BS EN ISO 11357-4:2014

BSI Standards Publication

Plastics - Differential scanning calorimetry (DSC)

Part 4: Determination of specific heat capacity

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National foreword

This British Standard is the UK implementation of EN ISO 11357-4:2014. It supersedes BS EN ISO 11357-4:2013 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Foreword

This document (EN ISO 11357-4:2014) 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 NBN.

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of an identical text or by endorsement, at the latest by December 2014, and conflicting national standards shall be withdrawn at the latest by December 2014.

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The text of ISO 11357-4:2014 has been approved by CEN as EN ISO 11357-4:2014 without any modification.

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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.

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 documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical and chemical properties*.

This second edition cancels and replaces the first edition (ISO 11357-4:2005). This minor revision contains the following changes:

a) all normative reference were changed into undated ones;

b) the term "pan" was replaced by "crucible" within the whole text;

c) the endothermic direction, a, was added in all figures and key.

ISO 11357 consists of the following parts, under the general title *Plastics — Differential scanning calorimetry (DSC)*:

— *Part 1: General principles*

— *Part 2: Determination of glass transition temperature and glass transition step height*

— *Part 3: Determination of temperature and enthalpy of melting and crystallization*

— *Part 4: Determination of specific heat capacity*

— *Part 5: Determination of characteristic reaction-curve temperatures and times, enthalpy of reaction and degree of conversion*

— *Part 6: Determination of oxidation induction time (isothermal OIT) and oxidation induction temperature (dynamic OIT)*

— *Part 7: Determination of crystallization kinetics*

Plastics — Differential scanning calorimetry (DSC) —

Part 4: Determination of specific heat capacity

1 Scope

This part of ISO 11357 specifies methods for determining the specific heat capacity of plastics by differential scanning calorimetry.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for the application of this document. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

ISO 11357-1, *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*

ISO 80000-1, *Quantities and units — Part 1: General*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 11357-1 and the following apply.

3.1
calibration material
material of known specific heat capacity

Note 1 to entry: Usually, α-alumina (such as synthetic sapphire) of 99,9 % or higher purity is used as the calibration material.

3.2
specific heat capacity (at constant pressure)
c_p
quantity of heat necessary to raise the temperature of unit mass of material by 1 K at constant pressure

Note 1 to entry: It is given by the following formula:

$$c_p = m^{-1}C_p = m^{-1}(dQ/dT)_p \quad (1)$$

where

m is the mass of material;

c_p is the heat capacity and is expressed in kilojoules per kilogram per K ($kJ \cdot kg^{-1} \cdot K^{-1}$) or in joules per gram per K ($J \cdot g^{-1} \cdot K^{-1}$); subscript p indicates an isobaric process;

dQ is the quantity of heat necessary to raise the temperature of the material by dT;

Note 2 to entry: This formula is valid in a temperature range where a material shows no first-order phase transition.

$$(dQ/dT) = (dt/dT) \times (dQ/dt) = (\text{heating rate})^{-1} \times (\text{heat flow rate}) \qquad (2)$$

Note 3 to entry: At phase transitions, there is a discontinuity in the heat capacity. Part of the heat is consumed to produce a material state of higher energy and it is not all used in raising the temperature. For this reason, the specific heat can only be determined properly outside regions of phase transitions.

