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Fibre-reinforced plastics — Moulding compounds and prepregs — Determination of mass per unit area and fibre mass per unit area

Plastiques renforcés de fibres — Mélanges à mouler et préimprégnés — Détermination de la masse surfacique et de la masse des fibres par unité de surface

Reference number
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Contents

Page

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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 10352:2010), which has been technically revised.

The main changes compared to the previous edition are as follows:

- determination of fibre mass per unit area by Method A, Method B, Method C, Method D and Method E have been added.

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.

Fibre-reinforced plastics — Moulding compounds and prepregs — Determination of mass per unit area and fibre mass per unit area

1 Scope

This document specifies a method for the determination of the mass per unit area. It also specifies five methods (Method A to Method E) for the determination of the fibre mass per unit area of moulding compounds and prepregs. The five methods are as follows:

- Method A: Extraction by Soxhlet;
- Method B: Extraction by immersion in solvent in a beaker;
- Method C: Decomposition by loss ignition;
- Method D: Extraction by wet combustion;
- Method E: Method by calculation.

This document is applicable to the following types of materials:

- moulding compound and preimpregnated unidirectional sheet, tape, fabric and mats;
- prepregs in which any type of reinforcement (aramid, carbon, glass, etc.) and any type of matrix (thermosetting or thermoplastic) has been used.

Typically, reinforcement fibres are coated with sizing or finishes. These normally dissolve with the resin and are, therefore, included in the resin content.

This document is not applicable to the following types of prepregs:

- those containing reinforcements which are soluble (or partly soluble) in the solvents used to dissolve the resin.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

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

ISO 4602, *Reinforcements — Woven fabrics — Determination of number of yarns per unit length of warp and weft*

3 Terms and definitions

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

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

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1
elementary unit
individual sample roll, sheet or pack which is intended for the measurement of the mass per unit area using this document

Note 1 to entry: Prepregs are usually supplied in rolls or in packs of sheets. In this context, an individual roll or pack of sheets is an elementary unit.

3.2
laboratory sample
sample taken from an *elementary unit* (3.1)

3.3
test specimen
specimen cut from a *laboratory sample* (3.2)

4 Principle

4.1 Determination of mass per unit area

The mass of a test specimen of known area is determined. Two different specimen sizes are specified, depending on the type of material. If the material has been manufactured using a solvent or if the volatile-matter content of the material is not negligible, the sample is conditioned in a specified atmosphere before test specimens are taken. The result is expressed as the mass per unit area.

4.2 Determination of fibre mass per unit area

4.2.1 Method A: Extraction in Soxhlet-type apparatus

Determination of mass by means of weighing to constant mass after extraction of the resin with suitable solvent in a Soxhlet-type apparatus. Solvent to use shall be able to extract all the resin component completely from the specimen.

4.2.2 Method B: Extraction by immersion in solvent in a beaker

Method B is similar to Method A (see 4.2.1) but faster. In case of dispute, Method A shall be applied.

4.2.3 Method C: Decomposition by loss on ignition

Determination of mass by means of weighing to constant mass after decomposition of the resin by ignition.

4.2.4 Method D: Extraction by wet combustion

Determination of mass by means of weighing to constant mass after extraction of the resin by acid digestion. Use a solution of concentrated sulphuric acid and hydrogen peroxide.

4.2.5 Method E: Method by calculation

If the linear density (in accordance with ISO 1889) and the number of yarns per unit (in accordance with ISO 4602) in use is known, the fibre mass per unit area can be simply calculated.

5 Apparatus and reagents

5.1 General

Normal laboratory equipment is required plus the following specific apparatus.

5.1.1 Balance, graduated in tenths of 0,1 mg and an accuracy of 0,5 mg.

5.1.2 Square template, with the dimensions given in Table 1 for the material being tested, with an accuracy of 0,1 mm on the length of each side.

5.1.3 Ancillary items, such as a sharp cutting tool and tweezers.

5.1.4 Desiccator, with containing a suitable drying agent (for example silica gel, calcium chloride or phosphorus pentoxide).

