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Plastics — Determination of specific volume as a function of temperature and pressure (pvT diagram) — Piston apparatus method

Plastiques — Détermination du volume spécifique en fonction de la température et de la pression (diagramme pvT) — Méthode utilisant un appareil à piston

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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.

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 17744 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

Introduction

The characterization of changes in volume of plastics, as a function of temperature and pressure, is necessary for the purpose of simulation studies and for optimizing polymer processing.

These thermophysical data may be used as they are or modelled in the form of suitable mathematical laws (see References [7] to [12] in the Bibliography).

In injection moulding, during the packing phase, most of the flow results from solidification. During solidification, if the plastic is semi-crystalline, the shrinkage is primarily due to crystallization. pvT data are used to model the volumetric shrinkage, which is translated into dimensional changes in the moulding.

It should be pointed out that, while all the techniques described hereafter are equivalent in their ability to characterize the melt state pvT behaviour, the isobaric cooling measurement is the only one which allows characterization of both the supercooling behaviour and the pressure dependency of the transition.

A list of references related to this International Standard is given in the Bibliography.

Plastics — Determination of specific volume as a function of temperature and pressure (pvT diagram) — Piston apparatus method

1 Scope

This International Standard describes procedures for determining the specific volume of plastics as a function of temperature and pressure in both the molten and solid states.

The standard specifies the use of a piston-equipped apparatus in which the test sample, held in a measurement cell, is pressurized by means of the piston. Measurements under conditions of constant pressure or constant temperature can be made. In the constant-pressure mode, the maximum heating and cooling rates permissible are restricted to 5 °C/min.

NOTE Higher heating and cooling rates can be used, but data will then have to be corrected for thermal gradients [13].

For the acquisition of data needed for processing design, it is recommended that the isobaric cooling method be used (see ISO 17282). The result of this measurement cannot be used directly for injection-moulding simulation.

By using these procedures, it is possible to obtain:

— pvT diagrams that represent the relationship which exists between pressure, specific volume and temperature for a given material;

— compressibility and volumetric thermal-expansion coefficients;

— information on first-order and glass transitions as a function of temperature and pressure.

Although thermoplastic polymers are currently tested down to room temperature using these procedures, it is emphasized that, at temperatures lower than T_g, the difficulty in achieving a true hydrostatic state is a source of uncertainty on the specific volume measurement.

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

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 7500-1, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 17282, *Plastics — Guide to the acquisition and presentation of design data*

NF T 51-561, *Plastiques — Détermination de la masse volumique en fonction de la température — Méthode par immersion*

3 Terms and definitions

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

3.1
specific volume
v
volume per unit mass of a material at a given temperature T and pressure p

NOTE Specific volume is expressed in cm^3/g.

3.2
density
ρ
mass per unit volume of a material at a given temperature T and pressure p

NOTE Density is expressed in g/cm^3.

3.3
preheating time
interval between the end of the cylinder-filling operation at the test temperature and the beginning of the measuring operation

3.4
pre-compression pressure
p_0
pressure applied during the pre-heating phase to achieve compaction of the sample

3.5
retention time
interval between the end of the cylinder-filling operation and the end of the measuring operation

3.6
volumetric thermal-expansion coefficient
α_v

$$\alpha_v = (1/v \times \mathrm{d}v/\mathrm{d}T)_p \qquad \text{(with } p \text{ constant)}$$

where

$\mathrm{d}v/\mathrm{d}T$ is deduced from the slope of the tangent to the $v = f(T)$ curve taken at a point on the curve;

α_v may be a function of pressure and temperature.

NOTE The volumetric thermal-expansion coefficient is expressed in $°C^{-1}$.

3.7
volumetric compressibility coefficient
β_v

$$\beta_v = -(1/v \times \mathrm{d}v/\mathrm{d}p)_T \qquad \text{(with } T \text{ constant)}$$

where

$\mathrm{d}v/\mathrm{d}p$ is deduced from the slope of the tangent to the $v = f(p)$ curve taken at a point on the curve;

β_v may be a function of pressure and temperature.

NOTE The volumetric compressibility coefficient is expressed in Pa^{-1}.

