INTERNATIONAL STANDARD

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Fourth edition
2022-07

Plastics — Acquisition and presentation of comparable multipoint data —

Part 2: Thermal and processing properties

Plastiques — Acquisition et présentation de données multiples comparables —

Partie 2: Propriétés thermiques et caractéristiques relatives à la mise en œuvre

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61 *Plastics*, Subcommittee SC 2, *Mechanical behaviour,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 11403-2:2012), which has been technically revised.

The main changes are as follows:

— the titles of Figure 1 and Figure 2 have been modified and Key tables have been added;

— the procedure for getting enthalpy/temperature curve (6.2) has been updated;

— the procedure for getting linear-expansion/temperature curve (6.3) has been updated;

— footnotes regarding transition temperatures have been added in Tables 2 and 3;

— an explanation that Clauses 4 and 5 do not apply in to enthalpy/temperature curve and melt shear viscosity, 6.2 and 6.4 respectively, has been added.

A list of all parts in the ISO 11403 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document has been prepared because users of plastics find sometimes that available data cannot be used readily to compare the properties of similar materials, especially when the data have been supplied by different sources. Even when the same standard tests have been used, they often allow the adoption of a wide range of alternative test conditions, and the data obtained are not necessarily comparable. The purpose of this document is to identify specific methods and conditions of test to be used for the acquisition and presentation of data in order that valid comparisons between materials can be made.

The ISO 10350 series is concerned with single-point data. Such data represent the most basic method for characterizing materials and are useful for the initial stages of material selection. This document identifies test conditions and procedures for the measurement and presentation of a more substantial quantity of data. Each property here is characterized by multipoint data which demonstrate how that property depends upon important variables such as time, temperature and environmental effects. Additional properties are also considered in this document. These data, therefore, enable more discriminating decisions to be made regarding a material's suitability for a particular application. Some data are also considered adequate for undertaking predictions of performance in service and of optimum processing conditions for moulding a component, although it should be recognized that, for purposes of design, additional data will often be needed. One reason for this is that some properties are strongly dependent upon the physical structure of the material. The test procedures referred to in this document employ, where possible, the multipurpose tensile bar and the polymer structure in this test specimen may be significantly different from that in specific regions of a moulded component. Under these circumstances, therefore, the data will not be suitable for accurate design calculations for product performance. The material supplier should be consulted for specific information on the applicability of data.

The ISO 10350 series and the ISO 11403 series, together, define the means for acquiring and presenting a core set of comparable data for use in material selection. Use of these International Standards results in a rationalization of effort and a reduction of cost associated with provision of these data. Furthermore, reference to these standards will simplify the development of data models for the computerized storage and exchange of data concerning material properties.

Where appropriate, values for test variables have been specified by this document. For some tests however, owing to the wide range of conditions over which different plastics perform, the standard gives guidance in the selection of certain test conditions so that they cover the operating range for that polymer. Because, in general, the properties and performance specifications for different polymers differ widely, there is no obligation to generate data under all the test conditions specified in this document.

Data on a wide range of properties are needed to enable plastics to be selected and used in the large variety of applications to which they are suited. ISO standards describe experimental procedures which are suitable for the acquisition of relevant information on many of these properties. For other properties, however, ISO standards either do not exist or exhibit shortcomings that complicate their use at present for the generation of comparable data (see Annex A). The ISO 11403 series has therefore been divided into parts so that each part can be developed independently. In this way, additional properties can be included as new or revised standards become available.

Plastics — Acquisition and presentation of comparable multipoint data —

Part 2: Thermal and processing properties

1 Scope

This document specifies test procedures for the acquisition and presentation of multipoint data on the following thermal and processing properties of plastics:

— enthalpy/temperature curve;

— linear-expansion/temperature curve;

— melt shear viscosity.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 293, *Plastics — Compression moulding of test specimens of thermoplastic materials*

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 295, *Plastics — Compression moulding of test specimens of thermosetting materials*

ISO 472, *Plastics — Vocabulary*

ISO 1133 (all parts), *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics*

ISO 2818, *Plastics — Preparation of test specimens by machining*

ISO 10724-1, *Plastics — Injection moulding of test specimens of thermosetting powder moulding compounds (PMCs) — Part 1: General principles and moulding of multipurpose test specimens*

ISO 20753, *Plastics — Test specimens*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Specimen preparation

In the preparation of specimens by injection moulding, the procedures described in ISO 294-1 or ISO 10724-1 shall be used. For compression moulding, the procedures described in ISO 293 or ISO 295 shall be used. The method of moulding and the conditions depends upon the material being moulded. If these conditions are specified in the International Standard appropriate to the material, then they shall be adopted, where possible, for the preparation of every specimen on which data are obtained using this document. For those plastics for which moulding conditions have not yet been standardized, the conditions employed shall be within the range recommended by the polymer manufacturer and shall, for each of the processing methods, be the same for every specimen. Where moulding conditions are not stipulated in any International Standard, the values used for the parameters in Table 1 shall be recorded with the data for that material.

