INTERNATIONAL STANDARD

ISO 11403-2

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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

Reference number
ISO 11403-2:1995(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11403-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

ISO 11403 consists of the following parts, under the general title *Plastics — Acquisition and presentation of comparable multipoint data*:

— *Part 1: Mechanical properties*

— *Part 2: Thermal and processing properties*

— *Part 3: Environmental influences on properties*

Annex A of this part of ISO 11403 is for information only.

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Introduction

This International Standard 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 International Standard 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.

ISO 10350 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. The present International Standard 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 standard. 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 standard 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.

ISO 10350 and the various parts of this International Standard together define the means for acquiring and presenting a core set of comparable data for use in material selection. Use of these standards should result 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 standard. 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 standard.

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 standard 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 part of ISO 11403 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 standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 11403. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 11403 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO 294:1995, *Plastics — Injection moulding of test specimens of thermoplastic materials.*

ISO 295:1991, *Plastics — Compression moulding of test specimens of thermosetting materials.*

ISO 2818:1994, *Plastics — Preparation of test specimens by machining.*

ISO 3167:1993, *Plastics — Multipurpose test specimens.*

ISO 10724:1994, *Plastics — Thermosetting moulding materials — Injection moulding of multipurpose test specimens.*

ISO 11403-1:1994, *Plastics — Acquisition and presentation of comparable multipoint data — Part 1: Mechanical properties.*

ISO 11443:1995, *Plastics — Determination of the fluidity of plastics using capillary and slit-die rheometers.*

ASTM E 831-93, *Test method for linear thermal expansion of solid materials by thermomechanical analysis.*

ASTM E 968-83(1993), *Practice for heat flow calibration of differential scanning calorimeters.*

3 Definition

For the purposes of this part of ISO 11403, the following definition applies.

3.1 multipoint data: Data characterizing the behaviour of a plastics material by means of a number of test results for a property measured over a range of test conditions.

4 Specimen preparation

In the preparation of specimens by injection or compression moulding, the procedures described in ISO 293, ISO 294, ISO 295 or ISO 10724 shall be used. The method of moulding and the conditions will depend 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 part of ISO 11403. 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.

5 Conditioning

Specimen conditioning shall be carried out at 23 °C ± 2 °C and (50 ± 5) % relative humidity for a minimum length of time of 88 h (see ISO 291) except where special conditioning is required as specified by the appropriate material standard. Reference to the use of any special conditioning shall be recorded with the data in the tables in clause 7.

6 Test requirements

6.1 General

In acquiring data for the properties included in this part of ISO 11403, the test procedures described in the test standard for each property indicated in the appropriate clause shall be followed.

Where data are recorded at selected temperatures, temperature values shall be chosen from the series of integral multiples of 10 °C, starting at − 40 °C and replacing 20 °C by 23 °C.

Table 1 — Moulding parameters

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

6.2 Enthalpy/temperature curve: ASTM E 968

Use differential scanning calorimetry to measure changes in enthalpy with temperature. Starting at a temperature near the maximum recommended processing temperature, cool the specimen at a rate of 10 °C/min to − 40 °C and immediately heat at 10 °C/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 in clause 7.)

6.3 Linear-expansion/temperature curve: ASTM E 831 (see note 1)

NOTE 1 For the acquisition of data for this part of ISO 11403, it is not necessary to use a reference material to calibrate the length-measuring equipment. The displacement-measuring device may be calibrated directly using a method that is traceable to standards for the measurement of length.

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

Use the central region of the type A multipurpose tensile specimen of ISO 3167. Use thermomechanical analysis to measure changes in the specimen length and width with changes in temperature whilst heating at a maximum rate of 5 °C/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 in clause 7.)

