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Thermal insulation — Test methods for specific heat capacity of thermal insulation for buildings in the high temperature range — Differential scanning calorimetry (DSC) method

Isolation thermique — Méthodes d'essai relatives à la capacité thermique massique de l'isolation thermique des bâtiments dans la plage de température élevée — Méthode par calorimétrie à balayage différentiel (DSC)

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Foreword

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This document was prepared by Technical Committee ISO/TC 163, *Thermal performance and energy use in the built environment*, Subcommittee SC 1, *Test and measurement methods*.

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Thermal insulation — Test methods for specific heat capacity of thermal insulation for buildings in the high temperature range — Differential scanning calorimetry (DSC) method

1 Scope

This document specifies test methods for specific heat capacity under high temperature conditions from the normal temperature range to 1 600 K for insulation materials for buildings using the differential scanning calorimetry (DSC) method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 11357-1:20—,[1] *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*

ISO 11357-4:2021, *Plastics — Differential scanning calorimetry (DSC) — Part 4: Determination of specific heat capacity*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11357-1, ISO 11357-4 and the following 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

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

3.1
specific heat capacity
c_p
quantity of heat necessary to raise the temperature of a unit mass of material by 1 K at constant pressure

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

$$c_p = \frac{1}{m} \times \left(\frac{\mathrm{d}Q}{\mathrm{d}T} \right)_p \tag{1}$$

where

c_p is the specific 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;

m is the mass of material, expressed in kilogram (kg) or gram (g);

1) Under preparation. Stage at time of publication: ISO/FDIS 11357-1.

$\left(\frac{dQ}{dT}\right)_p$ is the quantity of heat dQ necessary to raise the temperature of the material by dT, expressed in kilojoules per K (kJ·K^{-1}) or in joules per K (J·K^{-1}), measured at constant pressure.

3.2
specimen
item which is cut from thermal insulation material and processed into powder form or compression moulded for measurement by differential scanning calorimetry (DSC)

Note 1 to entry: See Annex B for further information on moulding procedure.

3.3
reference material
material of known *specific heat capacity* (3.1)

Note 1 to entry: See ISO 11357-4:2021, Annex A for further information.

3.4
calibration material
material of known temperature and heat of fusion

Note 1 to entry: See Annex D for further information on calibration materials.

3.5
three-step temperature control method
method that consists of isothermal maintenance at the start temperature, constant heating step at the middle temperature and isothermal maintenance at the end temperature

3.6
differential scanning calorimetry
DSC
method in which the difference in energy inputs into a substance and a reference material is measured as a function of temperature while the substance and reference material are subjected to a controlled temperature programme

[SOURCE: ISO/TS 80004-6:2021, 6.2.1]

4 Principles

4.1 General

DSC is a method in which the difference in energy inputs into a substance and a reference material is measured as a function of temperature while the substance and reference material are subjected to a controlled temperature programme.

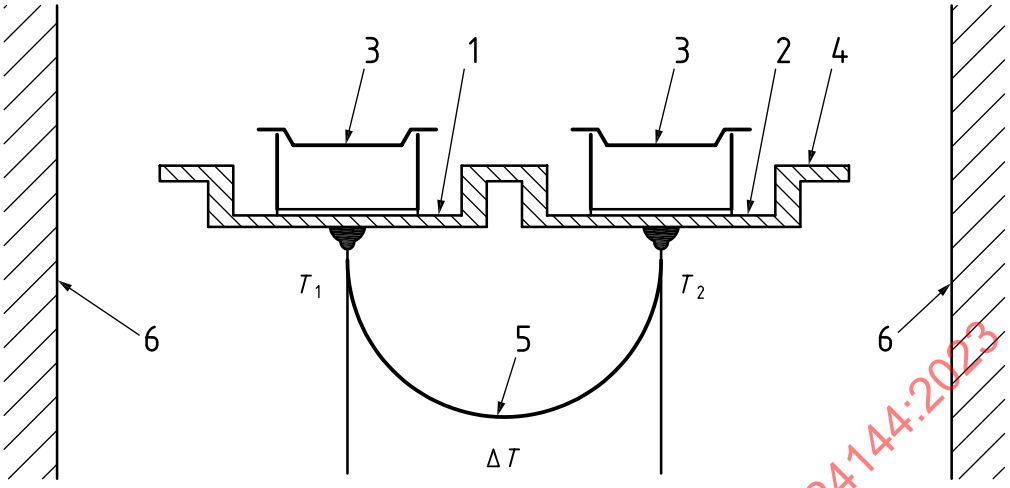
The difference between the rate of heat flow into a specimen and the rate of heat flow into a reference crucible is measured as a function of either temperature or time, or both, while the specimen and the reference are subjected to the same temperature-control programme under a specified atmosphere.

