GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 83.120

Q 23

GB/T 7193-2008

Replacing GB/T 7193.1~7193.6-1987, partly replacing GB/T 8238-1987

Test methods for unsaturated polyester resins

不饱和聚酯树脂试验方法

(ISO 2554:1997, ISO 584:1982, NEQ)

Issued on: June 30, 2008 **Implemented on: April 01, 2009**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;
Standardization Administration of the People's Republic of China.

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Test methods for unsaturated polyester resins

1 Scope

This Standard specifies the test methods, scope, principles, test samples, instruments and equipment, test procedures, test results and test reports for testing the properties of liquid unsaturated polyester resins.

This Standard is applicable to the determination of absolute viscosity, hydroxyl value, solid content, reactivity at 80 °C, heat stability at 80 °C, gel time at 25 °C and refractive index of liquid unsaturated polyester resins.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, all subsequent amendments (not including errata content) or revisions do not apply to this standard. However, parties to agreements that are based on this Standard are encouraged to study whether the latest versions of these documents can be used. For undated references, the latest edition applies to this Standard.

GB/T 2895, Determination for acid value of unsaturated polyester resins

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

3 Terms and definitions

The following terms and definitions are applicable to this Standard.

3.1

hydroxyl value

The number of milligrams of potassium hydroxide consumed to neutralize the acetic acid produced by the acetylation reaction of 1 g of unsaturated polyester resins.

3.2

acid value

The number of milligrams of potassium hydroxide consumed to neutralize 1 g of test sample under the test conditions.

3.3

total acid value

The number of milligrams of potassium hydroxide consumed to neutralize all carboxyl groups, free acids and free anhydrides in the polyester.

3.4

solid content

The mass fraction of non-volatiles contained in unsaturated polyester resins under specific test conditions.

3.5

heat stability at 80 °C

The time required for the liquid unsaturated polyester resins, at a temperature of 80 °C, from the start of the test to the appearance of gelation.

3.6

gel time

The time FROM the addition of the initiator to the resin TO the resin viscosity reaches 50 Pa·s.

4 Test methods

4.1 Viscosity

4.1.1 Principle

The drum or rotor rotates in the sample at a fixed speed. Due to the viscosity of the liquid, resistance is applied to the drum or rotor during rotation, generating torque, which is measured by a certain method.

The measurement process is achieved by the change of the digital pointer caused by the compression of the coil spring. The absolute viscosity is measured by the rotational viscometer by multiplying the reading by a coefficient that depends on the speed and the type of drum or rotor.

4.1.2 Test samples

4.1.2.1 Uniform, free of bubbles and impurities.

4.1.2.2 The quantity should be sufficient for viscometer measurement.

4.1.3 Instruments and apparatuses

4.1.3.1 Rotational viscometer: drum type or rotor type.

4.1.3.2 Constant temperature water bath: temperature control accuracy ±0.5 °C.

4.1.3.3 Thermometer: measuring range 0 °C ~ 50 °C, minimum graduation value 0.2 °C.

4.1.3.4 Container: shall meet the requirements of the viscometer.

4.1.3.5 Stopwatch.

4.1.4 Test procedures

4.1.4.1 Select the drum or rotor and speed of the rotational viscometer (see Appendix A) so that the measured reading falls between 20% and 90%, preferably between 45% and 90%, of the full scale value.

4.1.4.2 Place the test sample in a container; adjust the temperature to approximately 25 °C; then place the container in a constant temperature water bath at 25 °C ± 0.5 °C (or pour the sample into the measuring container of the viscometer), where the water bath surface shall be slightly higher than the sample surface.

4.1.4.3 Immerse the viscometer drum or rotor vertically into the center of the test sample. The immersion depth shall comply with the requirements of the viscometer. Start timing at the same time.

4.1.4.4 During the entire measurement process, the test sample temperature shall be controlled at 25 °C ± 0.5 °C. When the drum or rotor has been immersed in the test sample for 8 minutes, start the motor and read the reading after the drum or rotor rotates for 2 minutes. After reading the value, turn off the motor; wait for 1 minute, then turn on the motor; rotate for 1 minute and take the second reading.

4.1.4.5 Empty the container and repeat 4.1.4.2 ~ 4.1.4.4.

4.1.4.6 After measuring each test sample, the viscometer drum or rotor shall be cleaned using the solvent.

4.1.5 Test results

4.1.5.1 Each sample shall be measured twice. The readings shall be calculated according to the viscometer regulations and expressed as the arithmetic mean with three significant figures.

4.1.5.2 The measurement result is in Pa·s.

4.2 Hydroxyl value

4.2.1 Principle

4.2.3.4 Water bath, temperature controlled at 50 ℃ ± 1 ℃.

4.2.3.5 Pipettes, 5 mL and 10 mL (for acetylation solution).

4.2.3.6 Potentiometric titrator, equipped with a calomel electrode/glass electrode system and a titration stand.

4.2.3.7 Analytical balance, sensitivity 1 mg.

4.2.4 Procedures

4.2.4.1 Weigh approximately 5 mg of test sample containing equivalent hydroxyl groups (1 g of sample = 280 equal parts/hydroxyl value) into a 250 mL conical flask, accurate to 1 mg. If the approximate value of the hydroxyl value is unknown, perform preliminary tests first.

