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**Fibre-reinforced composites —  
Method for accelerated moisture  
absorption and supersaturated  
conditioning by moisture using sealed  
pressure vessel**

*Composites renforcés de fibres — Méthode pour l'absorption  
accélérée de l'humidité et le conditionnement sursaturé en humidité à  
l'aide d'un récipient sous pression scellé*

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Published in Switzerland

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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.

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](http://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](http://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](http://www.iso.org/iso/foreword.html).

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

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](http://www.iso.org/members.html).

## Introduction

Mechanical properties of fibre reinforced thermoset or thermoplastics with saturated moisture content under used conditions need to be determined for the specification of these materials. However, the moisture content of the fibre reinforced thermoset or thermoplastic materials immediately after production are usually lower than that during use, because the production process is under a higher temperature and uses dried ingredients for optimum quality control. The fibre reinforced materials absorb moisture under the used conditions and reach a saturated moisture content after a long period, for example after 6 months. Accordingly, the accelerated moisture absorption method (70 °C, 75 % RH) is specified in ASTM D5229/D5229M for determining the properties at saturation. However, the absorption time is still long (over three months) using this method.

This document specifies a method for obtaining practical saturated moisture absorption for effective and shorter research and development process of thermoset and thermoplastic materials. The method uses a sealed pressure vessel at a higher temperature with saturated water vapour condition (120 °C, 0,2 MPa of water vapour pressure).

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# Fibre-reinforced composites — Method for accelerated moisture absorption and supersaturated conditioning by moisture using sealed pressure vessel

## 1 Scope

This document specifies accelerated moisture absorption properties and supersaturated conditioning by moisture for fibre-reinforced plastics) using sealed pressure vessel at higher temperature than 100 °C with saturated water vapour at used heating temperature.

The purpose of this moisture absorbing procedure is to screen test specimens with moisture by mechanical or thermal properties.

This document applies to carbon-fibre materials and their products with either thermoset or thermoplastic matrices, with a  $T_g$  greater than 150 °C. This document can also apply to materials reinforced with other fibres (e.g. glass-fibres) with a  $T_g$  greater than 150 °C.

## 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*

## 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

#### **additional moisture content**

mass percentage of additionally absorbing moisture in the test material to total mass of the material and moisture already existing in this material

Note 1 to entry: See [Formula \(1\)](#).

### 3.2

#### **initial moisture content**

mass percentage of absorbed moisture in the test material at the initial time

Note 1 to entry: See [Formula \(3\)](#).

### 3.3

#### **supersaturated conditioning by moisture**

moisture absorbing conditions in the test material at room temperature cooled from the saturated moisture absorbing condition at higher temperature and higher humidity, such as saturated water vapour pressure at used higher temperature

### 3.4

#### **accelerated moisture absorption**

moisture absorption by process with specific conditions such as higher temperature (e.g. 120 °C) and higher pressure of moisture (0,2 MPa) to obtain *saturated moisture content* (3.5) during shorter time

### 3.5

#### **saturated moisture content**

moisture content from absorption condition with supersaturated moisture at lower temperature (e.g. room temperature) from higher temperature (e.g. 120 °C) where specimen has saturated moisture content

## 4 Principle

To obtain almost similar saturated moisture condition in a test material at room temperature for shorter time, the test material absorbs moisture at higher temperature and higher humidity conditions such as 120 °C and 0,2 MPa of saturated water vapour pressure without air (oxygen) gas.

Higher temperature causes higher diffusion rate of water molecules in materials and higher water vapour pressure causes higher frequencies of water molecules uptake at the surface of the material from outside. Therefore, equilibrium and saturated moisture condition to the materials is obtained during shorter time. However, if this material with saturated moisture is cooled to room temperature, this material absorbs supersaturated moisture because of slow diffusion rate inside of water molecules and slow releasing rate of water molecules at the surface.

In this way, supersaturated moisture condition for thermoset and thermoplastic matrices is obtained. It is important that this procedure does not cause degradation of the test material. Mechanical properties should be monitored to be stable during repeating these absorbing and desorbing (drying) processes.

In addition, this condition is simulated condition for real condition to obtain saturated equilibrium moisture content at room temperature. The purpose of this test method is to screen the optimal materials primarily during the research and development processes.