5.2 For Method A

5.2.1 Single thickness extraction thimble, nominal diameter 20 mm to 22 mm, nominal length 60 mm to 80 mm.

5.2.2 Electric oven, capable of maintaining a temperature of 105 °C with an accuracy of 5 °C.

5.2.3 Extraction apparatus of the Soxhlet type, comprising a condenser, siphon tube and flask and provided with an electric heating mantle.

5.2.4 Suitable solvent for extraction, (analytical grade), solvent to use shall be able to extract all the resin component completely from the specimen.

NOTE 1 General solvent is methyl-ethyl-ketone (MEK), tetra-hydro-furan (THF), acetone, N-methyl-pyrrolidone (NMP), denatured ethanol and others.

NOTE 2 Other solvents or the combination of more than one solvent can be used in order to extract the full all of the resin component from the specimen.

5.3 For Method B

5.3.1 Container 400 ml, Erlenmeyer flask or beaker.

5.3.2 Electric oven, capable of maintaining a temperature of 105 °C with an accuracy of 5 °C.

5.3.3 Suitable solvent for extraction, (analytical grade) solvent to use shall be able to extract all the resin component completely from the specimen.

NOTE 1 General solvent is methyl-ethyl-ketone (MEK), tetra-hydro-furan (THF), acetone, N-methyl-pyrrolidone (NMP), denatured ethanol and others.

NOTE 2 Other solvents or the combination of more than one solvent can be used in order to extract the full all of the resin component from the specimen.

5.3.4 Acetone (propanone)

5.4 For Method C

5.4.1 Crucible from porcelain or equivalent properties, with a capacity sufficient to completely contain the specimen.

5.4.2 Electric oven, capable of maintaining a temperature of 105 °C with an accuracy of 5 °C.

5.4.3 Muffle designed electric furnace, capable of maintaining a temperature of 565 °C with an accuracy of 30 °C.

5.4.4 Bunsen flame.

5.4.5 Heatproof gloves and safety screen for eye protection.

5.4.6 Laboratory exhausts fan and/or ventilation system.

5.5 For Method D

5.5.1 Erlenmeyer 250 ml double necked, pear-shaped flask, with 50 ml dropping funnel equipped with air inlet and a water pump.

5.5.2 Heat source, with suitable temperature control.

5.5.3 Container 400 ml, Erlenmeyer flask or beaker.

5.5.4 20 ml sintered glass crucible and suitable filtration assembly.

5.5.5 Electric oven, capable of maintaining a temperature of 105 °C with an accuracy of 5 °C.

5.5.6 Protective equipment, with protective clothing and rubber gloves resistant to hydrogen peroxide and sulphuric acid solutions, safety screen for eye protection.

5.5.7 Concentrated sulphuric acid, with specific gravity: 1,84 to 1,89.

5.5.8 Hydrogen peroxide solution, with concentration: 300 g/l to 500 g/l.

5.5.9 Acetone (propanone).

5.5.10 Distilled water.

6 Conditioning and testing

6.1 Conditioning

6.1.1 Materials for which no conditioning is required

Conditioning is not required for the following materials:

- those which are known to have been manufactured without using a solvent and to have been stored under conditions close to standard atmospheric conditions;

- those for which the volatile-matter content is known, from test data, to be negligible and which are known to have been stored under conditions close to standard atmospheric conditions;
- those for which the material specification states that no conditioning is required.

After the laboratory sample has been cut from an elementary unit, the sample shall be covered with a plastic film.

6.1.2 Conditioning of material stored at ambient temperature

With material which has been stored at ambient temperature, the laboratory sample cut from an elementary unit shall be conditioned in the same standard atmosphere as that which will be used for testing (see 6.2.1) for a minimum of 2 h, unless otherwise specified.

The laboratory sample shall be kept covered with its protective films in a solvent-resistant bag.

6.1.3 Conditioning of material stored at below ambient temperature

With material which has been stored at temperatures lower than ambient temperature, the material (generally an elementary unit), suitably packed in an airtight, solvent-resistant bag to prevent moisture pick-up, shall be allowed to reach ambient temperature over a period of time determined by the mass of the package. This time shall not be less than 8 h, and the actual time shall be recorded in the test report.

When the material has reached ambient temperature, the laboratory sample cut from the elementary unit shall be conditioned in the same standard atmosphere as that which will be used for testing (see 6.2.1) for a minimum of 2 h, unless otherwise specified.

