



**International
Standard**

ISO 19628

**Fine ceramics (advanced
ceramics, advanced technical
ceramics) — Thermophysical
properties of ceramic composites
— Determination of specific heat
capacity**

*Céramiques techniques — Propriétés thermophysiques des
composites céramiques — Détermination de la capacité
thermique massique*

**Second edition
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Contents

	Page
Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Method A – drop calorimetry	2
4.1 Principle.....	2
4.2 Apparatus.....	2
4.3 Standard reference materials.....	3
4.4 Containers.....	3
4.5 Test specimens.....	3
4.6 Calibration of the calorimeter.....	3
4.6.1 General.....	3
4.6.2 Electrical calibration.....	3
4.6.3 Calibration using standard reference material.....	4
4.7 Test procedures.....	4
4.7.1 General.....	4
4.7.2 Test without a container.....	4
4.7.3 Test with a container.....	5
4.7.4 Description of test.....	5
4.8 Calculations.....	6
4.8.1 General.....	6
4.8.2 Determination of the calorimetric calibration factor.....	6
4.8.3 Determination of mean specific heat capacity $\overline{C_p}$	6
4.8.4 Determination of the specific heat capacity C_p	7
5 Method B – differential scanning calorimetry	7
5.1 Principle.....	7
5.1.1 General.....	7
5.1.2 Stepwise heating method.....	8
5.1.3 Continuous heating method.....	8
5.2 Apparatus.....	9
5.3 Standard reference materials, SRM.....	9
5.4 Test specimens.....	9
5.5 Temperature calibration.....	9
5.6 Test procedure for the determination of C_p	10
5.6.1 General.....	10
5.6.2 Method 1: Measurements requiring the knowledge of the K factor.....	10
5.6.3 Method 2: measurements requiring the use of a reference standard material (SRM).....	12
5.7 Calculation of results.....	15
5.7.1 Method requiring the knowledge of the K factor.....	15
5.7.2 Method using an SRM.....	17
6 Test report	18
Annex A (Informative) Drop calorimetry – determination of the calibration factor using standard reference material	19
Annex B (informative) Standard reference material	21
Annex C (informative) Materials for calorimeter calibrations	27
Bibliography	28

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

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 184, *Advanced technical ceramics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 19268:2017), which has been technically revised.

The main changes are as follows:

- revised scope to extend the maximum temperature of use of Method A to 3 000 K;
- revised [Clause 4](#) by introducing the possibility to apply the drop calorimetry method for temperatures $T_1 > T_2$ (conventional drop calorimetry);
- relevant specifications added concerning the containers and thermometers to be used;
- description of in-situ calibration methods of the calorimeter and thermometers;
- addition of a paragraph dealing with the determination of specific heat capacity at given temperatures from measurements performed by drop calorimetry;
- updated list of references in the Bibliography.

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.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of specific heat capacity

1 Scope

This document specifies two methods for the determination of the specific heat capacity of ceramic matrix composites with continuous reinforcements (1D, 2D, 3D).

Unidirectional (1D), bi-directional (2D) and tridirectional (XD, with $2 < X \leq 3$).

The two methods are:

- method A: drop calorimetry;
- method B: differential scanning calorimetry.

The two methods are applicable from ambient temperature up to a maximum temperature that is method dependent: method A can be used up to 3 000 K, while method B is limited to 1 900 K.

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 19634, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Ceramic composites — Notations and symbols*

IEC 60584-1, *Thermocouples — Part 1: EMF specifications and tolerances*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 19634 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
amount of heat required to raise the temperature of a mass unit of material by 1 K at constant temperature and pressure

$$C_p = \frac{1}{m} \frac{dQ}{dT} \quad (1)$$

where Q is the heat required for a test-piece of mass m .

3.2

mean specific heat capacity

$$\bar{C}_p(T_1, T_2)$$

amount of heat required to raise the temperature of a mass unit of a material from temperature T_1 to temperature T_2 at a constant pressure, divided by the temperature increase ($T_2 - T_1$) expressed in K

3.3

representative volume element**RVE**

minimum volume which is representative of the material considered

4 Method A – drop calorimetry

4.1 Principle

In “conventional” drop calorimetry, the test piece is heated in a furnace at a constant temperature T_2 then dropped in a calorimeter at a constant temperature T_1 . The quantity measured is the amount of heat Q released in cooling the test piece to the calorimeter temperature T_1 .

In “inverse” drop calorimetry, the test piece is maintained in a conditioning chamber at a constant temperature T_1 near to room temperature and then dropped in a calorimeter heated at a constant temperature T_2 . The quantity measured is the amount of heat Q absorbed in heating the test piece to the calorimeter temperature T_2 .

Whatever the method, “conventional” drop calorimetry or “inverse” drop calorimetry, $T_2 > T_1$.

In both methods, the tested material must not undergo a phase transition in the temperature increment ($T_2 - T_1$).

Transfer of the test piece shall be done under conditions as close as possible to adiabatic conditions.