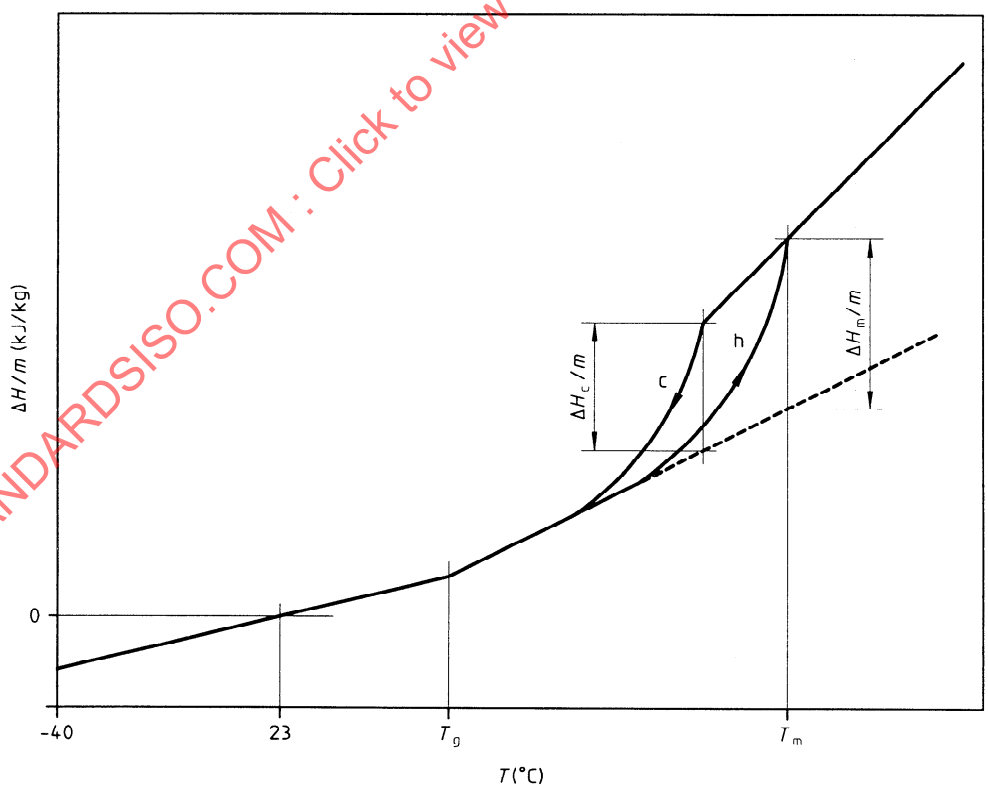

Figure 1 — Schematic diagram of the specific enthalpy difference $\Delta H/m$ of a semi-crystalline polymer against temperature T, showing the glass transition temperature T_g and the melting temperature T_m
(ΔH_m and ΔH_c are the enthalpies of melting and crystallisation, respectively, and c and h refer to the cooling and heating parts of the measuring cycle)

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. At each temperature, measure the shear viscosity over the range of shear-strain rates $\dot{\gamma}$ between 3 s^{-1} and 30 000 s^{-1}. Use true values of shear viscosity and shear-strain rate.

Record the shear viscosity at nine values of the shear-strain rate $\dot{\gamma}$, in reciprocal seconds (s^{-1}), given by $\lg \dot{\gamma} = 0{,}5\ k$ ($1 \leqslant k \leqslant 9$) and at each temperature T_i, as shown by figure 2, and in table 4 in clause 7. If melt fracture occurs, record the letter F at the appropriate strain rate (see note 2).

NOTE 2 For those materials where the onset of melt fracture means that the data recorded at the strain rates in table 4 do not accurately describe the dependence of viscosity on strain rate, then viscosity measurements may be recorded at additional strain rates.

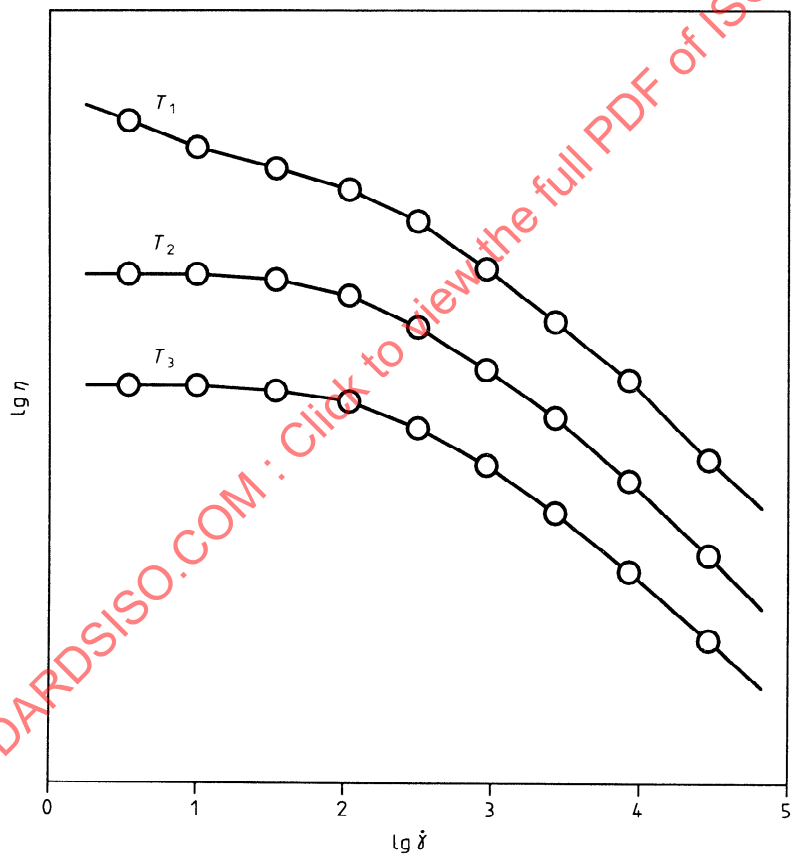

Figure 2 — Schematic diagram showing the variation of the melt-shear viscosity η, in pascal seconds (Pa·s), with shear strain rate $\dot{\gamma}$, in reciprocal seconds (s^{-1}), at three temperatures

7 Presentation of data

Record the results in the formats described by the following tables together with information that identifies the material.

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 the tables 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.

Table 2 — Specific enthalpy difference $\Delta H_i/m$ versus temperature T_i (see 6.2 and figure 1)

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

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 (see 6.3)

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

Table 4 — Shear viscosity values η in pascal seconds (Pa·s) at strain rates $\dot{\gamma}$ in reciprocal seconds (s^{-1}) and temperatures T_i (see 6.4 and figure 2)

i	T_i (°C)	lg $\dot{\gamma}$								
		0,5	1,0	1,5	2,0	2,5	3,0	3,5	4,0	4,5
1										
2										
3										
The letter F indicates that melt fracture has occurred.										