## 5 Apparatus

**5.1 Oven**, capable of heating to 200 °C for test specimens or sealed pressure vessels connected to pressure gauge and purge lines positioned outside the oven with controlling temperature system, ( $\pm 1$  °C).

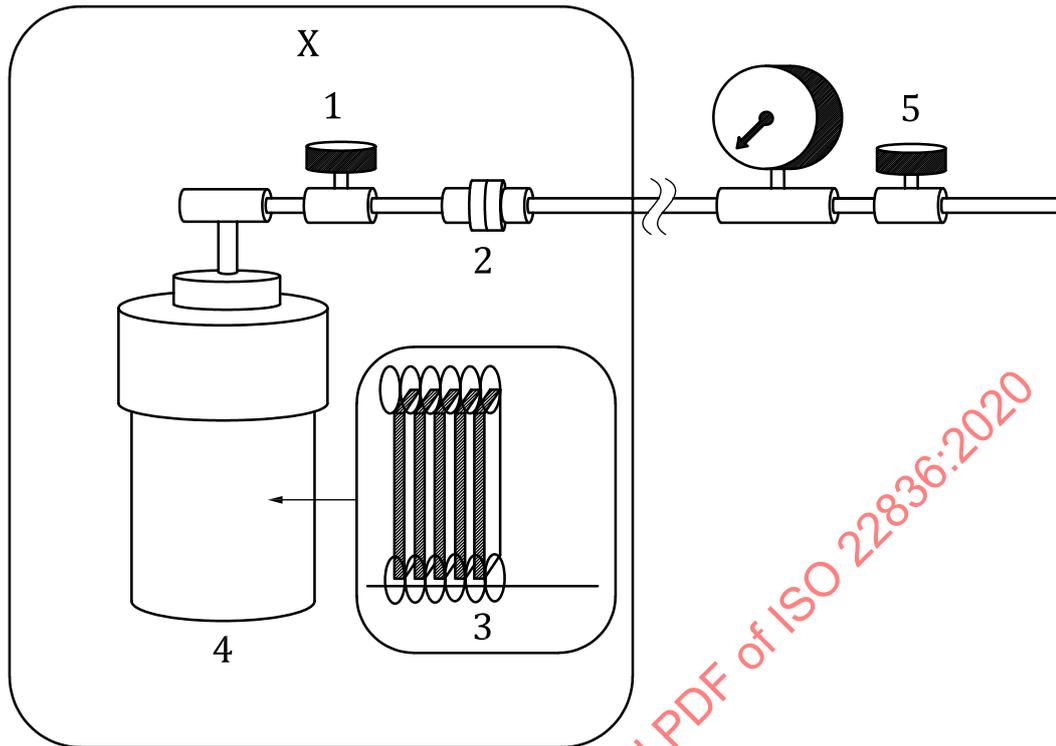
To control the temperature uniformly inside oven, air circulation heating system should be used.

If the conditions at 120 °C and 0,2 MPa of saturated water vapour pressure is used, a sterilizer (5.5) can be used instead of oven and sealed pressure vessels.

**5.2 Sealed pressure vessel**, capable of pressurization to 2,0 MPa) and connected to pressure gauge and purge line as indicated in [Figure 1](#).

The size of vessels is dependent on the size and numbers of specimens to be tested. For 10 specimens sized 10,0 mm × 100,0 mm × 2,0 mm, a 300 ml stainless steel vessel (autoclave) is needed.

A supporting frame should be used to hold the specimens fully immersed, while avoiding specimen surfaces contacting each other and minimising contact with the frame.

**Key**

- X oven
- 1 valve A
- 2 connector
- 3 water
- 4 sealed pressure vessel
- 5 valve

**Figure 1 — Supersaturated moisture condition obtaining system for CFRP or CFRTTP materials**

**5.3 Pressure gauge** (e.g. 0 to 1,0 MPa), to indicate the pressure in the vessel with resolution of 0,05 MPa.

**5.4 Relief valve or bursting disc**, operating at 3,5 MPa and installed in the line or pressure gauge to prevent higher pressures.

