

JIS

JAPANESE INDUSTRIAL STANDARD

**Testing Methods for
Carbon Fibers**

JIS R 7601 —1986

Translated and Published

by

Japanese Standards Association

Printed in Japan



Documents

Document Sharing Hub

21 S

Translation without guarantee
In the event of any doubt arising, the original
standard in Japanese is to be evidence

JAPANESE INDUSTRIAL STANDARD

J I S

Testing Methods for Carbon Fibers

R 7601-1986

1. Scope

This Japanese Industrial Standard specifies the testing methods for carbon fibers⁽¹⁾.

Note (1) The term "carbon fiber", used in the standard shall be fibrous carbon material essentially composed of carbon element only. For instance, such as material obtained by baking organic fibers.

Remark: In this standard the units in { } are those derived from the conventional unit system and are the standard value.

2. Definition of Terms

The definitions of the main terms used in this standard shall be as follows with exception of those listed in JIS L 0204, JIS L 0205, and JIS L 0208.

- (1) filament yarn Thread composed of plurality of continuous filaments. It is also called strand.
- (2) tow An untwisted bundle composed of plurality of filaments.
- (3) fineness Mass of carbon fiber per 1000 m.
- (4) twist Number of twists per unit length of carbon fiber.
- (5) water content This is the ratio of water contained in carbon fiber at an arbitrary condition to an absolute dry mass⁽²⁾.

Note (2) Refer to the Absolute Dry Condition in (7).

- (6) moisture equilibrium The state in which the sample has obtained a constant mass under the standard condition after pre-drying ($40 \pm 5^\circ\text{C}$) for 1 h. This moisture is called an equilibrium moisture.
- (7) absolute dry condition The condition in which the sample or test piece has obtained a constant mass at $110 \pm 2^\circ\text{C}$. The mass at standard temperature descended from the temperature of the absolute dry condition is called an absolute dry mass.
- (8) constant weight After weighing two times or more with the interval of 10 min or more in the case of an absolute dry condition, or 1 h or more in the case of a moisture equilibrium, the condition where the mass difference between weighings before and after of the interval has fallen in the limit of 0.03 percent to the mass weighed after.

- (9) tensile strength The maximum tensile stress applied to the test piece during tension test.
- (10) tensile modulus of elasticity The gradient at straight part, within the elastic limit, of a tensile stress-strain curve.
- (11) volume resistivity The electric resistance which, assuming a cube in carbon fiber, shows when applied with an electric voltage between two surfaces positioned opposite in the longitudinal direction of fibers.
- (12) amount of sizing The ratio of the amount of sizing to the absolute dry mass of carbon fiber.

3. Testing Items

Testing items shall be as follows:

- (1) Fineness
- (2) Twist
- (3) Density and Specific Gravity (Liquid displacement method and density gradient tube method)
- (4) Water Content
- (5) pH
- (6) Tensile Strength and Tensile Modulus of Elasticity (Single fiber and resin impregnated strand)
- (7) Volume Resistivity (Single fiber and strand)
- (8) Amount of Sizing (Extracting method by 2-butanone, washing method by sulfuric acid, and thermal-decomposition method)

4. Standard Condition of Testing Place

Principally, the standard condition of testing place shall be as specified in JIS Z 8703 such as; the temperature at standard condition is 23°C and classified as grade 5 of temperature (+5°C), and the humidity of standard condition is 50% and classified as Grade 20 of humidity (+20%).

5. Expression of Numerical Values of Test Results

5.1 Rounding Off of Numerical Values The test result shall be calculated to one more place than that of specified numerical value, and rounded off according to JIS Z 8401.

5.2 Average Value, Standard Deviation, and Coefficient of Variation Average value, standard deviation, and coefficient of variation shall be calculated according to the following formulae of (1), (2), and (3).

