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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*

Reference number
ISO 17744:2004(E)

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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 $\text{cm}^3/\text{g}$.

**3.2**
**density**
$\rho$
mass per unit volume of a material at a given temperature $T$ and pressure $p$

NOTE Density is expressed in $\text{g/cm}^3$.

**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**
$\alpha_v$

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

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;

$\alpha_v$ may be a function of pressure and temperature.

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

**3.7**
**volumetric compressibility coefficient**
$\beta_v$

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

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;

$\beta_v$ may be a function of pressure and temperature.

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

**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.

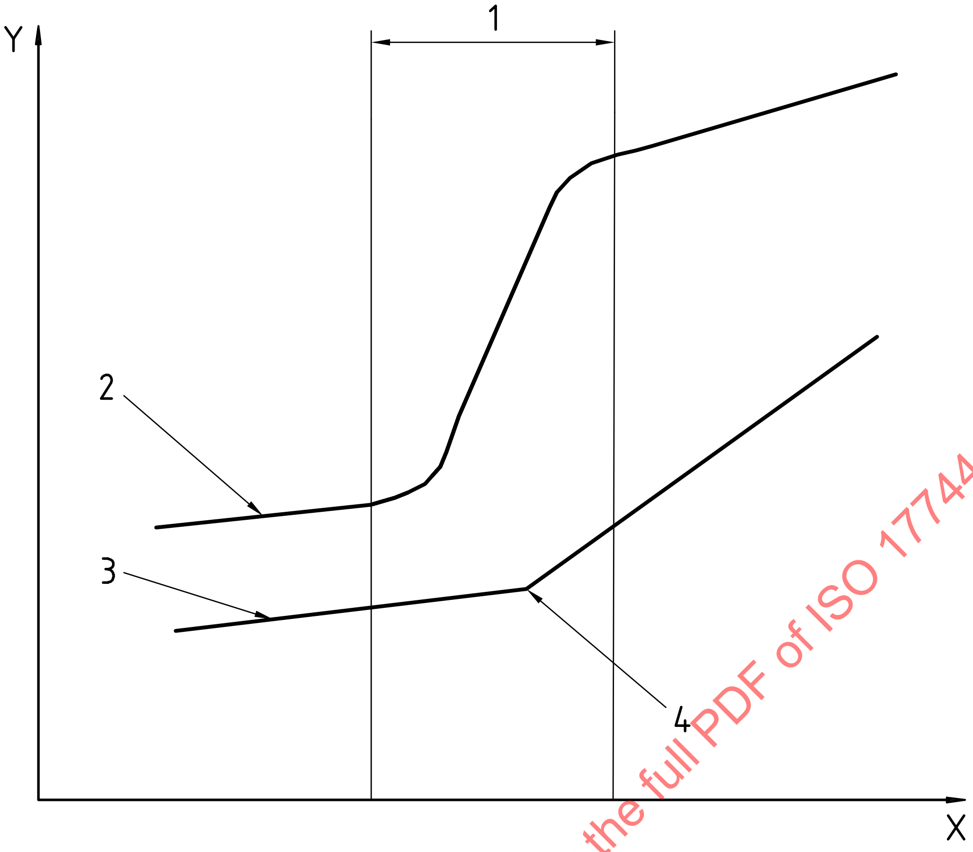
There are two measurement procedures:

