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JAPANESE INDUSTRIAL STANDARD

Testing Methods for
Polyethylenes

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Testing Methods for Polyethylenes

K 6760-1981

1. Scope

This Japanese Industrial Standard specifies testing methods for polyethylenes⁽¹⁾.

Further, this standard applies to polyethylenes added with adequate amount of antioxidant, lubricant and the like and to particular articles such as coloured articles, flame-proof articles, carbon-black-compounded articles, and filler-compounded articles.

Note ⁽¹⁾ Polyethylenes mean ethylene homopolymer, copolymer of ethylene and 1-olefin monomer of not more than 5 mol %, and copolymer of ethylene and non-olefin monomer of not more than 1 mol % having exclusively carbon, oxygen and hydrogen atoms as functional group.

Remarks 1. In this standard, the tests shall be carried out in three ranges divided as follows according to the density of polyethylene ⁽²⁾:

Low density polyethylene: density (g/cm³)
0.910 to 0.930 excl.

Medium density polyethylene: density (g/cm³)
0.930 to 0.942 excl.

High density polyethylene: density (g/cm³) 0.942 min.

Note ⁽²⁾ The density of particular articles is defined as the density containing no additive.

2. In this standard, the units and numerical values given in { } are in accordance with the International System of Units (SI) and appended for reference.

Applicable Standards and Reference Standard: See page 26.

2. Kind of Tests

The kind of tests shall be as follows:

- (1) Melt flow rate
- (2) Specific gravity and density
- (3) Tension test
- (4) Durometer D hardness test
- (5) Vicat softening point test
- (6) Cold resistance test
- (7) Test for environmental stress cracking under constant strain
- (8) Test for environmental stress rupture under constant stress
- (9) Dielectric dissipation factor and specific inductive capacity

3. General Conditions for Tests

The preparation of test pieces to be used for tests other than melt flow rate test, annealing, and conditioning of test pieces shall be as follows:

- (1) Preparation of Test Pieces The sample may be either of pellets, roll-kneadings, and granules. Insert the die containing the sampler in the press which is sufficiently heated to melt the sample, after moulded, pass the cooling water or transfer the die in other cooling press and cool down the die by applying adequate pressure, and then take out the moulded sheet. In this case, let the cooling speed ⁽³⁾ of the sample in the press be 20 to 30°C/min. However, in the case of sample of low-density polyethylene, specify no cooling speed. Take care on the appearance of the moulded sheet concerning bubbles, dusts, etc., and do not use the part where those exist as the test piece. Cut out, punch out, or machine-cut the test piece from the sheet to be specified shape and dimensions. When machine-cutting, take care sufficiently for the property of the material not to be changed by machining heat.

Note ⁽³⁾ Cooling speed is defined as the average cooling speed from 140°C to 80°C.

- (2) Annealing Annealing shall, as a rule, not be carried out except the low density polyethylene for measuring specific gravity and density. However, in the case where annealing is required specially, it shall be carried out according to the agreement between the parties concerned.
- (3) Conditioning of Test Pieces After the moulded or annealed test pieces have been conditioned at a temperature of $23 \pm 2^{\circ}\text{C}$ and a relative humidity of $50 \pm 5\%$ for 16 h, the test shall be carried out. However, the conditioning period shall, in the case of the test for environmental stress cracking under constant strain, 16 h to 24 h for low density and medium density polyethylenes, and 48 h to 72 h for high density polyethylene, and in the case of test for environmental stress rupture under constant stress, shall be 48 h to 72 h.
- (4) Standard Atmospheric Conditions for Testing Place The tensile test, durometer D hardness test, and the test for dielectric dissipation factor and specific inductive capacity shall be carried out in a room at a temperature of $23 \pm 2^{\circ}\text{C}$ and a relative humidity of $50 \pm 5\%$.
- (5) Expression of Numerical Values of Test Results Numerical values of test results shall be rounded-off to the specified places in accordance with JIS Z 8401.

