INTERNATIONAL STANDARD

ISO 18373-1

First edition
2007-07-15

Rigid PVC pipes — Differential scanning calorimetry (DSC) method —

Part 1: Measurement of the processing temperature

Tubes rigides en PVC — Méthode utilisant la calorimétrie différentielle à balayage —
Partie 1: Mesurage de la température de procédé

Reference number
ISO 18373-1:2007(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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 18373-1 was prepared by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, Subcommittee SC 5, *General properties of pipes, fittings and valves of plastic materials and their accessories - Test methods and basic specifications*.

ISO 18373 consists of the following parts, under the general title *Rigid PVC pipes — Differential scanning calorimetry (DSC) method*:

— *Part 1: Measurement of the processing temperature*

— *Part 2: Measurement of the enthalpy of fusion of crystallites*

Introduction

Studies have been undertaken at the international level to determine a method of measuring the B-onset or maximum processing temperature used during the production of rigid PVC pipes. These studies have demonstrated that a test using differential scanning calorimetry (DSC) fulfils these requirements.

The method involves taking small samples from the pipe wall and heating these in a differential scanning calorimeter. Small endotherms are used to detect the thermal history of the samples and the B-onset or maximum processing temperature is derived from these data.

The technique requires a good understanding of DSC instruments and techniques, particularly in relation to PVC. It is important that newcomers to the technique familiarize themselves with both the instrumentation and method prior to undertaking reportable tests.

It is intended that individual product standards will specify the requirements for B-onset or maximum processing temperature.

The method can also be suitable for other types of extruded rigid PVC products, but different sampling protocols might be required.

Rigid PVC pipes — Differential scanning calorimetry (DSC) method —

Part 1: Measurement of the processing temperature

1 Scope

This part of ISO 18373 specifies a method for the determination of the processing temperature of rigid PVC pipe samples. The method is based on the measurement of the thermal history using differential scanning calorimetry (DSC) and is suitable for all types of rigid PVC pipes.

2 Terms and definitions

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

2.1
baseline tilting
adjustment of the angle of the baseline to bring it to the horizontal

2.2
curve magnification
magnification of the differential scanning calorimetry (DSC) curve around A-onset and B-onset temperature ("zooming")

2.3
A-onset
indication of first crystallite melting

2.4
B-onset
indication of maximum processing temperature (T_p)

2.5
instrumental baseline
measurement with empty sample pan, i.e. background subtraction

2.6
position of sample
location in the product from where the sample was taken

2.7
purge gas
gas used to ensure an inert environment

2.8
repeat samples
samples from the same position

3 Principle

DSC is a well-established method for testing the melt temperature in PVC products (see References [1] and [2]).

The benefits with this test are that an accurate assessment can be made of the processing temperature, as well as the possibility of finding variations in the processing temperature in local areas of the product, due to the fact that only a small size of sample is required for the test. This enables the operator to cut samples from different locations around the pipe circumference. Thus, temperature variations in the pipe wall can be examined.

The characteristic B-onset temperature occurs because crystallites with melting points at or above the maximum processing temperature (T_p) will be annealed, thus slightly raising their melting point. Crystallites that do melt at T_p will recrystallize on cooling and therefore will have melting points below T_p. Thus, there are very few crystallites with melting temperatures in the immediate vicinity of T_p.

4 Apparatus

4.1 DSC instrument, and associated software, calibrated.

Calibration should be carried out using at least two different metals. An instrumental baseline must be obtained with an empty sample pan and reference pan in place, and with temperature settings and purge gas identical to the settings to be used for the sample analysis.

4.2 Aluminium sample pans.

4.3 Inert purge gas (e.g. N_2, Ar, etc.), of at least industrial quality.

4.4 Analytical balance, with an accuracy to within 0,01 mg.

4.5 Slow-speed saw (see Reference [3]), **knife**, or any other device introducing neither heat nor stress into the sample as it is cut.

5 Preparation of the test pieces and the test

5.1 Take at least four samples at the 0°, 90°, 180° and 270° positions, respectively, around the pipe circumference, with all the samples being taken from the centre of the pipe wall.

NOTE The samples are taken from the centre of the pipe wall because the processing temperature at the centre of the pipe wall is often lower than that close to the inner and outer surfaces, where additional shear occurs.