The measurements can be undertaken using two types of DSC: heat-flux DSC and power-compensation DSC.

4.2 Heat-flux DSC

The specimen and reference positions are subjected to the same temperature-control programme by a single heater. A difference in temperature, ΔT, occurs between the specimen position and the reference position because of the difference in heat capacity between the specimen and the reference. From this temperature difference, the difference in the rates of heat flow into the specimen and reference positions is derived and is normally recorded against the temperature of the reference, T_{ref}, or against time.

A schematic drawing of a heat-flux DSC instrument is shown in Figure 1.



Key

1	sample holder	6	heating furnace
2	reference holder	T_1	temperature at sample holder ($T_{specimen}$)
3	crucible	T_2	temperature at reference holder (T_{ref})
4	stage	ΔT	temperature difference between sample and reference holder
5	thermopile		

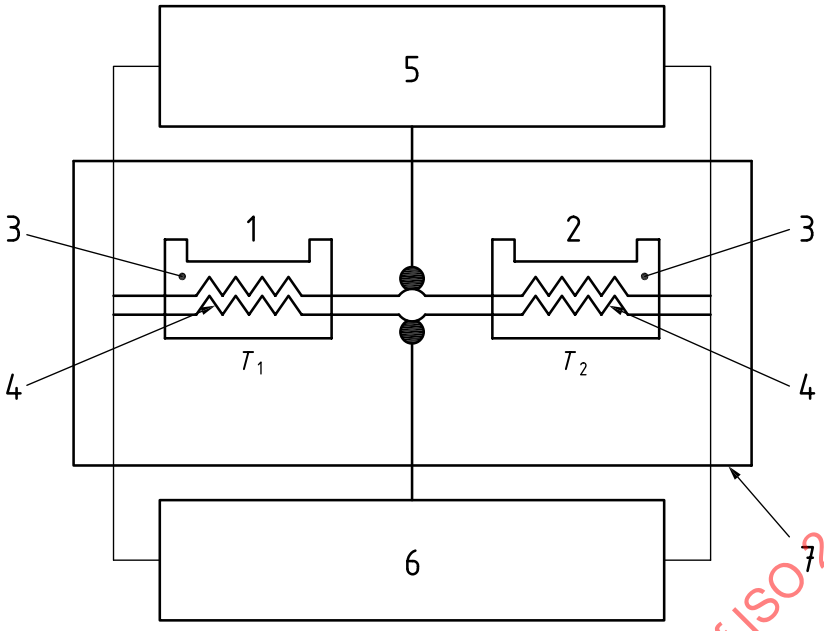
Figure 1 — Schematic diagram illustrating the basic principles of heat-flux DSC

4.3 Power-compensation DSC

In power-compensated DSC, individual heaters are used for the specimen and reference positions. The difference in electrical power required to maintain both the specimen position and the reference position at the same temperature is recorded against temperature or time, while each position is subjected to the same temperature-control programme.

For power-compensated isoperibolic calorimeters, the surrounding temperature (i.e. the temperature of the heat sink) shall be kept constant.

A schematic drawing of a power-compensation DSC instrument is shown in Figure 2.



Key

1	specimen position	6	heat-flux compensation circuit
2	reference position	7	surrounding heat sink
3	thermometers	T_1	temperature at specimen position ($T_{specimen}$)
4	individual heaters	T_2	temperature at reference position (T_{ref})
5	measurement circuit for $T_{specimen}$ and T_{ref}		

Figure 2 — Schematic diagram illustrating the basic principles of power-compensation DSC

5 Method

5.1 General

This document specifies methods for the measurement of specific heat capacity according to the heat-flux DSC method and the power-compensation DSC method, based on the three-step temperature control method.

