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Plastics — Thermoplastic polyurethanes for moulding and extrusion —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Polyuréthannes thermoplastiques pour moulage et extrusion —

Partie 2: Préparation des éprouvettes et détermination des propriétés

Reference number
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Foreword

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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 16365 consists of the following parts, under the general title *Plastics — Thermoplastic polyurethanes for moulding and extrusion*:

— *Part 1: Designation system and basis for specifications*

— *Part 2: Preparation of test specimens and determination of properties*

— *Part 3: Distinction between ether and ester polyurethanes by determination of the ester group content*

Introduction

Thermoplastic elastomer materials are classified into groups by the primary elastomeric property Hardness and with this as result of some relation with modulus, as shown in Figure 1. The classification on basis of hardness considers the special position of TPE's between the rubber materials on the one side and the plastic materials on the other.

Each class is subdivided in standard properties and special properties. The standard properties for a class are not only connected with the adjacent group(s) by many of the specified properties but also by special properties specified in the adjacent class(es). A standard property in a class can be a special property in an adjacent class and vice versa.

Special properties are those properties which are in wide use or of particular significance in the practical characterization of a specific material.

For each type of thermoplastic elastomer, refer to the relevant material standard.

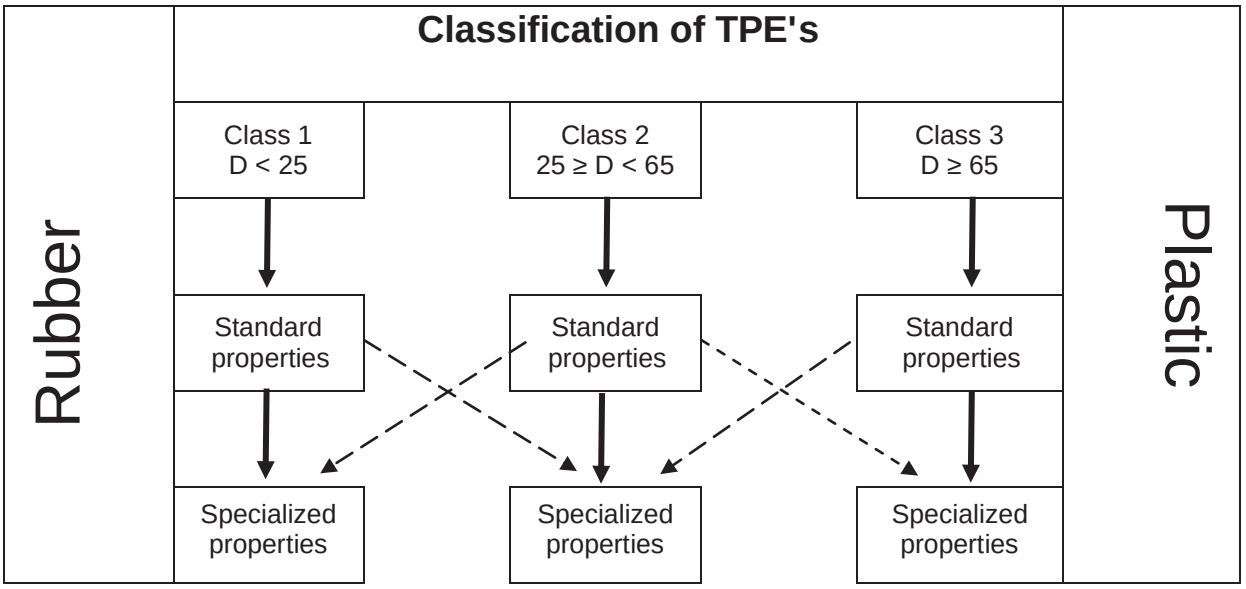

Figure 1 — Structure of thermoplastic elastomer (TPE) material standards

Plastics — Thermoplastic polyurethanes for moulding and extrusion —

Part 2: Preparation of test specimens and determination of properties

1 Scope

This part of ISO 16365 specifies the methods of preparation of test specimens and the standard test methods to be used in determining the properties of thermoplastic polyurethane moulding and extrusion materials. Requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing are also given.

Procedures and conditions for the preparation of test specimens in a specified state and procedures for measuring properties of the materials from which these specimens are given. Properties and test methods which are suitable and necessary to characterize thermoplastic polyester/polyurethane and polyether/polyurethane (TPU) moulding and extrusion materials, are listed.