4 Principle

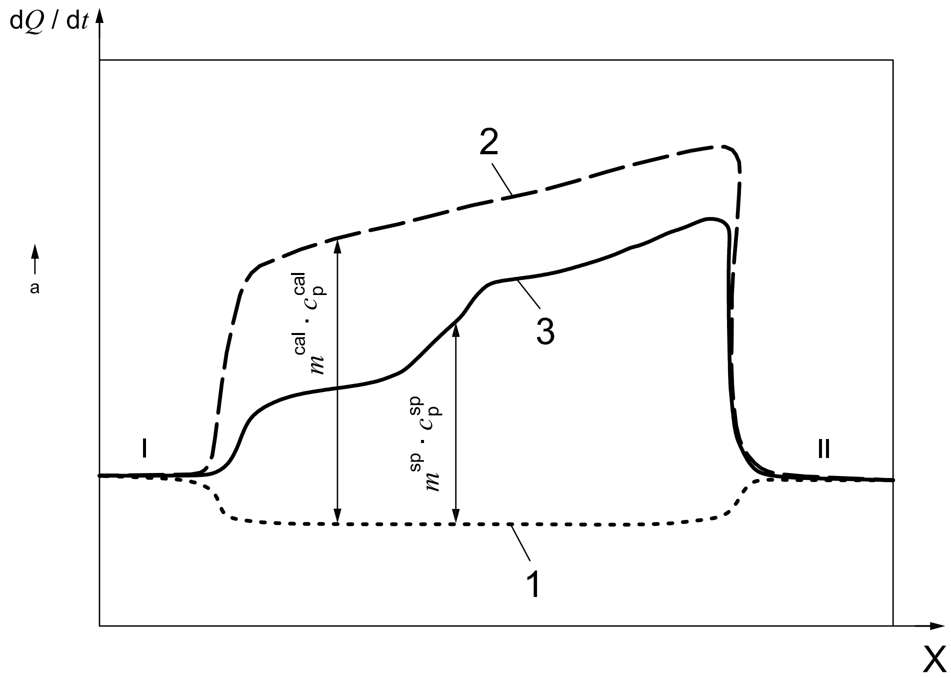
4.1 General

Each measurement consists of three runs at the same scanning rate (see Figure 1):

a) a blank run (empty crucibles in sample and reference holders);

b) a calibration run (calibration material in sample holder crucible and empty crucible in reference holder);

c) a specimen run (specimen in sample holder crucible and empty crucible in reference holder).



Key

X temperature T or time t
1 blank run
2 calibration run
3 specimen run
I isothermal baseline at start temperature T_s
II isothermal baseline at end temperature T_f
a Endothermic direction.

Figure 1 — Schematic drawing of typical DSC curves for specific heat capacity measurement (blank, calibration and specimen runs) after baseline adjustment

4.2 Continuous-scanning method

Based on the DSC principle (see ISO 11357-1) and the definition of specific heat capacity given in 3.2, the following relations can be obtained:

$$m^{sp} \cdot c_p^{sp} \propto P_{specimen\ run} - P_{blank\ run} \tag{3}$$

$$m^{cal} \cdot c_p^{cal} \propto P_{calibration\ run} - P_{blank\ run} \tag{4}$$

where P is the heat flow rate (dQ/dt); superscripts sp and cal represent specimen and calibration material, respectively (see Figure 1).

When $P_{specimen\ run}$, $P_{calibration\ run}$ and $P_{blank\ run}$ are measured, c_p^{sp} can be calculated using Formula (6), since the values of c_p^{cal}, m^{sp} and m^{cal} are known:

$$\frac{m^{sp} \cdot c_p^{sp}}{m^{cal} \cdot c_p^{cal}} = \frac{P_{specimen\ run} - P_{blank\ run}}{P_{calibration\ run} - P_{blank\ run}} \tag{5}$$

$$c_p^{sp} = c_p^{cal} \cdot \frac{m^{cal}\left(P_{specimen\ run} - P_{blank\ run}\right)}{m^{sp}\left(P_{calibration\ run} - P_{blank\ run}\right)} \tag{6}$$