3.8
isobaric measurement
procedure in which the pressure is maintained constant during a test, the temperature being modified continuously or stepwise by heating or cooling in a predefined manner

3.9
isothermal measurement
procedure in which the temperature is maintained constant during a test, the pressure being modified by either increasing or decreasing its value in a predefined manner

4 Principle

The pvT behaviour of a plastic material describes the specific volume as a function of temperature and pressure. The method described here consists of measuring, under given temperature and pressure conditions, the volume of a test sample, the mass of which is known and constant. The test sample is placed in a cylindrical measurement cell which is closed at one end by a moveable piston and sealed at the other end. The test sample is heated or cooled down in the cell and pressure is applied via the piston. Changes in the specific volume are determined from the movement of the piston.

There are two measurement procedures:

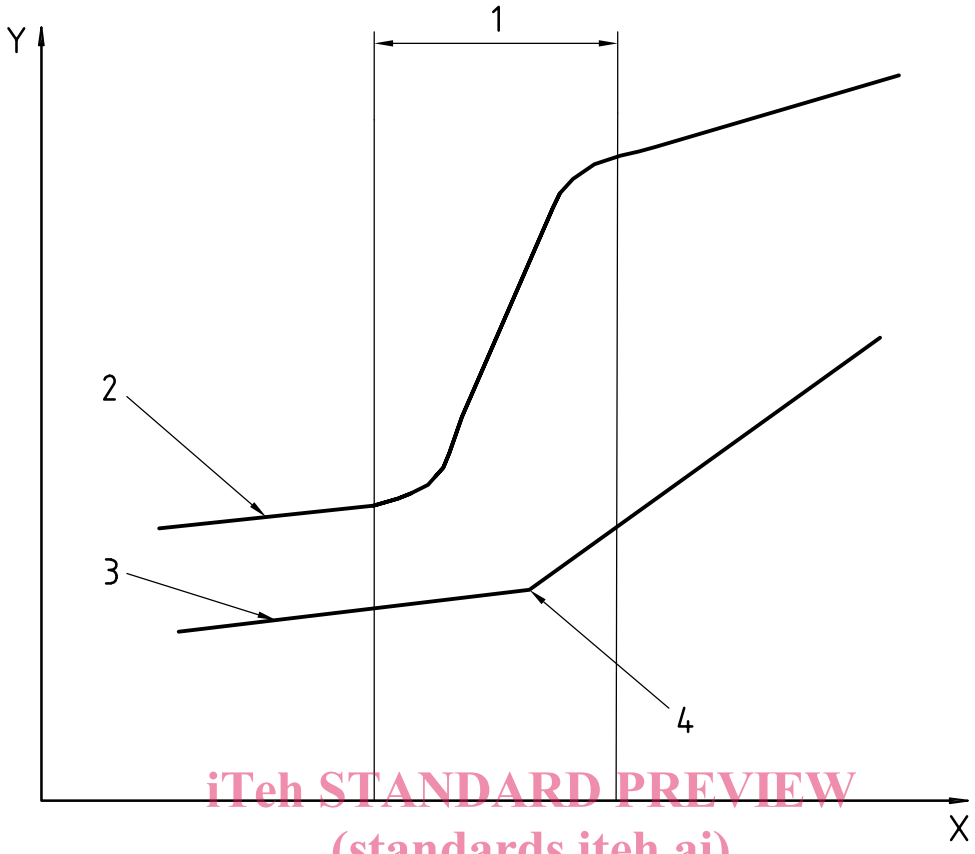
— at a constant pressure (isobaric measurement);

— at a constant temperature (isothermal measurement).

The choice between an increasing or a decreasing temperature profile for isobaric testing (or increasing or decreasing pressure for isothermal measurement) may have a significant effect on the results. It is important to specify the appropriate increasing or decreasing profile as well as the rate of change of the parameter.

When the temperature, the pressure (or applied force), the mass of the test sample, the cross-sectional area of the cell and the length of the test sample (derived from the piston position) are known, the pvT data can be obtained in absolute terms.

Schematic curves are shown in Figure 1.