Specific heat capacity and mean specific heat capacity are determined from the amount of heat absorbed or released by the test piece in the calorimeter depending on the drop calorimetry mode applied.

4.2 Apparatus

4.2.1 Drop calorimeter, there are several types of drop calorimeters. They include one (or more) conditioning chambers and measuring chambers, which can be operated under controlled atmosphere and which are all equipped with a temperature control system. It is recommended that these control systems enable a temperature stability better than 1 K below 1 300 K, better than 2 K from 1 300 K to 2 300 K and better than 4 K above 2 300 K.

The conditioning chamber shall have a homogeneous temperature zone size greater than the test specimen size. The measuring chamber shall have a homogeneous temperature zone of a sufficient length to accept several specimens and a sufficient thermal inertia to limit the temperature disturbance, due to the drop.

Heat transfer by radiation during the drop shall be avoided as far as possible.

4.2.2 Balance, with an accuracy of 0,1 mg for test pieces over 10 mg and an accuracy of 0,01 mg for test pieces below 10 mg.

4.2.3 Temperature detectors, thermocouples in accordance with IEC 60584-1 shall be used for the measurement of temperature up to 1 920 K.

For temperatures higher than 1 273 K, radiation thermometers (also named pyrometers) can be used.

Thermocouples and radiation thermometers shall be periodically calibrated in their operating temperature ranges as they can be subjected to drift over time.

Thermocouples may be calibrated by measurement either at a series of fixed-point temperatures (e.g. melting/freezing points) or by comparison with reference thermometers in thermally stabilised baths or furnaces.

NOTE 1 Guidelines on the Calibration of Thermocouples^[2] are available at the following address: <https://www.euramet.org/publications-media-centre/calibration-guidelines/>.

Pyrometers are usually calibrated in radiance temperature using reference blackbodies. In addition to these calibrations performed outside the drop calorimeter facility, it is recommended to perform in-situ calibrations of the pyrometers by using fixed-point temperatures^[3].

4.2.4 Data acquisition system, the sampling period during the test shall be less than 0,5 s.

4.3 Standard reference materials

Standard reference materials which can be used for calibration purposes are listed in [Annex B](#).

4.4 Containers

When containers are used, care shall be taken to choose suitable containers to avoid any chemical reaction or contamination of the specimen from the container material, in particular at high temperature.

4.5 Test specimens

The test specimens shall be representative of the material.

This criterion is generally met by test specimens containing the maximum number of representative volume elements compatible with the volume of the crucible. If this number is less than five, several solutions are possible:

- a) the test specimens should have an exact number of representative volume elements;
- b) the material should be cut into specimens; a number of similar test specimens should be tested and an average value determined.

4.6 Calibration of the calorimeter

4.6.1 General

Calibration of calorimeters may be done according to two different methods. The first consists of dissipating a known amount of thermal power using a calibrated resistor introduced in the second chamber of the calorimeter. In the second method, a reference specimen with known specific heat capacity is dropped according to the procedure described in [4.7](#).

4.6.2 Electrical calibration

The calibration factor is the ratio of a known amount of thermal power dissipated in the resistor to the steady-state calorimetric output signal, and is measured at temperature T_2 . It is recommended to let the calibrated resistor in the calorimeter during the electrical calibration and the tests, so that the experimental conditions during both steps remain strictly unchanged^[4,5].

NOTE 1 For the "inverse" drop calorimetry, the method using power dissipation in a resistor is limited to 1 350 K to avoid damaging the resistor at high temperature.

NOTE 2 This method can only be used if the sensitivity of the calorimeter is not affected by the filling of the measuring chamber.

4.6.3 Calibration using standard reference material

This calibration is called “drop calibration”. A specimen made of a standard reference material with a known specific heat capacity is dropped according to the test procedures described in 4.7. (See Annex B for standard reference material). The calibration factor is determined according to Annex A.

4.7 Test procedures

4.7.1 General

The test procedures described in sub-clauses 4.7.2 to 4.7.4 shall be applied depending on the experimental configuration (test performed with or without a container) and the calibration method (electrical calibration or calibration with standard reference material).

In the case of determination of the mean specific heat capacity $\overline{C_p}(T_1, T_2)$ (cf. sub-clause 4.8.3), the tests are performed for one couple of temperatures T_1 and T_2 .

For performing specific heat capacity C_p measurement (cf. sub-clause 4.8.4), the tests shall be carried out for different couples of temperatures T_1 and T_2 covering the temperature range of investigation.

- In “conventional” drop calorimetry, the tests are repeated by varying the temperature of the furnace T_2 , the temperature of the calorimeter T_1 being kept constant.
- In “inverse” drop calorimetry, the tests are repeated by varying the temperature of the calorimeter T_2 , the temperature of the conditioning chamber T_1 being kept constant.

4.7.2 Test without a container

4.7.2.1 Test with drop calibration

The test without a container and with drop calibration is done in the following order:

R, T, R, T, R, T, R

where

R is the test of standard reference material;

T is the test of test specimen.