**5.5 Sterilizer**, capable of heating at 105 °C to 135 °C with saturated water vapour pressure.

An air releasing system shall be installed at 100 °C during heating from room temperature to 120 °C.

**NOTE** Sterilizer is referred to as an “autoclave” in the biological and medical industrial fields. This autoclave is a different apparatus with a stainless-steel sealed pressure vessel (5.2) in the chemical industrial field

**5.6 Vacuum oven**, capable of heating to 200 °C under reduced pressure with temperature controlling to  $\pm 1$  °C.

**5.7 Balance**, capable of weighing the specimen with a resolution of at least 0,1 mg.

## 6 Test procedure

### 6.1 Weighing test specimens

Weigh the individual specimen using a balance (5.7). The specimen should be conditioned before weighing at 23 °C and 50 % RH for 1 day unless specified or agreed elsewhere. The specimen shall not be dried before moisture absorbing to avoid any degradation.

### 6.2 Obtaining supersaturated moisture absorption

Place the specimens inside a sealed pressure vessel using an inner specimen support frame so that specimen surfaces do not come into contact with each other or the container, and are fully immersed in the water.

Pour deionized water (10 ml to 20 ml in the case of 300 mL vessel) into the vessel.

Seal and place the pressure vessel inside the oven as indicated in [Figure 1](#). Open Valves A and B.

Set the oven temperature over 100 °C such as 120 °C. At around 100 °C, release steam (water vapour) at Valve B for a short time (for example, 1 min to 2 min) to release inside air. Then close valve B. Inside water vapour pressure is confirmed by a pressure gauge at determined temperature.

Continue heating for a determined period, such as 72 h.

### 6.3 Sampling of test materials with supersaturated moisture content

If only one vessel exists inside the oven, stop the heating and cool and vessel down to room temperature. Remove the specimens from the vessel.

If one of several vessels is removed from the oven, close valve A and disconnect the connector. Take the vessel out from the oven and cool it down to room temperature. Open valve A. Remove the specimens from this vessel.

A sterilizer [autoclave (5.5)] should be used instead of a sealed pressure vessel and oven for these methods. In this case, the procedure shall be followed by the operation manual of a sterilizer.

### 6.4 Confirming the saturated moisture absorbing

Weigh the absorbed specimens. Then repeat the procedures given in 6.2 (for an additional determined period such as 24 h) and 6.3. Weigh the repeated absorbed specimens.

Calculate additional moisture content,  $W_{ad}$  as indicated in [Formula \(1\)](#) (see [Annex A](#)). The mean value shall be used for at least duplicated test specimens.

$$W_{ad} = \frac{M_n - M_i}{M_i} \times 100 \quad (1)$$

where

$W_{ad}$  is the additional moisture content (%) of the test specimen under the saturated condition;

$M_n$  is the mass (g) of the test specimen under the saturated condition by confirming [Formula \(2\)](#);

$M_i$  is the mass (g) of the test specimen at the initial time of this absorbing process.

Confirm saturated moisture content for the above conditions as indicated in [Formula \(2\)](#).

$$|W_{n+1} - W_n| < 0,05 \% \quad (2)$$

where

$W_{n+1}$  is the additional moisture content (%) of the test specimen at the [n+1]-th number's measuring point;

$W_n$  is the additional moisture content (%) of the test specimen at the [n]-th number's measuring point.

Plot sample mass vs. time to confirm that saturated conditions have been reached (i.e. the mass reaches a maximum value that does not increase with further conditioning time – See [Figure A.1](#)). Determine the moisture content.

Weigh at least three absorbed specimens. These specimens are dried above 100 °C under reduced pressure. Constant dried weights shall be obtained. Moisture contents are calculated as indicated in [Formula \(3\)](#). The mean value shall be used for at least duplicated test specimens.

$$W_s = \frac{M_n - M_d}{M_d} \times 100 \quad (3)$$

where

$W_s$  is the moisture content (%) of the test specimen under the saturated condition;

$M_n$  is the mass (g) of the test specimen under the saturated condition by confirming [Formula \(2\)](#);

$M_d$  is the mass (g) of the test specimen after the drying process (e.g. 100 °C, under reduced pressure for 3 days).