4.2.4.2 Accurately add 10 mL of acetylation solution; place a magnetic stirring bar; plug the bottle; use ethyl acetate to moisten the bottle mouth; use a magnetic stirrer to stir the test sample to dissolve it. If the test sample cannot be completely dissolved after heating, add another 5 mL or 10 mL of acetylation solution.

4.2.4.3 Place the conical flask in a water bath at 50 ℃ ± 1 ℃; immerse to a depth of about 10 mm; keep it there for 45 minutes. (This time can be shortened, for example, to 30 minutes or less, as long as the test results are the same.)

4.2.4.4 Remove the conical flask; cool it to room temperature; add 2 mL of distilled water; use a magnetic stirrer to stir. When the solution is fully mixed, add 10 mL of pyridine/water mixture and stir for 5 min.

4.2.4.5 Use 60 mL of n-butanol/toluene mixture to rinse the stopper and the inner wall of the conical flask; add 5 drops of mixed indicator.

4.2.4.6 Use potassium hydroxide-methanol standard solution to titrate while stirring continuously. When the color changes, add (1~2) drops of mixed indicator. The solution changes from yellow to clear; record the milliliters of potassium hydroxide-methanol standard solution consumed (V_1); add one more drop of potassium hydroxide solution; the solution color turns blue; if it does not turn blue, record the burette reading and add one more drop of mixed indicator until blue appears.

4.2.4.7 The V_1 value used in the calculation of the result is the volume of the potassium hydroxide-methanol standard solution consumed before the drop that turns the solution blue.

4.2.4.8 Carry out a blank test under the same conditions and record the amount of potassium hydroxide-methanol standard solution consumed in milliliters (V_0).

The test sample shall be weighed from not less than 100 mL of sample that is uniform and free of mechanical impurities.

4.3.3 Instruments and apparatuses

4.3.3.1 Analytical balance: sensitivity 0.1 mg.

4.3.3.2 Electric constant temperature forced air drying oven: temperature control accuracy ±2 °C.

4.3.3.3 Dryer: Use anhydrous calcium chloride or color-changing silica gel as the desiccant.

4.3.3.4 Weighing bottle: 5 mL ~ 10 mL.

4.3.3.5 Petri dish or metal plate: 75 mm in diameter.

4.3.4 Test procedures

4.3.4.1 Mark a clean petri dish or metal plate; dry it in a drying oven at 150 °C ± 2 °C for 30 minutes. After removing it, place it in a sealed desiccator and cool it to room temperature. Weigh it on a balance (m_1) to the nearest 0.1 mg.

4.3.4.2 Shake the sample and pour it into a clean and dry weighing bottle.

4.3.4.3 Weigh 2 g ± 0.2 g of test sample (m_2) using the reduction method, accurate to 0.1 mg; place it in a Petri dish or metal plate; carefully flatten the entire bottom.

4.3.4.4 Place the Petri dish or metal tray horizontally on the top layer of a drying oven that has been pre-set to a constant temperature of 150 °C ± 2 °C and has air blowing.

4.3.4.5 After drying for 60 min ± 2 min, take out and immediately place in a desiccator to cool; after 20 min ~ 30 min, weigh (m_3) accurate to 0.1 mg.

4.3.5 Test results

4.3.5.1 Calculate the solid content according to Formula (2) and round to three significant figures.

$$SC = \frac{m_3 - m_1}{m_2} \times 100 \qquad \cdots\cdots (2)$$

Where:

SC – solid content (mass fraction) of unsaturated polyester resins, %;

m_3 – mass of the Petri dish and the residual sample, in grams (g);

m_1 – mass of the Petri dish, in grams (g);

4.4.4.2 Immediately pour the mixture into the test tube to a depth of 7 cm ~ 8 cm; insert the thermocouple into the center of the mixture; then place the test tube in a water bath at 80 ℃ ± 0.5 ℃, so that the sample liquid level is lower than the water bath surface.

4.4.4.3 A recorder, if used, shall be turned on and run throughout the reaction process. Alternatively, record the maximum temperature reached by the mixture and the time taken to rise from 65 °C to the maximum temperature. If the maximum temperature exceeds 90 °C, record the time taken to rise from 65 °C to 90 °C.

4.4.4.4 Repeat the test until the temperature difference between two consecutive measurements is no more than 5 °C and the time difference is no more than 10%.

4.4.5 Test results

The single value and average value of the maximum temperature of the two tests; the single value and average value of the time taken to increase the temperature from 65 °C to the maximum temperature; if the maximum temperature exceeds 90 °C, the single value and average value of the time taken to increase the temperature from 65 °C to 90 °C.