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n} \dots\dots\dots (1)$$

$$s = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \dots\dots\dots (2)$$

$$CV = \frac{s}{\bar{x}} \times 100 \dots\dots\dots (3)$$

where $\bar{x}$: average value

s : standard deviation

CV : coefficient of variation (%)

x_i : individual measured value

n : number of measurements

6. Testing Method

6.1 Fineness The test of fineness shall be as follows:

(1) Apparatus Apparatuses shall be as follows:

(1.1) Balance Use the balance capable of weighing to the nearest 0.1 mg.

(1.2) Length Meter Use the length meter capable of measuring to the nearest 1 mm.

(2) Procedure Procedure shall be as follows:

(2.1) Apply a load on the sample so far as it is stretched without any elongation or strain, and cut two test pieces at least, which measures the length as shown in Table 1 to the nearest 1 mm.

Table 1. Fineness and Length of Test Piece

Fineness tex	Length of test piece (m)
Under 50	2.0
50 to 50 excl.	1.5
500 or over	1.0

(2.2) Weigh the mass of the test piece to the nearest 0.1 mg.

(2.3) Calculate the fineness according to the following formula (4):

$$t = \frac{m}{L} \times 1000 \dots\dots\dots (4)$$

where t : fineness (tex)

m : mass of the test piece (g)

L : length of the test piece (m)

(2.4) Carry out the test on two test pieces, and calculate the average to ten's place.

6.2 Twist The test for twist shall be as follows:

(1) Apparatus The apparatus shall be a twist tester.

(2) Procedure Procedure shall be as follows:

(2.1) From a bobbin, pull out sample lest its twist should be changed, and make it a test piece after discarding first about 2 m.

(2.2) Attach a test piece to a twist tester so as to make the length between clamps 500 ± 1 mm.

(2.3) Rotate one clamp until the test piece is free from any twist, and read the number of rotation.

(2.4) The twist per 1 m of the sample is obtained by multiplying the rotation number twice.

(2.5) Carry out the test on two test pieces, and calculate the average to unit's place.

Remark: In case of 10 turns or less per 1 m, it is preferable to test with a twist tester of which length between two clamps is let be longer.

6.3 Density and Specific Gravity For measuring density and specific gravity, there are two methods as a liquid displacement method and a density gradient tube method, and either one may be used, but the method adopted shall be appended.

6.3.1 Measuring Condition Carry out the measuring of density and specific gravity under the standard at 23°C.

6.3.2 Measuring by Liquid Displacement Method The measuring by liquid displacement method shall be as follows:

(1) Apparatus and Tool Apparatuses and tools shall be as follows:

(1.1) Balance Use the balance capable of weighing to the nearest 0.1 mg.

- (1.2) Beaker
- (1.3) Base Dish or Fixed Supporter
- (1.4) Specific Gravity Hydrometer Use the hydrometer specified in JIS B 7525.
- (2) Immersion Liquid Immersion liquid shall be organic solvent having smaller specific gravity than that of the test piece and have high wettability, for instance ethyl alcohol⁽³⁾.
- Note⁽³⁾ Use the reagent specified in JIS K 8102.
- (3) Test Piece In order that no bubble may be attached on a test piece when being immersed, the test piece is preferably shaped for easy degasification in advance. The size of the test piece shall be suitable to the size of a beaker for immersion, and its mass shall be 0.1 to 5.0 g. Prepare two test pieces at least.
- (4) Procedure Procedure shall be as follows:
- (4.1) Tie the test piece by metal wire of 0.1 mm or less in diameter, and weigh its mass in the air of temperature at near 23°C to the nearest 0.1 mg. Previously, weigh the mass of metal wire in the air to the nearest of 0.1 mg.
- (4.2) Place the beaker filled with the immersion liquid that has been kept near 23°C on the base dish or fixed supporter so as not to let it touch a movable part of the balance.
- (4.3) Measure separately the specified gravity of the immersion liquid by using a specific gravity hydrometer.
- (4.4) Put the mark on the metal wire by which the liquid level, when the wire is immersed in liquid, can be recognized.
- (4.5) Immerse the test piece in the immersion liquid, and, after expelling bubbles completely owing to a method such as swing, weigh the mass to the nearest 0.1 mg with suspending the other end of the wire to the balance. Then, adjust the volume of the liquid to let the level of liquid surface be equal to the mark.
- (4.6) Weigh the mass of the metal wire to the nearest 0.1 mg when being immersed in the immersion liquid until the mark attached in (4.4).