## 6.5 Measuring mechanical or thermal properties

To confirm no degradation, additional curing or crystallization of test specimens, the mechanical and thermal properties shall be measured for specimens dried by reduced pressure to obtain constant weight before (giving the initial moisture content) and after the moisture absorbing procedure specified in this document.

Required mechanical or thermal properties of specimens with supersaturated moisture condition are measured generally at room temperature or controlling temperature.

## 7 Precision

The precision of this test method is not known because interlaboratory data are not available at the time of publication.

## 8 Test reports

The test report shall include at least the following information:

- a) a reference to this document, i.e. ISO 22836:2020;
- b) all information necessary for complete identification of the test specimens;
- c) absorbing conditions such as heating temperature, water vapour pressure and heating time;
- d) drying conditions such as heating temperature, pressure and drying time;
- e) masses of test specimens at initial, saturated and dried for absorbing procedure;
- f) additional moisture content and moisture content of test specimens after moisture absorbing process;
- g) storage period from this moisture absorbing to any properties measurement;
- h) date of application of this absorption procedure.

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## Annex A (informative)

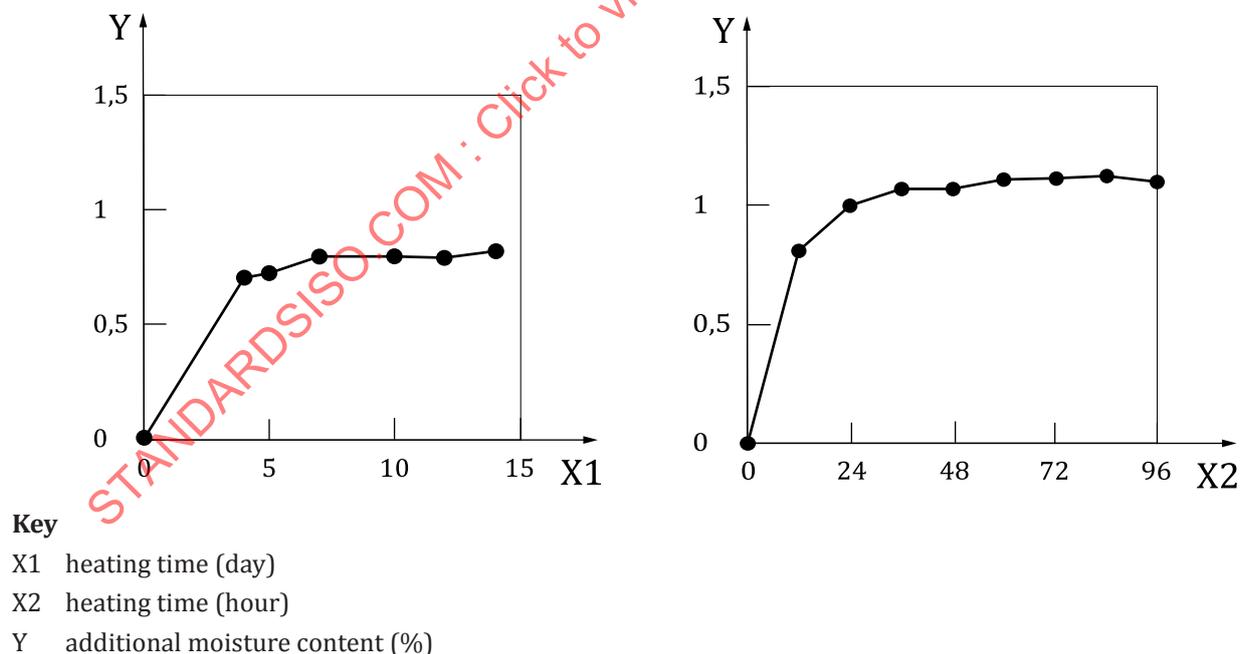
### Examples of CFRP and CFRTP specimens with supersaturated moisture condition