4.3 Stepwise-scanning method

In the stepwise-scanning method, the total temperature range to be scanned is divided into small intervals and a complete determination consisting of the three runs mentioned in 4.1 is performed for each temperature interval. Upon integration of the heat flow rate curve, the total heat ΔQ consumed in the interval can be obtained. Dividing ΔQ by the temperature interval ΔT and the mass of the specimen gives the specific heat [see Formula (1)]:

$$m^{\text{sp}} \cdot c_{\text{p}}^{\text{sp}} \propto \left(\frac{\Delta Q^{\text{sp}}}{\Delta T} \right)_{\text{p}} - \left(\frac{\Delta Q^{\text{blank}}}{\Delta T} \right)_{\text{p}} \tag{7}$$

$$m^{\text{cal}} \cdot c_{\text{p}}^{\text{cal}} \propto \left(\frac{\Delta Q^{\text{cal}}}{\Delta T} \right)_{\text{p}} - \left(\frac{\Delta Q^{\text{blank}}}{\Delta T} \right)_{\text{p}} \tag{8}$$

Keeping the temperature intervals ΔT constant, combining Formulae (7) and (8) results in:

$$c_{\text{p}}^{\text{sp}} = c_{\text{p}}^{\text{cal}} \cdot \frac{m^{\text{cal}}}{m^{\text{sp}}} \cdot \frac{\Delta Q^{\text{sp}} - \Delta Q^{\text{blank}}}{\Delta Q^{\text{cal}} - \Delta Q^{\text{blank}}} \tag{9}$$

5 Apparatus

5.1 DSC apparatus. See ISO 11357-1.

5.2 Crucibles. See ISO 11357-1.

The crucibles for the test specimen and the reference specimen (calibration material) shall be of the same shape and material and their masses shall not differ by more than 0,1 mg.

NOTE The same blank run and calibration run can be used for several measurements, if the instrument is sufficiently stable and the difference in mass between the calibration material crucible and the empty crucible is corrected for. An adequate correction can be obtained by adding the term $c_{\text{p, crucible}}(T)\beta\Delta m$ to the heat flow rate of the calibration run, where $c_{\text{p,crucible}}(T)$ is the specific heat capacity of the calibration crucible as a function of temperature, β is the heating rate and Δm is the difference in mass between the calibration crucible and the empty crucible. The same procedure can also be used for correcting differences in mass between the specimen run and the blank run.

5.3 Analytical balance. See ISO 11357-1.

6 Test specimen

See ISO 11357-1.

7 Test conditions and specimen conditioning

See ISO 11357-1.

8 Procedure

8.1 Selection of crucibles

Prepare three crucibles and their lids and weigh the crucibles together with their lids. The total mass shall not differ by more than 0,1 mg (see 5.2). In other respects, such as material, size, crucible type (open or sealed), the crucibles shall be identical.

8.2 Setting up the apparatus and adjustment of isothermal baselines

8.2.1 Place a pair of empty crucibles with lids in the DSC sample and reference holders.

8.2.2 If using a continuous-scan programme:

a) Set the start and end temperatures (T_s and T_f). The start temperature T_s should be at least 30 K lower than that at which data are first required.

NOTE 1 When more precise results are required over a wide temperature range, the overall range can be divided into two (or more) smaller ranges, each 50 K to 100 K wide. The start temperature T_s of the second range should be 30 K lower than the end temperature T_f of the first temperature range to ensure sufficient overlap.

b) Set the scanning rate.

c) Set the time interval between the isothermal stages I and II (see Figure 1) and allow the respective isothermal baselines to stabilize. This interval will usually be between 2 min and 10 min.

NOTE 2 Some calorimeters, e.g. those of the Calvet type, may need up to 30 min before the baseline stabilizes.

8.2.3 If using a stepwise-scan programme:

When the specific heat capacities of the samples do not significantly depend on the temperature, the stepwise-scanning method can be used in which the integration of the heat flow over small temperature intervals gives a set of individual specific heat values for the temperature intervals considered. Attention shall be paid to the following points:

a) The time interval between the isothermal stages shall be sufficiently long to obtain a stable baseline.

b) This method shall not be used over a temperature range in which first-order phase transitions occur.

The stepwise scan is performed as follows:

— Set the start and end temperatures (T_s and T_f).

