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# Test Method for the Identification of Recycled Polyethylene Terephthalate (PET) Fiber

循环再利用聚酯（PET）纤维鉴别方法

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**Standardization Administration of the People's Republic of China.**

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# Test Method for the Identification of Recycled Polyethylene Terephthalate (PET) Fiber

## 1 Scope

This Standard specifies the test method for the identification of recycled polyethylene terephthalate (PET) fiber (hereinafter referred to as recycled polyester).

This Standard is applicable to natural and colored recycled polyester. Other functional recycled polyester may take this as a reference.

## 2 Normative References

The following documents are indispensable to the application of this document. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 4146 (all parts) Textiles - Man-made Fibers

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

## 3 Terms and Definitions

What is defined in GB/T 4146, and the following terms and definitions are applicable to this document.

### 3.1 Recycled Polyethylene Terephthalate (PET) Fiber

Recycled polyethylene terephthalate (PET) fiber refers to polyethylene terephthalate fiber made from waste polyester (PET) polymer and waste polyester (PET) textile materials through recycling and processing.

## 4 Principle

Based on the essential difference between the processing process of recycled polyester and primary polyester, which causes certain characteristics to be different, process the specimen in accordance with the specified conditions, then, test it on a high performance liquid chromatograph. In accordance with the difference in the relative peak area of the specimen under different retention times, the purpose of qualitative identification is reached.

**5.1.5** Bench vice or other fixing device.

**5.1.6** Automatic pipetting device or pipette: 1 mL, 20 mL and 25 mL.

**5.1.7** Measuring cylinder: 50 mL and 200 mL.

**5.1.8** Volumetric flask: 2 L.

**5.1.9** 2 mL threaded sample bottle that can be used for automatic sample injection of the high performance liquid chromatograph, with PTFE liner.

**5.1.10** Syringe-driven filter, filter membrane 0.45 μm; made of PTFE.

**5.1.11** Reagent reservoir, with a stopper; made of glass.

**5.1.12** Centrifuge.

**5.1.13** Refrigerator with a refrigerating chamber.

### 5.2 Reagents and Materials

**5.2.1** Methanol, analytically pure.

**5.2.2** Methanol, chromatographically pure.

**5.2.3** Catalyst: zinc acetate, analytically pure.

**5.2.4** Transesterification solution: weigh-take 60 mg of zinc acetate; use methanol to dissolve it and dilute to 2 L.

**5.2.5** Sulfolane, analytically pure.

**5.2.6** Acetonitrile, analytically pure.

**5.2.7** Comply with Grade-2 water of GB/T 6682.

## 6 Test Procedures

### 6.1 Methanolysis

**6.1.1** Weigh-take 0.8 g (accurate to 0.1 mg) of the specimen to be tested.

**6.1.2** Place the specimen into the reaction tube; accurately add 25 mL of the transesterification solution. Use the bench vice to tighten the cap of the reaction tube (to ensure air tightness); vertically put it into the heating device. At (220 ± 5) °C, react for 2 h, then, take it out. Use tap water to cool it down to room temperature. Open the reaction tube; filter the supernatant through the syringe-driven filter to a 2 mL sample bottle. Place it in the refrigerating chamber of the refrigerator at 4 °C ~ 6 °C; maintain

for above 8 h. Then, filter it through the syringe-driven filter to another 2 mL sample bottle; let it stand, till it reaches the room temperature. This can be used for high performance liquid chromatography detection.

### 6.2 Swelling-extraction

**6.2.1** Weigh-take about 5 g of the same specimen to be tested; place it in a drying oven that is previously heated to 105 °C to dry for 30 min. Then, take it out and put it in a desiccator to cool it down; reserve it for later use.

**6.2.2** Use a measuring cylinder to respectively weigh-take 160 mL of sulfolane and 40 mL of acetonitrile. Put them into the reagent reservoir, put on the cap; shake it well and reserve it for later use.