Key

X temperature (°C)

Y specific volume (cm^3/g)

1 melting or crystallization zone

2 semi-crystalline polymer

3 amorphous polymer

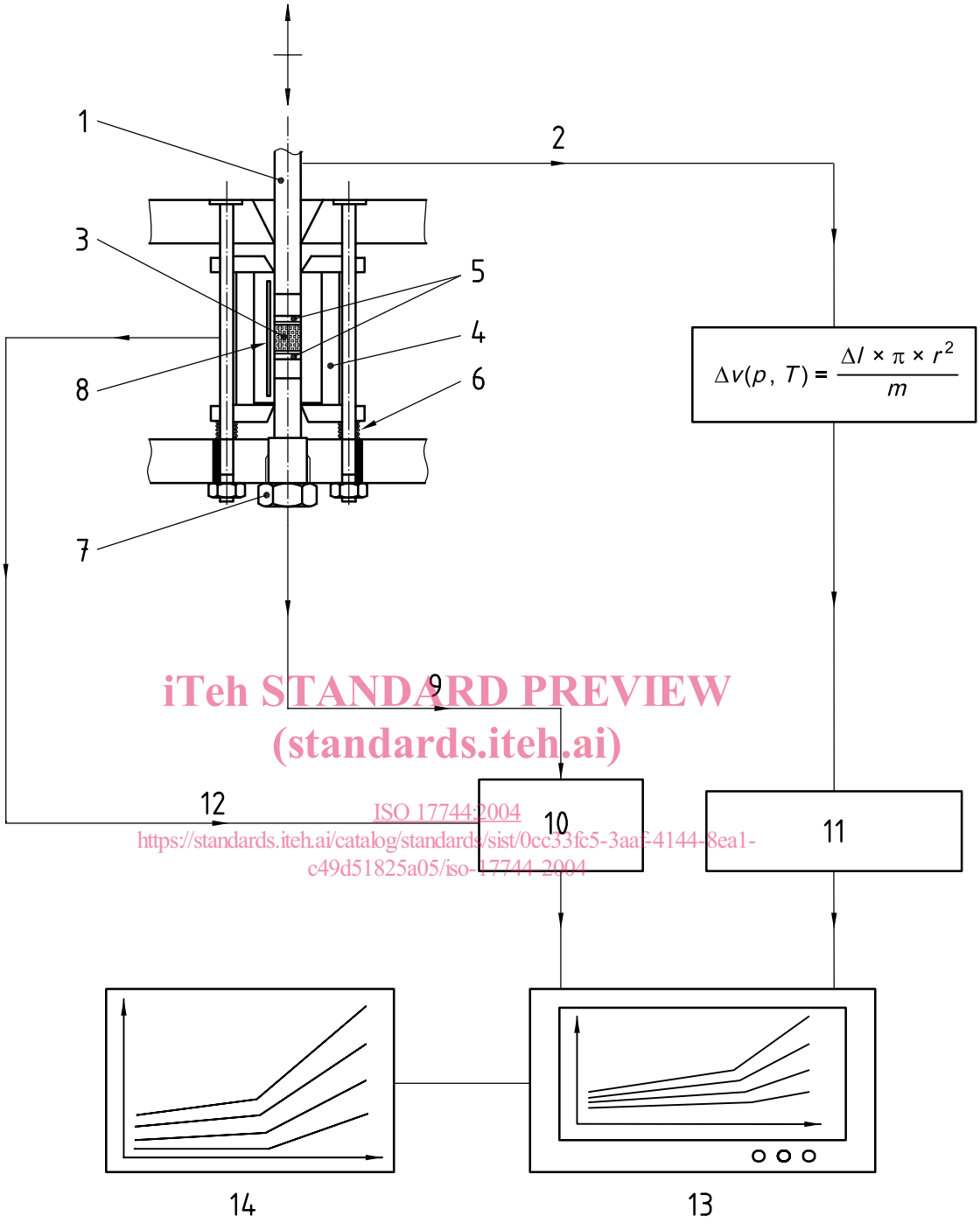
4 glass transition temperature

Figure 1 — Specific volume of semi-crystalline and amorphous polymers at a given pressure (isobaric mode) (the association of several such curves obtained at different pressures gives the pvT diagram)

5 Apparatus

5.1 General

The apparatus (see Figure 2) includes a cylinder (called a measurement cell), the bottom of which is closed, and a temperature-regulating device. Pressure is exerted on the test sample contained in the cylinder by means of a piston.



Key

1 piston of known radius, r
2 piston displacement, l
3 test sample
4 measurement cell of known diameter
5 seals
6 springs (optional)
7 end closure
8 heating/cooling device
9 pressure, p
10 AD/DA converter
11 displacement measurement
12 temperature, T
13 computer
14 printer

Figure 2 — Schematic diagram of a typical apparatus