The laboratory sample shall be kept covered with its protective films in a solvent-resistant bag.

6.2 Testing

6.2.1 Test atmosphere

The test shall be carried out in a standard atmosphere as specified in ISO 291.

6.2.2 Time interval between conditioning and testing

6.2.2.1 Materials not requiring conditioning

There is no time-interval requirement for the three categories of material described in 6.1.1.

6.2.2.2 Materials requiring conditioning

After conditioning, the test shall be carried out within 6 h unless otherwise specified, the laboratory sample being kept in the same standard atmosphere as that which will be used for testing until the test is carried out (see 6.1.2 or 6.1.3).

7 Test specimens

7.1 Shape and dimensions

The preferred test specimen shape is a square with the dimensions given in Table 1, which depend on the type of material. The various types of material given in Table 1 are defined as follows:

- Type Ia materials: relatively heavy materials, such as sheet moulding compounds, preimpregnated mats and woven prepregs, made without using a solvent;

- Type Ib materials: relatively heavy materials, such as sheet moulding compounds, preimpregnated mats and woven prepregs, made using a solvent;
- Type IIa materials: relatively light materials, such as unidirectional prepregs (e.g. for aerospace use), made without using a solvent;
- Type IIb materials: relatively light materials, such as unidirectional prepregs (e.g. for aerospace use), made using a solvent.

NOTE 1 A production method which uses a solvent is usually called a “wet” method and a production method which does not use a solvent is usually called a “dry” method or “hot melt” method.

NOTE 2 The designations type IIa and type IIb are usually used for relatively lightweight materials that require a more accurate determination.

For special applications, rectangular specimens, round specimens or square specimens of dimensions different from those given in Table 1 can be used by agreement between the interested parties. The surface area of these specimens should preferably be between 0,04 m^2 and 0,10 m^2 for type Ia and type Ib materials and between 0,01 m^2 and 0,04 m^2 for type IIa and type IIb materials.

Table 1 — Preferred specimen sizes

Type of material	**Description of material**	**Specimen size** m
Ia or Ib	Sheet moulding compounds, preimpregnated mats and heavy preimpregnated fabrics	0,20 × 0,20
IIa or IIb	Lightweight unidirectional prepregs and preimpregnated fabrics	0,10 × 0,10

7.2 Number

Unless otherwise required by the material specification or agreed between the interested parties, three test specimens shall be taken.

7.3 Preparation

Test specimens are usually taken from the laboratory sample cut from an elementary unit comprising a roll or a pack of sheets. If necessary, taking into account the roll dimensions and the number of sheets per pack, an agreement between the interested parties shall define either the size of the laboratory sample to be taken from each elementary unit or other information such as the number of specimens and their location within the laboratory sample.

The specimens shall be taken uniformly across the roll or sheet and cut perpendicularly to the edges of the sheet.

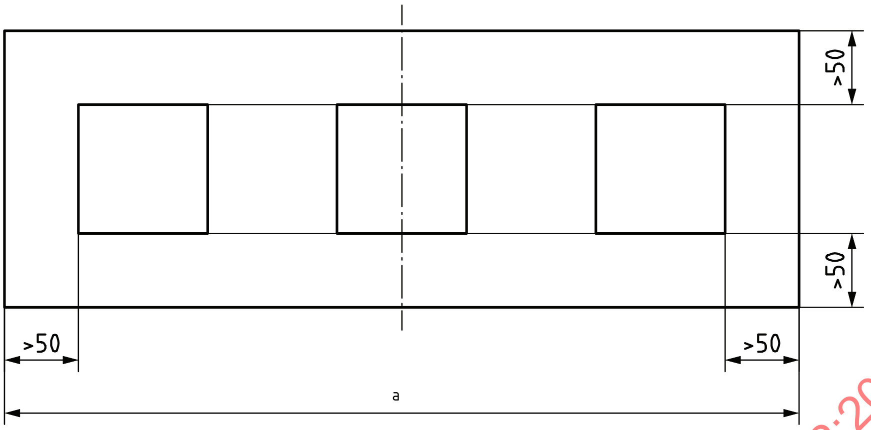
The specimens should be taken 50 mm away from the edges of the roll or sheet and from any folds or creases in the roll or sheet.