**6.2.3** Accurately weigh-take about 1.4 g (accurate to 0.1 mg) of the specimen treated in 6.2.1; put the specimen into the reaction tube. Use the pipetting device to add 35 mL of the solution described in 6.2.2; tighten the cap of the reaction tube (to ensure air tightness); place it into the heating device. At (180 ± 5) °C, react for 90 min, then, take it out. Use tap water to cool it down to room temperature.

**6.2.4** Pour the sample into a centrifuge tube, then, at a centrifugal speed of 4,000 r/min, centrifuge for 8 min. Take the supernatant; filter it through the syringe-driven filter to a 2 mL sample bottle. Then, place it in the refrigerating chamber of the refrigerator at 4 °C ~ 6 °C; maintain for above 8 h. Then, filter it through the syringe-driven filter to another 2 mL sample bottle; let it stand, till it reaches the room temperature. This can be used for high performance liquid chromatography detection.

### 6.3 High Performance Liquid Chromatography Detection

Respectively conduct high performance liquid chromatography detection on the treatment liquids treated in accordance with 6.1 and 6.2. After the chromatographic detection is completed, derive the original signal data of high performance liquid chromatography.

The test result depends on the instrument being used, and it is impossible to provide the common parameters of chromatographic analysis. The following operating conditions have been proven to be suitable for testing:

a) Chromatographic column: XDB $C_{18}$ (5 μm), 250 mm × 4.6 mm, or equivalent;

b) Flow rate: 1.0 mL/min;

c) Column temperature: 30 °C;

d) Injection volume: 10.0 μL;

e) Detector: diode array detector (DAD);

$n_2$---the number of other samples in the sample library;

$\mu_{i1k}$---the relative content of a specific fingerprint peak of each recycled polyester in the sample library;

$n_1$---the number of recycled polyester samples in the sample library.

In accordance with Formula (10), standardize $X_i$ value to $Z_{i1}$, then, query the standard normal distribution probability table to obtain the probability $F(Z_{i1})$ corresponding to $Z_{i1}$. In accordance with Formula (11), standardize $X_i$ value to $Z_{i2}$, then, query the standard normal distribution probability table to obtain the probability $F(Z_{i2})$ corresponding to $Z_{i2}$. When $\mu_{i1} > \mu_{i2}$, the precision rate $P_i$ shall be calculated in accordance with Formula (12); when $\mu_{i1} < \mu_{i2}$, the precision rate $P_i$ shall be calculated in accordance with Formula (13).

$$Z_{i1} = (X_i - \mu_{i1}) / \sigma_{i1} \quad \cdots\cdots (10)$$

$$Z_{i2} = (X_i - \mu_{i2}) / \sigma_{i2} \quad \cdots\cdots (11)$$

$$P_i = 1 - \frac{1 - F(Z_{i2})}{1 - F(Z_{i2}) + F(Z_{i1})} \quad \cdots\cdots (12)$$

$$P_i = 1 - \frac{F(Z_{i2})}{1 - F(Z_{i1}) + F(Z_{i2})} \quad \cdots\cdots (13)$$

Respectively calculate the discriminant function values $W_3$, $W_4$, $W_6$ and $W_7$ and the precision rates $P_3$, $P_4$, $P_6$ and $P_7$.

$P_3$, $P_4$, $P_6$ and $P_7$ respectively correspond to $W_3$, $W_4$, $W_6$ and $W_7$. From $P_3$, $P_4$, $P_6$ and $P_7$, choose the maximum value $P_{max}$; find the corresponding discriminant function value $W_i$. If $W_i$ is greater than 0, then, it is determined that “this sample contains recycled polyester component”, and $P_{max}$ is used as the precision rate of the conclusion that “this sample contains recycled polyester component”; if $W_3$, $W_4$, $W_6$ and $W_7$ are all smaller than 0, then, it is determined that “this sample is primary polyester”.

**NOTE:** this method is based on a great deal of benchmark sample data.

# 7 Test Report

The test report shall include the following contents:

a) Serial No. of this Standard;

b) Sample description;

c) Discriminant function values $W_3$, $W_4$, $W_6$ and $W_7$ and precision rates $P_3$, $P_4$, $P_6$ and $P_7$;

d) Conclusion of identification;

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