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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*.

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.html.

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.

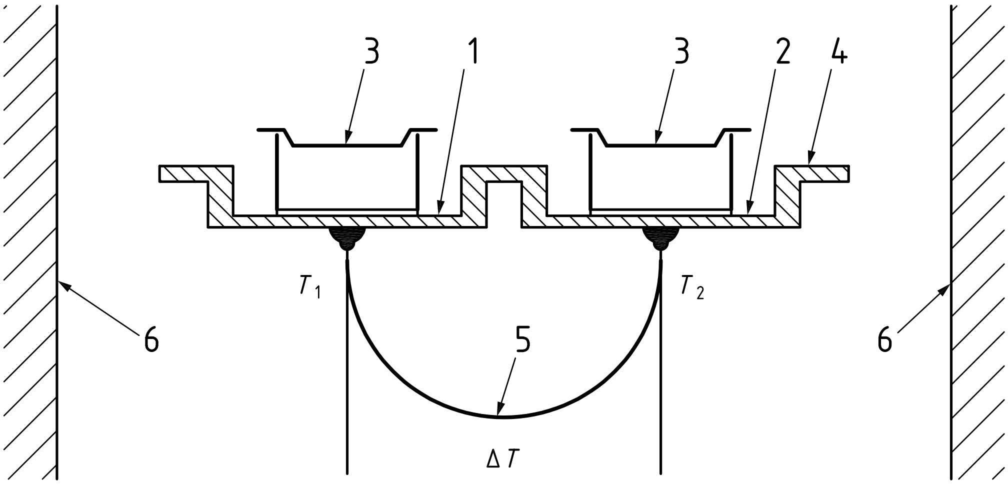
The difference between the rate of heat flow into a specimen and the rate of heat flow into a reference crucible is measured as a function of either temperature or time, or both, while the specimen and the reference are subjected to the same temperature-control programme under a specified atmosphere.

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

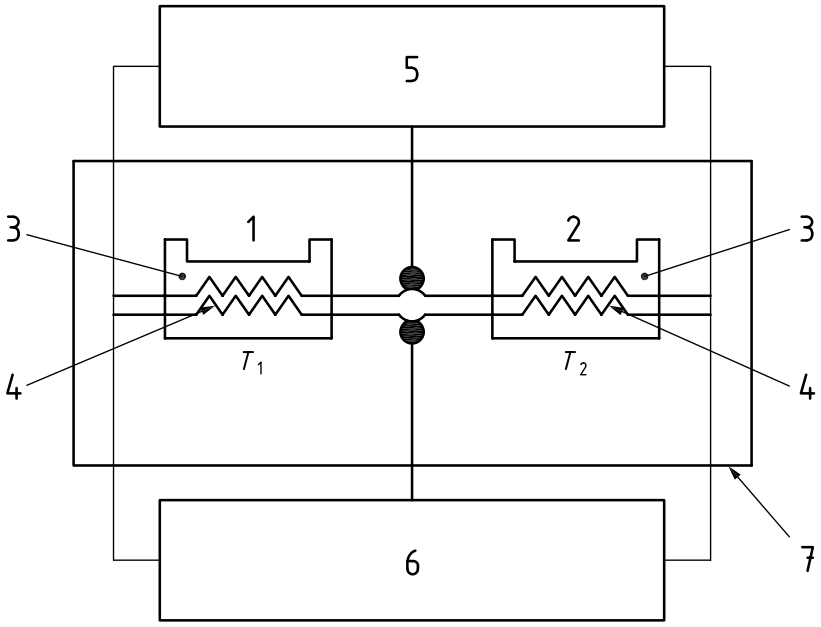
Figure 1 — Schematic diagram illustrating the basic principles of heat-flux DSC

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.

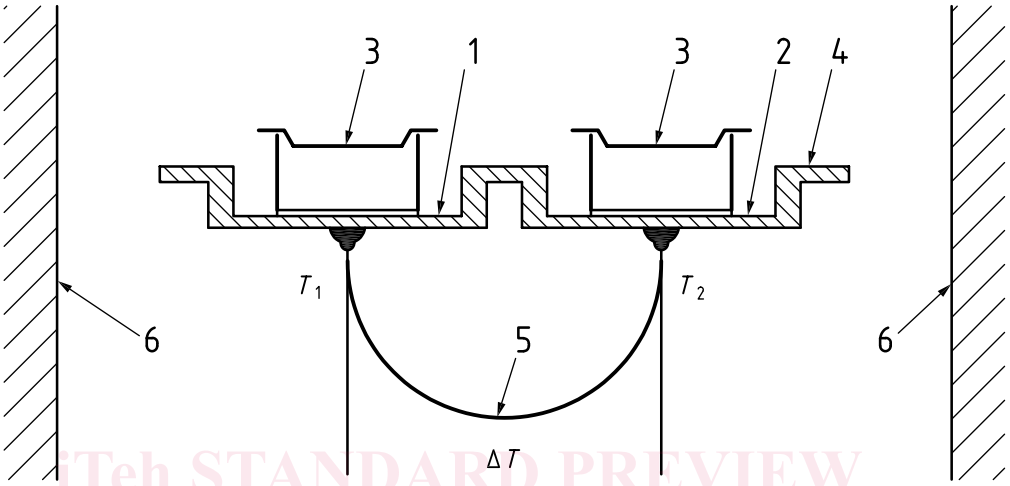
5.2 Basic procedure

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

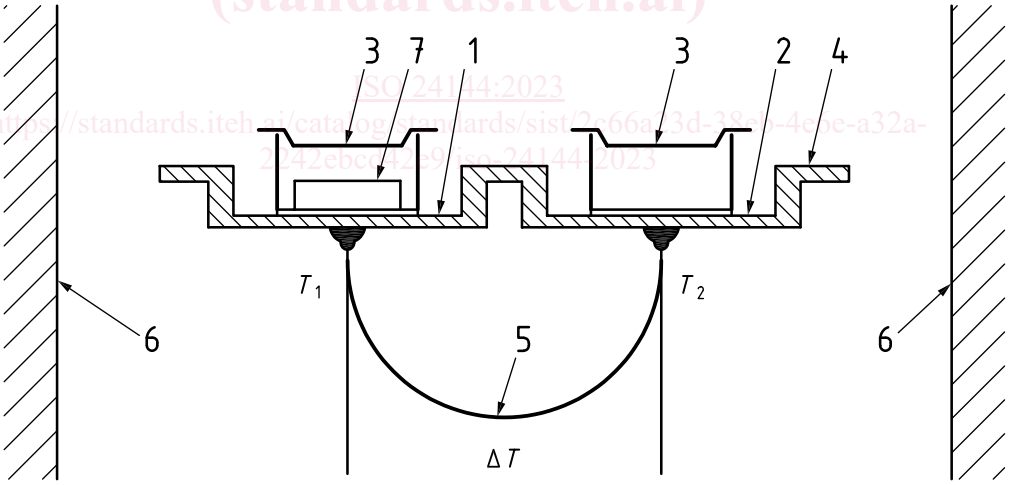
a) a blank run (empty crucibles in sample and reference holders);

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