The apparatus for both methods comprises two measuring cells (sample holders) housed in a furnace which provides overall system heating. One cell contains the test specimen within a crucible, and the other contains an empty crucible only.

a) Power-compensation DSC method

Each cell has an individual heater to compensate for temperature variations from the overall heating programme. The power which is supplied to either cell heater to maintain equal temperatures during heating is measured.

b) Heat-flux DSC method

Power is exchanged between each cell and its respective surrounding during the heating programme. The difference in power exchange between the two cells is measured.

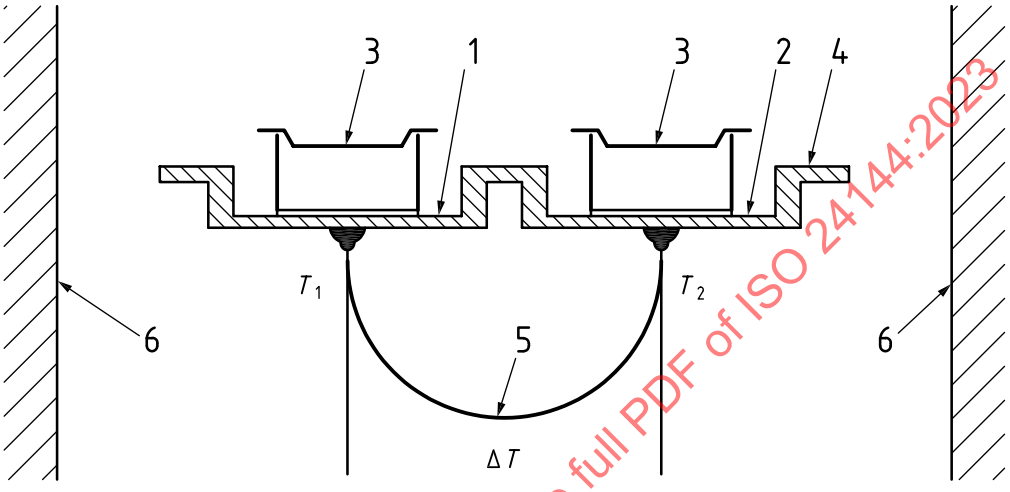
5.2 Basic procedure

Each measurement consists of three runs at the same scanning rate (see Figure 3 and Figure 4):

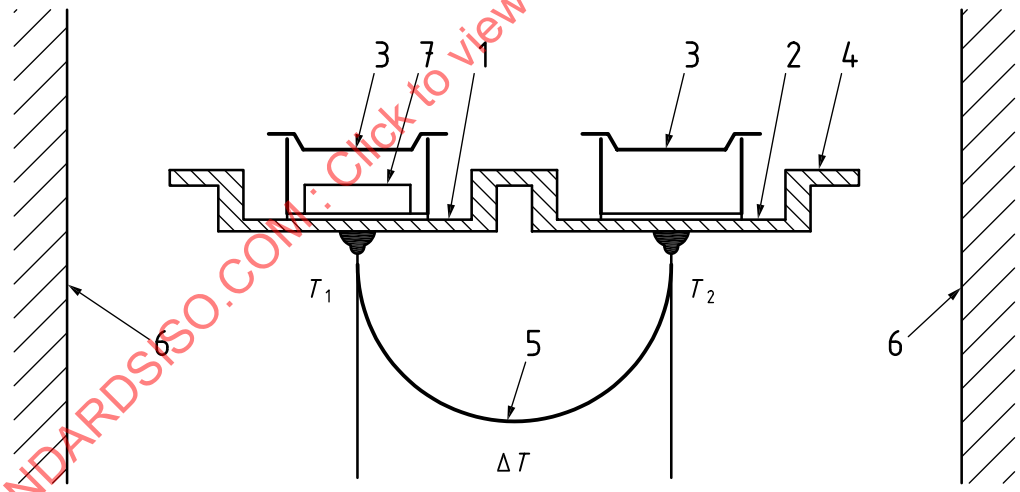
a) a blank run (empty crucibles in sample and reference holders);