Carry out each test as described in 4.7.4.

4.7.2.2 Test with electrical calibration

The test without a container and with calibration using power dissipation in a resistor is done in the following order:

- calibration of calorimeter;
- test on three test specimens.

Carry out each test as described in 4.7.4.

NOTE The avoidance of interaction between the test specimen and the calorimetric conditioning and measuring chambers can require the use of a sealed container.

4.7.3 Test with a container

4.7.3.1 General

The mass of all empty containers used for the test shall not differ by more than 5 %.

4.7.3.2 Test with drop calibration

The test with a container and with drop calibration is carried out in the following order:

C, C + R, C + T, C, C + R, C + T, C, C + R, C + T, C

where

C is the test with the empty container;

C + R is the test of container plus standard reference material;

C + T is the test of container plus test specimen.

Carry out each test as described in [4.7.4](#).

4.7.3.3 Test with electrical calibration

The test with a container and with calibration using power dissipation in a resistor is done in the following order:

- calibration of calorimeter;
- carry out the following sequence:

C, C + T, C, C + T, C, C + T, C

where

C is the test with the empty container;

C + T is the test with container plus test specimen.

Carry out each test as described in [4.7.4](#).

4.7.4 Description of test

The test piece (test specimen, standard material or empty container) and reference material shall be dried at (110 ± 5) °C until the difference in weight of two successive weighings is lower than 0,2 mg:

- measure the mass when a container is not used with an accuracy of $\pm 0,1$ mg or $\pm 0,1$ %, whichever is the smaller;
- when a container is used, measure the mass of each assembly dropped (empty container, container and standard reference material, container and test specimen);
- place the test piece (test specimen, standard material or empty container) in the conditioning chamber at temperature T_1 and wait for a sufficient period to reach thermal equilibrium of the test piece with its environment;
- measure T_1 and T_2 ;
- start recording the calorimetric signal before the test piece is dropped;
- drop the test piece;

— stop the recording when the steady-state output signal is reached.

4.8 Calculations

4.8.1 General

The change in heat Q corresponding to the drop of the test piece is related to the area A under the calorimetric output signal by Formula (2).

$$Q = K \cdot A \quad (2)$$

where K is the calorimeter calibration factor.

4.8.2 Determination of the calorimetric calibration factor

4.8.2.1 Electrical calibration

The calibration factor K at a temperature T is determined by dividing the amount of heat Q dissipated by the calibrated resistor inside the calorimeter maintained at temperature T by the area A under the calorimetric output signal.

$$K = \frac{Q}{A} \quad (3)$$

where

Q is the amount of heat dissipated;

A is the area under the calorimetric output signal.

4.8.2.2 With standard reference material

See [Annexes A](#) and [B](#).

4.8.3 Determination of mean specific heat capacity $\overline{C_p}$

The mean specific heat capacity is determined using Formula (4):

$$\overline{C_p}(T_1, T_2) = \frac{1}{m_i} \frac{Q_i(T_1, T_2)}{(T_2 - T_1)} \quad (4)$$

where

T_1 is the calorimeter temperature when using “conventional” drop calorimetry, or the initial temperature at which test pieces are conditioned in case of “inverse” drop calorimetry;

T_2 is the initial temperature at which test pieces are conditioned when using “conventional” drop calorimetry, or the calorimeter temperature in case of “inverse” drop calorimetry;

$Q_i(T_1, T_2)$ is the amount of heat to increase the temperature of the test specimen from T_1 to T_2 ;

m_i is the mass of the test piece, determined by weighing;

$\overline{C_p}(T_1, T_2)$ is the mean specific heat capacity at temperature $T = (T_1 + T_2) / 2$.

The subscript i has a different meaning depending on the type of test piece:

— $i = c$ for an empty container;

- $i = t$ for a test piece;
- $i = t + c$ for a test piece and container.

without container

$$\overline{C}_{pt} = \frac{K \cdot A_t}{m_t (T_2 - T_1)} \quad (5)$$

with container

$$\overline{C}_{pt} = \frac{K(A_{c+t} - A_c)}{m_t (T_2 - T_1)} \quad (6)$$

where

- A_t is the value of integration of calorimetric output signal of test specimen;
- A_c is the value of integration of calorimetric output signal of container;
- A_{c+t} is the value of integration of calorimetric output signal of test specimen plus container.