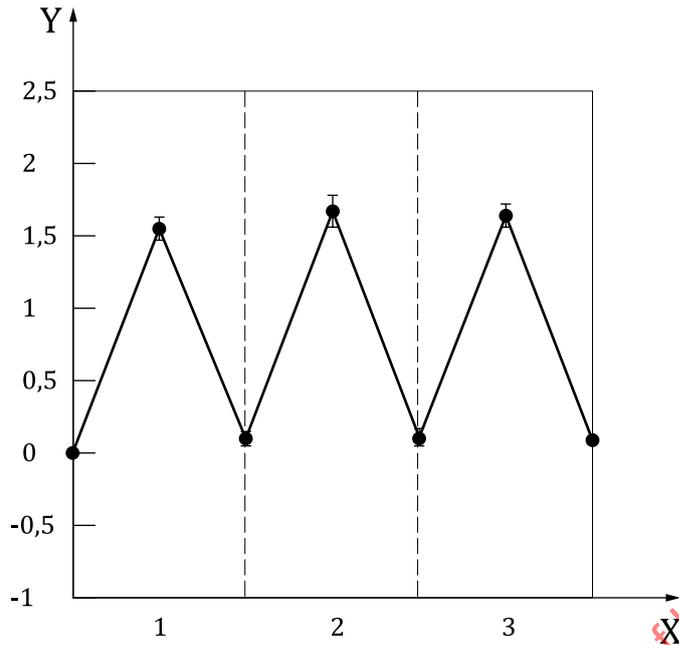
#### A.1 CFRP specimens

CFRP specimens, composed of epoxy resin and 8 layers of unidirectional 2- $\mu\text{m}$ -diameter CF oriented at +45, 90, -45, 0, 0, -45, 90 and +45° (fibre content of 72 mass % and thickness: 1,5 mm) were prepared under 180 °C, 0,7 MPa over 2 h. CFRP specimens for bending strength measurement were cut to 15 mm  $\times$  100 mm size.

#### A.2 Moisture content and bending strength of CFRP specimens with supersaturated moisture condition

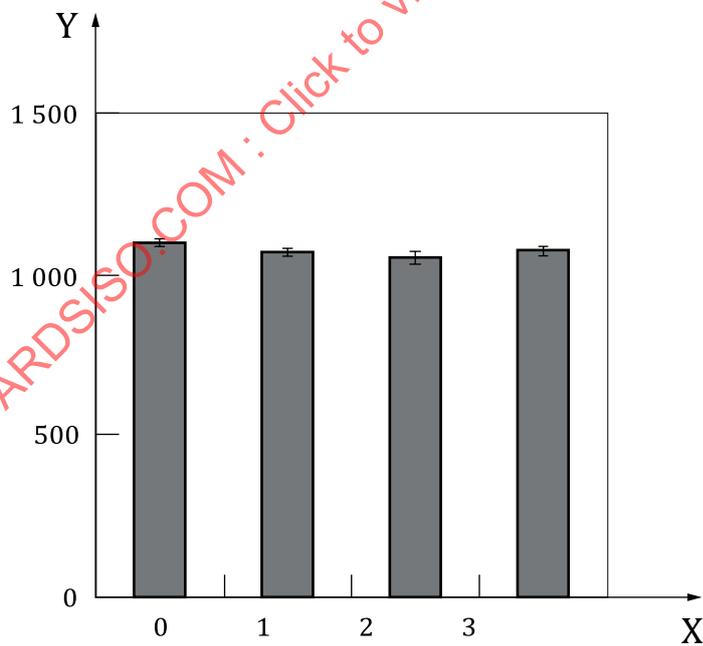
[Figure A.1](#) shows that the moisture content of the specimens depends on the heating time in the autoclave at 105 °C and 120 °C with saturated moisture condition. Specimens were taken at different continuous heating times with different specimens used for wet mass measurements in order to avoid the heat cycles between high and room temperature at mass measuring. It was confirmed that the moisture saturation (0,80 % and 1,14 %) was reached after 14 d and 60 h at 105 °C and 120 °C as indicated in [Table A.1](#).





**Key**  
X number of cycles  
Y moisture content (%)

**Figure A.2 — Moisture content of dry CFRP specimens during repetitive moisture absorption at 120 °C for 72 h and drying at 100 °C for 72 h under a reduced pressure**



**Key**  
X number of cycle(s)  
Y bending strength (MPa)

**Figure A.3 — Bending strength of CFRP specimens during repetitive moisture absorption at 120 °C and drying**