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

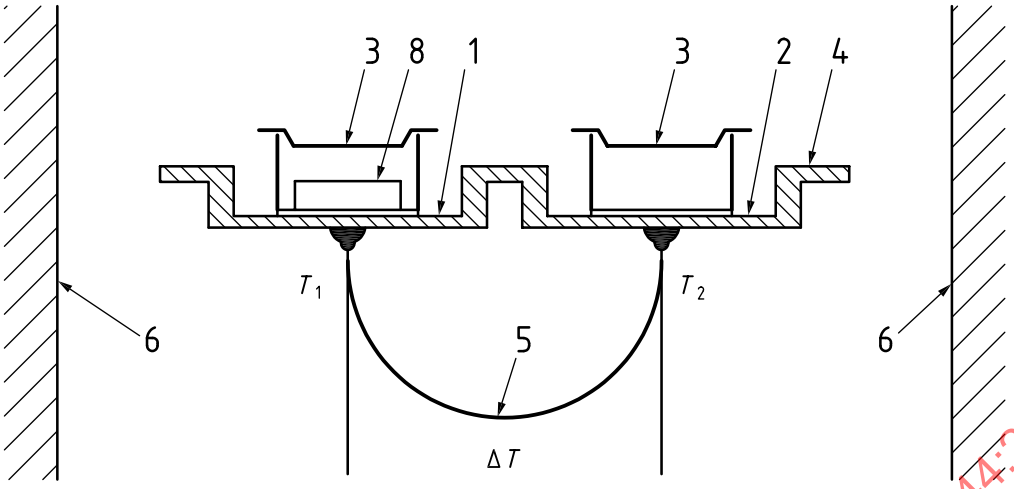
c) a specimen run (specimen in sample holder crucible and empty crucible in reference holder).



