# Guideline for Sample Preparation and Acquisition of Physical and Structural Data for Registration in the Recycled Material Data Bank

**2nd Edition: November 17, 2025**

## 1 Scope

This guideline specifies the procedures for the preparation of samples and the test methods for obtaining physical and structural data of virgin polypropylene (PP) and recycled PP intended for registration in the Recycled Material Data Bank.

All values specified in this guideline shall be stated in SI units.

This guideline does not purport to address all safety concerns, if any, associated with its use. It is the responsibility of the user of this guideline to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

## 2 Normative References

The following referenced standards are indispensable for the application of this guideline.

For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including amendments) applies.

2.1 JIS Standards

- JIS K 7111-1, Plastics — Determination of Charpy impact properties — Part 1: Non-instrumented impact test
- JIS K 7112-1, Plastics — Determination of density of non-foamed plastics — Part 1: Immersion method, liquid pyknometer method, and titration method
- JIS K 7120, Plastics — Thermogravimetric analysis (TGA)
- JIS K 7121, Plastics — Differential scanning calorimetry (DSC) — Determination of transition temperatures
- JIS K 7139, Plastics — Test specimens
- JIS K 7171, Plastics — Determination of flexural properties
- JIS K 7210-1, Plastics — Determination of melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method
- JIS Z 8722, Method of color measurement — Reflecting and transmitting objects

# 3 Terms and Definitions

Terms defined in the referenced JIS standards listed in Clause 2 shall apply. The definitions of terms specific to this guideline are as follows.

3.2.1 PP crystallinity

The ratio of the infrared (IR) absorbance of the polypropylene reference band at 973 $cm^{-1}$ to that of the PP crystalline band at 998 $cm^{-1}$, obtained by Fourier transform infrared spectroscopy (FT-IR).

3.2.2 Polyethylene (PE) index

The ratio of the IR absorbance of the polypropylene reference band at 973 $cm^{-1}$ to that of the polyethylene band at 719 $cm^{-1}$, obtained by FT-IR.

3.2.3 Polystyrene (PS) index

The ratio of the IR absorbance of the polypropylene reference band at 973 $cm^{-1}$ to that of the polystyrene band at 699 $cm^{-1}$, obtained by FT-IR.

3.2.4 Integrated scattering intensity

The integral of scattering intensity as a function of the scattering vector $q$ obtained from synchrotron X-ray scattering measurements.

**Note**—The scattering vector $q$ is defined by $q = \frac{4\pi}{\lambda}\sin\theta$, where $\lambda$ is the wavelength and 2θ is the scattering angle.

3.2.5 Foreign matter content

The ratio of the inorganic residue mass after TG-DTA measurement to the initial mass of the sample at the start of the test.

3.2.6 Onset decomposition temperature

The temperature at which the true sample mass (initial mass minus final residue) decreases by 1 wt% or 2.5 wt% during TG-DTA measurement.

# 4 Sample Preparation Procedures

## 4.1 Film Samples

4.1.1 Apparatus

Two types of heat-press equipment shall be used:

(1) a vacuum hot press capable of reaching temperatures sufficient to melt plastics and equipped with a vacuum chamber, and (2) a secondary heat press for crystallization treatment. Metal plates and molds suitable for forming plastic pellets or flakes into film specimens shall be used; molds made of phosphor bronze are preferred. A schematic representation of the mold assembly is shown in Figure 1.

4.1.2 Conditions for Sample Preparation

The temperatures of the vacuum hot press and the heat press for crystallization treatment shall be set to 230 °C and 80 °C, respectively.

4.1.3 Dimensions of prepared film

The film specimen shall have a width and length of 80 mm × 80 mm, and a thickness of 0.5 mm.

4.1.4 Procedure

The procedure for film preparation should be as follows.