4.8.4 Determination of the specific heat capacity C_p

The specific heat capacity is determined by applying the following procedure:

- Plot the amounts of heat $Q(T_1, T_2)$ obtained for different couples of temperatures T_1 and T_2 as a function of T_2 ;
 In “conventional” drop calorimetry, the amounts of heat $Q(T_1, T_2)$ are equal to $K(T_1) \cdot (A_{c+t} - A_c)$ with a container or to $K(T_1) \cdot A_t$ without container;
 In “inverse” drop calorimetry, the amounts of heat $Q(T_1, T_2)$ are equal to $K(T_2) \cdot (A_{c+t} - A_c)$ with a container or to $K(T_2) \cdot A_t$ without container.
- Determine the mathematical model giving the amounts of heat $Q(T_1, T_2)$ versus T_2 , which best fits the obtained experimental data;
- Differentiating this mathematical model with respect to temperature and dividing the result by the mass of the test piece leads to the following relationship giving the specific heat capacity $C_p(T)$ as a function of temperature T .

$$C_p = \frac{1}{m} \frac{dQ}{dT} \quad (7)$$

5 Method B – differential scanning calorimetry

5.1 Principle

5.1.1 General

The method consists in measuring the difference in power needed to raise the temperature of the test specimen in its crucible and of an empty identical crucible using the same heating programme, which may be stepwise heating or continuous heating.

Stepwise heating allows only the determination of the mean specific heat capacity $\overline{C}_p(T_1, T_2)$ over a temperature range (T_1, T_2) , whereas continuous heating allows determination of the specific heat capacity C_p at a given temperature.

Whatever the heating programme, the tested material must not undergo a phase transition in the temperature range of investigation.

5.1.2 Stepwise heating method

The mean specific heat capacity $\overline{C_p}(T_1, T_2)$ is measured in a temperature interval defined by two isothermal levels, T_1 and T_2 . The heat, Q_E , which is necessary to change the temperature from T_1 to T_2 is determined by integrating the thermal power, P_E , with respect to time. The corresponding heat, Q_E , is:

$$Q_E = \int_0^t P_E dt = (m_t \overline{C_p}(T_1, T_2) + C_c + C_o)(T_2 - T_1) \quad (8)$$

where

- m_t is the mass of the test specimen;
- $\overline{C_p}(T_1, T_2)$ is the mean specific heat capacity of the test specimen;
- C_o is the heat capacity of the calorimeter;
- C_c is the heat capacity of the crucible.

Another experiment for the determination of the baseline is performed using an identical imposed heating sequence with the empty crucible. The corresponding heat, Q_B , is given by:

$$Q_B = \int_0^t P_B dt = [C_c + C_o](T_2 - T_1) \quad (9)$$

From the above formulae, the mean specific heat capacity can be calculated as:

$$\overline{C_p}(T_1 - T_2) = \frac{Q_E - Q_B}{m_t(T_2 - T_1)} \quad (10)$$

5.1.3 Continuous heating method

Temperature is increased linearly versus time at a constant heating rate β . Using the same notation as in [5.1.2](#), the thermal power P_E supplied to the system at every moment is:

$$K \cdot S_{c+t} = (m_t C_p + C_c + C_o) \beta \quad (11)$$

Another experiment for the determination of the baseline is performed with the empty crucible. The corresponding thermal power is given by

$$K \cdot S_c = (C_c + C_o) \beta \quad (12)$$

The specific heat capacity can be calculated from:

$$C_p = \frac{K(S_{c+t} - S_c)}{m_t \beta} \quad (13)$$

where

- K is the calibration factor;
- S_c, S_{c+t} are the output signals;
- $K \cdot S_c$ and $K \cdot S_{c+t}$ are the thermal powers supplied to the system.

5.2 Apparatus

5.2.1 Differential scanning calorimeter.

5.2.1.1 There are two types of differential scanning calorimeters operating on power compensation and heat flux principles, both designed to operate under adiabatic conditions.

Both comprise two measuring cells housed in a furnace which provides overall system heating. One cell contains the test specimen and its crucible, the other contains an empty crucible only.

5.2.1.2 Power compensation type: each cell has an additional heater to compensate for the temperature variations from the overall heating programme. The power which is supplied to either cell heater to maintain equal temperatures during heating is measured.

5.2.1.3 Heat flux type: 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.

5.2.2 **Balance**, with an accuracy better than 0,1 mg.

5.2.3 **Temperature detectors**, thermocouples in accordance with IEC 60584-1 shall be used for the measurement of temperature.

5.2.4 **Data acquisition system**, the time duration between two successive measurements shall be less than 0,5 s.

5.3 Standard reference materials, SRM

Standard reference materials shall be used for calibration. An example is given in [Annex B](#).

5.4 Test specimens

The test specimens shall be representative of the material.

This criterion is generally met by test specimens containing the maximum number of representative volume elements compatible with the volume of the crucible. If this number is less than five, several solutions are possible:

- a) the test specimens should have an exact number of representative volume elements;
- b) the material should be cut into pieces, and a number of similar test pieces should be tested and an average value determined.

5.5 Temperature calibration

A temperature calibration curve for the furnace using the same heating rate as for the determination of the specific heat capacity is established by using the melting points of standard reference materials (see [Annex C](#) for an example).

Thermocouples shall be calibrated in accordance with IEC 60584-1.

5.6 Test procedure for the determination of C_p

5.6.1 General

Depending on the necessity or not of using a calibration factor K for the calorimeter, two methods can be used:

- a) Method 1: measurements requiring the knowledge of the K factor; in this case, care shall be taken in order to ensure that the calibration is valid for all the measurements to be taken.