CAUTION — Taking samples from the locus of a spider line can lead to an increase in the spread of results.

5.2 Prepare the test pieces having a mass of (20 ± 10) mg in a way that maximizes the contact surface between the pan and the test piece.

NOTE Maximizing the contact surface between the pan and the test piece reduces the resistance to heat flow through the DSC temperature sensors and results in maximum peak sharpness and resolution.

5.3 The best test piece shapes for optimum performance are thin disks placed on the bottom of the pan. Test pieces may be conveniently prepared by cutting out sections with a slow-speed saw or with a razor or knife (4.5). A hole punch or cork borer may be used if the sample is very thin.

6 Procedure

6.1 Ensure that the scanning device is calibrated.

6.2 Encapsulate the test piece in an aluminium pan with cover.

6.3 It is important that the test piece does not move in the pan during the measurement. The most common method to immobilize the test piece is to crimp the pan cover in place with a crimper. This yields a tightly, but not hermetically, sealed pan without movement of the test piece in the pan during the measurement. Other methods of pan closure that immobilize the test piece may be used.

6.4 Using the following test parameters, perform and register the scan:

a) start temperature: (35 ± 15) °C;

b) end temperature: 225 °C;

c) heating rate: (20 ± 1) °C/min;

d) purge gas (4.3): (20 ± 5) °C/min.

7 Expression of results

If necessary, magnify the relevant part of the curve using the zooming feature of the DSC instrument. Determine the B-onset by taking tangents to the DSC curve at the points of maximum slope just before and just after this point of inflection in the curve, as shown in Figure 1.

NOTE 1 A typical curve consists of two endotherms between approximately 100 °C and approximately 200 °C where the B-onset corresponds very closely to the maximum processing temperature, Tp. The change in energy levels is usually quite small. See Figure 2 for an example.

If the individual results from three consecutive tests on samples taken from the same location (i.e. the same angular location) within a pipe differ by more than 3 °C, then further tests shall be made and/or the instrument re-calibrated.

NOTE 2 If the glass transition values, Tg (typically at 70 °C to 80 °C), do not vary by this amount, then the differences reflect real differences in the sample.

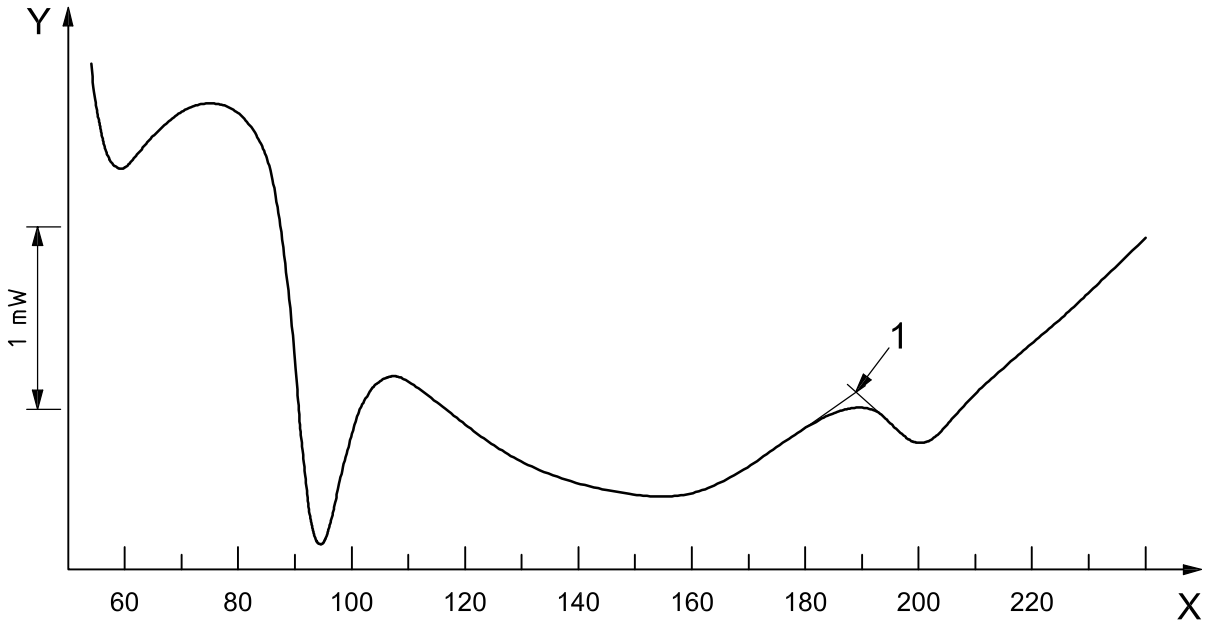
Uneven or irregular scan data shall be discarded.