a) Blank run



b) Calibration run

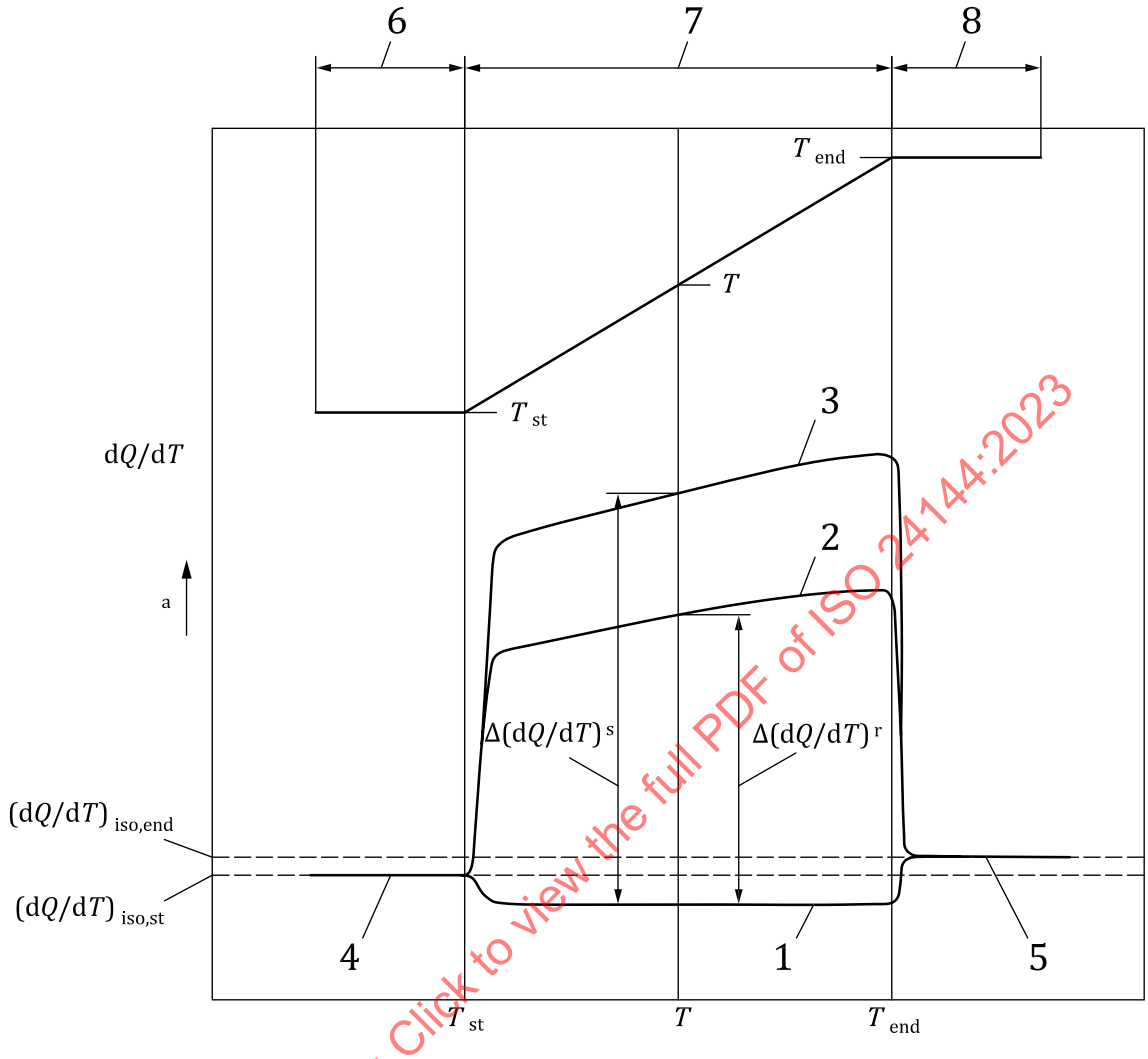


c) Specimen run

Key

1	sample holder	5	thermopile
2	reference holder	6	heating furnace
3	crucible	7	reference material
4	stage	8	specimen
T_1	temperature at sample holder ($T_{specimen}$)		
T_2	temperature at reference holder (T_{ref})		
ΔT	temperature difference between sample and reference holders		

Figure 3 — Three scanning runs



Key

1	empty run	7	temperature-rise region II
2	calibration specimen run	8	isothermal region III
3	test specimen run	a	Endothermic direction.
4	isothermal start lines	T	temperature
5	isothermal end lines	dQ/dT	heat flow rate
6	isothermal region I		

Figure 4 — DSC curves for heat flow rate

6 Apparatus and materials

6.1 DSC apparatus

The specification of the DSC apparatus is the following:

a) A symmetrical crucible holder assembly which has holders for the specimen and reference crucibles. Both holders are made of the same material and of equal mass.

b) The capability to generate constant heating rates suitable for the intended measurements.

c) The capability to maintain the test temperature constant to within ±0,3 K or less for at least 60 min.

d) The capability to carry out step heating.

NOTE 1 Normally, this is achieved by a suitable combination of linear heating and constant temperature regimes.

e) The capability to maintain a constant purge gas flow rate controllable to within ±10 % over a range of flow rates (e.g. 10 ml·min^{-1} to 100 ml·min^{-1}).

NOTE 2 The actual gas flow rate depends on the design of the instrument used.

f) A temperature range in line with the experimental requirements.

g) A recording device capable of automatically recording the measured curve of heat flow rate against temperature and time.

h) The capability to measure temperature signals with a resolution of ±0,1 K.

i) The capability to measure time with a resolution of ±0,5 s and an accuracy of ±1 s or better.

j) The capability to measure heat flow rates with a resolution of ±0,5 µW and an accuracy of ±2 µW or better.

DSC apparatus for high temperature range shall be according to Annex A.

6.2 Crucibles

6.2.1 General

Crucibles shall be according to ISO 11357-1.

6.2.2 Shape, material and mass

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 2 %.

6.2.3 Measurement temperature range

The material of a crucible shall resist the upper limit of the measurement temperature. As a guide, the upper limit of the operating temperature for a crucible shall be as indicated below. Refer to ISO 11357-1:20—[2], Annex D for the appropriate crucible types.

In the measurement temperature range, the sample and the crucible shall not react with each other, and the crucible shall not be welded to the DSC sensor. If necessary, place an alumina disk between the bases of the cell crucibles to prevent welding to the sensor with the following specifications:

— aluminium crucible: 873 K (600 °C);

— platinum-rhodium crucible: 1 873 K (1 600 °C);

— alumina crucible (sheet or liner): 1 873 K (1 600 °C).

7 Test specimen

7.1 General

The specimen shall be cut from a product of thermal insulation material to a suitable size. Specimen masses should be between 10 mg to 100 mg.

2) Under preparation. Stage at time of publication: ISO/FDIS 11357-1.

The specimen shall be representative of the sample being examined and shall be prepared and handled with care. Particular care shall be taken to avoid any contamination of the specimen.

The bottom of the crucible shall not have any deformation.

Good thermal contact between the specimen and crucible and between the crucible and holder shall be ensured.

7.2 Sampling

The procedure for selecting the sample should normally be specified in the material specification. The selection of the specimen from the sample may be partly specified in the material specification.