— Set the temperature increment preferably to 5 K to 10 K.

— Set the temperature-scanning rate to 5 K·min^{-1} or 10 K·min^{-1}.

— Set the time interval between the isothermal stages, usually to between 2 min and 10 min.

8.2.4 Set the sensitivity of the heat flow rate in order to obtain an ordinate span of at least 80 % of full scale (see Figure 1).

8.2.5 Adjust the apparatus so that the isothermal baselines before and after the heating stage are at the same ordinate level.

If microcomputer-based systems are used, the isothermal baselines can be adjusted to the same ordinate level after the data has been acquired. However, it is strongly recommended that baseline adjustment is done before any measurements are made in order to improve the accuracy of the results. If a conventional pen recorder is used, proper apparatus adjustment is crucial to minimize differences in isothermal baseline level.

Check that adjustment of the baselines of the respective DSC curves results in the same ordinate level. If the baseline reproducibility is poor, readjust the apparatus and repeat the determination.

NOTE Other reasons for poor baseline reproducibility can be contamination of the sample crucible, the position of the lid, the stability of the purge gas flow rate, sample decomposition, sample evaporation, chemical reaction between crucible and sample, etc.

8.2.6 Execute the temperature programme set as described in 8.2.2 or 8.2.3. Figure 2 shows a typical DSC curve obtained in the continuous-scanning mode whereas Figure 3 shows a DSC curve obtained in the stepwise-scanning mode.

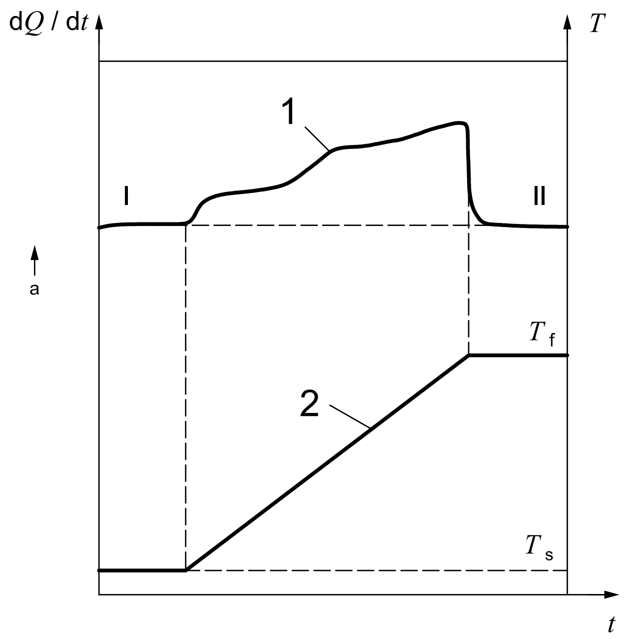
8.3 Measurement of specific heat capacity of calibration material

Using an analytical balance, weigh a calibration material, such as *α*-alumina (synthetic sapphire) of 99,9 % or higher purity, into one of the crucibles prepared in 8.1. Put the crucible containing the calibration material, with the lid, in the sample holder and perform a DSC run.

NOTE 1 Small differences in the masses of the crucibles used for the specimen, calibration and blank runs can be corrected for as indicated in the Note to 5.2.

NOTE 2 The heat capacity of the calibration material should match that of the specimen to be analysed as closely as possible in order to minimize systematic errors.

For the blank run, use another of the empty crucibles prepared in 8.1. Carry out the same measurement(s) as described in 8.2. The nominal values of the specific heat capacity of α-alumina at various temperatures are given in Table A.1.