NOTE Generally, this can be done by running a test using a test specimen with well-known properties.

- b) Method 2: measurements requiring the use of a reference standard material during a series of tests.

5.6.2 Method 1: Measurements requiring the knowledge of the K factor

5.6.2.1 Determination of the K factor

The calibration factor K is obtained by electrical calibration. It is determined from the ratio of a known amount of power dissipated in a resistor to the steady-state calorimetric output signal.

5.6.2.2 Measurements with the specimen for the determination of the C_p

5.6.2.2.1 General

A series of measurements shall always be referenced to a baseline measurement performed under experimental conditions identical to the other measurements in the series. The type of crucible used depends on the type of test specimen and on the temperature range, and shall be the same for the series of measurements. The mass of all empty crucibles used in the series shall not differ by more than 5 %.

5.6.2.2.2 Test sequence for the stepwise heating method (see [Figure 1](#))

5.6.2.2.2.1 Generation of the baseline

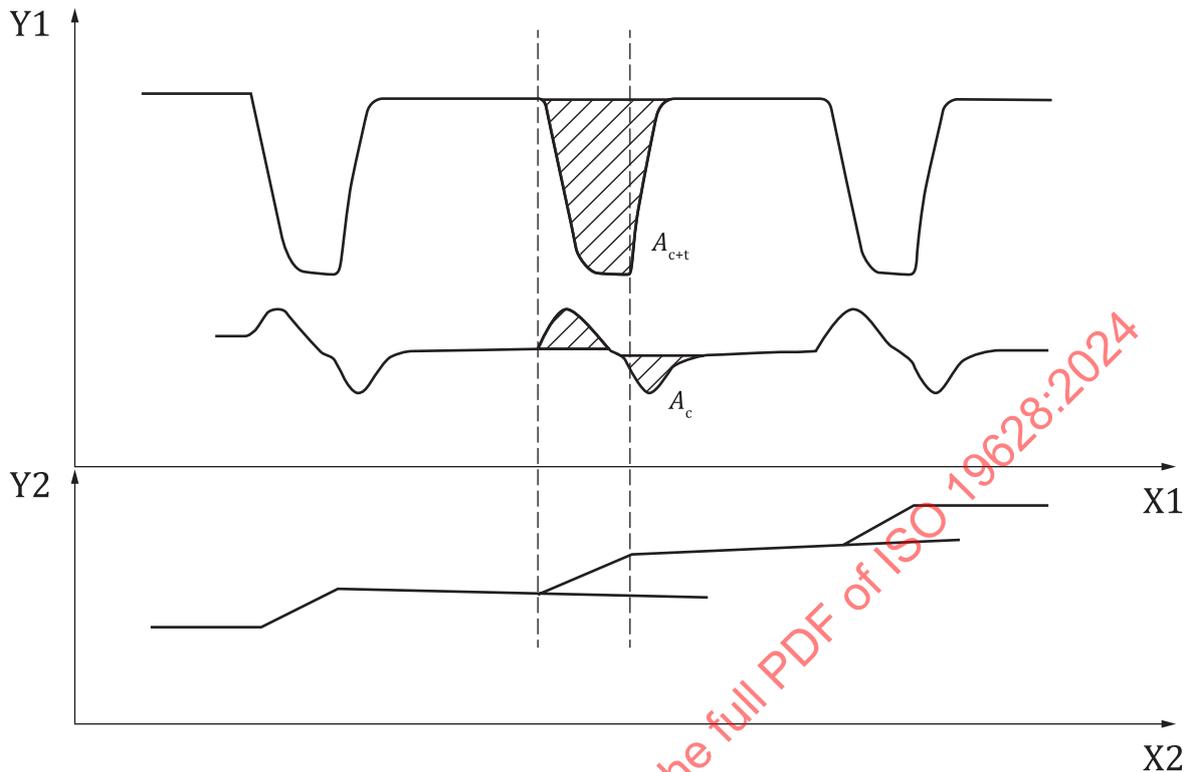
See [Figure 1](#).

- a) weigh the two empty crucibles to the nearest 0,1 mg;
 - b) place the two crucibles in the calorimeter;
 - c) set the calorimeter heating rate, initial and final temperature, and cooling rate;
- NOTE Generally, the heating rate is in the range 1 K/min to 20 K/min.
- d) heat to an initial temperature, and wait for the temperature to be stabilized at the initial temperature;
 - e) heat at a constant rate to final temperature of the first step while recording the calorimeter output signal, until the final temperature is reached and stabilized in order to obtain a baseline;
 - f) repeat c) to e) for the number of steps required;
 - g) cool down to initial temperature;
 - h) remove the crucibles from the measurement cell.

5.6.2.2.2.2 Measurements using a test specimen

See [Figure 1](#).

Weigh the test specimen and place it in the crucible to be located in the measurement cell. Repeat operations b) to h) from 5.6.2.2.2.1 on generation of the baseline. Repeat this procedure for a minimum of three test specimens.



