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JAPANESE INDUSTRIAL STANDARD

Testing Methods for Polyamides (Nylon) Molding Materials

JIS K 6810 —1977

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JAPANESE INDUSTRIAL STANDARD

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Testing Methods for Polyamides (Nylon)
Molding MaterialsK 6810-1977
(Reaffirmed: 1980)1. Scope

This Japanese Industrial Standard specifies the testing methods for polyamides (nylon) molding materials ⁽¹⁾, hereinafter referred to as the "molding materials".

Remark: The units and numerical values shown in braces { } in this standard are in accordance with the International System of Units (SI), and are appended for reference.

Note ⁽¹⁾ The polyamides (nylon) molding materials described herein are of pellet form of not more than 12 mm in maximum length and do not contain those of powder state and solution state.

2. Testing Items

The testing items shall be as follows:

- (1) Specific gravity and density
- (2) Melting point
- (3) Water content
- (4) Relative viscosity
 - (a) Method by sulfuric acid solution
 - (b) Method by formic acid solution
- (5) Contents of monomer and polymer of low molecular weight
- (6) Tensile strength
- (7) Elongation
- (8) Bending modulus of elasticity
- (9) Impact strength
- (10) Temperature of thermal deformation

Applicable Standards and Reference Standard: See page 27.

3. General Matters of Tests

3.1 Method for Sampling For the method for sampling, carry out the reasonable one for each lot of molding materials capable of being considered to be the same quality.

3.2 Method for Making Test Pieces The molding conditions for preparing the test piece from the sample shall be in accordance with the instructions of the manufacturer for molding materials. However, the lot of the sample used for molding shall be in accordance with the agreement between the parties concerned.

3.3 Testing Conditions Unless otherwise specified in this standard, the testing conditions shall be $23 \pm 5^{\circ}\text{C}$ in temperature and $65 \pm 5\%$ in relative humidity.

3.4 Conditioning for Test Piece Unless otherwise specified in this standard, condition the test piece for a specified period of time immediately after molding in the room where the temperature is $23 \pm 5^{\circ}\text{C}$ and the relative humidity is $65 \pm 5\%$ before test.

3.5 Number of Tests Unless otherwise specified in this standard, carry out five tests and allow the arithmetic mean value of these to be the test result. Obtain the mean value to the one place lower than the specified value and round off that in accordance with JIS Z 8401.

4. Testing Methods

4.1 Specific Gravity and Density

4.1.1 Sample For the sample, use the pellets (not to contain blow holes or foreign matters).

4.1.2 Conditioning for Sample Dry the sample in the drying vessel of constant temperature at $50 \pm 5^{\circ}\text{C}$ for 48 h before test. Cool that in the desiccator containing calcium chloride.

4.1.3 Measuring Method of Specific Gravity with Pycnometer

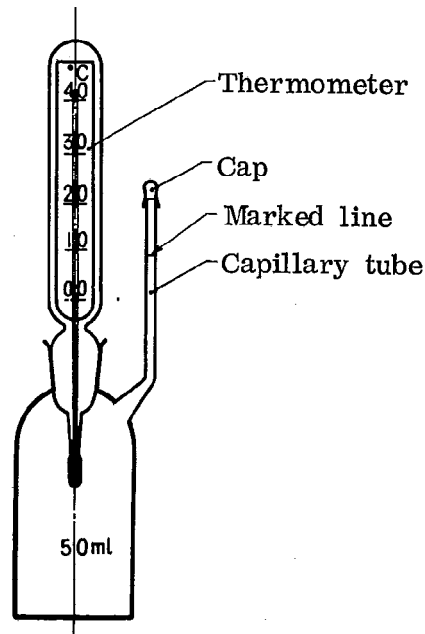
(1) Apparatus The apparatus shall be as follows:

(a) Pycnometer For the pycnometer, use the specific gravity bottle with the thermometer of Gay-Lussac type in JIS R 3503.

(b) Chemical Balance 1 mg in reciprocal sensitivity.