The properties have been selected from the general test methods in ISO 10350. Other test methods in wide use or of particular significance to these moulding and extrusion materials are also included in this part of ISO 16365, as are the designatory properties specified in ISO 16365-1.

In order to obtain reproducible and comparable test results, it is necessary to use the methods of preparation and conditioning, the specimen dimensions, and the test procedures specified in this part of ISO 16365. Values determined are not necessarily identical to those obtained using specimens of different dimensions or prepared using different procedures.

NOTE This part of ISO 16365 has been developed on the basis of ISO 10350-1 as at the time of publication a standard on 'acquisition and presentation of comparable single point data' for thermoplastic elastomers' does not exist yet. After acceptance and publication of this part of ISO 16365, it is the intension to develop ISO 10350-3, based on the two International Standards mentioned before, as starting point for the development of thermoplastic elastomer material standards.

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 34-1, *Rubber, vulcanized or thermoplastic — Determination of tear strength — Part 1: Trouser, angle and crescent test pieces*

ISO 37, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain 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 294-1, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens*

ISO 294-3, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates*

ISO 294-4, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 4: Determination of moulding shrinkage*

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 815, (all parts), *Rubber, vulcanized or thermoplastic — Determination of compression set*

ISO 846, *Plastics — Evaluation of the action of microorganisms*

ISO 868, *Plastics and ebonite — Determination of indentation hardness by means of a durometer (Shore hardness)*

ISO 1133-2, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 2: Method for materials sensitive to time-temperature history and/or moisture*

ISO 1183 (all parts), *Plastics-Methods for determining the density and relative density of non-cellular plastics*

ISO 4649, *Rubber, vulcanized or thermoplastic — Determination of abrasion resistance using a rotating cylindrical drum device*

ISO 15512, *Plastics — Determination of water content*

ISO 16365-1, *Plastics — Thermoplastic polyurethanes for moulding and extrusion — Part 1: Designation system and basis for specifications*

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

IEC 60093, *Methods of test for volume resistivity and surface resistivity of solid electrical insulation materials*

IEC 60112, *Method for determining of the proof and the comparative and the proof tracking indices of solid insulation materials under moist conditions*

IEC 60243-1, *Electric strength of insulating materials-Test methods-Part 1:Tests at power frequencies*

IEC 60250, *Recommended methods for the determination of the permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelengths*

IEC 60695-11-10, *Fire hazard testing — Part 11-10 Flammability—11-10: Test flames — 50 W horizontal and vertical flame*

3 Preparation of test specimens

3.1 Treatment of the material before moulding

The granules/moulding compound shall have reached room temperature and the moisture content shall not exceed 0,02 % (m/m).

For drying a dehumidified air dryer is recommended, but an oven with circulating air can also be used provided that the drying temperature is increased by 20 °C. For a dehumidified air dryer 3 h at 100 °C might be sufficient, but for several products longer times up to 1 day and a temperature of 110 °C can be used for drying without checking the water content for each sample, if proven to give a moisture content < 0,02 % (m/m). If colour master batches, pigments, or other additives are added, it

is recommended to prepare a premix with the granules before drying. If the material tends to become sticky it might be necessary to choose a lower temperature in order to be able to handle the material.

The water content can be measured after evaporation (see ISO 62 and ISO 10101) or extraction (see ASTM E1064 and ISO 12937), of the water from the sample using solvents like water-free alcohols and subsequent water determination e.g. by Karl Fisher titration, ISO 15512 or any other suitable method to determine moisture content in the range of 0,02 % (m/m) accurately. The moisture content of filled or reinforced materials shall be expressed as a percentage of the total mass of the compound.

To ensure that the moisture content remains low, it is recommended that the sample material in the feed hopper of the injection-moulding machine be blanketed with a suitable gas (dried air, nitrogen, or argon, for example). Better results can be obtained using a dehumidifier hopper drier. Drying for the preparation of test specimen is recommended if the container has been opened several times after delivery of dry product or drying.

Excessive moisture content in the granule can lead to degradation during preparation of test specimen and thus to wrong results. Foaming and formation of gas bubbles are indications that the moisture content is excessively high.

Thermoplastic polyurethanes absorb moisture from air. Storage of granules in dry conditions is therefore recommended before processing. In order to prevent condensation on the granule if storing at low temperatures, material should be brought to room temperature before opening the container. Containers should be tightly closed after use. The granules should be exposed to the surrounding air only as long as absolutely essential.

