BS ISO 18263-2:2015



BSI Standards Publication

Plastics — Mixtures of polypropylene (PP) and polyethylene (PE) recyclate derived from PP and PE used for flexible and rigid consumer packaging

Part 2: Preparation of test specimens and determination of properties



BS ISO 18263-2:2015

National foreword

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Plastics — Mixtures of polypropylene (PP) and polyethylene (PE) recyclate derived from PP and PE used for flexible and rigid consumer packaging —

Part 2:

Preparation of test specimens and determination of properties

Plastiques — Recyclat mixte de polypropylène (PP) et polyéthylène (PE) —

Partie 2: Préparation des éprouvettes et détermination des propriétés



BS ISO 18263-2:2015 **ISO 18263-2:2015(E)**



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

ISO 18263 consists of the following parts, under the general title *Plastics* — *Mixtures of polypropylene* (*PP*) and polyethylene (*PE*) recyclate derived from *PP* and *PE* used for flexible and rigid consumer packaging:

- Part 1: Designation system and basis for specification
- Part 2: Preparation of test specimens and determination of properties

Plastics — Mixtures of polypropylene (PP) and polyethylene (PE) recyclate derived from PP and PE used for flexible and rigid consumer packaging —

Part 2:

Preparation of test specimens and determination of properties

1 Scope

This part of ISO 18263 specifies methods of preparation of test specimens and the standard test methods to be used in determining the properties of mixtures of polypropylene (PP) and polyethylene (PE) recyclate derived from PP and PE used for flexible and rigid consumer packaging for moulding and extrusion. Recyclate from packaging used for the transport, handling, or storage of hazardous goods is not to be used. Since it is impossible to get the information about stabilizer levels of recyclate, it is necessary to add stabilizer as needed. Requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing are given in this part of ISO 18263.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 178, Plastics — Determination of flexural properties

ISO 179-1, Plastics — Determination of Charpy impact properties — Part 1: Non-instrumented impact test

ISO 179-2, Plastics — Determination of Charpy impact properties — Part 2: Instrumented impact test

ISO 291, Plastics — Standard atmospheres for conditioning and testing

ISO 472, Plastics — Vocabulary

ISO 527-1, Plastics — Determination of tensile properties — Part 1: General principles

ISO 527-2, Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics

ISO 1133-1, Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method

ISO 1183-1, Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pyknometer method and titration method

ISO 1183-2, Plastics — Methods for determining the density of non-cellular plastics — Part 2: Density gradient column method

ISO 1183-3, Plastics — Methods for determining the density of non-cellular plastics — Part 3: Gas pyknometer method

ISO 1873-2, Plastics — Polypropylene (PP) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties

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ISO 10350-1, Plastics — Acquisition and presentation of comparable single-point data — Part 1: Moulding materials

ISO 15270, Plastics — Guidelines for the recovery and recycling of plastics waste

ISO 18263-1, Plastics — Mixtures of polypropylene (PP) and Polyethylene (PE) recyclate derived from PP and PE used for flexible and rigid consumer packaging — Part 1: Designation system and basis for specifications

ISO 20753, Plastics — Test specimens

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 15270 apply.

4 Preparation of test specimens

The test specimens shall be prepared in accordance with ISO 1873-2 and shall be tested in accordance with each test method indicated in <u>Table 1</u>. It is essential that specimens are always prepared by the same procedure using the same processing conditions.

5 Conditioning of test specimens

Test specimens for the determination of mechanical properties and density shall be conditioned in accordance with ISO 291 for 40 h to 96 h at 23 $^{\circ}$ C \pm 2 $^{\circ}$ C.

6 Determination of ratio of PP and PE

The ratio of PP and PE shall be determined in accordance with traceable documents from the supplier.

NOTE In regard to the procedures etc. needed for the traceable documents on the recycled plastics, it is recommended to use EN 15343 or ISO/IEC 17050-1 and ISO/IEC 17050-2 as references. It is known that the ratio of PP and PE can be determined by FTIR spectrometer, but sufficient precision data, especially including other materials, is not yet available (see Annex A).

7 Determination of properties

In the determination of properties and the presentation of data, the standards, supplementary instructions, and notes given in ISO 10350-1 shall be applied. All tests shall be carried out in the standard atmosphere of 23 $^{\circ}$ C \pm 2 $^{\circ}$ C, unless specifically stated otherwise in Table 1 and Table 2.

Table 1 is compiled from ISO 10350-1 and the properties listed are those which are appropriate for the mixtures of PP and PE recyclate for moulding and extrusion materials. These properties are those considered useful for comparisons of data generated for different thermoplastics.

