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Textile glass - Determination of density

玻璃纤维 密度的测定

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Textile glass - Determination of density

1 Scope

This document specifies the humidity conditioning and test environment, test method and test report for determination of glass fiber density.

This document describes three test methods for determining the density of glass fibers, namely:

- Method A: Gas pycnometer method;
- Method B: Liquid pycnometer method;
- Method C: Buoyancy method.

Method A is the arbitration test method.

This document applies to the determination of glass fiber density.

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.

GB/T 6682, Water for analytical laboratory use - Specification and test methods

GB/T 9914.2, Test method for reinforcement products - Part 2: Determination of combustible-matter content for glass fibre

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 density; ρ

The mass of a substance per unit volume at a specified temperature.

NOTE: Density is measured in kilograms per cubic meter (kg/m³), kilograms per liter (kg/L) or grams per cubic centimeter (g/cm³).

- **5.1.2.2** Constant temperature water bath. The temperature of the sample chamber can be maintained at $23^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$.
- **5.1.2.3** Analytical balance. It is readable to 0.1mg. The maximum allowable error is 0.5mg. The measuring range is 200g.
- **5.1.2.4** Measuring gas. It is recommended to use helium with a purity of not less than 99.99%. Other non-corrosive and non-adsorbed gases can also be used, such as nitrogen with a purity of not less than 99.99%.
- **5.1.2.5** Suitable tools, such as scissors or knife.
- **5.1.2.6** Vacuum pump (optional).

5.1.3 Specimen

Unless otherwise agreed, the specimen shall be free of wetting agent. To remove the wetting agent, the burning method specified in GB/T 9914.2 can be used. If the interested parties agree, the test specimen containing wetting agent can also be used.

Take a specimen of appropriate mass according to the volume of the sample chamber. Cut the fibers with a suitable tool (5.1.2.5) to no higher than the height of the sample chamber.

5.1.4 Operation

- **5.1.4.1** Check the gas specific gravity meter (5.1.2.1) to ensure that there is no gas leakage in the instrument.
- **5.1.4.2** Weigh the mass (m_1) of the sample chamber with the analytical balance (5.1.2.3), accurate to 0.1mg. Put the fiber in the sample chamber. Fill to more than 60% of the sample chamber capacity. Weigh the mass (m_2) of the sample chamber and the specimen, accurate to 0.1mg.
- **5.1.4.3** Put the weighed sample chamber and specimen into the gas specific gravity meter. Seal the sample chamber. Adjust the constant temperature water bath (5.1.2.2) to keep the temperature of the sample chamber at $23^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$. Access with the gas (5.1.2.4). Multiple pulse purges to remove air from instrument sample chamber and additional chamber. The vacuum pump (5.1.2.6) can also be used to remove air from the instrument sample chamber and additional chamber.
- **5.1.4.4** Open all valves. Fill the sample chamber and additional chamber with gas at standard atmospheric pressure. The pressure sensor should now read zero.
- **5.1.4.5** Close the valve of the additional chamber. Fill the sample chamber with gas until the desired pressure is reached. 1 standard atmosphere is recommended. Close the intake valve. The pressure sensor reading is recorded as p_1 .

- **5.2.2.4** Distilled water, which shall comply with the provisions of GB/T 6682.
- **5.2.2.5** Pycnometer. The capacity is 50mL.
- **5.2.2.6** Analytical balance. It is readable to 0.1mg. The maximum allowable error is 0.5mg. The measuring range is 200g.
- **5.2.2.7** Ultrasonic cleaner with flat bath. The power is not less than 400W.
- **5.2.2.8** Electric furnace, laboratory use, commercially available.
- **5.2.2.9** Constant temperature water bath. The temperature control accuracy is ± 0.1 °C.

5.2.3 Specimens

Specimens should be free of wetting agent. To remove the wetting agent, the burning method specified in GB/T 9914.2 can be used. Weigh 35g±2g of specimen. After removing the wetting agent, use an agate mortar to grind it to a length of 1mm~3mm. Transfer it to a beaker. Use 3 times the specimen volume of absolute ethanol (5.2.2.1) to wash. Place still. Pour out the solvent. Repeat washing 2~3 times. Dry in the blast drying oven (5.2.2.2) at a temperature of 105°C±2°C for not less than 1h. Cool to room temperature in the desiccator (5.2.2.3).