Where specimens are prepared by machining from sheet, the machining shall be performed in accordance with ISO 2818.

This clause does not apply to 6.2 and 6.4.

Table 1 — Moulding parameters

Type of moulding material	Moulding method and standard (where applicable)	Moulding parameters
Thermoplastic	Injection, ISO 294-1	Melt temperature Mould temperature Injection velocity
Thermoplastic	Compression, ISO 293	Moulding temperature Moulding time Cooling rate Demoulding temperature
Thermosetting	Injection, ISO 10724-1	Injection temperature Mould temperature Injection velocity Cure time
Thermosetting	Compression, ISO 295	Mould temperature Moulding pressure Cure time

5 Conditioning

Specimens shall be conditioned in accordance with the International Standard appropriate to the material. Reference to the use of any special conditioning shall be recorded with the data in Tables 2 to 4. If no materials standard is available, condition test specimens at 23 °C ± 2 °C and (50 ± 10) % RH for a minimum length of time of 88 h according to ISO 291.

This clause does not apply to 6.2 and 6.4.

6 Test requirements

6.1 General

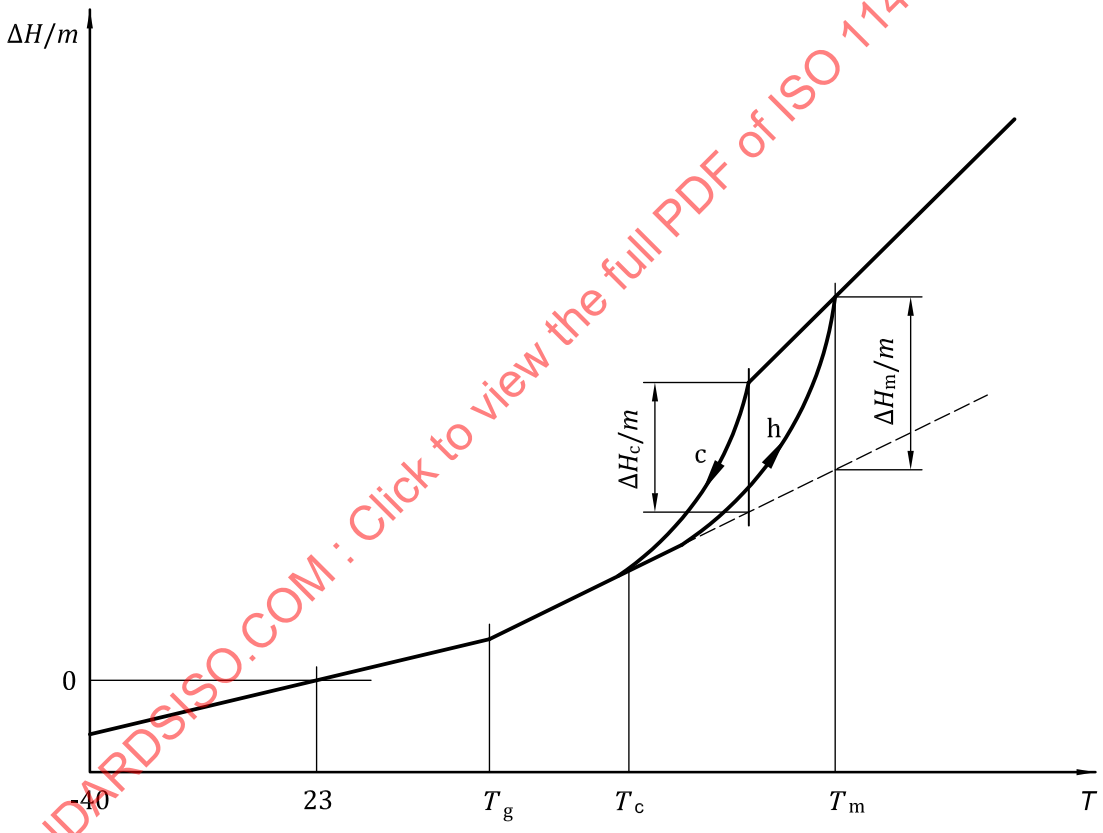
In acquiring data for the properties included in this document, the test procedures described in the test standard for each property indicated in the appropriate clause shall be followed.