- 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

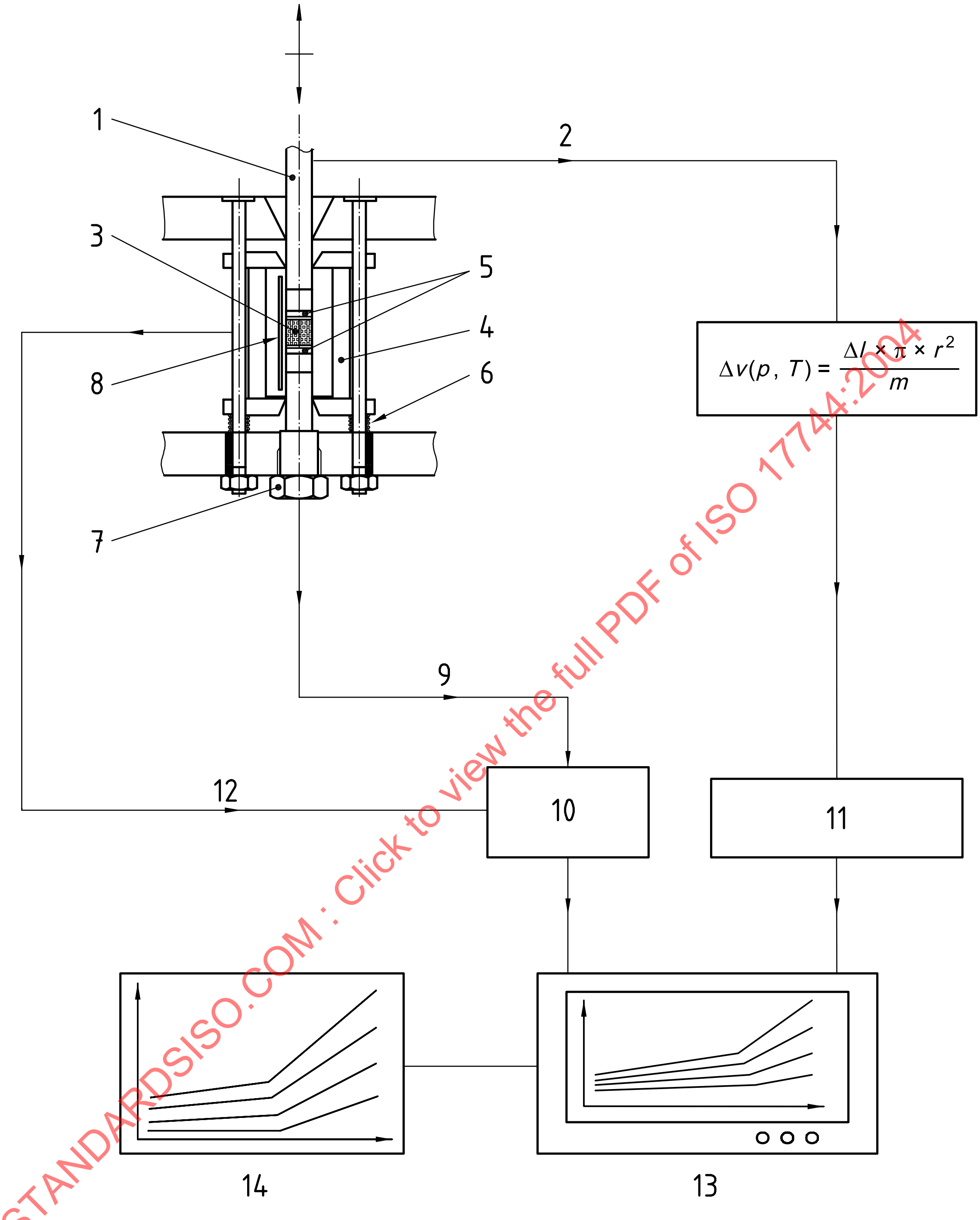
4 glass transition temperature

**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**

### 5.2 Measurement cell

The cylindrical measurement cell shall be as smooth as possible on the inside surface, i.e. free of any visible scratches or defects. It shall have an inside diameter between 5 mm and 12 mm, constant to within ± 0,01 mm along its whole length. It shall be made of a material which is resistant to wear and corrosion up to the maximum temperature produced by the heating device. It may include an opening at the side or in the base into which a pressure sensor can be inserted. It shall be manufactured using a method that produces an inside surface with a Vickers hardness of at least 800 HV 30 (see ISO 6507-1) and a roughness $Ra$ of less than 0,25 µm (arithmetical mean deviation) (see ISO 4287).

### 5.3 Piston

The piston shall be fitted with a seal made of a material which is inert to the test sample and suitable for use at the test temperature. The hardness of the piston shall be less than that of the cylinder, without, however, being less than 375 HV 30 (see ISO 6507-1). The piston shall be designed so that there is minimum friction between it and the cylinder wall.

NOTE 1 A suitable seal will minimize the likelihood of leaks during the test.

NOTE 2 Examples of suitable seal materials are polytetrafluoroethylene up to 280 °C and polyimide for higher temperatures.

### 5.4 Temperature-regulating device

The temperature-regulating device shall be designed so that:

- for isothermal measurements, the temperature can be controlled so that the maximum allowable temperature differences given in Table 1 are not exceeded during the test;
- for isobaric measurements, the heating or cooling rate can be controlled at a value between 1 °C/min and 20 °C/min (see 8.2.1.1).