In the case of an edge-trimmed roll or sheet without folds or creases, the specimens may be taken from the edge of roll or sheet (see Figure 3).

Typical cutting plans are shown in Figures 1, 2 and 3.

NOTE In practice, the locations from which the specimens are taken are usually specified by the material specification or agreed between the interested parties.

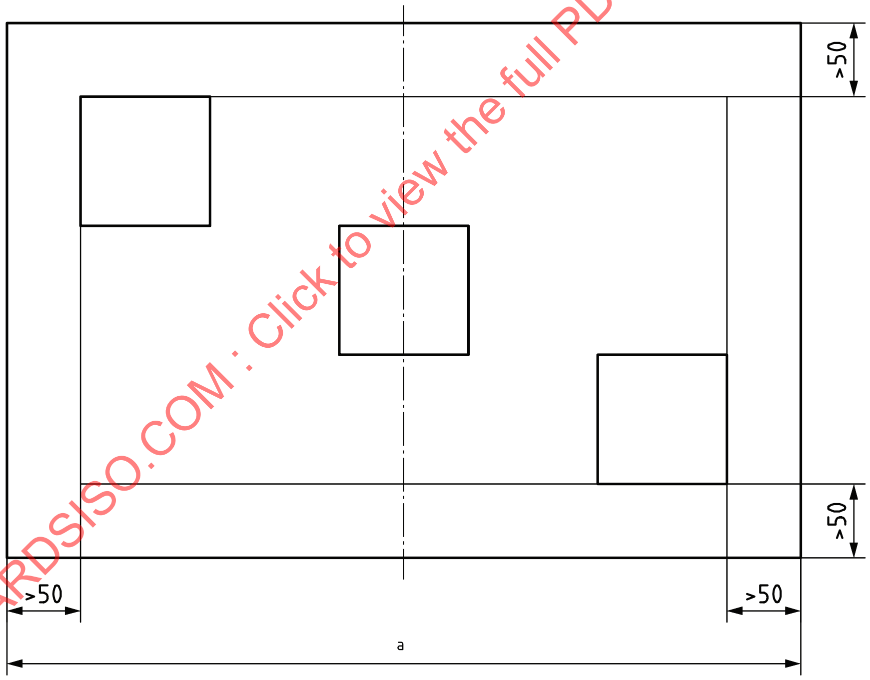
Dimensions in millimetres



a Width of material.

Figure 1 — Example of locations of specimens across the width of the material

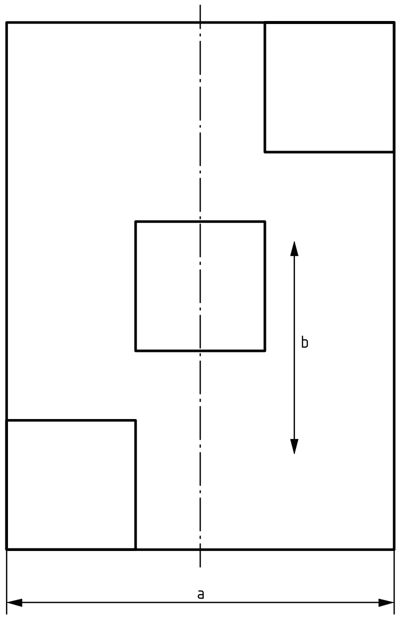
Dimensions in millimetres



a Width of material.

Figure 2 — Example of locations of specimens along an axis inclined at an angle across the material

Dimensions in millimetres



[a] Width of material.

[b] Fibre direction.

Figure 3 — Example of diagonal positioning of test specimens in unidirectional sheet or continuous tape

8 Procedure

8.1 Determination of mass per unit area

8.1.1 Materials made without using a solvent

8.1.1.1 Cut the test specimens from each laboratory sample using the template (5.1.2) and a suitable cutting tool (5.1.3).

8.1.1.2 To ensure consistency in the specimen dimensions, it is important that precise instructions concerning the cutting operation be given to the operator.