- (4.7) Calculate the specific gravity according to the following formula (5):

$$d = \frac{m_a}{m_a - m_i} \times d_i \dots\dots\dots (5)$$

where d : specific gravity

m_a : mass of the test piece in air (g)

$$m_a = m_1 - m_2$$

where m_1 : mass of the test piece tied with metal wire in air (g)

m_2 : mass of the metal wire in air (g)

m_i : mass of the test piece in immersion liquid (g)

$$m_i = m_3 - m_4$$

where m_3 : mass of the test piece tied with metal wire in immersion liquid (g)

m_4 : mass of the metal wire immersed until the mark (g)

d_i : specific gravity of the immersion liquid

- (4.8) Carry out the test on two test pieces, and calculate the average to the second decimal place.

6.3.3 Measuring by Density Gradient Tube Method The measuring by density gradient tube method shall be as follows:

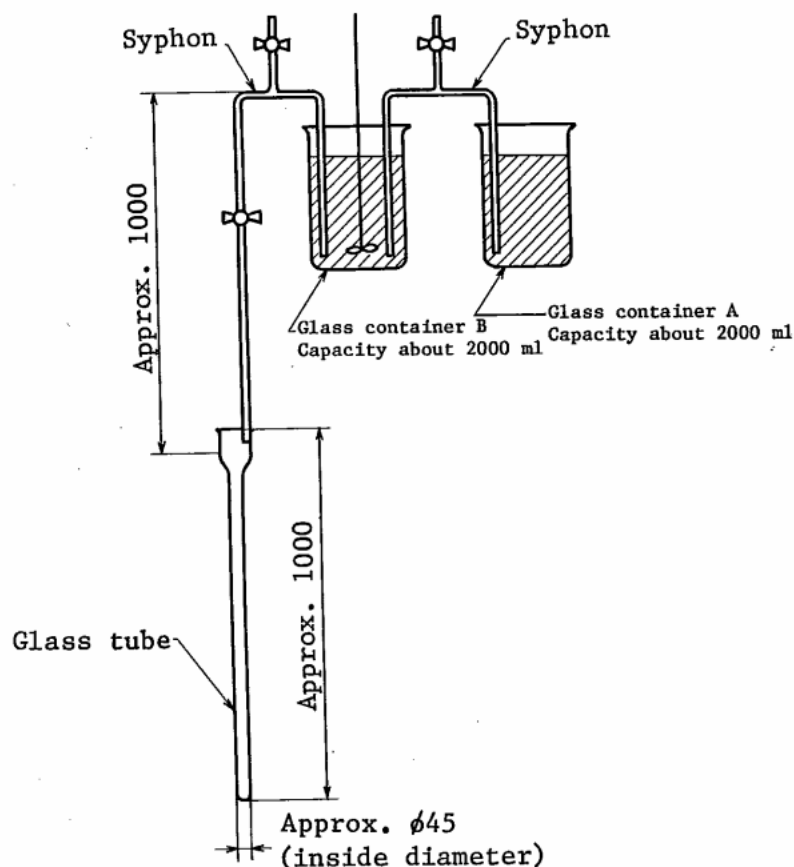
- (1) Apparatus and Tool Apparatuses and tools shall be as follows:

- (1.1) Glass Cylinder This glass tube measures about 1 m in length, about 4.5 cm in inside diameter, and is graduated by 1 mm division over 85 cm long at least and equipped with a ground cap at its top. A glass tube having other size than the above may be used if the same result is anticipated.
- (1.2) Thermostatic Water Bath This is large enough to contain the glass tube, and has a device to control the temperature at $23 \pm 0.5^\circ\text{C}$.
- (1.3) Glass Container Use two glass containers of the same diameter. These containers have preferably the capacity of about 2000 ml, respectively.