For enthalpy and linear thermal expansion measurements, results shall be recorded at temperatures T_i at intervals of 10 K starting at −40 °C and replacing 20 °C by 23 °C.

6.2 Enthalpy/temperature curve: ISO 11357-3

Use differential scanning calorimetry to measure changes in enthalpy with temperature. According to ISO 11357-3, measure the melting and crystallization temperatures. Print out the temperature/time curve, the enthalpy/time curve and DSC/time curve, if necessary, afterwards. From these graphs, read enthalpy in the required temperature (see Figure 1). Starting at a temperature near the maximum recommended processing temperature, cool the specimen at a rate of 10 K/min to −40 °C and immediately heat at 10 K/min back to the starting temperature.

During the cooling part of the cycle, record the differences in enthalpy per unit mass $\Delta H_i/m$, in kilojoules per kilogram (kJ/kg), between temperatures T_i and the reference temperature of 23 °C at intervals of 10 °C in the temperatures T_i. The mass of the specimen is m, in kilograms. Repeat using values obtained during the heating part of the cycle. (See Figure 1 and Table 2.)



Key

T	temperature
$\Delta H/m$	specific enthalpy difference, in kJ/kg
ΔH_m and ΔH_c	enthalpies of melting and crystallization, respectively
c and h	cooling and heating parts of the measuring cycle
T_g	glass transition temperature
T_c	crystallization temperature
T_m	melting temperature

Figure 1 — Schematic diagram showing an example of the specific enthalpy difference $\Delta H/m$ of a semi-crystalline polymer against temperature T, showing the glass transition temperature T_g and the melting temperature T_m

6.3 Linear-expansion/temperature curve: ISO 11359-2

For those materials that absorb water, data shall be presented for this property in the dry state. The presentation of additional data for the polymer in a state of equilibrium water content at 23 °C and 50 % relative humidity shall be decided by the data supplier.

Use the central region of the type A multipurpose tensile specimen of ISO 20753. Prepare the test specimen for the length measurement by cutting from the length of the type A specimen direction. From the width direction of the type A specimen, prepare the test specimen for the width measurement by cutting. Use thermomechanical analysis to measure changes in the specimen length and width with changes in temperature whilst heating at a maximum rate of 5 K/min.

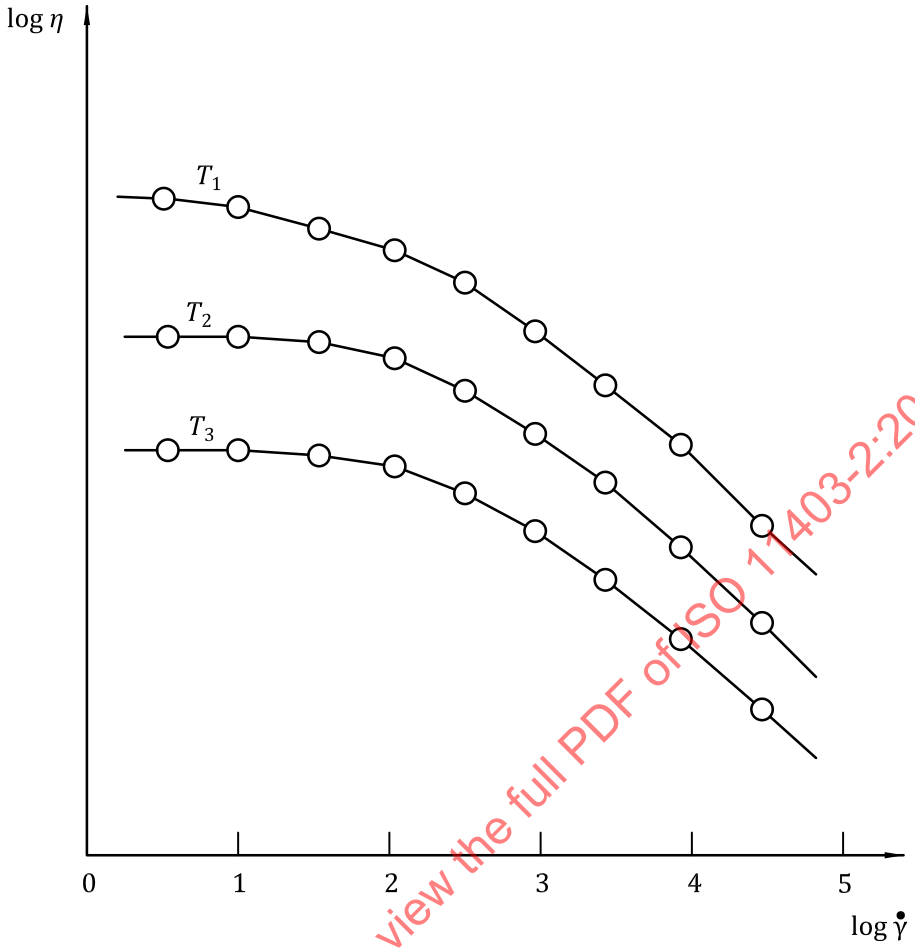
Starting at −40 °C, record the normalized length difference $(l_i - l_r)/l_r$, where l_i and l_r are the specimen lengths at temperatures T_i and a reference temperature of 23 °C, respectively, at intervals of 10 °C in the temperatures T_i. Record also the normalized changes in specimen width $(b_i - b_r)/b_r$ at the same temperatures T_i, where b_i and b_r are the specimen widths at temperatures T_i and 23 °C, respectively (see Table 3).

