

European standard

French standard

NF EN ISO 11403-2
August 2004

Classification index: **T 50-064-2**

ICS: 83.080.01

Plastics

Acquisition and presentation of comparable multipoint data

Part 2: Thermal and processing properties

F : Plastiques — Acquisition et présentation de données multiples comparables —
Partie 2 : Propriétés thermiques et caractéristiques relatives à la mise en œuvre
D : Kunststoffe — Ermittlung und Darstellung von vergleichbaren
Vielpunktennwerten — Teil 2: Thermische und Verarbeitungseigenschaften

French standard approved

by decision of the Director General of AFNOR on July 20, 2004 taking effect on August 20, 2004.

It replaces the approved standard NF EN ISO 11403-2 dated October 1999.

Correspondence

The European standard EN ISO 11403-2:2004 has the status of French standard. It reproduces in full the international standard ISO 11403-2:2004.

Analysis

This document specifies operational test regimes in view to acquire and present multiple data relating to certain thermal properties of plastics and certain characteristics relating to their processing.

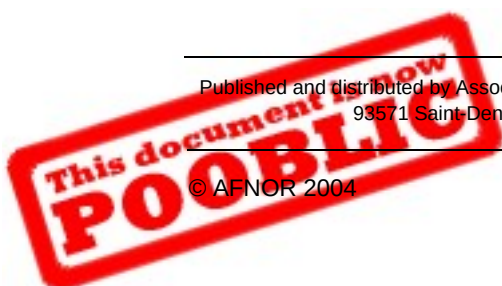
Descriptors

Technical International Thesaurus: plastics, data, tests, specimen preparation, viscosity, data display.

Modifications

With respect to the replaced document, revision of the ISO standard.

Corrections



National foreword

References to French standards

The correspondence between the standards figuring in the clause "Normative references" and the identical French standards is as follows:

ISO 291	: NF EN ISO 291 (classification index: T 51-014)
ISO 293	: NF EN ISO 293 (classification index: T 58-003)
ISO 294-1	: NF EN ISO 294-1 (classification index: T 51-294-1)
ISO 295	: NF EN ISO 295 (classification index: T 58-005)
ISO 1133	: NF EN ISO 1133 (classification index: T 51-038)
ISO 2818	: NF EN ISO 2818 (classification index: T 58-001)
ISO 3167	: NF EN ISO 3167 (classification index: T 58-004)
ISO 10724-1	: NF EN ISO 10724-1 (classification index: T 51-713-1)

The correspondence between the standards figuring in the clause "Normative references" and French standards having the same scope, but which are not identical, is as follows:

ISO 11357-3	: XP T 51-507-3
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The other standards mentioned in the clause "Normative references" that do not have any correspondence in the collection of French standards are as follows (they may be obtained from AFNOR):

ISO 11359-2
ISO 11443



EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 11403-2

March 2004

ICS 83.080.01

Supersedes EN ISO 11403-2:1999

English version

**Plastics - Acquisition and presentation of comparable multipoint
data - Part 2: Thermal and processing properties
(ISO 11403-2:2004)**

Plastiques - Acquisition et présentation de données
multiples comparables - Partie 2: Propriétés thermiques
et caractéristiques relatives à la mise en oeuvre
(ISO 11403-2:2004)

Kunststoffe - Ermittlung und Darstellung von
vergleichbaren Vielpunktkennwerten - Teil 2: Thermische
und Verarbeitungseigenschaften
(ISO 11403-2:2004)

This European Standard was approved by CEN on 1 March 2004.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

EN ISO 11403-2:2004 (E)

Foreword

This document (EN ISO 11403-2:2004) has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2004, and conflicting national standards shall be withdrawn at the latest by September 2004.

This document supersedes EN ISO 11403-2:1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 11403-2:2004 has been approved by CEN as EN ISO 11403-2:2004 without any modifications.

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ISO 11403-2:2004(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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

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

This second edition cancels and replaces the first edition (ISO 11403-2:1995), which has been technically revised.

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*



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 referenced documents are indispensable for the application 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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 293, *Plastics — Compression moulding 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 1133, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*

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

ISO 3167, *Plastics — Multipurpose test specimens*

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 11357-3, *Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization*

ISO 11359-2, *Plastics — Thermomechanical analysis (TMA) — Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*

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

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3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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 294-1, ISO 295 or ISO 10724-1 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.

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 Moulding 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 the tables in Clause 7. If no materials standard is available, condition test specimens at $23\text{ °C} \pm 2\text{ °C}$ and $(50 \pm 10)\text{ \% RH}$ for a minimum length of time of 88 h (see ISO 291).

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.

For enthalpy and linear thermal expansion measurements, results shall be recorded at temperatures T_i at intervals of 10 °C 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. 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 (see Note).