- (1.4) Standard Float Use the hollow glass bulb having 3 to 5 mm in diameter.
- (1.5) Syphon
- (1.6) Burette Use the burette capable of measuring 5 to 50 ml.
- (2) Preparation of Density Gradient Tube Either of two following methods is available, but the method adopted is appended.
- (2.1) Method to Use Syphon The method to use a syphon shall be as follows. An example of a density gradient tube is shown in Fig. 1.

Fig. 1 Density Gradient Tube

Unit: mm



- (2.1.1) In order to prepare a required density gradient tube, obtain ρ_A according to the following formula (6):

$$\rho_A = \rho_1 - \frac{2(\rho_1 - \rho_2) V_B}{V} \dots\dots\dots (6)$$

where ρ_A : initial density of the liquid in the glass container A in Fig. 1 (g/cm³)

ρ_1 : initial density of the liquid in the glass container B in Fig. 1, which should be 0.005 g/cm³ larger than the maximum density of the standard float required (g/cm³)

ρ_2 : density of the liquid at the top of a density gradient tube, which should be 0.01 g/cm³ smaller than the minimum density of the standard float required (g/cm³)

V_B : capacity of initial liquid in the glass container B in Fig. 1 (ml)

V : volume of total liquid in the density gradient tube (ml)

- (2.1.2) Put the liquid of ρ_A in a glass container A in Fig. 1 and the liquid of ρ_1 in a glass container B, and after making the level of both liquids the same, connect them by a syphon.
- (2.1.3) While agitating the glass container B by a stirrer, pour the liquid in the container by a syphon into a glass cylinder at the rate of 20 ml/min or less in the manner as letting it run along the surface of a wall.
- (2.1.4) Because the liquid level in the glass container B is lowered by this operation, the liquid in a glass container A is gradually transferred into the glass container B. Accordingly, the liquid in the glass tube shows a continuous density gradient.
- (2.1.5) The operation from (2.1.2) to (2.1.4) shall be carried out at near 23°C.
- (2.1.6) Put gently the glass tube in which the liquid has been poured completely in a thermostatic bath.

- (2.1.7) Then, after washing a standard float with methyl alcohol or the like, place gently the standard float wetted with the minimum density liquid into the cylinder, and make it as the density gradient tube. Keep the tube in a thermo-static bath at $23 \pm 0.5^\circ\text{C}$. The number of standard floats is suitable if putting one or more per 0.01 g/cm^3 of density difference, and in the case of putting in the glass cylinder, they should preferably be arranged at the interval of 10 to 20 cm at least.
- (2.1.8) After the lapse of 24 h, read the height of the center of the standard float in the tube to the nearest 1 mm by using the graduation on the tube, and prepare a correction curve by plotting the density of the standard float and the graduation on the tube.
- (2.1.9) When the correction curve shows a zigzag or serious bow shape, repeat the procedure from (2.1.1) to (2.1.3).
- (2.2) Method of Stacking Liquid The method of stacking liquid shall be as follows:
- (2.2.1) In order to prepare a required density gradient tube, make n kinds of liquids whose densities are $\rho_1, \rho_2, \dots, \rho_n$. Let the density ρ_1 become 0.005 g/cm^3 larger than the density of the maximum standard float, and ρ_n 0.01 g/cm^3 smaller than the density of the minimum standard float. Decide the number of liquids n in order that each liquid may occupy 1 to 2 cm height in the cylinder and total height becomes to the required height of the gradient tube. It is necessary to let the density of each liquid show exactly equal interval.
- (2.2.2) Pour each liquid prepared in (2.2.1) from the higher density, that is ρ_1 , into a glass tube successively by using a burette in the manner to let it run along the surface of a wall.
- (2.2.3) Carry out the procedure in (2.2.1) and (2.2.2) at a temperature near 23°C .
- (2.2.4) Carry out the procedure hereinafter similarly to (2.1.6) to (2.1.9).
- (3) Immersion Liquid Immersion liquid shall be in accordance with Table 2.