8.1.1.3 After removing the protective film(s), weigh each specimen to the nearest milligram (m_0) for type Ia or IIa materials.

8.1.2 Materials made using a solvent

8.1.2.1 On completion of conditioning, cut the test specimens from each laboratory sample using the template (5.1.2) and a cutting tool (5.1.3).

8.1.2.2 To ensure consistency in the specimen dimensions, it is important that precise instructions concerning the cutting operation be given to the operator.

8.1.2.3 Weigh each test specimen with its protective film(s) to the nearest milligram (m_1) for type Ib or type IIb materials.

8.1.2.4 Remove, then weigh, the protective film(s) and record the mass (m_2) to the nearest 0,1 g for type Ib materials or to the nearest 1 mg for type IIb materials. Ensure that all the film is removed from a specimen before weighing the film together with any material attached to it.

8.2 Determination of fibre mass per unit area

8.2.1 Method A: Extraction in Soxhlet-type apparatus

8.2.1.1 Dry the extraction thimble (5.2.1) in the oven (5.2.2) at 105 °C, for 2 h. Cool to room temperature in the desiccator (5.1.4) and weigh. However, if there is a known drying condition, the drying operation may be carried out in that condition. Repeat this blank test until the mass is constant to 1 mg (m_3). This operation only needs to be carried out once as long as the thimble is dry and clean in each subsequent test.

8.2.1.2 Remove the protective films. Cut the specimen into pieces and place them into the thimble, taking care that no fragments are lost.

8.2.1.3 Place the thimble in the siphon tube of the extraction apparatus (5.2.3). Fit the condenser, siphon tube and flask together and add a suitable quantity of solvent (5.2.4).

8.2.1.4 Regulate the heating so that the siphoning rate is compatible with the total extraction of the resin during 1 h. Carry out the test for 1 h minimum or for another period as agreed between the user and manufacturer.

8.2.1.5 Remove the thimble, dry in the oven and then cool in the desiccator and weigh to the nearest milligram (m_4).

8.2.1.6 Repeat 8.2.1.3 to 8.2.1.5 until the difference in mass on two successive weights is less than 1 mg.

8.2.1.7 Clean and dry the thimble.

8.2.2 Method B: Extraction by immersion in solvent

8.2.2.1 Take a clean container (5.3.1). Dry it for 10 min in the oven (5.3.2) at 105 °C. Cool in a desiccator (5.1.4) and weigh to the nearest milligram (m_5).

8.2.2.2 Remove the protective films. Place the specimen in the container.

8.2.2.3 Wash the specimen in at least 50 ml of suitable solvent (5.3.3) until all the resin dissolves. A mechanical shaker should be used for accelerating the extraction. Carefully decant the solution from the container ensuring that no fibres are allowed to escape.

8.2.2.4 Repeat the operation and rinse several times with acetone (5.3.4). Allow to dry.

8.2.2.5 Dry in the oven, then allow cool in the desiccator and weigh the container with the dry residue (fibre or woven fabric) to the nearest milligram (m_6).

8.2.3 Method C: Decomposition by loss on ignition

8.2.3.1 Use heatproof gloves (5.4.5) for burn-out. The burn-out shall be carried out behind the safety screen for eye protection at laboratory exhaust fan and/or ventilation system.

8.2.3.2 Take a clean crucible (5.4.1). Dry it for 10 min in the oven (5.4.6) at 105 °C. Cool in a desiccator (5.1.4) and weigh to the nearest milligram (m_7).

8.2.3.3 Remove the protective films. Place the specimen in the crucible (5.4.6).

8.2.3.4 Place each specimen in a desiccated crucible and place into a preheated muffle furnace (5.4.3) at 500 °C, or lower, depending upon the composite system (a temperature below which the matrix system will spontaneously ignite). To avoid spontaneous ignition in the furnace, specimens should be pre-ignited under a controlled heat flame (5.4.4) before entrance into the furnace.

8.2.3.5 Heat the furnace to 565 °C, or other temperature compatible with the composite system that will burn-off the matrix and leave the reinforcement unaltered. The maximum time for burn-off should be 6 h; shorter times are dependent on the system and specimen size.