3.2 Injection moulding

Injection-moulded specimens shall be prepared in accordance with ISO 294-1, using the conditions specified in Table 1. The specimens shall be prepared by injection moulding from dry granules. Test specimen can be cut out from the sample plates using cutting blades.

It is essential that specimens are always prepared by the same procedure using the same processing conditions. The material shall be kept in moisture-proof containers until it is required for use.

Table 1 — Conditions for injection moulding of test specimens

Material Shore D hardness	Heating zone temperature °C	Nozzle temperature °C	Melt temperature °C
Soft: D < 25	200–220	210–230	205–225
Medium: 25 ≥ D < 65	205–225	215–235	210–230
Rigid: D ≥ 65	210–230	220–240	215–235
Injection pressure: 10–100 MPa Holding pressure: 10–100 MPa Back pressure: 0,5–2 MPa			

The processing parameters and temperatures are chosen in a way to minimize differences of mechanical properties in flow direction and crosswise. If the differences are higher than 5 % (mean value) the direction needs be indicated together with the test results.

The flow behaviour of the melt is important for optimizing the processing parameters. The melt flow rate/melt volume rate, using the specified temperature and load in Table 3 can be used for determination of differences in melt flow behaviour (see ISO 1133-2). Only MFR/MVR data measured under exactly the same conditions are comparable. Load and temperatures are chosen in a way to obtain a MVR in the range between 5 cm^3/10 min and 100 cm^3/10 min, but preferably between 10 cm^3/10 min and 40 cm^3/10 min.

4 Conditioning of test specimens

Moulded parts made of thermoplastic polyurethane require several weeks of storage at room temperature to attain full mechanical properties. To achieve optimal functional properties in a shorter period, annealing of the finished parts or test specimens is necessary. The recommended duration and temperature of annealing is 20 h at 100 °C. The annealing treatment can be carried out in a circulating air oven immediately after moulding. The tests can be carried out after the test specimen have been stored 24 h at 23 °C and 50 % relative humidity (standard atmosphere).

NOTE During annealing articles or test specimen with low dimensional stability should be stored in a way that deformation is avoided.

5 Determination of properties

5.1 General

In order to obtain reproducible and comparable test results, it is necessary to use the methods of preparation and conditioning, the specimen dimensions and the test procedures specified in this part of ISO 16365. Values determined will not necessarily be identical to those obtained using specimens of different dimensions or prepared using different procedures.

All tests shall be carried out in the standard atmosphere of (23 ± 2) °C and (50 ± 10) % relative humidity unless specifically stated otherwise in Tables 2 and 3.

Table 2 is compiled from ISO 10350-1 (see Clause 1) and the properties listed are those, which are appropriate to thermoplastic polyester/ester and polyether/ester moulding and extrusion materials. These properties are those considered useful for comparisons of data generated for different thermoplastic elastomers.

Tables 3 and 5 contain those properties that are considered to be standard properties for the relevant hardness class. Tables 4 and 6 contain those properties that are considered a special property for that relevant hardness class, in wide use and/or of particular significance in the practical characterization of the thermoplastic elastomer moulding and extrusion material (see Introduction).

Tables 3 and 4 comprise the properties for both hardness classes 1 and 2, up to and including shore D ≤ 65. Tables 5 and 6 comprise the properties for the hardness class 3, Shore D > 65.

Thermoplastic polyurethanes are used for very different applications. Therefore, type and required values can differ considerably depending on the specific application.

As an example, the following data can be represented by curves or sets of values not by single values.

Content of legally restricted substances, rigidity, torsion behaviour, tear strength, long-term behaviour, creep behaviour, thermal expansion, low-temperature behaviour, softening temperatures, aging, thermal stability, flame classes and burning behaviour, influence of solvents such as swelling, chemical stability, hydrolysis, volatile matter or fogging, electrical properties, undesired substances for a certain application, mould flow behaviour, crystallization behaviour, permeability, etc.

5.2 Biological resistance

The resistance of polyurethanes against microorganisms is dependent on the content of ester bonds in TPU and chemical structure.

For evaluation of the stability against microorganisms ISO 846-Method D (biologically active earth) can be used.

NOTE Since this test takes several months it can be substituted by analysing the content of ester bonds in accordance with ISO 16365-3.