As sampling is beyond the scope of this method, when the sampling aspect is not covered by a material specification, reference shall be made to appropriate documents.

7.3 Moulding

The moulding procedure of the test specimen shall be carried out as specified in Annex B.

The form of the specimen should be moulded into a form that fits into the crucible used for the measurement.

It is necessary to prepare a sufficient volume of sample to make a compressed specimen.

The mass and the thickness of the compressed specimen shall be measured.

When it is difficult to compress the specimen, the sample shall be crimped to fit the crucible.

8 Test conditions and specimen conditioning

8.1 Test conditions

The instrument shall be maintained and operated in an atmosphere suitable for the intended test.

It is recommended that the instrument be protected from air draughts, exposure to direct sunlight and abrupt changes in temperature, humidity, pressure or mains voltage.

Measurement of heat capacity (baseline measurement, standard specimen measurement and specimen measurement) should be completed in one day.

8.2 Conditioning of specimens

Specimens shall be conditioned prior to the measurement run as specified in the relevant material specification or by a method agreed between the interested parties.

Unless otherwise specified, specimens should be dried to a constant mass before performing measurements.

Care should be taken to choose suitable drying conditions to prevent physical changes or changes in the crystallinity of the specimens.

9 Calibration

9.1 General

Calibration shall be according to ISO 11357-1.

9.2 Calibration materials

The temperature and enthalpy of transition of a selection of calibration materials are shown in Annex D.

When performing temperature calibration, the entire measurement temperature range shall be included.

Normally, changes in the enthalpy of substances due the fusion shall be considered in the calibration. However, α-alumina or sapphire can be used (see ISO 11357-4:2021, Annex A).

10 Procedure

10.1 Setting up the apparatus

Setting up the apparatus shall be according to ISO 11357-1.

10.2 Loading the specimen into the crucible

10.2.1 General

Do not handle the specimen, sample material or crucibles with bare hands. Use either tweezers or gloves.

10.2.2 Selection of crucibles

Use only clean crucibles of volume and material appropriate for the intended measurements.

Unless excluded by the particular type of test, use closed, ventilated crucibles to obtain quantitative results and to allow sufficient contact with the purge gas. The crucibles shall be such that they will not become deformed during loading and closing so that good thermal contact is ensured between the specimen and the instrument.

Select two identical crucibles, one for a blank reference material or the specimen and the other (normally empty) for the reference crucible.

10.2.3 Weighing the specimen crucible

Weigh the specimen crucible together with its lid, to the nearest 0,1 mg.

10.2.4 Loading the specimen

Load the specimen into the specimen crucible. The specimen mass depends on the thermal effect being investigated and is specified in more detail in ISO 11357-1.

If necessary, homogenize the sample to be investigated to obtain a representative specimen.

If the specimen contains silicon carbide (SiC), an alumina liner shall be used.

If the reactivity between a sample and the crucible is unknown, it is recommended that it should be checked beforehand by using another heating furnace. As an example, in the case where the reactivity between a sample and a platinum-rhodium crucible is determined, a small amount of sample is placed on a platinum foil and heated. If traces are left on the platinum foil after heating, reactivity between the two materials exists. Therefore, some measure such as laying a thin alumina liner inside the platinum-rhodium sample crucible for use is required.

10.2.5 Determination of the mass of the specimen

Weigh the crucible containing the specimen and calculate the mass of the specimen by subtracting the mass of the empty crucible determined in 10.2.3.

The specimen under investigation should preferably not contain volatile substances, which should be eliminated by appropriate conditioning. However, it should be taken into account that conditioning can change the specimen by inducing chemical reactions, removing volatile substances, ageing or changing the morphology or crystallinity. If volatile substances are an important part of the specimen under investigation, gas- and pressure-tight crucibles or a pressure DSC instrument should be used.

10.3 Performing measurements

Performing measurements shall be according to ISO 11357-1.

10.4 Post-run checks

Post-run checks shall be according to ISO 11357-1.

11 Determination of specific heat capacities

11.1 General

Based on the recorded measurement data, calculate the specific heat capacities by the following procedure.

11.2 Calculation of specific heat capacities

11.2.1 In case of near-match with isothermal baselines of DSC curves

If the isothermal baselines of the three DSC curves given in Clause 10 almost match, draw a figure by parallel movement of the T_i and T_f isothermal baselines in the vertical axis direction to overlap.