- (c) Thermostat The temperature being $23 \pm 0.1^\circ\text{C}$.
- (d) Vacuum Desiccator

Fig. 1. Pycnometer



(2) Operation

- (a) Accurately weigh the mass of the pycnometer, which is preliminarily washed and dried, to the nearest 0.1 mg.
- (b) Accurately fill the pycnometer of (a) with distilled water to the marked line at a temperature of $23 \pm 0.1^\circ\text{C}$ and accurately weigh the mass to the nearest 0.1 mg.
- (c) Then, after drying the pycnometer, take 1 to 5 g of the sample and accurately weigh that to the nearest 0.1 mg again. Obtain the mass of the sample by subtracting the result of (a).
- (d) After allowing the sample to be covered by adding distilled water to the pycnometer containing the sample, remove the air included in the distilled water within the vacuum desiccator.

- (e) Then, fill the pycnometer with distilled water near to the marked line and keep that in the thermostat until the temperature of that reaches $23 \pm 0.1^\circ\text{C}$.
- (f) After regulating the quantity of distilled water in the pycnometer so that the quantity of distilled water accurately fill to the marked line, accurately weigh the mass to the nearest 0.1 mg.
- (g) Calculate the specific gravity S ($23/4^\circ\text{C}$) from the following formula:

$$S = \frac{a \times d}{b - c + a}$$

where a : mass of sample (g)

b : mass of the pycnometer filled to the marked line with distilled water (g)

c : mass of the pycnometer containing the sample filled to the marked line with distilled water (g)

d : specific gravity of distilled water at 23°C
(0.99756)

4.1.4 Measuring Methods of Density by Density Gradient Column

(1) Apparatus The apparatus shall be as follows:

- (a) Glass Cylinder The glass cylinder of about 1 m in length, 4.5 to 6.0 cm in inner diameter, having the graduation with intervals of 1 mm over the length of 85 cm at least, and with a ground cap at its upper part.
- (b) Thermostatic Water Tank The tank capable of being put with the glass cylinder, furthermore, of being regulated at a temperature of $23 \pm 0.1^\circ\text{C}$.
- (c) Beaker Two pieces of the same diameter, with about 2000 ml in capacity.
- (d) Standard Float Hollow glass bulb of 3 to 5 mm in diameter, for the density of 1.12 to 1.16 (g/cm^3).
- (e) Siphon The siphon as shown in Fig. 2.
- (f) Magnetic Stirrer

(2) Reagents The reagents shall be as follows:

(a) Carbon Tetrachloride That corresponding to JIS K 8459.

(b) Toluene That corresponding to JIS K 8680.

(3) Method for Making Density Gradient Column

(a) Mix an appropriate quantity of toluene and carbon tetrachloride in a beaker to prepare two kinds of liquids of the heavy liquid and the light liquid ⁽²⁾.

Note ⁽²⁾ Prepare for allowing the density between the graduation of 40 cm of the density gradient column to be 1.12 to 1.16 (g/cm³). For this purpose, it is preferable that the density of the heavy liquid should be about 1.180 (g/cm³) and the density of the light liquid, 1.100 (g/cm³). It is preferable to prepare in accordance with Table 1 as a standard.

Table 1

Unit: ml

	Carbon tetrachloride	Toluene
Light liquid	322	678
Heavy liquid	431	569

- (b) Put the light liquid into the beaker A and put the heavy liquid into the beaker B as shown in Fig. 2. Allow both liquids to reach at the same height, connect those with a siphon.
- (c) During stirring the beaker B with the magnetic stirrer, pour the liquid in the beaker with a siphon into the glass cylinder at a speed of not more than 10 ml/min along the wall of the beaker.
- (d) Because the height of the liquid in the beaker decreases by this operation, the liquid in the beaker A tends to flow gradually into the beaker B, and then to be poured into the glass cylinder. Therefore, the liquid in the glass cylinder becomes to show the continuous density gradient.
- (e) Carry out the operation from (b) to (d) at a temperature of near 23°C.