<u>Table 2</u> contains those properties which are in wide use or of particular significance in the practical characterization of mixtures PP and PE recyclate for moulding and extrusion materials.

NOTE This recyclate is derived from PP and PE used for flexible and rigid consumer packaging for moulding and extrusion. Therefore, there is no information about a stabilizer of recyclate and it may be necessary to add a stabilizer as needed.

Table 1 — General properties and test conditions (selected from ISO 10350-1)

Property	Unit	Standard	Specimen type (dimensions in mm)	Test conditions and supplementary instruction				
Rheological propertion	Rheological properties							
Melt mass-flow rate (MFR)	g/10 min	ISO 1133-1	Moulding compound	Temperature 230 °C, load 2,16 kg				
Mechanical propertie	es							
Tensile yield stress	MPa		Injection-moulded	Test speed 50 mm/min				
Tensile stress at break	%	ISO 527-1 ISO 527-2	type A1 test specimen as defined in ISO 20753	Test speed 5 mm/min Only to be quoted if strain at break is under 10 %				
Flexural modulus Flexural strength	MPa MPa	ISO 178	Injection-moulded type B2 test specimen as defined in ISO 20753 (80 × 10 × 4)	Test speed 2 mm/min				
Charpy unnotched impact strength		ISO 179-1 ISO 179-2	Injection-moulded type B2 test specimen	Edgewise impact				
Charpy notched impact strength	kJ/m ²		as defined in ISO 20753 (80 × 10 × 4)	Also record type of failure (see ISO 179-1)				

 $Table\ 2-Additional\ properties\ and\ test\ condition$

Property	Unit	Standard	Specimen type	Test conditions and supplementary instruction					
Basic properties	Basic properties								
PP/PE composition (by mass)	%	ISO 18263-1	Moulding compound	See <u>Clause 6</u>					
Density	g/cm ³	ISO 1183-1, ISO 1183-2, or ISO 1183-3	Strand after measured MFR	The methods specified in ISO 1183-1, ISO 1183-2 and ISO 1183-3 are regarded as equivalent for the purposes of this part of ISO 18263					
Colour		ISO 18263-1	Moulding compound	Visual inspection specified in ISO 18263-1					
Form		ISO 18263-1	Moulding compound	Visual inspection specified in ISO 18263-1					

Annex A

(informative)

Determination of the ratio of PP and PE by the FTIR spectrometer

A.1 General

This annex shows the relationship between the ratio of IR absorbance at 1 370 cm⁻¹ and 1 464 cm⁻¹ and a PP and PE blend ratio of the mixture of virgin PP and PE obtained by using FTIR. This relationship is useful for presuming the ratio of PP and PE in a recyclate.

NOTE Since the recyclate included a PP/PE copolymer etc., the PP/PE ratio calculated from this relationship is just a reference and it cannot be used for deciding the composition indicated in <u>Table 2</u>.

A.2 Samples

The samples were blended by test roll machine under about 200 °C.

PE-LLD: MFR = 1.0 g/10 min, density = 0.926 g/cm^3

PP (Homo-PP): MFR = 7.7 g/10 min

Table A.1 — Prepared samples

mass %

	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	No. 11
PE	100	95	90	85	75	50	25	15	10	5	0
PP	0	5	10	15	25	50	75	85	90	95	100

A.3 IR measurements

All samples were measured by the FTIR with the following condition.

<Conditions>

ATR method: Using Zn-Se crystal

Wave number regions: 4 000 - 650 cm⁻¹

Cumulated number: 64 times

Resolution: 4 cm⁻¹

A.4 Result

Plot the ratio of A (1 370 cm⁻¹)/A (1 464 cm⁻¹) against the ratio of PP and PE (see Figure A.1).

A.5 Estimation of ratio of PP and PE

Calculate the ratio of PP and PE using the formula in Figure A.1.

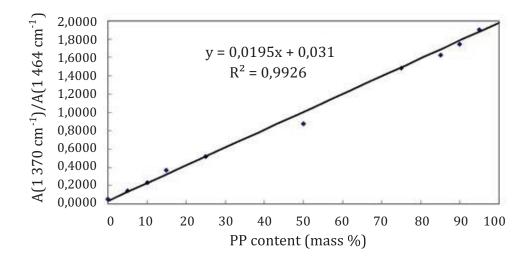


Figure A.1 — Relationship between A (1 370 cm $^{-1}$)/A (1 464 cm $^{-1}$) and PP content for PP and PE blend

Annex B

(informative)

The properties of the PP and PE mixture

B.1 General

This annex shows the relationship between PP/PE blend ratio of mixture and tensile properties (strength and elongation) or morphology using mixture of virgin PP and PE. From this diagram, it is useful for estimation of the tensile properties of a recyclate. This annex shows the relationships schematically. Therefore, these data shall not be used for calculating the composition of the recyclates.

