

---

---

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

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

STANDARDSISO.COM : Click to view the full PDF of ISO 22314:2006



**PDF disclaimer**

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

STANDARDSISO.COM : Click to view the full PDF of ISO 22314:2006

© ISO 2006

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

Published in Switzerland

## 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 22314 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

STANDARDSISO.COM : Click to view the full PDF of ISO 22314:2006

## 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).

STANDARDSISO.COM : Click to view the full PDF of ISO 22314:2006

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

## 1 Scope

This International Standard 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 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 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*

## 3 Terms and definitions

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

## 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**, capable of maintaining a temperature of  $625\text{ °C} \pm 25\text{ °C}$ .

**5.2 Crystallizing dish**, diameter 80 mm to 110 mm.

**5.3 Ultrasonic device**.

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

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.

Calcine the material at  $625\text{ °C}$  in accordance with ISO 1172 for 1 h 30 min and allow to cool.

**NOTE** The ash obtained is mainly made up of glass fibres and possibly also mineral fillers. The fibres are very fragile and all subsequent handling must be conducted with extreme caution in order not to break them and therefore invalidate the results.

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.

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

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.

Then place the crystallizing dish in an oven (5.8) preheated to  $130\text{ °C}$  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.

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

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

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 lengths the fibres, and comparing the reading with the gauge length. The recommended tolerance is 0,01 mm.

Calibrate the microscope as frequently as necessary.

### 6.3 Examination and measurements

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.

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  $100 \pm 20$  fibres appear on the screen.

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  $100 \pm 20$  fibres in this way from each of three images, for a total of  $300 \pm 60$  fibres.

NOTE 1 This manual measurement method can be replaced by a semi-automatic method. However, it must 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 2 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.

### 6.4 Expression of results

The results to be reported are:

- $L_n$ , the mean fibre length, expressed in micrometres, calculated from the following equation:

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

where

$L_i$  is the length of the  $i$ th fibre,

$n$  is the number of fibres measured;

- $\sigma$ , the standard deviation of the individual values;
- a histogram plotted for individual ranges over the entire measurement range (e.g. 50 ranges of  $40 \mu\text{m}$  each over a measurement range extending from  $0 \mu\text{m}$  to  $2\,000 \mu\text{m}$ );
- $L_p$ , a weighted mean length, expressed in micrometres, calculated from the following equation:

$$L_p = \frac{\sum_i n_i L_i^2}{\sum_i n_i L_i} \text{ where } n_i \text{ is the number of fibres of length } L_i;$$

- the ratio  $L_p/L_n$ .

## 7 Test report

The test report shall contain the following information:

- a) a reference to this International Standard, i.e. ISO 22314:2006;
- b) all details necessary for identification of the sample;
- c) the quantity of material sampled, the diameter of the crystallizing dish and the quantity of water added;
- d) the magnification used;
- e) the results obtained as described in Clause 6, as well as the minimum and maximum lengths measured;
- f) the date of the measurement;
- g) all procedural details not specified in this method.

STANDARDSISO.COM : Click to view the full PDF of ISO 22314:2006