NOTE A heating rate of 20 °C/min has been recommended for a revised version of ISO 11357-3. The lower rate of 10 °C/min is specified here because it can give higher precision in measurements, and the associated higher resolution in enthalpy changes can be better for discriminating between materials.

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.)

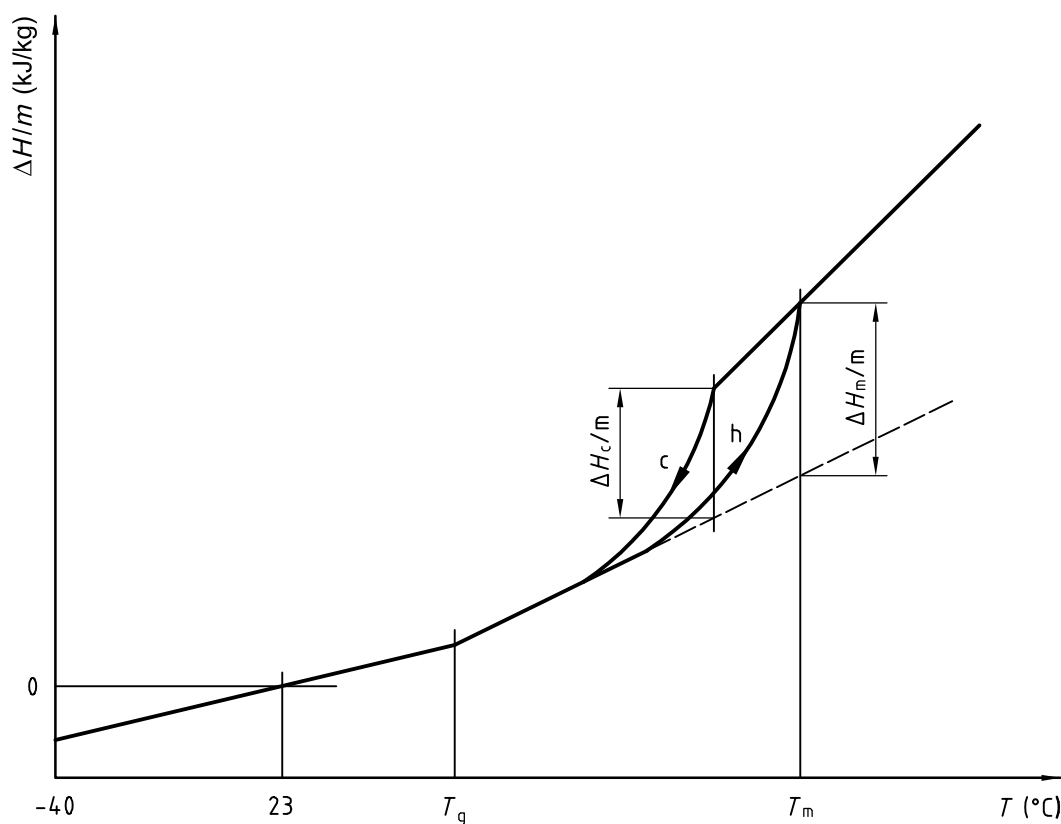


Figure 1 — Schematic diagram showing an example of the specific enthalpy difference $\Delta H/m$ of a semi-crystalline polymer against temperature T , showing the glass transition temperature T_g and the melting temperature T_m (ΔH_m and ΔH_c are the enthalpies of melting and crystallization, respectively,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

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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 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.)

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 ISO 1133. At each temperature, measure the shear viscosity over the range of shear-strain rates between 3 s^{-1} and $30\,000 \text{ s}^{-1}$. 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 $\log \dot{\gamma} = 0,5k$ ($1 \leq k \leq 9$) and at each temperature T_i as shown by Figure 2 and in Table 4. Where viscosity values at the specified strain rates are determined by interpolation, the measured viscosity data shall be no more than half a decade in strain rate from the strain rate at which the interpolated viscosity value is determined. If melt fracture occurs, record the letter F at the appropriate strain rate in Table 4.

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 shall be recorded at additional strain rates.