B.2 Preparation of samples

PP and PE were melt blended in a laboratory twin screw type batch mixer at the compositions 0 %, 30 %, 50 %, 70 %, and 100 % of PE by mass. Sheets of 150 mm \times 150 mm \times 0,5 mm were prepared by compression moulding.

<Moulding conditions>

Pre heat: 220 °C, 5 min

Press: 220 °C, 10 MPa, 5 min

Cooling: 30 °C, 10 MPa, 5 min

<Materials>

PE-HD: MFR = 0,36 g/10 min (at 190 °C × 2,16 kg), density = 0,956 g/cm³

PP: MFR = 3.1 g/10 min (at 230 °C × 2.16 kg), Homo-PP

Table B.1 — Prepared samples

mass %

No	1	2	2	4	_	
Materials	1		3	4	3	
PE	100	70	50	30	0	
PP	0	30	50	70	100	

The tensile test specimens, ISO 527-3 Type 2 (width = 15 mm), were prepared by cutting from each blend sheet. The specimens for tensile test were conditioning at (23 ± 2) °C for at least 4 h.

B.3 Tensile test

B.3.1 Test conditions

Test speed: 50 mm/min

Initial distance between grips: 100 mm

Test condition: (23 ± 2) °C

B.3.2 Results

The results are shown in Table B.2.

Table B.2 — Results of tensile test

Item	Tensile yield stress	Tensile stress at break	Tensile strain at break
Samples (PE/PP)	MPa	MPa	%
No 1 (100/0)	24,2	18,0	1 000
No 2 (70/30)	27,0	25,9	130
No 3 (50/50)	28,9	27,6	25
No 4 (30/70)	30,9	23,1	300
No 5 (0/100)	30,2	32,4	2 500

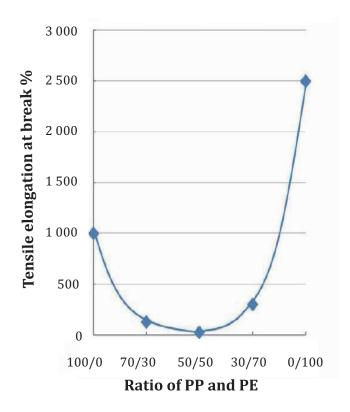


Figure B.1 — Ratio of PP and PE VS tensile elongation at break

According to Reference [5], the same tensile strain tendency is shown.

B.4 Transmission Electron Microscope (TEM) observation

The samples were observed by TEM after all samples stained with ruthenium tetrachloride.

B.4.1 Results

PP and PE = 70/30 and PP and PE = 50/50 samples have a typical sea (PP) island (PE) structure. From the result, the much amount of PE or PP have better compatibility than equal amount of PE and PP.

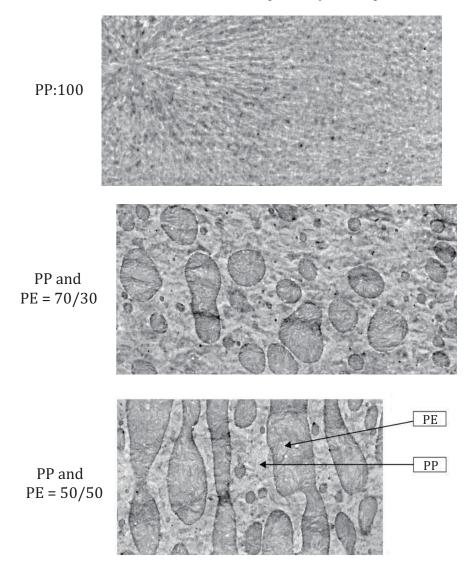


Figure B.2 — Electron micrographs of PP sheet and PP and PE blends sheets

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- [1] ISO 527-3, Plastics Determination of tensile properties Part 3: Test conditions for films and sheets
- [2] ISO/IEC 17050-1, Conformity assessment Supplier's declaration of conformity Part 1: General requirements
- [3] ISO/IEC 17050-2, Conformity assessment Supplier's declaration of conformity Part 2: Supporting documentation
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- [5] NAKAMURA, S., TOKUMITSU, K., KITAMURA, M., MIYAGAWA, E., KANZAWA T. AND TANAKA, A. Morphology and Mechanical Properties of PE/PP blends with Compatibilizer. *Resources processing.* 2008, 55(2), 56–65





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