Table 2

Immersion liquid	Density limit (g/cm ³)
Zinc chloride ⁽⁴⁾ /water	1.00 to 2.00
Carbon tetrachloride ⁽⁵⁾ /ethylene bromide	1.60 to 2.18
Carbon tetrachloride ⁽⁵⁾ /bromoform	1.60 to 2.89
Ethyl alcohol ⁽³⁾ /bromoform	0.81 to 2.89

Notes ⁽⁴⁾ Use the reagent specified in JIS K 8111.

⁽⁵⁾ Use the reagent specified in JIS K 8459.

Remark: In the case of using a zinc chloride-water system, it is admissible to add methyl alcohol or ethyl alcohol by the rate of 0.1 % as a wetting agent for easily eliminating of bubbles.

For preparing of this liquid, instead of distilled water, the use of 1% hydrochloric acid is preferable. In this case, use the methyl alcohol specified in JIS K 8891, ethyl alcohol in JIS K 8102, and hydrochloric acid in JIS K 8180.

- (4) Test Piece In order that no bubble is attached on the test piece when being immersed, the test piece should be preferably shaped for easy degasification in advance.

The shape of the test piece shall be either one of following, and its weight shall be 1 to 200 mg.

- (4.1) Cut the sample to the length of 3 to 5 mm.
- (4.2) After crushing the sample, pass the sample through the standard sieve of 149 μ m specified at JIS Z 8801.
- (4.3) Make loop type the single fiber of about 30 mm long.

- (5) Procedure Procedure shall be as follows:

- (5.1) Wet the test piece with such as low density liquid, methyl alcohol, or ethyl alcohol, and then place quietly it in the density gradient tube.
- (5.2) When the test piece has stood in the liquid at equilibrium position⁽⁶⁾, read accurately the height shown at its center with the graduation on the tube to the nearest 1 mm.

Note⁽⁶⁾ Usually, 1 h or more is necessary.

- (5.3) Compare the result of (5.2) with the correcting curve, and read out the density corresponding to each measured result.
- (5.4) Carry out the test on two test pieces, and round off the average of results to the second decimal place.

Remark: If the density gradient correcting curve is seriously deviated from a linear relation, renew the density gradient tube.

6.4 Water Content The test for water content shall be as follows:

(1) Apparatus and Tools Apparatuses and tools shall be as follows:

- (1.1) Balance Use the balance capable of weighing to the nearest 0.1 mg.
- (1.2) Oven Use the hot air circulating-type oven, hereinafter referred to as the "oven", controllable at a temperature $110 \pm 2^\circ\text{C}$.

- Remarks 1. Instead, a reduced pressure oven or an infrared-ray oven is available.
2. In case of the sample with sizing whose weight may be easily changed by such as decomposition due to above-mentioned drying temperature, apply lower temperature than the above, and append the condition.

(1.3) Desiccator Use the desiccator containing desiccant, (hereinafter referred to as the "desiccator").

(2) Procedure Procedure shall be as follows:

(2.1) Measuring of Water Content

- (2.1.1) Take two test pieces weighing respectively about 10 g from the sample previously prepared by 20 g at least, and weigh each mass to the nearest 1 mg. Then, weigh the absolute dry mass to the nearest 1 mg.
- (2.1.2) Calculate the water content according to the following formula (7).

$$w = \frac{m - m_d}{m_d} \times 100 \dots\dots\dots (7)$$

where w : water content (%)

m : mass of the test piece when being taken (g)

m_d : absolute dry mass of the test piece (g)