5.3 TPU properties and test conditions

Table 2 — Overview of standard properties and special properties for the characterization of thermoplastic polyurethane materials

Properties	Test method	Shore D ≤ 25		25 < Shore D ≤ 65		Shore D > 65	
Rheological		Standard	Special	Standard	Special	Standard	Special
MVR/MFR	ISO 1133-2	X		X		X	
Mechanical		Standard	Special	Standard	Special	Standard	Special
Hardness, shore A, D	ISO 868	X		X		X	
Tensile modulus	ISO 527-1/ ISO 527-2	X		X		X	
Tensile stress							
at 20 %, 100 %, 300 % strain		X		X		X	
at > 300 % strain			X		X		X
Stress at break		X				X	
Stress at break/hot water treatment			X		X		X
Strain at break		X		X			X
Strain at break/hot water treatment			X		X		X
Tensile creep modulus	ISO 899-1		X		X		X
Charpy notched impact strength	ISO 179		X		X		X
Tear strength	ISO 34-1; B/b	X		X			X
Compression set	ISO 815	X		X			X
Abrasion loss	ISO 4649A	X		X			X
Biological resistance	ISO 846		X		X		X
Thermal		Standard	Special	Standard	Special	Standard	Special
Glass transition temperature	ISO 11357-1 and ISO 11357-2		X		X	X	
Burning behaviour	IEC 60695–11–10		X		X		X
Electrical		Standard	Special	Standard	Special	Standard	Special
Surface resistivity	IEC 60093		X		X		X
Relative permittivity	IEC 60250		X		X		X
Volume resistivity	IEC 60093		X		X		X
Dissipation factor tan	IEC 60250		X		X		X
Dielectrical strength	IEC 60243–1		X		X		X
Comparative tracking index CTI	IEC 60112		X		X		X
Other		Standard	Special	Standard	Special	Standard	Special
Density	ISO 1183	X		X		X	
Moisture content	ISO 15512	X		X		X	

Table 3 — Standard properties and test conditions[a] — Shore hardness classification D ≤ 65

Property	Units	Standard	Specimen type mm	Specimen preparation[a]	Test conditions and supplementary conditions
Rheological properties					
MFR/MVR	g/10 min/ cm^3/10 min	ISO 1133–2	Moulding compound	Dry, moisture content < 0,03 % (m/m)	Load 2,16 kg, 5 kg, 10 kg, 21,6 kg T-melt + 10 °C = T-MFR
Mechanical properties					
Hardness	Shore D	ISO 868	≥ 80 × ≥ 10 × ≥ 6	M	5 measurements ≥ 9 mm from any edge, ≥ 6mm apart. 3 specimens of 2 mm stacked
Tensile modulus	MPa	ISO 527-2	1A (equal ISO 3167A)	M	Speed 1 mm/min
Tensile stress at x % elongation	MPa		1BA or 5A thickness 2 mm	M	20 %, 100 %, 300 % elongation
Stress at break	MPa			M	Speed 200 mm/min
Strain at break	%			M	Speed 200 mm/min
Tear strength	kN/m	ISO 34-1	ISO 31-1 method B procedure A	M	Speed 500 mm/min
Compression set	%	ISO 815	Ø 13 mm × 6mm	M	23°C, 70°C
Abrasion loss	mm^3	ISO 4649 A	Thickness 6 mm	M	
Other properties					
Density	g/cm^3	ISO 1183			
Moisture content	%	ISO 15512	Granules/moulding compound	Before and/or after drying	

[a] M: Injection moulding

Table 4 — Specialized properties and test conditions — Shore hardness classification D ≤ 65

Property	Unit	Standard	Specimen type mm	Specimen preparation[a]	Test conditions and supplementary instructions
Mechanical properties					
Tensile stress at > 300 % strain	MPa	ISO 37 or ISO 527-2	Type to be specified and documented	M	To be agreed between stakeholders. Elongation and applied standard to be specified and documented
Tensile creep	MPa	ISO 899	ISO 3167A	M	To be agreed between customer and supplier. Times to be specified and documented
Charpy notched Impact Strength	kJ/m^2	ISO 179	80 × 10 × 4 V-notch r=1	M	Method 1eA, edgewise impact
Biological resistance		ISO 846 variant D and ISO 527-2	ISO 527-2 type 1BA Thickness 2 mm	M	Tensile test for comparison purposes for ester containing TPU, but not needed for 100 % TPU-ARET or TPU-ALET
Thermal properties					
Glass transition temperature		ISO 11357-1 and ISO 11357-2	Moulding compound	M	Record midpoint temperature Use 10 °C/min
Burning behaviour		IEC 60695–11–10	125 × 13 × 3	M	0,91 mm, 3,0 mm
Electrical properties					
Relative permittivity		IEC 60250	≥ 60 × ≥ 60 × 2	M	100 Hz, 1 MHz
Dissipation factor tan		IEC 60250		M	100 Hz, 1 MHz
Volume resistivity	Ω m	IEC 60093		M	
Surface restivity	Ω	IEC 60093		M	
Dielectrical strength	kV/mm	IEC 60243–1	≥ 60 × ≥ 60 × 1	M	
Comparative tracking index CTI	V	IEC 60112	≥ 20 × ≥ 20 × 4	M	