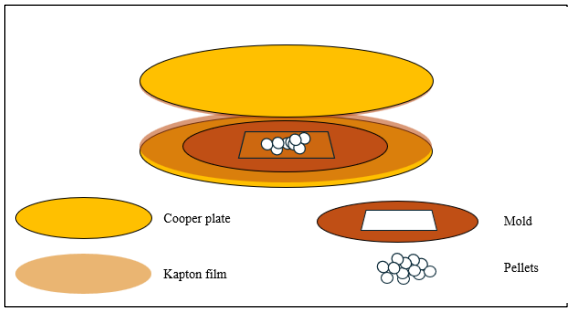


Figure 1 — Press setup during film preparation

1) The plastic pellets or flakes shall be dried in a vacuum oven maintained at 60 °C for not less than 4 h under reduced pressure.

2) The sample set shall be assembled in the order shown in Figure 1:

   copper plate – Kapton film – mold – pellets (3.2 g per film) – Kapton film – copper plate.

3) The assembled set shall be inserted into the vacuum hot press preheated to 230 °C.

   The chamber door shall be closed, and evacuation shall be initiated.

   The press gap shall be reduced to the minimum clearance such that pressure is not applied to the sample.

4) As the pellets melt and the material volume changes, the platen height shall be continuously adjusted to maintain the minimum non-contact condition.

   The vacuum pump shall remain ON until sample removal.

5) When the internal pressure reaches a vacuum level of −0.1 MPa or lower (as indicated by the sealed-type gauge), timing shall be started, and the sample shall be held for 1 min.

6) The hydraulic jack shall then be operated to apply cyclic pressure as follows:

   10 MPa → 0 MPa → 10 MPa, repeated rapidly for 30 s.

7) After the cyclic pressing, the sample shall be held at 10 MPa for 1 min, and the chamber shall then be returned to atmospheric pressure.

8) Using pliers, the sample assembly shall be transferred to the heat press preheated to 80 °C within 3 s.

   Immediately after insertion, the press gap shall be reduced and a weak pressure (≤ 1 MPa) shall be applied.

   **Note**—Any sample for which this transfer exceeds 3 s shall be discarded and the film preparation shall be repeated.

9) The sample shall be held in the 80 °C press for 10 min, removed, air-cooled, and then demolded.

# 5 Test Methods

## 5.1 Lightness (L*)

This clause specifies the method for determining the lightness of PP pellets or flakes.

### 5.1.1 Apparatus

A colorimeter capable of outputting measurement results in the CIE L*a*b* color space shall be used.

For the SOM input parameters and grading, the lightness (L*) obtained under the SCI geometry shall be used; however, the chromaticity coordinates a* and b* shall also be recorded as part of the measurement results. For acquisition of data for the Recycled Material Data Bank, a Konica Minolta CM-26dG colorimeter was used.

### 5.1.2 Test Conditions

The test shall be conducted in accordance with JIS Z 8722.

### 5.1.3 Procedure

- The pellets shall be spread uniformly in a plastic container with an internal diameter of 70 mm or larger.
- The measuring head of the colorimeter shall be placed directly on the surface of the pellets, and the measurement shall be performed without applying external pressure.

## 5.2 Melt Flow Rate (MFR)

This clause specifies the method for determining the melt mass-flow rate (MFR) of PP pellets or flakes.

### 5.2.1 Apparatus

A melt indexer compliant with JIS K 7210 shall be used. For data acquisition for the Recycled Material Data Bank, a Toyo Seiki Manufacturing Co., Ltd., Melt Indexer, Model G-02 was used.

### 5.2.2 Test Conditions

The test shall be performed using Method B. The test temperature and applied load shall be 230 °C and 2.16 kg, respectively.

### 5.2.3 Procedure

- The procedure shall be conducted in accordance with Method A of JIS K 7210.
- For each pellet or flake type, three replicate measurements shall be performed.

## 5.3 Young’s Modulus, Yield Stress, and Elongation at Break

This clause specifies the method for determining the tensile modulus, yield stress, and elongation at break of the film specimens prepared in 4.1.