Calculate c_{ps}, in $J \cdot g^{-1} \cdot K^{-1}$, by using Formula (2).

$$c_{ps}(T) = \frac{D_s(T)}{D_r(T)} \times \frac{m_r}{m_s} \times c_{pr}(T) \tag{2}$$

where

$c_{ps}(T)$ is the specific heat capacity of the specimen at temperature T, $JK^{-1}g^{-1}$;

$c_{pr}(T)$ is the specific heat capacity of the reference (standard specimen) at temperature T, $JK^{-1}g^{-1}$;

$D_s(T)$ is the signal displacement of the DSC curve of the specimen measured based on the measurement of the empty crucible, mW;

$D_r(T)$ is the signal displacement of the DSC curve of the reference material measured based on the measurement of the empty crucible, mW;

m_s is the mass of the specimen, g;

m_r is the mass of the reference (standard specimen), g.

Determine $c_{pr}(T)$ by using the data shown in ISO 11357-4:2021, Annex A and interpolation of its data which exist above and below temperature T.

11.2.2 In case of discordance with isothermal baseline of DSC curves

When the isothermal baselines of the three DSC curves given in Clause 10 are in discordance (i.e. when the isothermal baselines for each DSC curve, either the maximum signal displacement of the specimen or the reference material, do not agree by more than 0,5 %), consider discordance of the isothermal baseline and calculate the heat capacity using the procedure in Annex C.

11.3 Numerical rounding of the results

Round the specific heat capacity values thus obtained to the second decimal place, using the method specified, in accordance with ISO 11357-4.

12 Test report

The test report shall include the following items:

a) General

   1) a reference to this document, i.e. ISO 24144:2023;

   2) the date of the test and name of the testing laboratory;

b) Specimen

   1) the mass of the specimen (the values before and after measurement);

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

   3) the manufacturer, model and type of product used as the test specimen, including the shape, dimensions and mass;

c) Conditions

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

   2) the materials of the crucible and lid (if used), and the details of the alumina liner (if used);

   3) the test atmosphere and the flow rate and type (e.g. purity) of the inflow gas;

   4) the calibration details including the material, the issuing body or source, the geometry of the material, the mass used and other characteristics relevant to the calibration;

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

   6) the temperature programme parameters, i.e. the start temperature, the heating rate, the end temperature and the time interval between the isothermal stages;

   7) calibration of isothermal baseline correction and its details;

d) Results

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

   2) the data sheet of each DSC curve line and the tabulated data (empty, reference specimen and test specimen);

e) Data handling

   1) averaging process and its details;

2) name of analysis software and its details;

f) Other

1) Any additional details related to the measurement to be noted, if any.

Annex A
(normative)

DSC apparatus for high temperature range

The DSC apparatus for the high temperature range shall be the following:

— not furnished with a hot bath.

NOTE For structural reasons, DSC apparatuses for the low temperature range (generally, under 700 °C), contain a hot bath.

Additional specifications of the DSC apparatus for the high temperature range are the following:

— furnished with a determined thermal resistance between the specimen and reference material;

— accuracy of measurement of enthalpy shall be within ±3 %.

Annex B
(normative)

Moulding procedure of test specimen

B.1 General

This annex specifies the moulding procedure of the test specimen.

B.2 Crushing procedure

The cut specimen shall be crushed into powder, grain or shortened fibres in a mortar (see Figure B.1).

B.3 Compression moulding procedure

The compression moulding procedure shall be applied to the crushed specimen in the following order:

a) Clean all metal mould parts: Spacer (1), Body (2), Upper lid (3), Bottom lid (4) in Figure B.2.

b) Set the metal mould in the compressor (see Figure B.3).

c) Fill the crushed specimen into the mould and put the upper lid mould on the body mould (see Figure B.4).

d) Load with a sufficient pressure for a sufficient time to compress and mould the specimen.

   In case the specimen is Alumina [Al_2O_3 (75 %) and SiO_2 (25 %)], load at around 3 kN to 5 kN by the compressor for 10 min to 15 min (see Figure B.5).

e) Remove the mould and recover the compressed specimen.

WARNING — Pay attention when removing the metal mould. During decompression, the compressed specimen can pop out in some cases.

Figure B.1 — Crushing operation

Figure B.2 — Metal mould

Figure B.3 — Compressor