4.5 Heat stability at 80 °C

4.5.1 Instruments and apparatuses

4.5.1.1 Electric blast drying oven: temperature control accuracy ±2 °C.

4.5.1.2 Ground-mouth white wide-mouth bottle: 125 mL.

4.5.1.3 Balance: sensitivity 0.2 g.

4.5.2 Test procedures

4.5.2.1 Weigh 100 g ± 1 g of the test sample into a clean, dry, white wide-mouth bottle; tightly stopper it with a glass stopper; mark it. Each two test samples constitute a group.

4.5.2.2 Place the white wide-mouth bottle containing the test sample into an electric forced air drying oven maintained at a constant temperature of 80 °C ± 2 °C; start the test and record the time.

4.5.2.3 Invert the white wide-mouth bottle every 2 hours to check for bubbles. When the bubbles are no longer flowing and the sample becomes sticky and clumpy, the sample has gelled. Record the time when gelling occurs.

4.5.3 Test results

The heat stability of the sample at 80 °C is measured in hours, and the shorter time value in each group is used as the test result.

4.6 Gel time at 25 °C

4.6.1 Principle

At 25 °C and a certain formulation (resin, accelerator, initiator), the time it takes for the resin viscosity to reach 50 Pa·s (usually considered the viscosity of the gel state).

4.6.2 Reagents

The type and amount of initiator and accelerator can be selected according to the type and application of the resin.

It is recommended to use cyclohexanone peroxide dibutyl phthalate paste or methyl ethyl ketone peroxide as the initiator, and cobalt naphthenate styrene solution or cobalt naphthenate solution as the accelerator. The usage amount of initiator and accelerator is 4%.

4.6.3 Instruments and apparatuses

4.6.3.1 Constant temperature water bath: temperature control accuracy ±0.5 °C.

4.6.3.2 Stopwatch.

4.6.3.3 150 mL straight cylindrical beaker.

4.6.3.4 Thermometer: minimum graduation value 0.1 °C.

4.6.3.5 Balance: maximum weighing capacity 200 g, reading accuracy 0.2 g.

4.6.3.6 Pipette: capacity 5 mL, minimum graduation value 0.05 mL.

4.6.3.7 Dropping bottle: 50 mL.

4.6.4 Test procedures

4.6.4.1 Adjust the water bath temperature to 25 °C ± 0.5 °C.

4.6.4.2 Use a beaker as a container; weigh 100 g of sample and initiator using a balance, accurate to ±0.2 g. Place the beaker in a water bath (the sample liquid level is 2 cm below the water surface) to maintain a constant temperature; stir carefully.

4.6.4.3 When the test sample temperature is 25 °C ± 0.5 °C, accurately add the accelerator using a pipette. When the last drop is added, start the stopwatch and stir the sample thoroughly.

4.6.4.4 Observe every 30 seconds; use a glass rod to test the flow of the sample, until a wire drawing state appears; stop the stopwatch; record the time shown on the stopwatch, which is the gel time.

4.6.5 Test results

4.7.2.1.1 Abbe refractometer or other refractometer capable of producing equivalent results. The measuring range of the instrument is from 1.300 to 1.700, and the measuring accuracy is not less than 0.000 3. A temperature control device needs to be provided for the sample and the prism.

4.7.2.1.2 White light source or sodium lamp light source.

4.7.2.1.3 Thermostat, accurate to ±0.1 °C.

Note: Circulating water shall be distilled water or deionized water.

4.7.2.2 Materials

Contact liquid, α-bromonaphthalene.

4.7.3 Test procedures

4.7.3.1 Calibration instruments. Check the refractometer's sight regularly using the standard block that comes with the instrument. If the reading differs from the value of the standard block by more than the measurement accuracy, adjust the instrument according to the instruction manual. When using an Abbe refractometer, it can be calibrated with a standard block or double distilled water. The refractive index of water is 1.333 0 at 20 °C and 1.332 0 at 30 °C. The temperature coefficient is -0.000 1/°C.

4.7.3.2 Place the refractometer in a well-lit location; connect it to a thermostat; adjust the temperature of the refractometer prism to 20 °C. Keep the water circulating to ensure the required temperature and maintain it within ±0.1 °C.

4.7.3.3 Before each test, clean the prism surface of the instrument according to the instruction manual. If not otherwise specified, use anhydrous ethanol and lens cleaning paper to clean the prism; immediately use dry lens cleaning paper to wipe the prism clean. Wait 2 minutes for the temperature to stabilize before placing the sample on the prism.

4.7.3.4 Use a round-ended glass rod or pipette to add the sample to the surface of the refracting prism so that the prism fits perfectly. Wait 2 minutes for the temperature to stabilize.

4.7.3.5 Adjust the light source so that the divergent light shines on the prism. Adjust the instrument's knob until the field of view appears. If the instrument is not equipped with a compensating prism, adjust the light source to make the field of view boundary clear and the contrast between light and dark maximized.

4.7.3.6 If the instrument is equipped with a compensating prism, adjust the compensating prism until the field of view appears with light and dark parts and the color fringes disappear.

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