5.3.1 Apparatus

A universal testing machine equipped with tensile grips shall be used. Since the maximum load during testing is typically below 50 N, a load cell with a capacity in the range of 100 N to 1 kN is recommended. For data acquisition for the Recycled Material Data Bank, a Shimadzu AGS-X universal testing machine was used.

5.3.2 Test Conditions

5.3.2.1 Test Specimens

Dumbbell-shaped specimens equivalent to Type A15 specified in JIS K 7139 shall be punched out from the film samples prepared in 4.1. Figure 3 shows the preparation of dumbbell specimens using a Thomson-type cutting die. A cutting mat shall be placed beneath the film sample and die, and a roller press shall be used to punch out the specimens. The specimen dimensions in the parallel section shall be measured at three points, and the average width and thickness shall be used to calculate the cross-sectional area. Although the dimensions may be measured with a micrometer, a non-contact dimensional measurement instrument is recommended for measuring the width.

5.3.2.2 Testing Conditions

The crosshead speed shall be 50 mm/min. Strain shall be calculated based on the displacement of the crosshead. All tests shall be conducted at room temperature (25 °C), and five measurements shall be performed for each sample type.

5.3.3 Analysis Procedure

- The determination of tensile properties—namely Young's modulus, yield stress, tensile strength at break, and elongation at break—shall be carried out in accordance with JIS K 7161-1.

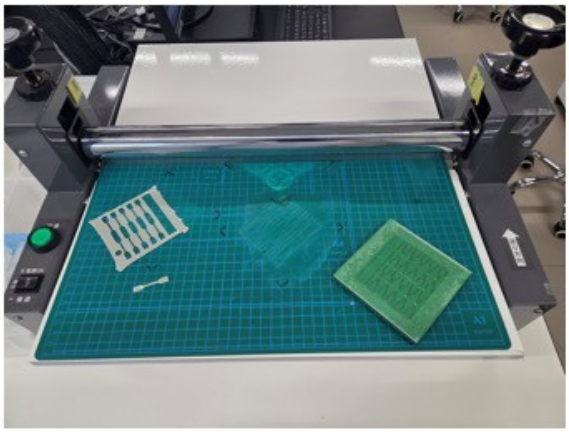

Figure 3— Preparation of dumbbell-shaped film specimens using a Thomson punch and roller press

## 5.4 Density

This clause specifies the method for determining the density of the film specimens prepared in 4.1.

5.4.1 Apparatus

An electronic balance, immersion vessel, thermometer, and a sinker (used when the density of the specimen is lower than that of the immersion liquid) compliant with Method A (immersion method) of JIS K 7112-1 shall be used. For data acquisition for the Recycled Material Data Bank, a Mettler Toledo analytical balance (Model MS105) and density measurement kit (Part No. 30553760) were used. Ultra-pure water supplied by

Fujifilm Wako Pure Chemical Corporation (Product No. 210-01303, JAN 4987481446748) was used for the immersion liquid.

5.4.2 Test Conditions

Distilled or deionized water shall be used as the immersion liquid, and the water temperature shall be maintained at 25 °C. Film specimens prepared in accordance with 4.1 shall be cut into 15 mm square pieces, and three pieces shall be prepared for each sample.

5.4.3 Procedure

- The test shall be conducted in accordance with Method A of JIS K 7112-1.
- For each sample type, three measurements shall be performed.

## 5.5 Melting Temperature and Crystallization Temperature

This clause specifies the method for determining the melting temperature and crystallization temperature of the film specimens prepared in 4.1.

5.5.1 Apparatus

A differential scanning calorimeter (DSC) compliant with JIS K 7121 shall be used. For data acquisition for the Recycled Material Data Bank, a Shimadzu DSC-60 Plus was used.

5.5.2 Temperature Calibration

Temperature calibration shall be performed in accordance with JIS K 7121.

