
**Plastics — Glass-fibre-reinforced
products — Determination of fibre
length**

*Plastiques — Produits renforcés de fibres de verre — Détermination
de la longueur des fibres*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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 document 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 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

This second edition cancels and replaces the first edition (ISO 22314:2006), which has been technically revised.

The main changes are as follows:

- the requirements of muffle furnace and ultrasonic device have been modified;
- the description of the calcination time has been added;
- the description of the number of fibre measurements has been added;
- the meaning of L_p / L_n has 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.

Introduction

There is global interest in the determination of the length of glass fibres in all types of composite, to predict their characteristics (essentially mechanical ones). For these determinations, three steps are necessary:

- separation of the fibres from the composite;
- dispersion of the fibres to obtain individual fibres;
- measurement of their length.

After considering all the existing methods for separating the fibres from the resin, it was decided to develop the proposed method only for short glass fibres from thermoplastic resins, extracted by calcination.

The principles of the method are probably suitable for other composites, but it would need more development to obtain a method suitable for other conditions (thermoset resins, long fibres in thermoplastic or thermoset resins, carbon fibres).

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1 Scope

This document specifies a method of determining the length of the fibres present in a fibre-reinforced product. The method is applicable to moulding materials and to moulded parts. The test conditions specified limit the application of this method to thermoplastics reinforced with short glass fibres (less than 1 mm long), i.e. fibres whose length is less than or equal to 7,5 mm prior to incorporation in the moulding material and moulding.

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 472, *Plastics — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 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/>

4 Principle

The fibres are separated from the reinforced polymer and their length measured from a magnified image on a screen.

5 Apparatus

5.1 Muffle furnace, located under a ventilated hood and capable of maintaining a temperature of $625\text{ °C} \pm 20\text{ °C}$.

5.2 Crystallizing dish, diameter 80 mm to 110 mm.

5.3 Ultrasonic device, that enables the effective dispersion of fibres under ultrasonic vibration.

NOTE A basic ultrasonic device, like those used in laboratories for cleaning purposes, has been found suitable.

5.4 Microscope or stereoscope (with fixed or variable magnification factors), equipped with a video camera, giving at least $\times 50$ magnification.

5.5 Image acquisition device, that enables the image to be displayed on a TV monitor.

5.6 Reference micrometer, to calibrate the images at the selected magnification.

5.7 Data-processing equipment, with suitable image analysis software.

5.8 Drying oven, capable of maintaining a temperature of $130\text{ °C} \pm 5\text{ °C}$.

5.9 Laboratory and observation accessories (crucibles, evaporation dishes, spatulas, glass microscope slides).

6 Procedure

6.1 Preparation of the test specimen

6.1.1 From the sample to be examined, take the quantity of material necessary to obtain a concentration of fibres such that each image displayed on the screen contains a hundred or so fibres. This quantity will depend on the fibre content of the material and on the test conditions, in particular on the diameter of the crystallizing dish (the water depth in the crystallizing dish does not have any influence on the concentration of the fibres deposited on the glass slide, as the fibres precipitate in a horizontal plane).

EXAMPLE For a 30 % glass-fibre-reinforced polyamide, around 0,006 g of material are required when a 90 mm diameter crystallizing dish is used.

NOTE This assumes that the fibre length is less than 1 mm.

6.1.2 Calcine the material at 625 °C , in accordance with ISO 1172, for 1 h 30 min and allow to cool. If the material is not sufficiently calcined within 1 h 30 min, the calcination time can be extended appropriately. The ash obtained is mainly made up of glass fibres and possibly also mineral fillers. The fibres are very fragile and all subsequent handling shall be conducted with extreme caution in order not to break them and therefore invalidate the results.

6.1.3 Place a previously degreased glass microscope slide in a crystallizing dish (5.2) and pour in a quantity of demineralized water (containing a small amount of surfactant) just sufficient to cover the slide. The quantity of water is limited in order to avoid the formation of convection currents during subsequent evaporation, which lead to a selection of the fibres on the basis of their mass and therefore of their size.

6.1.4 Pour the ash into the crystallizing dish prepared as described above (or transfer a sufficient quantity on the tip of a spatula).

6.1.5 Place the crystallizing dish containing the ash in the ultrasonic device in order to disperse the fibres without any mechanical action. The time required for this dispersion is in the region of a few seconds to 1 min.

6.1.6 Place the crystallizing dish in an oven (5.8) preheated to 130 °C and leave it there for approximately 1 h, in order to eliminate the water. Allow to cool down. This procedure may be omitted if heating tends to cause the fibres to stick together.

6.1.7 Place the crystallizing dish beside the microscope or stereoscope (5.4). Take the slide covered with fibres and place it under the microscope or stereoscope lens. If necessary, wipe off any fibres present on the underside of the slide.

6.2 Calibration

6.2.1 Calibrate the system using a reference gauge; a 1 mm or 1,5 mm length micrometric glass slide has been found suitable.

6.2.2 Calibration can be carried out by measuring the length of the reference gauge, pointing to the two ends as would be done when measuring the length of the fibres, and comparing the reading with the gauge length. The recommended tolerance is 0,01 mm.

6.2.3 Calibrate the microscope as frequently as necessary.

6.3 Examination and measurements

6.3.1 The fibres are examined directly, without microscope cover glass or mounting fluid, in reflected or transmitted light, in the light-field or dark-field (annular illumination) mode.

6.3.2 Adjust the overall magnification chain (optics and projection) in order to obtain on the screen an image magnified between 50 and 100 times. The magnification shall be such that the whole lengths of 100 ± 20 fibres appear on the screen.

6.3.3 Measure manually the lengths of all the complete fibres appearing on the screen by clicking with the mouse on the ends of each fibre. Measure 100 ± 20 fibres in this way from each of three images, for a total of 300 ± 60 fibres.

6.3.4 This manual measurement method can be replaced by a semi-automatic method. The number of fibres measured by the semi-automatic method is recommended to be no less than the upper limit of the number of fibres measured by the manual method, i.e. greater than or equal to 360 fibres. However, it shall be realized that such methods are biased towards the smaller fibres and that the results obtained will therefore be systematically lower than those obtained by manual measurement.

NOTE While outside the scope of this method, if longer fibres up to 5 mm in length are measured, the magnification can be reduced to as little as 15, provided the quality of the optical system is good enough to permit satisfactory measurement.

6.3.5 If there is a significant difference between the measurements of the three images, the number of fibre measurements shall be increased.

6.4 Calculation and expression of results

6.4.1 The mean fibre length, L_n , expressed in micrometres, shall be calculated using [Formula \(1\)](#):

$$L_n = \frac{\sum_{i=1}^{i=n} L_i}{n} \quad (1)$$

where

L_i is the length of the i th fibre, in micrometres;

n is the number of fibres measured.

6.4.2 The standard deviation of the individual values, σ .