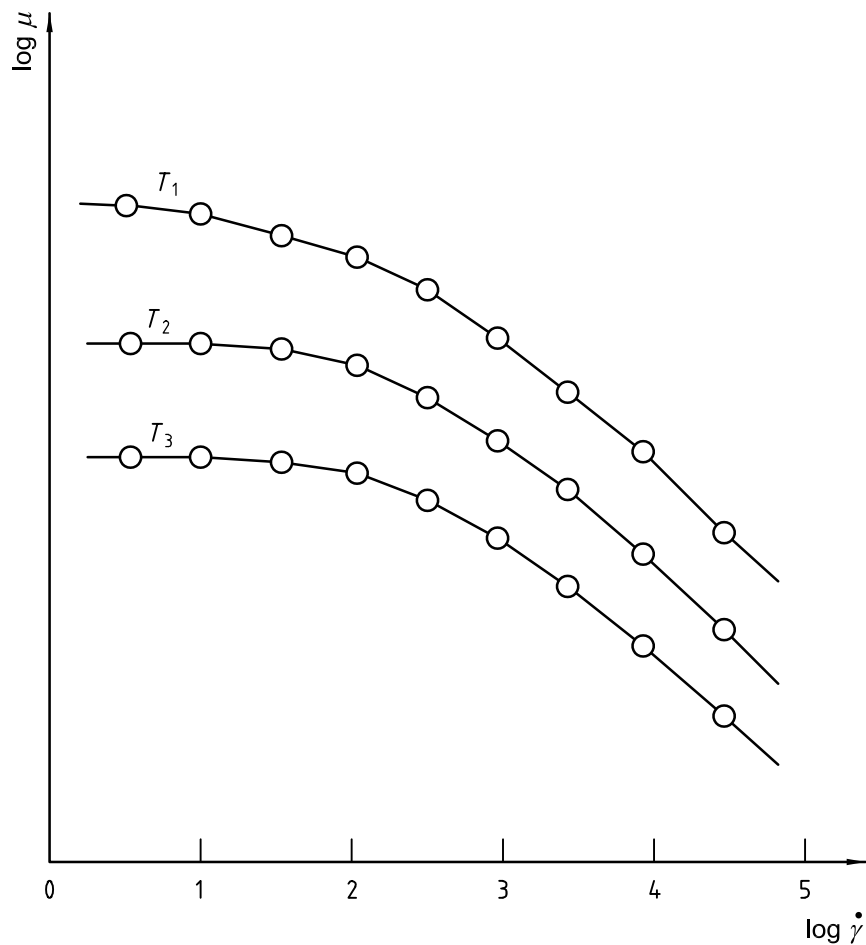


Figure 2 — Schematic diagram showing the variation of the melt-shear viscosity η , in pascal seconds (Pa·s), with shear strain rate $\dot{\gamma}$, in reciprocal seconds (s⁻¹), at three temperatures

7 Presentation of data

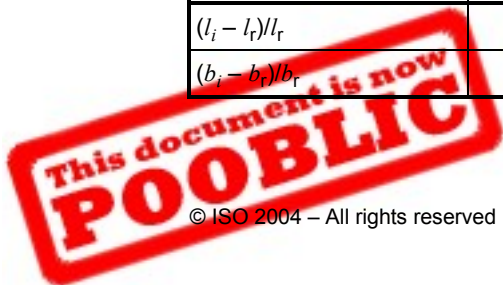
Record the results in the formats described by the following tables together with information that identifies the material.

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		



ISO 11403-2:2004(E)**Table 4 — True shear viscosity values η , in pascal seconds (Pa·s), at true strain rates $\dot{\gamma}$, in reciprocal seconds (s⁻¹), at temperatures T_i (see 6.4 and Figure 2)**

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										

The letter F indicates that melt fracture has occurred.

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

- The method of preparation of the specimen.
- 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.
- Any special conditioning procedure referred to in Clause 5.
- 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.



Annex A (informative)

Other properties

A.1 General

In this annex, some properties are listed which are considered sufficiently important that mention of them should be included in this part of ISO 11403. These properties have been excluded for the time being by the absence of adequate standards to refer to for details on conducting the tests. The purpose of this annex is to emphasize the scope for adding further properties to this part of ISO 11403 when International Standards are available.

A.2 Shrinkage

An International Standard is available for the measurement of shrinkage after injection moulding: ISO 294-4, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 4: Determination of moulding shrinkage*. The inclusion of this test method in this edition of ISO 11403-2 was considered with mould thickness and cavity pressure at hold as test variables but it is too early to obtain agreement on preferred values for the test variables.

A.3 Thermal conductivity of melt

There is no adequate International Standard for this property. Currently, only a few organizations generate data, but an International Standard is needed to validate the methods used.

A.4 Pressure-volume-temperature data

An International Standard is currently under development: ISO 17744, *Plastics — Determination of specific volume as a function of temperature and pressure (pVT diagram) — Piston apparatus method*.



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Bibliography

- [1] ISO 10350-1:1998, *Plastics — Acquisition and presentation of comparable single-point data — Part 1: Moulding materials*
- [2] ISO 10350-2:2001, *Plastics — Acquisition and presentation of comparable single-point data — Part 2: Long-fibre-reinforced plastics*