5.5.3 Measurement Conditions

5.5.3.1 Temperature Program

The measurement shall be conducted over the temperature range of 20 °C to 200 °C. The heating and cooling rates shall both be 10 °C/min.

5.5.3.2 Atmosphere

The measurement shall be performed under a nitrogen atmosphere, with a nitrogen flow rate of 50 mL/min.

5.5.3.3 Sample Pans

Sample pans shall be made of aluminum or another material with sufficiently high thermal conductivity.

5.5.3.4 Test Specimens

Specimens shall be cut from the film prepared in 4.1 to a size that fits into the DSC pan without deformation. The specimen mass shall be approximately 8 mg.

5.5.4 Test and Analysis Procedure

- The test and subsequent analysis shall be performed in accordance with JIS K 7121.
- One measurement shall be conducted for each sample type.
- The baseline for calculating the melting enthalpy shall be defined as a linear function connecting 80 °C (lower limit) and 180 °C (upper limit). If the linear baseline differs significantly from the actual dataset, the baseline may be adjusted accordingly.
- The melting enthalpy shall be determined by area integration.

- A peak search shall be performed within this temperature range to determine the melting temperature of PP.
- When an endothermic peak appears near 100 °C, a peak search shall be performed in the 80–125 °C range to determine the melting temperature of PE.
- For the cooling curve, the baseline shall be drawn between 140 °C (upper limit) and 70 °C (lower limit). The total crystallization enthalpy and the crystallization temperature of PP shall be obtained.
- If a secondary small peak is present in addition to the PP crystallization peak, a peak search shall be performed in that local region to determine the crystallization temperature of PE.

## 5.6 PP Crystallinity Index, PE Index, and PS Index

This clause specifies the method for determining the PP crystallinity index, PE crystallinity index, PE index, and PS index of the film specimens prepared in 4.1.

### 5.6.1 Apparatus

A Fourier-transform infrared spectrophotometer (FT-IR) shall be used. For data acquisition for the Recycled Material Data Bank, a Shimadzu IRSpirit-T was used.

### 5.6.2 Measurement Conditions

Attenuated total reflectance (ATR) shall be used, employing a diamond ATR prism. Measurements shall be conducted at room temperature under the following conditions:

- Aperture: none
- Number of scans: 45
- Spectral resolution: 4 $cm^{-1}$
- Wavenumber range: 400–4600 $cm^{-1}$

Film specimens prepared in accordance with 4.1 shall be used, and five measurements shall be performed for each sample type.

### 5.6.3 Analysis Procedure

- ATR Correction: ATR spectra shall be corrected to compensate for the wavelength-dependent depth of penetration and refractive-index mismatch between the ATR crystal and the polymer specimen. The correction shall follow the general principles described in ISO 10640 (Quantitative FT-IR analysis) and ASTM E1252 (Standard Practice for General Techniques for Obtaining FT-IR Spectra), such that the corrected spectrum approximates an equivalent transmission spectrum.
- Baseline Correction: A linear baseline shall be applied. The baseline shall be defined such that the absorbance at the following wavenumbers becomes zero:

  400, 417, 494, 566, 623, 652,773, 862, 921, 1076, 1193, 1529,1602, 1911, 1999, 2479, 3676, 3957, 4600

- Absorbance Measurement: The absorbance at wavenumbers listed in Table 1 shall be recorded.

Table 1 — Absorption bands used for FT-IR analysis and their assignments
(A: amorphous band, C: crystalline band)

| Wavenumber ($cm^{-1}$) | 699 | 719 | 730 | 841 | 973 | 998 |
|---|---|---|---|---|---|---|
| Polymer species | PS | PE | PE | PP | PP | PP |
| Assignment | A | A/C | C | A/C | A/C | C |

- Index Calculation: Each crystallinity index (PP crystallinity index, PE crystallinity index, PE index, PS index) shall be calculated using the equations specified in Table 2.