[a] M: Injection moulding

Table 5 — Standard properties and test conditions[a] — Shore hardness classification D > 65

Property	Units	Standard	Specimen type mm	Specimen preparation[a]	Test conditions and supplementary conditions
Rheological properties					
MFR / MVR	g/10 min/ cm^3/10 min	ISO-1133–2	Moulding compound	Dry, moisture content < 0,03 % (m/m)	Load 2,16 kg, 5 kg, 10 kg, 21,6 kg T-melt + 10 °C = T-MFR
Mechanical properties					
Hardness	Shore D	ISO 868	≥ 80 × ≥ 10 × ≥ 6	M	5 measurements ≥ 9 mm from any edge, ≥ 6mm apart. 3 Specimens of 2 mm stacked
Tensile modulus	MPa	ISO 527-2	1A (equal ISO 3167A)	M	Speed 1 mm/min
Tensile stress at x % elongation	MPa		1BA thickness 2 mm	M	20 %, 100 %, 300 % elongation
Stress at break	MPa			M	Speed 200 mm/min
Tensile elongation at break	%			M	Speed 200 mm/min
Stress at break	MPa			M	Speed 200 mm/min
Thermal properties					
Glass transition temperature		ISO 11357-1 and ISO 11357-2	Moulding compound	M	Record midpoint temperature Use 10 °C/min
Other properties					
Density	g/cm^3	ISO 1183		M	
Moisture content	%	ISO 15512	Granules/moulding compound	Before and/or after drying	

[a] M: Injection moulding

Table 6 — Specialized properties and test conditions — Shore hardness classification D > 65

Property	Unit	Standard	Specimen type mm	Specimen preparation[a]	Test conditions and supplementary instructions
Mechanical properties					
Strain at break	%				Speed 200 mm/min
Tensile creep	MPa	ISO 899	ISO 3167A	M	To be agreed between customer and supplier. Times to be specified and documented
Charpy notched impact Strength	kJ/m^2		80 × 10 × 4 V-notch r=1	M	Method 1eA, Edgewise impact
Tear strength	kN/m	ISO 34-1	ISO 31-1 Bb		Speed 500 mm/min
Compression set	%	ISO 815	Ø 13 mm × 6 mm		23°C, 70°C
Abrasion loss	mm^3	ISO 4649 A	Thickness 6 mm		
Biological resistance		ISO 846 variant D and ISO 527-2	ISO 527-2 type 1BA thickness 2 mm	M	Tensile test for comparison purposes for ester containing TPU, but not needed for 100 % TPU-ARET or TPU-ALET
Thermal properties					
Burning behaviour		IEC 60695–11–10	125 × 13 × 3	M	0,91 mm, 3,0 mm
Electrical properties					
Relative permittivity		IEC 60250	≥ 60 × ≥ 60 × 2	M	100 Hz, 1 MHz
Dissipation factor tan		IEC 60250		M	100 Hz, 1 MHz
Volume resistivity	Ω m	IEC 60093		M	
Surface restivity	Ω	IEC 60093		M	
Dielectrical strength	kV/mm	IEC 60243–1	≥ 60 × ≥ 60 × 1	M	
Comparative tracking index CTI	V	IEC 60112	≥ 20 × ≥ 20 × 4	M	

[a] M: Injection moulding

Annex A
(informative)

Material identification

For material identification of thermoplastic polyurethane materials, infrared spectra can be used. Thermogravimetry can also be used and can be combined with density determination. Generally, this gives less information, but can easily be applied even in presence of carbon black without any sample preparation.

For quality control, other methods related to the molar mass of the polymer can be used in addition as solution viscosity measurements or gel permeation chromatography (GPC).

NOTE DSC usually is not considered as an adequate method for identity control of thermoplastic polyurethanes, because this method depends not only on the material used but also on processing and annealing conditions.

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