NOTE In the cooling mode, it is necessary to check that the pre-set minimum temperature is compatible with the cooling rate used, taking into account the available cooling power of the cooling device used.

### 5.5 Test temperature measuring device

The test temperature is measured with a sensor (platinum resistance thermometer or thermocouple), the end of which shall be in contact either with the molten material or, should that not be possible, with the metal inner wall of the cell at a point adjacent to the test sample, preferably mid-way along its length. A heat-conducting fluid may be used in the thermometer well to improve conduction.

### 5.6 Device for measuring the distance travelled by the piston

This device shall be capable of determining the position of the piston with an accuracy of ± 5 µm.

### 5.7 Pressure-measuring device

The pressure may be measured by:

- a pressure sensor in the hydraulic circuit;
- a pressure sensor in the measurement cell;
- a load cell firmly attached to the piston.

The device shall only be used within a range of 10 % to 90 % of its nominal rating.

# 6 Equipment calibration

## 6.1 Temperature-measuring device

For all temperatures that can be set, the temperature control shall be designed so that, over the whole region occupied by the test sample, the temperature difference measured at the wall during the test does not exceed the relevant value given in Table 1.

**Table 1 — Maximum allowable temperature differences as functions of distance and time**

| Test temperature, $T$<br>°C | Temperature difference<br>°C | |
|---|---|---|
| | as a function of distance | as a function of time |
| $\leqslant 200$ | ± 1,0 | ± 0,5 |
| $200 < T \leqslant 300$ | ± 1,5 | ± 1,0 |
| $> 300$ | ± 2,0 | ± 1,5 |

The temperature-measuring device used for the test shall have a resolution of 0,1 °C and be calibrated by means of a device with error limits of ± 0,1 °C. The calibrating device shall have a heat sink effect on the instrument that is similar to that of the piston when in its working position.

## 6.2 Pressure sensor

An external hydraulic bench may be used to calibrate the pressure sensor. The maximum permissible error is less than or equal to 1 % of the measurement range. It is preferable to calibrate the pressure sensor at a temperature representative of that of the test.

If a load cell is used, it shall be of class 1 as defined in ISO 7500-1.

## 6.3 System

The measurement cell, piston and seals have their own $pvT$ behaviour. This $pvT$ behaviour shall be determined by calibration measurements. The results of the calibration measurements shall be subtracted in an appropriate way from the results obtained with a test sample.

To prevent changes in the seals, e.g. degradation, from occurring and affecting the results, it is recommended that the seals be replaced before each test.

## 6.4 Measurement cell

The diameter of the measurement cell shall be measured at three places along the part of its length in which the test sample is held. The variation in the diameter shall be less than ± 0,01 mm from the nominal value.

## 6.5 Piston displacement measuring device

The device for measuring the piston displacement shall be calibrated using one or more suitable gauges of known length and made of a material which has a very low coefficient of expansion (e.g. ceramics). Insert a gauge into the measurement cell, move the piston up to the gauge and use the displacement-measuring device to record the height of the gauge. Carry out the measurement with the piston pressing against the gauge with a small predefined force. Repeat the measurement. The displacement indicated by the measuring device shall not vary by more than ± 1 increment on the scale of the measuring device. The accuracy of the device shall be such that the position of the piston can be determined to within ± 5 µm.

The same predefined force shall be used for all calibrations (for example, the force can be 1 % of the maximum load).

## 7 Test sample

### 7.1 Preparation

The test sample may come in any form which enables it to be introduced into the cylinder, such as powder, granules, pellets, strips of film or moulded parts. In some cases, it may be preferable to transform the sample into, for instance, pellets (see Annex A, Clause A.1).

The test sample shall have a volume such that, once melted, its height in the cell is between 10 mm and 20 mm.

NOTE It is advisable to fill the measurement cell progressively, compressing the test sample manually several times during filling.

### 7.2 Conditioning

In cases where humidity speeds up degradation or affects the repeatability of the measurements, the material shall be dried. When prior conditioning is required, use the conditions prescribed in the material specification. If no material specification exists, consult the manufacturer for recommendations. Materials containing volatile components, substances which react chemically or substances which have special characteristics are likely to require special conditioning methods.

