~~2025-01~~

ISO/FDIS 17744~~:2024(E)~~

ISO/TC 61/SC 5~~/WG 9~~

Secretariat: DIN

Date: 2025-02-20

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

FDIS stage

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

This second edition cancels and replaces the first edition (ISO 17744:2004), which has been technically revised.

The main changes are as follows:

— ——procedure for measuring amourphous samples with a lower height of the sample once melted has been added;

— ——a specification for the balance to determine specific volume or density has been added;

— ——a specification for temperature calibration and position of measurement has been added;

— ——a specification for further measurement of density in the melt has been added;

— ——the presentation of results has been revised.

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.htmlwww.iso.org/members.html.

Introduction

The characterization of changes in specific 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 can be used as they are or modelled in the form of suitable mathematical laws (see References [7] to [12]).[7] to [12]).

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. For materials with high shrinkage and fast cooling rates, material can loselose wall contact especially at low holding pressures. *pvT* data are used to model the volumetric shrinkage, which is translated into dimensional changes in the moulding. Also, critical stress areas are detectable with a loss of wall contact.

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.

~~Plastics — Determination of specific volume as a function of temperature and pressure, *pvT* diagram — Piston apparatus method~~

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

1 Scope

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

This document 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.

NOTE For the acquisition of data needed for processing design, the isobaric cooling method is found to be more useful, see ISO 17282. The result of this measurement cannot be used directly for injection-moulding simulation.

This document is ~~applicable~~used to obtain:

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

— ~~—~~volumetric compressibility and volumetric thermal-expansion coefficients;

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

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

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

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

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

Note 1 to entry: Specific volume is expressed in cm^3/g.

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

Note 1 to entry: Density is expressed in g/cm^3.

3.3 ~~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 ~~3.4~~
pre-compression pressure
p_0
pressure applied during the pre-heating phase to achieve compaction of the sample

3.5 ~~3.5~~
volumetric thermal-expansion coefficient
α_v
coefficient defined by the formula

$$\alpha_v = (1/v \times dv/dT)_p \quad \text{(with } p \text{ constant)}$$

where

- dv/dT is the slope of the tangent to the $v = f(T)$ curve taken at a point on the curve;
- v is the specific volume;
- p is the pressure;
- T is the temperature

Note 1 to entry: The volumetric thermal-expansion coefficient can be a function of pressure and temperature.

Note 2 to entry: The volumetric thermal-expansion coefficient is expressed in K^{-1}.

3.6 ~~3.6~~
volumetric compressibility coefficient
β_v
coefficient defined by the formula

$$\beta_v = -(1/v \times dv/dp)_T \quad \text{(with } T \text{ constant)}$$

where

- dv/dp is the slope of the tangent to the $v = f(p)$ curve taken at a point on the curve;
- v is the specific volume;
- p is the pressure;
- T is the temperature

Note 1 to entry: The volumetric compressibility coefficient can be a function of pressure and temperature.

Note 2 to entry: The volumetric compressibility coefficient is expressed in Pa^{-1}.

3.7 ~~3.7~~
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

3.8 3.8

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 specified here consists of measuring, under given temperature and pressure conditions, the specific 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:

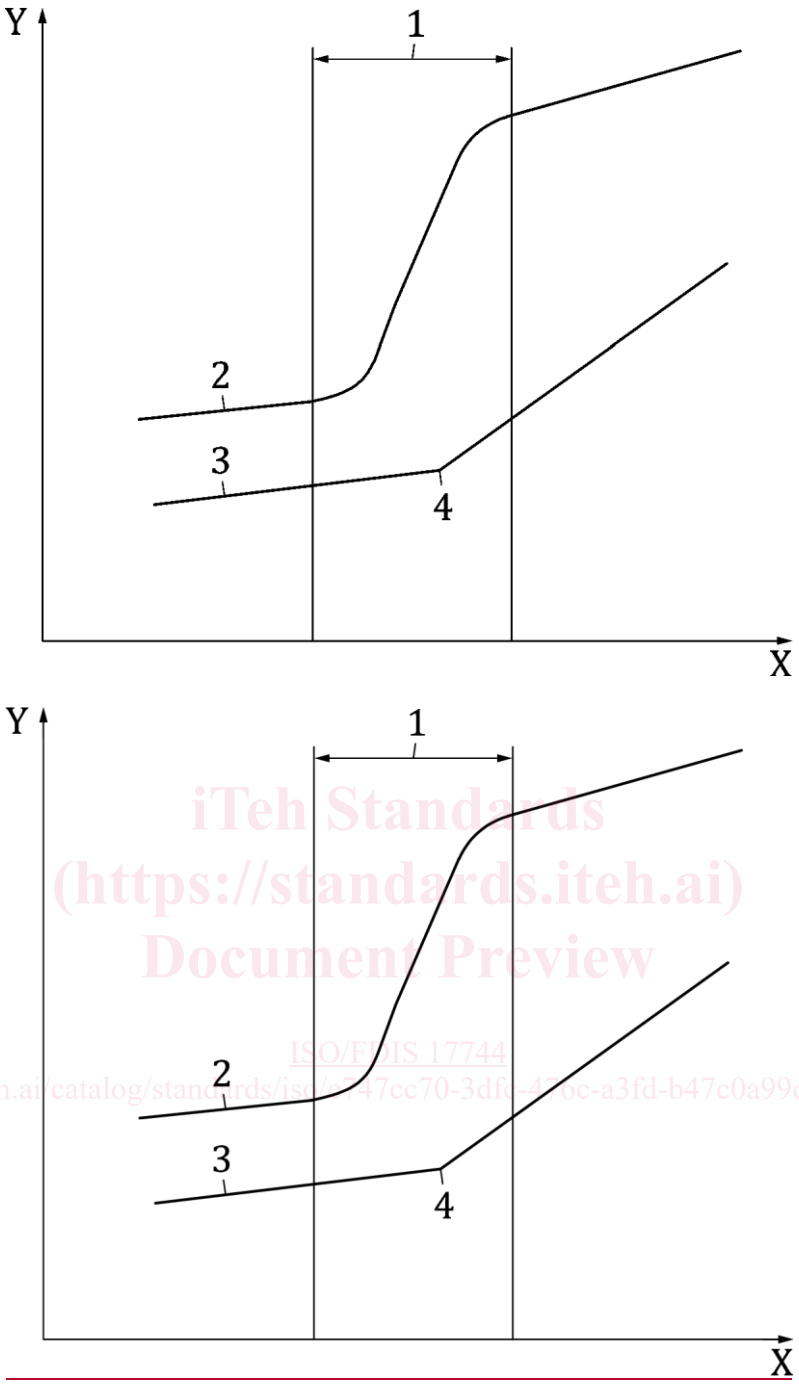
— ——at a constant pressure (isobaric measurement);

— ——at a constant temperature (isothermal measurement).

Both measurement procedures are generally plotted in isobaric curves but the choice between an increasing or a decreasing temperature profile for isobaric testing or increasing or decreasing pressure for isothermal measurement can 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.

Figure 1Figure 1 shows schematic curves of a semi crystalline and amorphous polymer. Mainly the transition zone is influenced by the choice of temperature profile and pressure order during measurement. The association of several such curves obtained at different pressures gives the *pvT* diagram.



Key

X	temperature (°C)	2	semi-crystalline polymer
Y	specific volume (cm^3/g)	3	amorphous polymer
1	melting or crystallization zone	4	glass transition temperature
~~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 in isobaric mode