**Note**: In cases where inorganic fillers such as talc or silica (glass) are present, the polypropylene (PP) absorption bands at 973 $cm^{-1}$ and 998 $cm^{-1}$ may disappear due to spectral interference. However, the PP band near 834 $cm^{-1}$, as well as the characteristic absorption bands of PE and PS, are often still observable. Therefore, when the internal reference bands at 973 $cm^{-1}$ cannot be used, the band at 843 $cm^{-1}$ shall be employed as an alternative internal reference. The PE index obtained using the 843 $cm^{-1}$ reference shall then be converted to the conventional 973 $cm^{-1}$–based PE index using the corresponding conversion equation. The calibration curve used to determine the conversion coefficient is shown in Figure 4.

Table 2 — Calculation methods for indices used in the Recycled Material Data Bank

| index | Formula | Formula (with inorganic additives) |
|---|---|---|
| PP crystallinity | $A_{998}\,\mathrm{cm}^{-1}/A_{973}\,\mathrm{cm}^{-1}$ | Not possible |
| PE crystallinity | $A_{730}\,\mathrm{cm}^{-1}/A_{719}\,\mathrm{cm}^{-1}$ | $A_{730}\,\mathrm{cm}^{-1}/A_{719}\,\mathrm{cm}^{-1}$ |
| PE amount | $A_{719}\,\mathrm{cm}^{-1}/A_{973}\,\mathrm{cm}^{-1}$ | $A_{719}\,\mathrm{cm}^{-1}/A_{843}\,\mathrm{cm}^{-1} \times 0.6949$ |
| PS amount | $A_{699}\,\mathrm{cm}^{-1}/A_{973}\,\mathrm{cm}^{-1}$ | $A_{699}\,\mathrm{cm}^{-1}/A_{843}\,\mathrm{cm}^{-1} \times 0.6616$ |

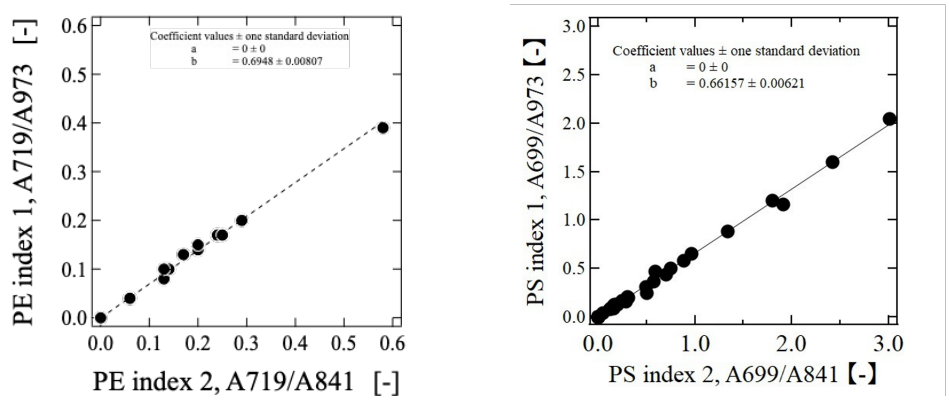


Figure 4 — Correlation of PE (left) and PS (right) indices obtained using different internal reference bands

### 5.7 Small-Angle X-Ray Scattering (SAXS) Integrated Intensity

This clause specifies the method for determining the SAXS integrated scattering intensity of the film specimens prepared in 4.1.

5.7.1 Apparatus

For data acquisition for the Recycled Material Data Bank, measurements were performed using the 3-GeV high-brilliance synchrotron radiation facility NanoTerasu, beamline BL08-SAXS.