Key
X1 time
X2 time
Y1 output signal
Y2 temperature

Figure 1 — Stepwise heating method (with calibration factor)

5.6.2.2.3 Test sequence for the continuous heating method

5.6.2.2.3.1 Generation of the baseline

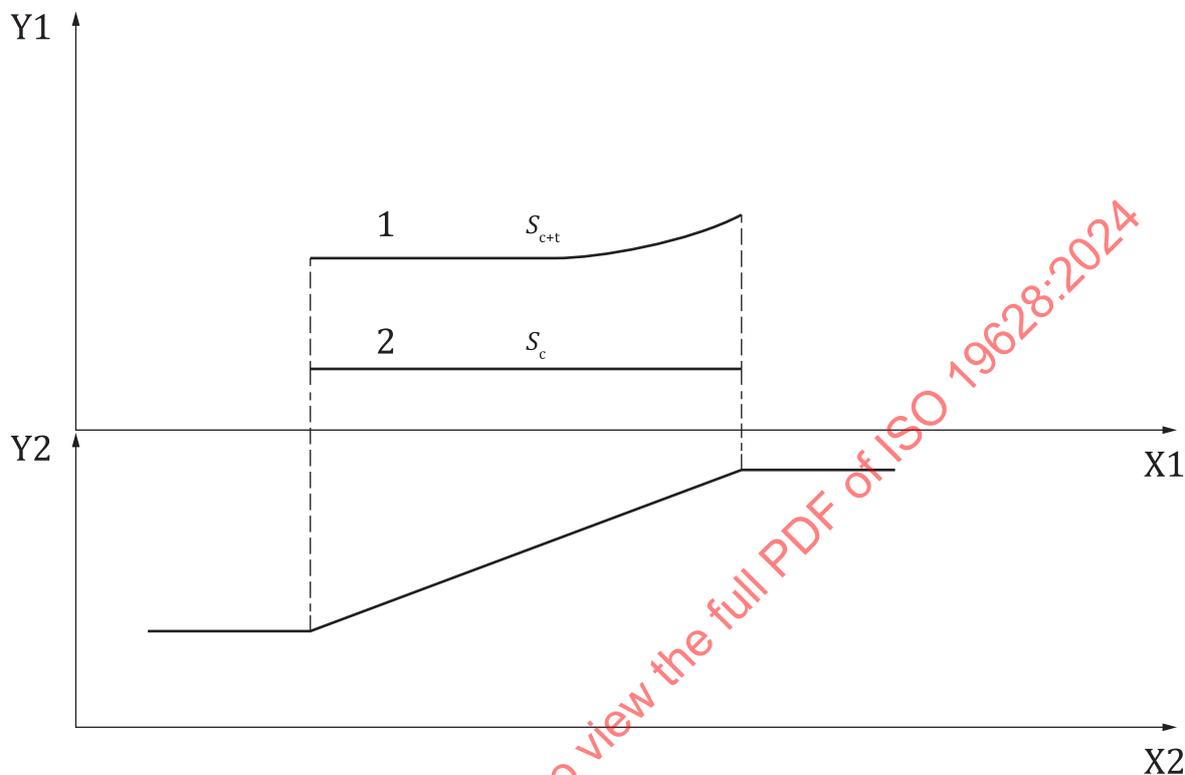
See [Figure 2](#).

- weigh the two empty crucibles to the nearest 0,1 mg;
- place the two crucibles in the calorimeter;
- set the calorimeter heating rate, initial and final temperature, and cooling rate;
NOTE Generally, the heating rate is in the range 1 K/min to 20 K/min.
- heat to an initial temperature, and wait for the temperature to be stabilized at the initial temperature;
- heat at a constant rate to final temperature of the first step, while recording the calorimeter output signal, until the final temperature is reached and stabilized in order to obtain a baseline;
- cool down to initial temperature;
- remove the crucibles from the measurement cell.

5.6.2.2.3.2 Measurements using a test specimen

See [Figure 2](#).

Weigh the test specimen and place it in the crucible to be located in the measurement cell. Repeat operations b to g of the above paragraph on generation of the baseline. Repeat this procedure for a minimum of three test specimens.



Key

X1	time	Y2	temperature
X2	time	1	test piece
Y1	output signal	2	baseline

Figure 2 — Continuous heating method (with calibration factor)

5.6.3 Method 2: measurements requiring the use of a reference standard material (SRM)

5.6.3.1 General

The two methods described in [5.6.3.2](#) and [5.6.3.3](#) each require:

- measurements with two empty crucibles for the generation of the baseline;
- measurements with one empty crucible and one crucible with the SRM;
- measurements with one empty crucible and one crucible with the test specimen.

5.6.3.2 Test sequence for the stepwise heating method

5.6.3.2.1 Generation of the baseline

See [Figure 3](#).