## 8 Procedure

### 8.1 Preliminary phase

It is important to know the thermal stability of the polymer over the range of test conditions used. The test shall be completed before an unacceptable amount of degradation of the polymer occurs. The operations to be carried out prior to each series of measurements are as follows:

a) Select the seals in the apparatus to suit the maximum temperature (see 5.3) and, if necessary, lubricate the cell with an agent which is inert to the material to be tested (see Annex A, Clause A.3).

b) Bring the cell to the required temperature.

c) Prepare the test sample in accordance with Clause 7 and weigh it to the nearest 1 mg.

d) Introduce the test sample into the cell in such a way as to avoid the presence of bubbles (see Annex A, Clause A.1).

e) Wait for the material to soften completely, activate the piston so as to compress the sample and note the initial pressure $p_0$ (minimum 20 MPa). Compression may be preceded by extrusion of the material should the apparatus allow this.

f) Bring the test sample, at the initial pressure $p_0$, to the first measurement temperature.

## 8.2 Measurements

### 8.2.1 Isobaric measurements

### 8.2.1.1 Continuous method

In this method, the pressure on the test sample is kept constant and the temperature is either increased or decreased at a predefined rate. The position of the piston is measured during the temperature scan. When the whole temperature range has been scanned, the same series of measurements may be performed at another pressure. Set the following pressure and wait for it to stabilize. When the whole pressure range has been covered, repeat the measurements made at the first pressure used in order to make sure that there has been no ageing or degradation of the test sample. The recommended maximum heating or cooling rate is 5 °C/min to ensure a sufficiently uniform temperature distribution within the test sample. The rate may be selected to suit the internal diameter of the measurement cell used. Heating or cooling rates of up to 20 °C/min may be used provided that a correction for the effects of thermal gradients in the sample is made[13].

NOTE It may be desirable to use higher heating or cooling rates, particularly for unstable materials that would otherwise degrade excessively if lower rates, and hence longer test times, were used.

It is recommended that measurements be made at pressure intervals of 20 MPa.

### 8.2.1.2 Stepwise method

In this method, the pressure of the test sample is kept constant and the temperature is increased or decreased stepwise. The position of the piston is measured after the temperature readout has stabilized and the sample has reached temperature equilibrium. The recommended minimum waiting time is 5 min, but this may be longer for bigger temperature steps, sample masses and measurement cell diameters. The recommended step size is between 1 °C and 10 °C, depending on the desired accuracy and the temperature range to be covered.

After the whole temperature range has been covered, the same series of measurements can be made at another pressure, waiting for the pressure to stabilize each time a new pressure is set.

When the whole pressure range has been covered, repeat the measurements at the first pressure used in order to make sure that there has been no ageing or degradation of the test sample.

NOTE 1 The stepwise method may be useful if measurements are required closer to thermal equilibrium.

NOTE 2 Because of the longer residence time at higher temperatures, this method is not suitable for use with samples that degrade.

### 8.2.2 Isothermal measurements

In this method, the temperature of the test sample is kept constant and the pressure is modified stepwise, being either increased or decreased. Once the pressure has stabilized at each value, the position of the piston is measured. When the whole pressure range has been scanned, the same series of measurements may be performed at another temperature. Set the following temperature and wait for thermal equilibrium to be reached (minimum 5 min). When the whole temperature range has been covered, repeat the measurements at the first temperature used in order to make sure that there has been no ageing or degradation of the test sample.

Within the limits of the transition zone, the measurements would be valid only if the sample did not undergo any significant change during the measurements (crystallization for instance). However, this may not be the case. Isothermal measurements should therefore be limited to measurements outside of the transition zones.

It is recommended that measurements be made at temperature intervals of 20 °C to 30 °C.

### 8.3 Final stage

After testing, remove the solid test sample and weigh it to the nearest milligram. This value will be used to calculate the specific volume and shall be compared with the mass prior to testing in order to check that there have not been any excessive leaks. The loss in mass shall be less than 2 %. If a higher mass loss is found, it may be necessary to repeat the test using different seals or, alternatively, carry out the test at a lower temperature and/or pressure.

Check the test sample, noting the presence of any bubbles, any degradation of the material and any other changes in the appearance of the test sample.

Do not reuse test samples after removing them from the instrument.

### 8.4 Further measurements

It is advisable to measure the density of the test sample as a function of temperature at atmospheric pressure to have a reference curve.

In some cases, two values can be sufficient, at room temperature and in the melt. They can be obtained using ISO 1183. If more values are required, NF XT 51-561 is recommended.