4. Testing Methods

4.1 Melt Flow Rate The measurement of melt flow rate shall be carried out in accordance with JIS K 7210. In this case, the test conditions shall, as a rule, be Condition 4 specified in Table 1 of JIS K 7210 (testing temperature, 190°C ; testing load, 2.16 kgf {21.18 N}), and, as required, Condition 5 (testing temperature, 190°C ; testing load, 5.00 kgf {49.03 N}), Condition 6 (testing temperature, 190°C ; testing load, 10.00 kgf {98.07 N}) and Condition 7 (testing temperature, 190°C ; testing load, 21.60 kgf {211.82 N}) may be employed.

4.2 Specific Gravity and Density The measurement of specific gravity and density shall be carried out in accordance with JIS K 7112. In this case, the testing temperature shall, as a rule, be $23 \pm 0.1^{\circ}\text{C}$. In the case where the sample is low density polyethylene, the measurement shall be carried out after annealing by the following method: Take the test piece into a 1000 ml beaker, fill it with distilled water, cover with a watch glass or aluminium foil, and heat with a mantle heater. After boiling period of 60 min from beginning of boiling of distilled water, put the beaker on the wooden stand, and leave it cool. The period of time necessary to cool down to room temperature shall be more than 60 min. In this case, the amount of boiling distilled water after 60 min boiling shall be 500 ml approximately, and the test piece shall be immersed into the approximately central portion of the water so as not to come in contact with the beaker and the water surface.

Remark: In the case of converting the density measured at the range of 20 to 23°C to the density at 23°C, it shall be calculated from the following formula:

$$\rho_{23} = \rho_t - 0.0006(23 - t)$$

where ρ_{23} : density at 23°C (g/cm³)

ρ_t : density measured at t °C (g/cm³)

t : testing temperature (°C)

4.3 Tension Test The tension test shall be carried out in accordance with JIS K 7113. In this case, the test piece to be used shall be No. 2 test piece. The thickness of the test piece shall be 2.0 ± 0.2 mm, the testing speed, speed F (50 mm/min ± 10 %) for high density and medium density polyethylenes, and speed H (200 mm/min ± 10 %) for low density polyethylene. If the test piece breaks at the flaw caused during the test or breaks outside the mark line, the result shall be discarded and the retests same as the discarded number shall be carried out. However, if the test piece is cold elongated remarkably and the broken portion is the part proceeding in cold elongation or cold elongated part, the results may be employed.

4.4 Durometer D Hardness Test The durometer D hardness test shall be carried out in accordance with JIS K 7215.

4.5 Vicat Softening Point Test The Vicat softening point test shall be carried out in accordance with JIS K 7206. In this case, the testing load shall be 1.00 to 1.05 kgf {9.807 to 10.30 N}, and the results shall be reported in integer.

4.6 Cold Resistance Test The cold resistance test shall be carried out in accordance with the following:

- (1) Apparatus, Measuring Device, Heat Transfer Medium, Temperature Regulation of Heat Transfer Medium and Testing Speed These shall be in accordance with JIS K 7216. In this case, test piece grips and the percussion hammer shall be Type A specified in 5.1.1 of JIS K 7216.
- (2) Test Piece The dimensions of the test piece shall be Type A specified in 4.1 of JIS K 7216, and the number of test pieces shall be five pieces or more for every testing temperature.

(3) Procedure

- (a) Preliminarily, keep the heat transfer medium in the testing bath at the specified temperature uniformly. After immersing the test piece fixed by the grip in accordance with 7.2 of JIS K 7216 in the heat transfer medium for 3 ± 0.5 min, apply one impact by the percussion hammer, and examine the existence of breakage of test piece.
- (b) The breakage means the fact that the test piece breaks in two or more parts completely, and generation of fissures or crazings is not defined as breakage.
- (c) Obtain the testing temperature and the number of test pieces broken.
- (d) The testing temperature shall be determined by agreement between the parties concerned, and be -76°C at the lowest.
- (e) Test results shall be reported on the number of test pieces, number of breakages and the testing temperature.