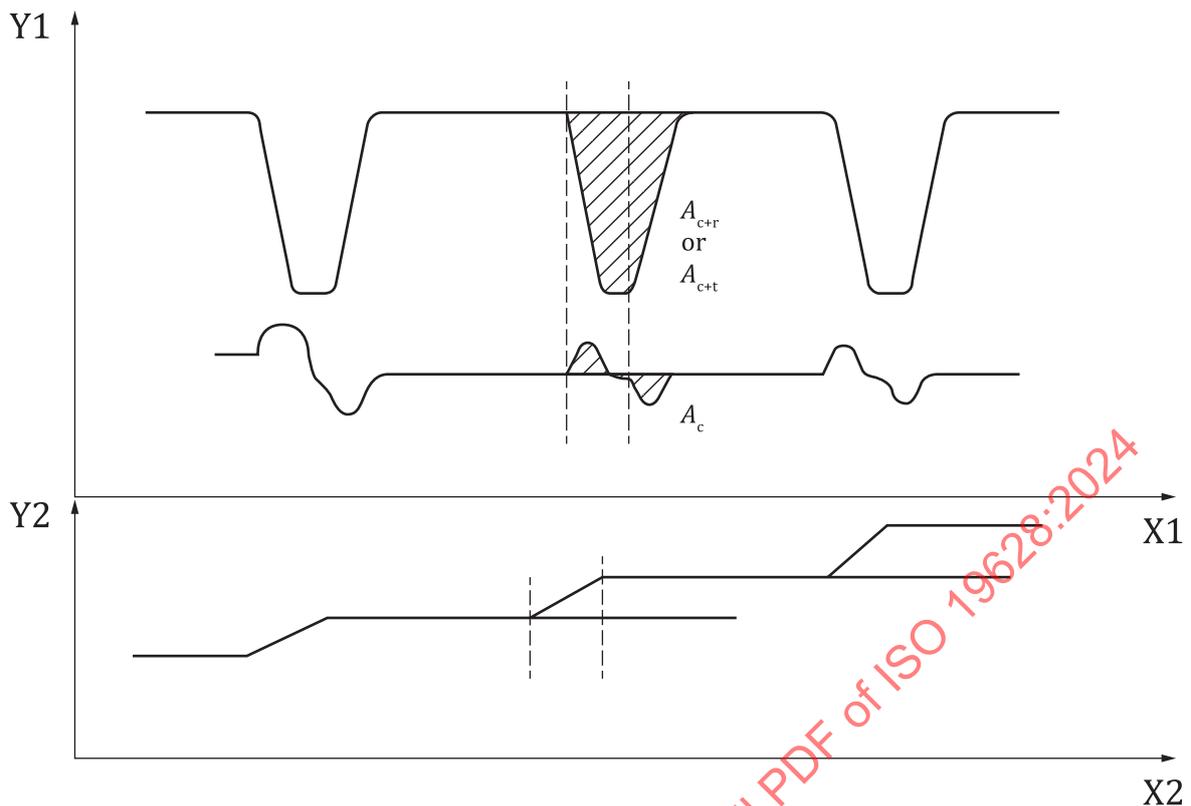
- a) weigh the two empty crucibles to the nearest 0,1 mg;
- b) place the two crucibles in the calorimeter;
- c) set the calorimeter heating rate, initial and final temperature, and cooling rate;
NOTE Generally, the heating rate is in the range 1 K/min to 20 K/min.
- d) heat to an initial temperature, and wait for the temperature to be stabilized at the initial temperature;
- e) heat at a constant rate to final temperature of the first step, while recording the calorimeter output signal, until the final temperature is reached and stabilized in order to obtain a baseline;
- f) repeat c) to e) for the number of steps required;
- g) cool down to initial temperature;
- h) remove the crucibles from the measurement cell.

5.6.3.2.2 Measurement with a test specimen or with a SRM

See [Figure 3](#).

Weigh the test specimen or the SRM and place it in the crucible to be located in the measurement cell. Repeat operations b) to h) from [5.6.3.2.1](#) on generation of the baseline. Repeat this procedure for a minimum of three test specimens.

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**Key**

- X1 time
 X2 time
 Y1 output signal
 Y2 temperature

Figure 3 — Stepwise heating method (with standard reference material)