## 9 Expression of results

The results are expressed in sets of three values (pressure, specific volume, temperature) grouped together in tabular form. They may be transformed directly into $pvT$ diagrams representing either the specific volume as a function of temperature at different pressures, or the specific volume as a function of pressure at different temperatures. The volumetric thermal-expansion or volumetric compressibility coefficients of the material studied may also be calculated and included in the table and/or plots.

## 10 Precision

No precision data are currently available.

## 11 Test report

### 11.1 General and test conditions

a) a reference to this International Standard;

b) the date of the test;

c) details of the test sample:

   1) preparation (see 7.1),

   2) designation of the material,

   3) type of material,

   4) shape,

   5) conditioning, as applicable (see 7.2),

   6) mass before and after testing;

d) a description of the apparatus used with an indication of:

   1) its make,

   2) the type of instrument (application of pressure or force),

   3) the cell diameter;

e) details of the test:

   1) the temperature and initial pressure $p_0$ used in the preliminary phase (see 8.1),

   2) the type of measurement made (whether $p$ or $T$ varied and whether increased or decreased),

   3) the length of the test,

   4) for the isobaric method: the heating or cooling rate,

   5) for the stepwise isobaric method: the heating or cooling rate and the size and duration of the temperature steps,

   6) for the isothermal method (if possible): the time taken for equilibrium to be established at each pressure,

   7) the total length of time for the whole sequence of measurements,

   8) any observations concerning the appearance of the test sample after testing (see 8.3),

   9) details of any abnormal behaviour observed during the test,

   10) whether a correction was applied to the data to allow for thermal gradients and, if so, details of the kind of correction (raw data may also be presented in addition to the corrected data);

f) details of any further measurements (see 8.4).

## 11.2 Presentation of the results

Include the test results as a table of values of pressure (in bars or Pa), specific volume (in cm$^3$/g) and temperature (in °C).

## 11.3 Presentation in the form of a graph (see example in Annex B)

If prepared, include a graph of the specific volume as a function of temperature (one curve per pressure) or a graph of the specific volume as a function of pressure (one curve per temperature).

# Annex A
(normative)

# Sources of error when specific volume is measured as a function of temperature and pressure

## A.1 Presence of voids, bubbles or heterogeneity in the test sample

This is a possible cause of non-reproducibility of results, and indicates insufficient initial compression.

A visual examination of the test sample after the test, including cutting the sample in half and examining the cut surface, using a microscope if necessary, will help detect the presence of voids.

In order to avoid this problem, it is advisable at the beginning of a test to fill the measurement cell progressively, compressing the test sample manually several times during filling. Moreover, degradation or degassing of the test sample may be the cause of bubbles. In this case, the use of an inert-gas atmosphere prior to or during filling, or preliminary treatment (e.g. stoving) of the test sample, may minimize this problem.

## A.2 Sensor drift

The pressure or force sensor, displacement-measuring device or temperature sensor shall be verified in accordance with a quality-control plan.

## A.3 Friction

In the case of measurements on test samples in the solid state using a force sensor, the force due to the static friction between the piston (or seal attached to the piston) and the cell wall shall be taken into account when calculating the applied pressure. To do this, a test can be carried out without a test sample and the frictional force measured at different temperatures. It is then sufficient to subtract this force from the applied force to calculate the pressure at the temperature in question. Friction between the test sample and the cell wall is more difficult to assess. To minimize it, it is recommended that the height of a solid test sample be limited to five times its diameter or to make use of an inert lubricant such as a mould release agent.

## A.4 Sensor compliance and apparatus expansion

**A.4.1** If a force sensor is used, it may prove necessary to correct the recorded position of the piston to allow for the compliance of the sensor. This correction is more important at higher pressures.

**A.4.2** Compressibility and thermal expansion of the apparatus are sources of error in the volume of the test sample and the measurement of its height. It is possible to correct the error if the values of the compressibility coefficients and thermal-expansion coefficients of the materials of which the apparatus is made, including the seals, are known or by making measurements to quantify the error.

## A.5 Non-uniformity of the temperature distribution

In view of the poor thermal conductivity of polymers, it is difficult to know the exact temperature distribution within a test sample.

With isobaric measurements, at heating and cooling rates lower than 5 °C/min, the effect is likely to be small and correction is unlikely to be necessary. However, higher rates affect the results due to the existence of significant temperature gradients. Should a heating or cooling rate greater than 5 °C/min be used, a correction for thermal-gradient effects shall be made.