4.7 Test for Environmental Stress Cracking under Constant Strain The test for environmental stress cracking under constant strain shall be carried out according to the following:

(1) Apparatus and Devices

- (a) Constant Temperature Bath The constant temperature bath capable of maintaining the water temperature at $50 \pm 0.5^{\circ}\text{C}$ shall be used.
- (b) Hard Glass Test Tube (with Stopper) The test tube of approx. 32 mm in cylinder diameter and approx. 200 mm in height as shown in Fig. 1 shall be used.
- (c) Test Piece Fixing Device The device made of copper alloy ⁽⁴⁾ or stainless steel ⁽⁵⁾ as shown in Fig. 2 shall be used.

Notes ⁽⁴⁾ The copper alloy shall be used by selecting the adequate material from those specified in JIS H 3250.

⁽⁵⁾ The stainless steel shall be used by selecting the adequate material from those specified in JIS G 4303.

- (d) Nicking Jig The nicking jig is a device to notch the central portion of test piece surface, and an example is shown in Fig. 3.
- (e) Test Piece Bending Jig The test piece bending jig is a device to bend the test piece, and an example is shown in Fig. 4.

- (f) Test Piece Transfer Jig The test piece transfer jig is a device to attach the bent test piece to the test piece fixing device and an example is shown in Fig. 5.
- (g) Blade The blade is a sharp blade to cut the specified notch and its shape and dimensions are shown in Fig. 6.
- (h) Aluminium Foil The aluminium foil is employed to wrap the stopper.
- (i) Stopwatch or Watch The stopwatch or watch to be used shall be able to measure to 1 sec.
- (j) Trip Balance The trip balance specified in JIS B 7601 having an accuracy of reciprocal sensibility of 0.5 g or superior shall be used.

Fig. 1. Hard Glass Test Tube (with Stopper)

Unit: mm

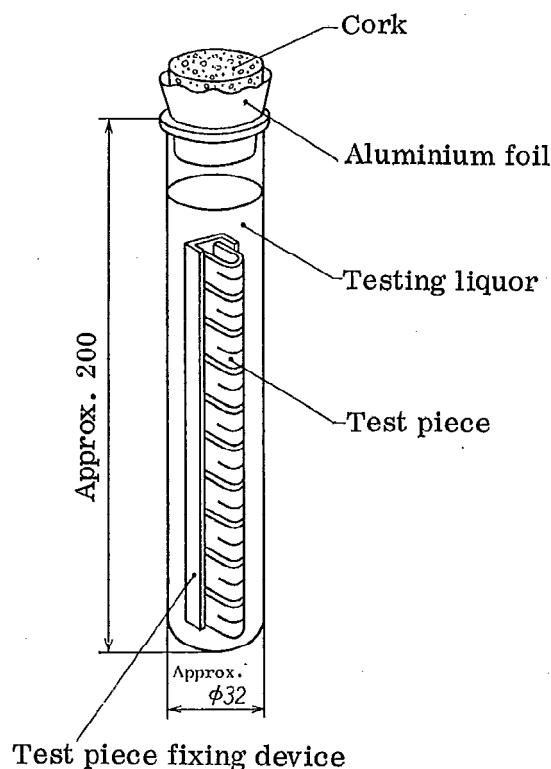


Fig. 2. Test Piece Fixing Device

Unit: mm

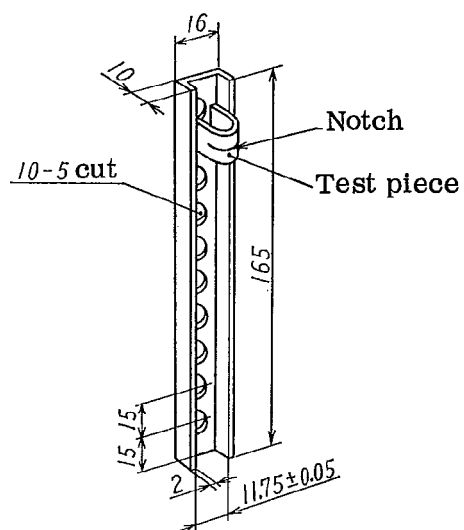


Fig. 3. An Example of Nicking Jig

