in grams per cubic centimetre, of the glass of which the roving is made (see ISO 2078 for definitions of the various types of glass) — unless otherwise specified, use the following values:

2,61 for E glass,
2,71 for E-CR glass,
2,55 for R glass,
2,49 for S glass;

ρ_l is the exact linear density, in tex, of the roving.

5.5.2 Modulus of elasticity in tension

The modulus of elasticity in tension E is given, in megapascals, by the formula

$$\frac{F \times \rho}{10^{-3} \rho_l} \times \frac{L_0}{\Delta L}$$

where

F is the measured force, in newtons, which gives an elongation ΔL;

L_0 is the gauge length, in millimetres, of the extensometer;

ΔL is the elongation, in millimetres, produced by the force F;

ρ and ρ_l are as defined in 5.5.1.

5.6 Test report

The test report shall include the following particulars:

a) a reference to this International Standard and the method used (reference method);

b) all details necessary for identification of the roving tested;

c) the impregnation system used;

d) the exact linear density of the roving (and the standard deviation);

e) the percentage by mass of glass in the impregnated roving (and the standard deviation);

f) the breaking stress (and the standard deviation);

g) the tensile modulus of elasticity of the glass (and the standard deviation);

h) the type of break observed with each test specimen;

i) the date of impregnation of the roving and the date of the test;

j) details of any operation not specified in this International Standard, as well as any incident liable to have affected the results.

6 Fast method

This method is similar to the reference method described in 5.1 to 5.6, but incorporates modifications designed to permit the test to be carried out more rapidly. Note that the fast method is valid only for the determination of the breaking stress.

6.1 Apparatus

Ordinary laboratory apparatus, plus the following:

6.1.1 Impregnation apparatus, as specified in 5.1.1 but taking account of the following differences:

The ratio by mass of rosin to beeswax in the rosin/beeswax mixture shall be 35 % ± 10 %.

The former on to which the impregnated roving is wound may be a circular mandrel 160 mm to 200 mm in diameter.

If necessary (see note 1 in 5.3.3), the apparatus may also include a cold-air blower.

6.1.2 Test machine, as specified in 5.1.2, but in which the clamps are replaced by suitable grips (see ISO 3341) which do not impart to the impregnated roving a curvature of radius less than 30 mm.

6.1.3 Conditioning apparatus (see 5.2.2).

6.2 Sampling and conditioning

See 5.2.

6.3 Test specimens

6.3.1 Type and dimensions

Each test specimen shall consist of a length of impregnated roving without tabs.

For the tensile test, the length of each test specimen shall be equal to the distance between the grips (500 mm) plus at least 50 mm at each end to allow the specimen to be held securely in the grips.

For the determination of the loss on ignition, the test specimens shall be 2 m to 5 m in length, depending on the mass per unit length of the roving.

6.3.2 Number

Prepare at least 20 specimens for the tensile test and two for the determination of the loss on ignition. These last two specimens shall be cut one from each end of the roving from which the test specimens for the tensile test were taken.

6.3.3 Impregnation procedure

The diameter of the die used shall be

1 mm for 800 tex;

1,2 mm for 1 200 tex;

1,6 mm for 2 400 tex;

2,4 mm for 4 800 tex.

The impregnation system shall be a mixture of 75 % rosin and 25 % beeswax.

The impregnation shall be carried out at a temperature between 125 °C and 135 °C.

The impregnation system shall be replaced after a utilization time at 125 °C of 48 h.

As the impregnated roving remains tacky until the impregnation mixture has cooled sufficiently, the follow-