Key

1 DSC curve

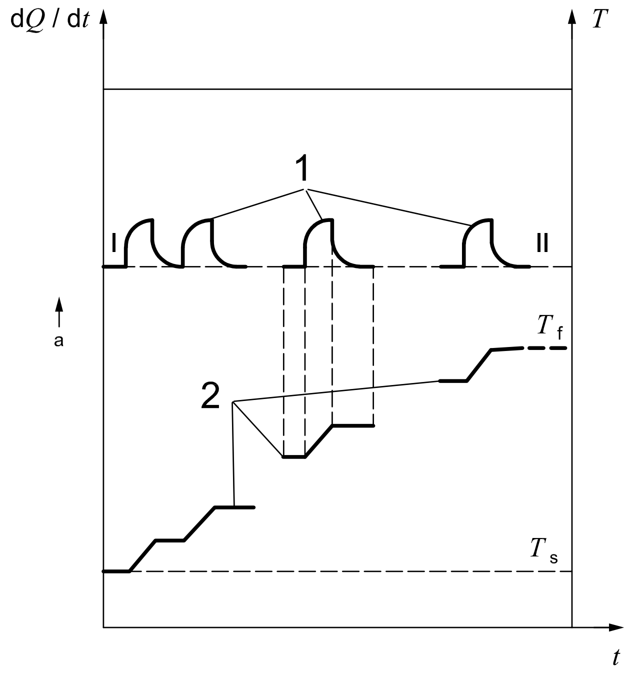
2 temperature curve

I isothermal baseline at start temperature T_s

II isothermal baseline at start temperature T_f

a Endothermic direction.

Figure 2 — Schematic drawing of a continuous-scan DSC curve



Key

1 DSC curve

2 temperature curve

I isothermal baseline at start temperature T_s

II isothermal baseline at start temperature T_f

a Endothermic direction.

Figure 3 — Schematic drawing of a stepwise-scan DSC curve

8.4 Specimen run

Weigh the test specimen into the sample crucible. Put the crucible containing the test specimen, with the lid, into the sample holder and perform a DSC run as for the calibration material. A large mass of sample is recommended.

The same blank run as was used for the calibration run in 8.3 can also be used for the specimen run.

9 Determination of specific heat capacities

9.1 Calculation of specific heat capacities

Calculate c_p^{sp}, in $J \cdot g^{-1} \cdot K^{-1}$, using Formula (6) for continuous heating or Formula (9) for the stepwise method.

9.2 Numerical rounding of the results

Round the specific heat capacity values thus obtained to the second decimal place, using the method specified in ISO 80000-1.

10 Precision and bias

The precision and bias of the methods described are not known because of a lack of interlaboratory testing. A precision statement will be added as soon as sufficient data are available.

11 Test report

The following items shall be reported:

a) a reference to this part of ISO 11357;

b) the date of the test;

c) all details necessary for complete identification of the sample tested, including the thermal history;

d) the manufacturer, model and type (power compensation or heat flux) of DSC apparatus used;

e) the shape, dimensions and materials of the crucible and lid;

f) the test atmosphere and the flow rate of the inflow gas;

g) the calibration material, including information on the issuing body, the nature of the material, the mass used and other characteristics relevant to the calibration;

h) the shape, dimensions and mass of the test specimen;

i) details of sampling and the conditioning of the test specimen;

j) the temperature programme parameters, i.e. the start temperature, the heating rate, the end temperature, the time interval between the isothermal stages and, in the stepwise-heating method, the temperature increment, as well as the cooling rate in conditioning, if carried out;

k) the test results, including the specific heat capacities and the respective temperatures;

l) other items required, if any.

Annex A
(informative)

An approximate expression of the specific heat capacityof pure α-alumina [3] to [5]