5.7.2 Measurement Conditions

- X-ray wavelength: 1.54 Å
- Camera length: 1600 mm
- Exposure time: 10 s

5.7.3 Measurement and Analysis Procedure

- Film specimens prepared in accordance with 4.1 shall be cut into 7-mm-diameter circular samples. Five such specimens shall be prepared for each material.
- For calibration of the integrated scattering intensity ratio used in the Recycled Material Data Bank, five reference specimens of FY-4, supplied by Japan Polychem, shall be prepared and measured on the same day under identical measurement conditions.
- From each SAXS profile, the integrated intensity shall be calculated over the scattering-vector range: $0.05 < q < 1.0\ \mathrm{nm}^{-1}$
- The SAXS integrated scattering intensity shall be defined as the ratio of the integrated intensity of the sample to that of FY-4. Both the ratio and the coefficient of variation (CV) shall be recorded in the Recycled Material Data Bank.
- Corrections shall be applied for instrumental background, including air scattering and dark current.However, for the BL08W-SAXS beamline of NanoTerasu, which operates under full vacuum, air-scattering contributions are negligible and may be disregarded.

## 5.8 Foreign Matter Content and Onset Decomposition Temperature

5.8.1 Apparatus

A simultaneous thermogravimetric–differential thermal analysis instrument (TG–DTA) compliant with JIS K 7120 shall be used. For data acquisition for the Recycled Material Data Bank, a Shimadzu DTG-60A was used.

5.8.2 Temperature Calibration

Temperature calibration shall be performed in accordance with JIS K 7121.

5.8.3 Measurement Conditions

5.8.3.1 Temperature Program

The measurement shall be conducted in the temperature range of 40 °C to 500 °C, with both heating and cooling rates set to 10 °C/min.

5.8.3.2 Atmosphere

The measurement shall be performed under a nitrogen atmosphere, with a flow rate of 100 mL/min.

5.8.3.3 Sample Pans

Sample pans shall be made of aluminum or another material with sufficiently high thermal conductivity.

5.8.3.4 Test Specimens

Specimens shall be cut from the film samples prepared in 4.1 to a size that fits in the TG–DTA pan without deformation. The specimen mass shall be approximately 12 mg.

5.8.4 Test and Analysis Procedure

The test and analysis shall be carried out in accordance with JIS K 7120.

- Foreign matter content

  The foreign matter content shall be calculated as: $\text{Foreign matter content} = \dfrac{\text{residual mass at } 500\,^{\circ}\text{C}}{\text{initial sample mass}}$

- Onset decomposition temperature

  The onset decomposition temperature shall be defined as the temperature at which the true sample mass (initial mass minus final residue) decreases by 1 wt% and 2.5 wt%, respectively.

## 5.9 Charpy Impact Test

This clause specifies the method for determining the Charpy impact strength of the injection-molded specimens prepared in 4.2.

5.9.1 Apparatus

A notching tool and an impact tester compliant with JIS K 7111 shall be used. For data acquisition for the Recycled Material Data Bank, a Toyo Seiki Notching Tool A-4E and a Toyo Seiki Impact Tester (Type IT) were used.

5.9.2 Test Conditions

A V-notch (r = 0.25 mm) shall be machined into each specimen, and the test shall be conducted in accordance with JIS K 7111. The pendulum shall be selected such that the absorbed energy upon fracture falls within 10–80% of the pendulum capacity. Specimen dimensions shall be measured using a micrometer, and the remaining ligament at the notch shall be measured using an optical microscope. The test temperature shall be room temperature.

5.9.3 Procedure

- The test procedure shall follow JIS K 7111.
- For each sample type, five measurements shall be performed.

## 5.10 Flexural Test

This clause specifies the method for determining the flexural modulus and flexural strength of the injection-molded specimens prepared in 4.2.

5.10.1 Apparatus

A universal testing machine and three-point bending fixture compliant with JIS K 7171 shall be used. For data acquisition for the Recycled Material Data Bank, a Shimadzu AGS-X universal testing machine was used.

5.10.2 Test Conditions

The test shall be conducted in accordance with JIS K 7171.