5.2.4 Operation

- **5.2.4.1** Use distilled water (5.2.2.4) to rinse the pycnometer (5.2.2.5). Dry to constant weight together with the stopper at 105°C±2°C. Cool to room temperature in the desiccator. Use the analytical balance (5.2.2.6) to weigh its mass (m), accurate to 0.1mg.
- **5.2.4.2** Put the $8g\sim10g$ of specimen in the pycnometer through the funnel. Wipe off the powder stuck to the outside and mouth of the pycnometer. Weigh the mass (m_1) of the pycnometer and the specimen, accurate to 0.1mg.
- **5.2.4.3** Fill 3/4 pycnometer with distilled water. Eliminate air bubbles by cleaning with the ultrasonic cleaner (5.2.2.7) or heating with the electric furnace (5.2.2.8). After removing the bubbles, place the pycnometer in the constant temperature water bath (5.2.2.9) at $23^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$ for at least 1h. Fill with $23^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$ distilled water. Use a stopper to plug. Set the volume to constant. Dry and weigh its mass (m₂), accurate to 0.1mg.
- **5.2.4.4** Remove the specimen from the pycnometer. Filled with distilled water after debubbling. Place in a constant temperature water bath at 23°C±0.1°C for at least 1h. Take out. Use 23°C±0.1°C distilled water to set the volume tot constant. Dry and weigh its mass (m₃), accurate to 0.1mg.
- **5.2.4.5** The density of the specimen is calculated according to formula (2):

- 0.5mg. The measuring range is 200g.
- **5.3.2.8** Thermometer, with an accuracy of 0.1°C, capable of measuring the temperature of water.
- **5.3.2.9** Beaker. Appropriate volume (250mL~750mL). It can be placed in the balance. The gondola or wire loop specimen holder can be immersed in distilled water.
- **5.3.2.10** Support frame. Place the beaker across the balance tray.
- **5.3.2.11** Hanging basket, connected with the balance tray. The gondola is made of degreased wire with a diameter of less than 0.2mm.

5.3.3 Specimens

Perform sampling during glass fiber production drawing. Use the nickel-plated crucible tongs (5.3.2.1) to cut a section of glass rod directly from under the bushing. The mass is $2g\pm1g$. Place in the specimen dish (5.3.2.2) to cool naturally. Repeat the interception to get multiple backups. Pick at least 5 glass rods that are free of visible bubbles, stones, streaks and so on. Use the file (5.3.2.3) to trim both ends into a smooth and flat specimen with slightly rounded edges. After repeated washing with distilled water (5.3.2.4) 2~3 times, put it in the blast drying oven (5.3.2.5) with a temperature of 105° C \pm 2°C and dry it for not less than 1h. Cool to room temperature in the desiccator (5.3.2.6).

5.3.4 Operation

- **5.3.4.1** Use the analytical balance (5.3.2.7) to weigh the mass (m_1) of the glass specimen, accurate to 0.1mg.
- **5.3.4.2** Place the beaker (5.3.2.9) with distilled water and the thermometer (5.3.2.8) inserted on the balance support (5.3.2.10). Measure and record the temperature of the water in the beaker. The temperature of the water should be $23^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$.
- **5.3.4.3** Place the specimen in the hanging basket (5.3.2.11). Immerse the basket and the specimen into the water in the beaker. Weigh the mass of the specimen in water (m₂), accurate to 0.1mg. During the test, the hanging basket and the specimen should not touch the beaker.
- **5.3.4.4** The density of the specimen is calculated according to formula (3):

$$\rho = \frac{m_1 \times \rho_k}{m_1 - m_2} \qquad \cdots \qquad (3)$$

Where,

 ρ - The glass fiber density, in grams per cubic centimeter (g/cm³);

Annex A

(normative)

Calibration of pycnometer sample chamber and additional chamber volumes

- **A.1** Before starting the calibration procedure, open all valves to purge the instrument with gas. Purge the sample chamber and additional chamber. Fill both chambers with gas at standard atmospheric pressure. Make the pressure sensor read zero.
- **A.2** Close the valve of the additional chamber. Fill the sample chamber with gas until the desired pressure is reached. 0.1MPa is recommended. Close the intake valve. The pressure sensor reading is recorded as p^0 .
- **A.3** Open the valve of the additional chamber. Let the gas in the sample chamber enter the additional chamber. The pressure sensor reading is recorded as p_2^0 .
- **A.4** Put a standard sample of known volume (V_c) in the sample chamber. Repeat A.2 and A.3. Fill the sample chamber with gas again until the desired pressure is reached. The pressure sensor reading is recorded as p_1^* . After the gas enters the additional chamber, the pressure sensor reading is recorded as p_2^* .
- **A.5** The volume of the sample chamber and the additional chamber can be calculated by formula (A.1) and formula (A.2), respectively:

$$V_{\text{meas}} = \frac{V_{\text{c}}(p_{1}^{*} - p_{2}^{*})}{(p_{1}^{*} - p_{2}^{*}) - (p_{1}^{0} - p_{2}^{0}) p_{2}^{*}/p_{2}^{0}} \dots (A.1)$$

$$V_{\text{exp}} = V_{\text{meas}} \frac{p_{1}^{0} - p_{2}^{0}}{p_{2}^{0}} \dots (A.2)$$

Where,

 $p_1^0 \cdot p_2^0$ - The pressure of the empty sample chamber before and after the gas diffuses into the additional chamber, in Pascals (Pa);

 p_1^*, p_2^* - The pressure of the sample chamber where the standard sample is placed before and after the gas diffuses into the additional chamber, in Pascals (Pa);

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