When carrying out isothermal measurements, it is necessary to wait until thermal equilibrium is reached before beginning to take measurements (5 min is the minimum).

Finally, during changes in temperature or pressure, an endothermal or exothermal change may occur in the test sample which is likely to modify the temperature distribution within the sample. Should any anomalies be noticed, thermal analysis of the test samples should be considered in order to confirm that this is happening.

NOTE In the transition zone for semi-crystalline materials, kinetic effects are important factors. The transition temperature depends not only on pressure but also on the entire thermal history of the material (especially the cooling rate). So it appears the best way of obtaining reliable results is to measure the specific volume evolution in the isobaric cooling mode. Thus, thermal-history influences can be analysed by controlling the cooling rate. Nevertheless, in such conditions, because of the poor thermal conductivity of polymers, a significant thermal gradient appears in the test sample. So it is essential to extract the thermal-gradient effect from the experimental results in order to obtain the intrinsic specific volume of the polymer and to analyse it in terms of equation-of-state and crystallization kinetics models [13].

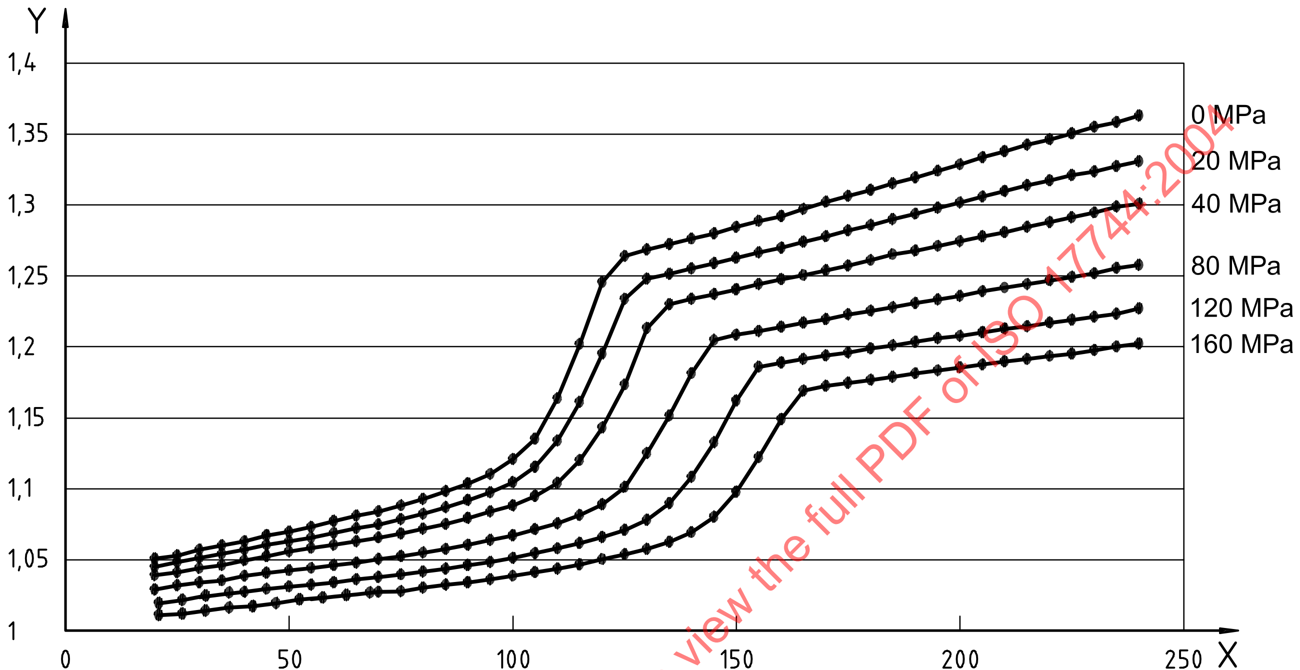
## A.6 Errors concerning the mass

Knowing the mass of the test sample on which the measurement is being carried out requires:

- leaks to be limited to as little as possible or that their extent be known;
- weighing to be carried out with sufficient accuracy.

# Annex B
(informative)

## Examples of *pvT* diagrams



**Key**

X temperature (°C)

Y specific volume ($cm^3/g$)

Figure B.1 — *pvT* diagram for PE (specific volume versus temperature)