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**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

SH/T 1827-2019

**Plastics. Determination of crystallinity. X-ray diffraction
(English Translation)**

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009 this standard was proposed by China Petrochemical Corporation this standard is under the jurisdiction of the Petrochemical Plastic Resin Products Branch of the National Plastics Standardization Technical Committee (SAC/TC15/SC1) Responsible for drafting this standard. China National Petroleum Corporation Petrochemical Research Institute, Beijing Yanshan Petrochemical High-tech Technology Company Limited liability company Participated in the drafting of this standard. Tianjin University, Shanghai Jiaotong University,

China Petroleum & Chemical Corporation Beijing Research Institute of Chemical Industry,
China Petroleum & Chemical Corporation Zhenhai Refining & Chemical Company, China
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Instrument Co., Ltd., Dandong Tongda Technology Co., Ltd.
Co., Ltd., Shanghai Ruoyi New Material Technology Co., Ltd.

1 Scope

This standard specifies the method for determining the crystallinity of polyethylene (PE) and polypropylene (PP) by X-ray diffraction this standard is applicable to the determination of crystallinity of granular or powdered polyethylene and polypropylene (alpha crystal form, beta+alpha crystal form) compression-molded or injection-molded specimens this standard does not apply to the determination of the crystallinity of filled polyethylene and polypropylene Note. The determination of the crystallinity of polyethylene and polypropylene products can refer to this standard. The high ethylene content ethylene-propylene copolymer polypropylene can also refer to this standard.

2 Normative references

The following documents are indispensable for the application of this document. All dated reference documents only the dated version applies to this article the latest version (including all amendments) of undated reference documents is applicable to this document
GB/T 1845.2 Plastic Polyethylene (PE) Molding and Extrusion Materials Part 2: Sample Preparation and Performance Determination
GB/T 2546.2 Plastic polypropylene (PP) Moulding and extrusion materials Part 2: Sample preparation and performance measurement
GB/T 2918 Standard environment for condition adjustment and testing of plastic samples
JJG 629-2014 Polycrystalline X-ray Diffractometer

3 Terms and definitions

The following terms and definitions apply to this document
3.1 Crystallinity (X-ray diffraction method) the degree of crystallinity calculated based on the percentage of the contribution of the crystal region in the total area of the X-ray diffraction intensity peaks of the partially crystalline polymer[1]Xc

4 Method summary

The crystallinity of the polymer is proportional to the integrated area of the X-ray diffraction peaks of the crystalline phase. Collect 2theta at an angle of 5° under the specified test conditions.

The copper Kalpha X-ray diffraction data of the sample within the range of 35° is obtained

by using the chemometric peak analysis software to obtain the measured crystal plane diffraction peak and amorphous scattering the peak 2theta angle and peak area count value are calculated by the ratio of the sum of the peak area of the crystalline peak and the sum of the sum of the peak area of the amorphous peak.

Kind of crystallinity

5 Reference materials

Powder alpha-SiO₂ or Si standard material that meets the technical requirements of JJG 629-2014

6.1 X-ray diffractometer

The X-ray diffractometer that meets the technical requirements of JJG 629-2014 is equipped with a copper target (K α radiation). The rotating sample stage instrument is as per JJG the stability of the diffraction intensity measured in 629-2014 for 8 hours is not more than 1.5% (expressed as relative range) Note. If the instrument's optical system configuration or adjustment is improper, the scattered light will enter the detector at a low angle and a bun peak will appear on the background, and the instrument cannot be completed.

Determination of crystallinity

6.2 Software

X-ray diffraction data processing software with Pearson VII peak splitting program

7.1.1 Preparation of PE sample

Use compression molding (Q) or injection molding (M) methods to prepare specimens according to GB/T 1845. The specimen thickness is 1.0mm $\pm$ 0.1mm

7.1.2 Preparation of PP sample

Use compression molding (Q) or injection molding (M) methods to prepare specimens according to GB/T 2546. The specimen thickness is 1.0 mm $\pm$ 0.1 mm

7.2 Sampling

Use punching equipment or ruler and blade to cut a test piece in the middle of the sample prepared by compression molding (Q) or injection molding (M) method. The test piece is required.

Flat as a mirror surface without bending The test surface is clean and free from dust and other mechanical impurities and kept dry. The size of the test piece is as specified by the

instrument used.

Note: When sampling from the product, machine processing or other suitable methods can be used to prepare a slice of $1.0\text{mm} \pm 0.1\text{mm}$ from the product and then sample according to the above method

8 Condition adjustment

According to GB/T 2918, the state is adjusted at a temperature of $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$. The state adjustment time is in accordance with GB/T 1845.2 and GB/T 2546.2.

9.1 Test conditions

Test according to GB/T 2918 at a temperature of $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$

9.2 Calibration instrument

Start the X-ray diffractometer (6.1) and use the standard material (5) to verify that the diffraction intensity of the instrument is not greater than the stability of the instrument 1.5%/8 hours (expressed in relative range)

9.3 Set the working conditions of the instrument

Set the working conditions of X-ray diffractometer (see Table 1)

9.4 Test

Do two tests in parallel, install the debugging film on the X-ray diffractometer sample table, close the sample room and start the test

10.1 Get the 2theta value of the peak position and the count value of the peak area

Use the data processing software configured with the X-ray diffractometer to first perform the Save-Golay 9 points or the X-ray diffraction data of the sample.

11-point parabolic smooth stripping Kalpha2 subtracts the background (B.G.), and then uses the Pearson-VII peak splitting program to perform peak shape fitting.

The peak shape of the radiation curve is divided into the crystal diffraction peak and the amorphous scattering peak to obtain the 2theta value of the peak position of the crystal diffraction peak and the amorphous scattering peak of the tested sample.

Amorphous scattering peaks and symmetrical crystal diffraction peaks should be symmetrically fitted to the peak area count value. If the measured crystal diffraction peaks are asymmetric the asymmetric fitting is performed according to its peak shape