8.2.3.6 Remove the crucible from the furnace and evaluate the completeness of dissolution by examining the specimen residue. The reinforcement content should be easily separable, with no evidence of binding action between fibres. If the specimen residue does not meet this condition, the test result shall be rejected and the test repeated under a more vigorous condition (longer time, increased temperature, or both).

8.2.3.7 Allow to cool in the desiccator and weigh the crucible with the residue (fibre or woven fabric) to the nearest milligram (m_8).

8.2.4 Method D: Extraction by wet combustion

8.2.4.1 Use protective clothing and rubber gloves (5.5.6) for handling hydrogen peroxide and sulphuric acid solutions. The reactions shall be carried out behind the safety screen for eye protection.

8.2.4.2 Remove the protective films and weigh the specimen to the nearest milligram (m_9), place it into the Erlenmeyer (5.5.1) and carefully pour 20 ml of concentrated sulphuric acid (5.5.7) into the flask. Fit the funnel to one neck and the water pump to the other.

8.2.4.3 Pour a suitable amount (e.g. 20 ml of hydrogen peroxide at 500 g/l or 40 ml of hydrogen peroxide at 300 g/l) of the hydrogen peroxide solution (5.5.8) into the dropping funnel and heat (5.5.2) the flask gently to (160 ± 10) °C until the acid starts to fume. Discontinue heating of the flask and allow the hydrogen peroxide solution to drip into the acid at the rate of about one drop every 2 s, increasing to one drop per second after 5 min. If, after all the hydrogen peroxide solution has been used, the solution is still brown, add further 5 ml portions of hydrogen peroxide solution at the latter rate and heat until the solution becomes clear.

8.2.4.4 Stop heating. When the neck of the Erlenmeyer is cool enough to handle, ensure the air inlet is open, turn the water pump off, remove the attachments from the Erlenmeyer and cool the Erlenmeyer and contents down to ambient temperature in running water.

8.2.4.5 Pour the contents of the Erlenmeyer into a 400 ml beaker (5.5.3) containing 100 ml of distilled water, wash with distilled water any fibres adhering to the Erlenmeyer into this beaker.

8.2.4.6 Filter the contents of the beaker through the sintered glass crucible (5.5.4), washing the beaker and contents of the crucible with distilled water (5.5.10) until the washings are free from acid, followed by 10 ml of acetone (5.5.9).

8.2.4.7 Dry the crucible and contents in the oven (5.5.5), allow to cool in a desiccator (5.1.4) and weigh to the nearest milligram (m_{10}).

8.2.5 Method E: Method by calculation

8.2.5.1 Obtain from the linear density (*Tt*) determined in accordance with ISO 1889, as by certificates.

8.2.5.2 Obtain from the number of yarns per unit (N) determined in accordance with ISO 4602, as by certificates.

N_{UD} is the number of 0° yarns in preimpregnated unidirectional sheet, in a given distance/(10 mm or 25 mm).

N_{WARP} is the number of warp yarns, in a given distance in preimpregnated fabric/(10 mm or 25 mm).

N_{WEFT} is the number of weft yarns, in a given distance in preimpregnated fabric/(10 mm or 25 mm).

9 Expression of results

9.1 Mass per unit area

9.1.1 Materials made without using a solvent

The mass per unit area, ρ_A, expressed in grams per square metre, is given by Formula (1):

$$\rho_A = \frac{m_0}{A} \qquad (1)$$

where

m_0 is the mass, in grams, of the test specimen without its protective film(s);

A is the surface area, in square metres, of the test specimen (see 7.1).

Report as the test result the arithmetic mean of the values of ρ_A obtained for all the test specimens cut from that particular laboratory sample (i.e. originating from a particular elementary unit).

9.1.2 Materials made using a solvent

The mass per unit area, ρ_A, expressed in grams per square metre, is given by Formula (2):

$$\rho_A = \frac{m_1 - m_2}{A} \qquad (2)$$

where

m_1 is the mass, in grams, of the test specimen with its protective films;

m_2 is the mass, in grams, of the protective films;

A is the surface area, in square metres, of the test specimen (see 7.1).

Report as the test result the arithmetic mean of the values of ρ_A obtained for all the test specimens cut from that particular laboratory sample (i.e. originating from a particular elementary unit).