5.10.3 Test Procedure

The procedure shall follow JIS K 7171, and five measurements shall be performed for each sample type.

- Measurement of specimen dimensions

  Thickness: Measured using a micrometer at nine locations: three locations near the center in the longitudinal direction, plus measurements at the thickest (ends) and thinnest (center) regions. The average of the nine values shall be taken as the specimen thickness.

  Width: Measured using a micrometer at two locations near the center of the specimen. The average value shall be taken as the specimen width.

- Determination of flexural modulus

  The flexural modulus shall be calculated from the slope of the stress–strain curve obtained under the initial crosshead speed of 2 mm/min, up to a strain of 0.3%. The modulus shall be determined from the linear region corresponding to the strain range of: $0.05\% \leq \varepsilon \leq 0.25\%$

- Determination of flexural strength

  Flexural strength shall be determined using the data obtained under the later-stage bending speed of 10 mm/min. The stress at a deflection of 6 mm shall be taken as the specified-deflection flexural stress, and this value shall be reported as the flexural strength.

# 6 Reporting Requirements

The test report shall include the items specified for each test method.

6.1 Color Measurement

a) All available information required to identify the test material (material type, supplier, production lot number, and material history)

b) Date and time of measurement

c) Measuring instrument

d) Test results: **L*, a*, b* values** obtained in SCI modes

6.2 Melt Mass-Flow Rate (MFR)

a) All available information required to identify the test material (material type, supplier, production lot number, and material history)

b) Date and time of measurement

c) Measuring instrument

d) Test results: **Average melt mass-flow rate**

6.3 Tensile Test

a) All available information required to identify the test material

b) Date and time of measurement

c) Measuring instrument

d) Test results:

— **Tensile modulus (average and coefficient of variation, CV)**

— **Yield stress (average and CV)**

— **Fracture strain (average and CV)**

6.4 Density Measurement

a) All available information required to identify the test material

b) Date and time of measurement

c) Measuring instrument

d) Test results: **Density (average and CV)**

6.5 Differential Scanning Calorimetry (DSC)

a) All available information required to identify the test material

b) Date and time of measurement

c) Measuring instrument

d) Mass of test specimen

e) Test results:

— **PP melting temperature**

**— PE melting temperature (if present)**

**— Total melting enthalpy**

**— PP crystallization temperature**

**— PE crystallization temperature**

**— Total crystallization enthalpy**

6.6 FT-IR Measurement

a) All available information required to identify the test material

b) Date and time of measurement

c) Measuring instrument

d) Test results:

**— PP crystallinity index**

**— PE crystallinity index**

**— PE index**

**— PS index**

6.7 SAXS Integrated Scattering Intensity

a) All available information required to identify the test material

b) Date and time of measurement

c) Measuring instrument

d) Test results:

**— Integrated scattering-intensity ratio (average and CV)**

6.8 TG–DTA

a) All available information required to identify the test material

b) Date and time of measurement

c) Measuring instrument

d) Mass of test specimen

e) Test results:

**— Foreign matter content**

**— Onset decomposition temperatures corresponding to 1% and 2.5% mass loss**

6.9 Charpy Impact Test

a) All available information required to identify the test material

b) Date and time of measurement

c) Measuring instrument

d) Specimen dimensions: shape, remaining ligament at the notch, thickness, and length

e) Nominal pendulum energy

f) Test results (summarized for each individual specimen):

1) Fracture mode, classified into the three standard categories:

C: complete break (including hinge break, H), P: partial break, N: non-break

2) **Charpy impact strength (average and CV),** reported only when the predominant fracture mode is C or P

6.10 Flexural Test

a) All available information required to identify the test material

b) Date and time of measurement

c) Measuring instrument

d) Specimen dimensions: shape, width, thickness, and length

e) Test results:

— **Flexural modulus (average and CV)**

— **Flexural strength (average and CV)**