Possible sources of error are given in Annex C.

The correlation between B-onset temperature and time to stress rupture is given in Annex D.

NOTE 3 The appearance of DSC curves can differ depending upon whether the curve is displayed as “exo up” (see Figures 1 and 2) or “exo down”. The latter type produce “inverted” curves compared with Figures 1 and 2. Examples of the different types of presentation are shown in Annex A.

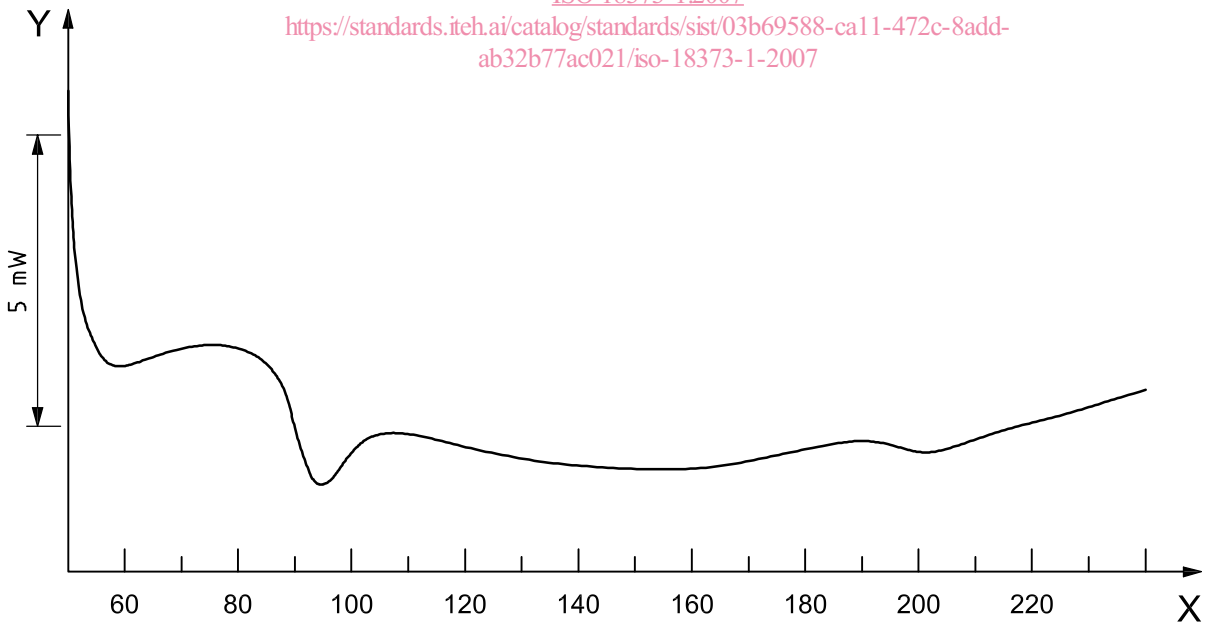
NOTE 4 The presence of some additives can also be detected by DSC and so extra peaks can appear in the DSC curves as a result. Examples of these are shown in Annex B.



Key

X temperature (°C)
Y heat flow (mW)
1 extrapolated peak at 188,36 °C

Figure 1 — DSC result curve after zooming



Key

X temperature (°C)
Y heat flow (mW)

Figure 2 — Typical DSC curve from a PVC pipe

8 Test report

The test report shall contain the following information:

a) reference to this part of ISO 18373;

b) sample reference number (e.g. production code number of the pipe);

c) location from where the samples were taken (e.g. 0°, 90°, 180°, 270° around the circumference of the pipe);

d) mean and standard deviation of B-onset temperature;

e) number of repeat samples tested;

f) minimum value of B-onset measured in any single scan;

g) any factors that could have affected the results, such as any incidents, test interruptions or any operating details not specified in this standard;

h) date(s) of test.