The specific heat capacity values in Table A.1 are approximated by the following formulas:

$$c_p = A_0 + A_1 x + A_3 x^2 + A_3 x^3 + A_4 x^4 + A_5 x^5 + A_6 x^6 + A_7 x^7 + A_8 x^8 + A_9 x^9 + A_{10} x^{10} \quad (A.1)$$

$$x = (T\,\mathrm{K} - 650\,\mathrm{K})/550\,\mathrm{K} \quad (A.2)$$

$$= (\theta\,°\mathrm{C} - 376{,}85\,°\mathrm{C})/550\,°\mathrm{C} \quad (A.3)$$

$$\theta\,°\mathrm{C} = T\,\mathrm{K} - 273{,}15\,\mathrm{K} \quad (A.4)$$

where

$100\,\mathrm{K} \le T \le 1\,200\,\mathrm{K}$

and

$A_0 = 1{,}127\ 05$

$A_1 = 0{,}232\ 60$

$A_2 = -0{,}217\ 04$

$A_3 = 0{,}264\ 10$

$A_4 = -0{,}237\ 78$

$A_5 = -0{,}100\ 23$

$A_6 = 0{,}153\ 93$

$A_7 = 0{,}545\ 79$

$A_8 = -0{,}478\ 24$

$A_9 = -0{,}376\ 23$

$A_{10} = 0{,}344\ 07$

and

c_p and A_i (i = 1, 2, ...) are in $\mathrm{J \cdot g^{-1} \cdot K^{-1}}$;

T is in K;

θ is in °C

The coefficients in Formulae (A.2) and (A.2a) are for normalizing the temperature variables T and θ.

The standard deviation from the values in Table A.1 is 0,000 13 $J \cdot g^{-1} \cdot K^{-1}$.

The maximum deviation is 0,071 % at 140 K.

The standard deviation at temperatures higher than 300 K is less than 0,02 %.

Table A.1 — Specific heat capacity of pure α-alumina in the temperature region from 120 K to 780 K [3] to [5]

Temperature		Specific heat capacity	Temperature		Specific heat capacity
K	°C	J·g−1·K−1	K	°C	J·g−1·K−1
120,00	−153,15	0,196 9	440,00	166,85	0,987 5
130,00	−143,15	0,235 0	450,00	176,85	0,997 5
140,00	−133,15	0,274 0	460,00	186,85	1,007 0
150,00	−123,15	0,313 3	470,00	196,85	1,016 0
160,00	−113,15	0,352 5	480,00	206,85	1,024 7
170,00	−103,15	0,391 3	490,00	216,85	1,033 0
180,00	−93,15	0,429 1	500,00	226,85	1,040 8
190,00	−83,15	0,465 9	510,00	236,85	1,048 4
200,00	−73,15	0,501 4	520,00	246,85	1,055 6
210,00	−63,15	0,535 5	530,00	256,85	1,062 6
220,00	−53,15	0,568 2	540,00	266,85	1,069 2
230,00	−43,15	0,599 4	550,00	276,85	1,075 6
240,00	−33,15	0,629 2	560,00	286,85	1,081 6
250,00	−23,15	0,657 6	570,00	296,85	1,087 5
260,00	−13,15	0,684 5	580,00	306,85	1,093 1
270,00	−3,15	0,710 1	590,00	316,85	1,098 6
280,00	6,85	0,734 2	600,00	326,85	1,103 8
290,00	16,85	0,757 1	610,00	336,85	1,108 8
300,00	26,85	0,778 8	620,00	346,85	1,113 6
310,00	36,85	0,799 4	630,00	356,85	1,118 2
320,00	46,85	0,818 6	640,00	366,85	1,122 7
330,00	56,85	0,837 2	650,00	376,85	1,127 0
340,00	66,85	0,854 8	660,00	386,85	1,131 3
350,00	76,85	0,871 3	670,00	396,85	1,135 3
360,00	86,85	0,887 1	680,00	406,85	1,139 2
370,00	96,85	0,902 0	690,00	416,85	1,143 0
380,00	106,85	0,916 1	700,00	426,85	1,146 7
390,00	116,85	0,929 5	720,00	446,85	1,153 7
400,00	126,85	0,942 3	740,00	466,85	1,160 4
410,00	136,85	0,954 4	760,00	486,85	1,166 7
420,00	146,85	0,966 0	780,00	506,85	1,172 6
430,00	156,85	0,977 0			

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