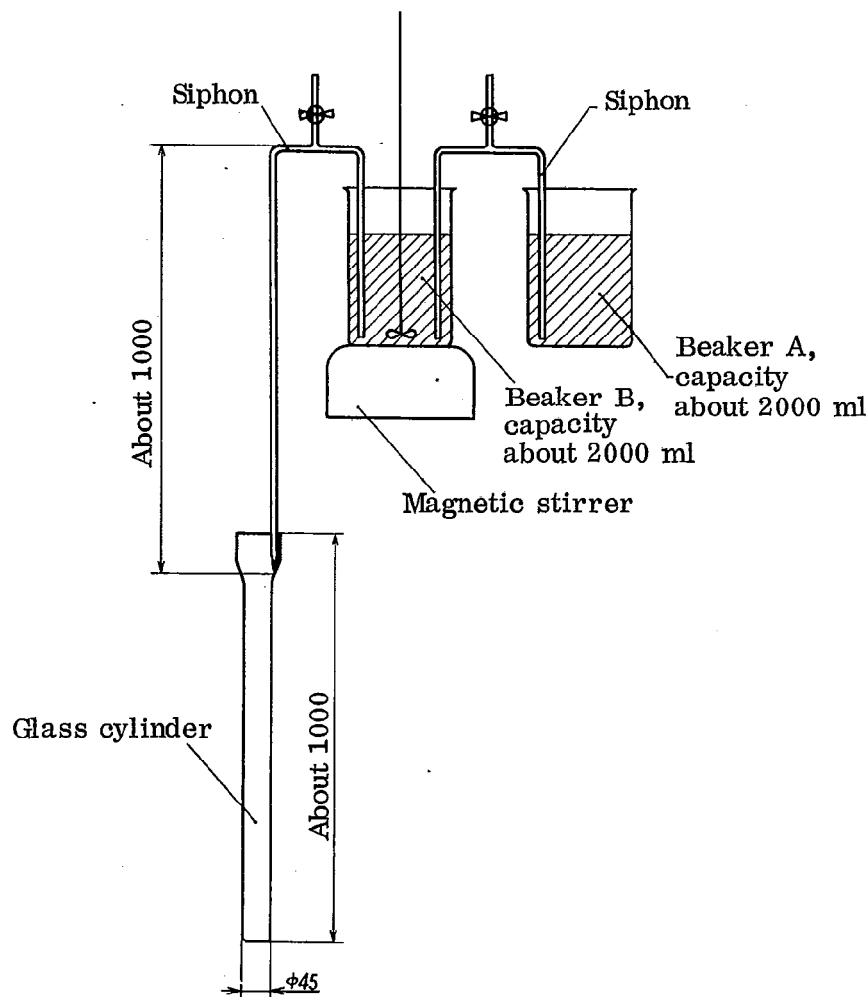
- (f) Slowly put the glass cylinder poured out with the liquid into the thermostatic water tank.
- (g) Then, after getting the standard float wet with the liquid (light liquid) in the beaker A, slowly put it into the glass cylinder to make it the density gradient column.
- (h) Keep the density gradient column at a temperature of $23 \pm 0.1^{\circ}\text{C}$ in the thermostatic water tank.
- (i) Use the standard floats of 1.12 to 1.16 in density, and not less than one piece for the difference of 0.01 g/cm^3 in density should be suitable.
- (j) After 24 h passed, make the calibration curve between the density of the standard floats in the density gradient column and the graduation of the density gradient column.
- (k) When this calibration curve shows zigzag or remarkable bow-shape, repeat the operation from (a) to (i) again.

(4) Operation

- (a) Cut the sample in the thickness of 20 to 50 μm so that blow hole portions may not be included in the small piece of the sample.
- (b) After letting three samples be cut into small pieces, wet by the light liquid, and then slowly insert those into the density gradient column.
- (c) After three test pieces have reached the equilibrium state and stood still in the liquid, accurately read the heights of those centers of gravity to the nearest 1 mm from the graduation of the density gradient column.

Fig. 2. Method for Making Density Gradient Column

Unit: mm



- (d) Compare the result of (c) with the calibration curve. Obtain the specific gravity corresponding to each measured result to the three places of decimals. Round off the mean value of three results to the three places of decimals and allow that to be the density.
- (e) Slowly scoop up the standard float and the sample with a wire net at a speed of not more than 1 cm/min.