6.4 Melt shear viscosity: ISO 11443

Use the capillary rheometer method. Select three temperatures T_i which span the recommended melt processing temperature range of the polymer. One of these temperatures shall be the same as that specified in the materials standard in connection with the determination of melt flow rates for the material using the relevant part of ISO 1133. At each temperature, measure the shear viscosity over the range of shear-strain rates between 3 s^{-1} and 30 000 s^{-1}. Determine true values of the shear viscosity and shear-strain rate.

Record the true shear viscosity at nine values of the true shear-strain rate $\dot{\gamma}$, in reciprocal seconds (s–1), given by log $\dot{\gamma}$ = 0,5 k (1 ≤ k ≤ 9) and at each temperature Ti as shown by Figure 2 and in Table 4. Where shear viscosity values at the specified shear strain rates are determined by interpolation, the measured shear viscosity data shall be no more than half a decade in shear strain rate from the shear strain rate at which the interpolated shear viscosity value is determined. If melt fracture occurs, record the letter F at the appropriate shear strain rate in Table 4.

For those materials where the onset of melt fracture means that the data recorded at the shear strain rates in Table 4 do not accurately describe the dependence of shear viscosity on shear strain rate, then shear viscosity measurements shall be recorded at additional shear strain rates.



Key

η melt shear viscisity, expressed in pascal seconds (Pa·s)

$\dot{\gamma}$ shear strain rate, expressed in reciprocal seconds (s^{-1})

Figure 2 — Schematic diagram showing the variation of the melt shear viscosity η at three temperatures (T_1, T_2, T_3)

7 Presentation of data

Record the results in the formats described by Tables 2 to 4, together with information that identifies the material.

Table 2 — Specific enthalpy difference $\Delta H_i/m$ versus temperature T_i [a]

T_i (°C) [b]		−40	−30	−20	−10	0	10	23	. . .
$\Delta H_i/m$ [a] (kJ/kg)	Cooling							0	
	Heating							0	

[a] See 6.2 and Figure 1.

[b] Add T_g, T_c, T_m to the table if necessary.

Table 3 — Normalized length difference $(l_i - l_r)/l_r$ and normalized width difference $(b_i - b_r)/b_r$ versus temperature T_i [a]

T_i (°C) [b]	−40	−30	−20	−10	0	10	23	30	. . .
$(l_i - l_r)/l_r$ [a]							0		
$(b_i - b_r)/b_r$ [a]							0		

[a] See 6.3.

[b] Add T_g, T_c, T_m to the table if necessary.

Table 4 — True shear viscosity values η, in pascal seconds (Pa·s), at true shear strain rates $\dot{\gamma}$, in reciprocal seconds (s^{-1}), at temperatures T_i [a]

i	T_i (°C)	log $\dot{\gamma}$								
		0,5	1,0	1,5	2,0	2,5	3,0	3,5	4,0	4,5
1										
2										
3										

NOTE The letter F indicates that melt fracture has occurred.

[a] See 6.4 and Figure 2.

Where appropriate, the following additional information shall be included with each table:

a) the method of preparation of the specimen;

b) a reference to the International Standard which gives the processing conditions used to prepare the specimen, if this was prepared by injection or compression moulding. If these are not given in any standard, then record the appropriate conditions identified in Table 1;

c) any special conditioning procedure referred to in Clause 5;

d) the number of specimens tested.

8 Precision

For information on the precision of the test methods used to generate the data recorded in Tables 2 to 4 in Clause 7, the appropriate ISO test standard should be consulted. However, not all of these standards contain a precision clause and, furthermore, the precision of the data from some tests will depend on the test conditions and on the behaviour of the material under those conditions.