5.6.3.3 Test sequence for continuous heating method

5.6.3.3.1 Generation of the baseline

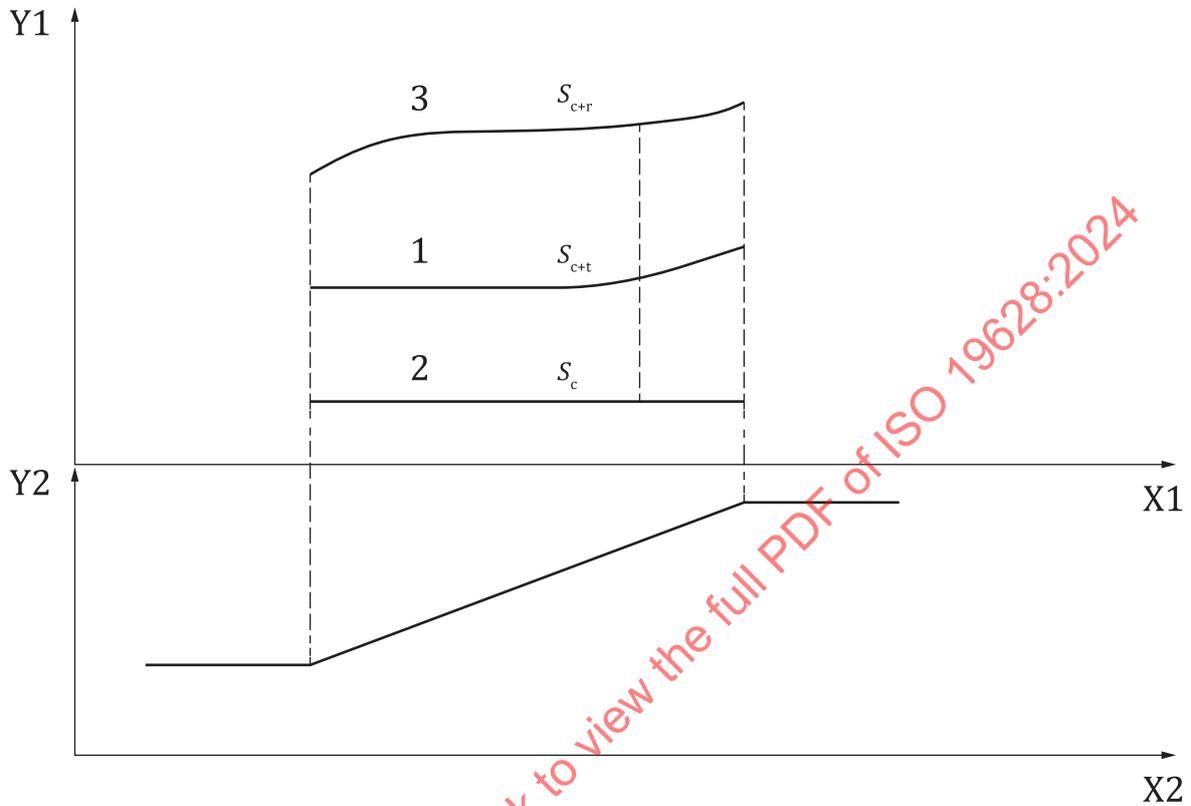
See [Figure 4](#).

- a) weigh the two empty crucibles to the nearest 0,1 mg;
- b) place the two crucibles in the calorimeter;
- c) set the calorimeter heating rate, initial and final temperature, and cooling rate;
 NOTE Generally, the heating rate is in the range 1 K/min to 20 K/min.
- d) heat to an initial temperature, and wait for the temperature to be stabilized at the initial temperature;
- e) heat at a constant rate to final temperature of the first step, while recording the calorimeter output signal, until the final temperature is reached and stabilized in order to obtain a baseline;
- f) cool down to initial temperature;
- g) remove the crucibles from the measurement cell.

5.6.3.3.2 Measurements using a test specimen or an SRM

See [Figure 4](#).

Weigh the test specimen or the SRM and place it in the crucible to be located in the measurement cell. Repeat operations b) to g) from [5.6.3.3.1](#) on generation of the baseline. Repeat this procedure for a minimum of three test specimens.



Key

X1	time	1	test piece
X2	time	2	baseline
Y1	output signal	3	reference
Y2	temperature		

Figure 4 — Continuous heating method (with standard reference material)

5.7 Calculation of results

5.7.1 Method requiring the knowledge of the *K* factor

5.7.1.1 Stepwise heating method

See [Figure 1](#).

The use of a computer with adapted software greatly simplifies data acquisition and treatment, and is recommended.

For the considered temperature interval, the shaded areas, *A*, are the integrals of the output signal, *s*, with respect to time.

For the baseline run with an empty crucible, one obtains:

$$\bar{K} \cdot A_c = (C_c + C_o)(T_2 - T_1) \quad (14)$$

For crucible plus test specimen:

$$\bar{K} \cdot A_{c+t} = (m_t \bar{C}_{pt} + C_c + C_o)(T_2 - T_1) \quad (15)$$

From these two Formulae one obtains:

$$\bar{C}_{pt}(T_1, T_2) = \frac{\bar{K}(A_{c+t} - A_c)}{m_t(T_2 - T_1)} \quad (16)$$

where

\bar{K} is the mean calorimeter calibration factor for the temperature range T_1 to T_2 , in watts per volt ($\text{W} \cdot \text{V}^{-1}$);

C_o is the heat capacity of the calorimeter, in joules per kelvin ($\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$);

C_c is the heat capacity of the crucible, in joules per kelvin ($\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$);

\bar{C}_{pt} is the mean specific heat capacity of the test specimen, in joules per kilogram and per kelvin ($\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$);

m_t is the mass of the test specimen, in kilograms (kg).

5.7.1.2 Continuous heating method

See [Figure 2](#).

Using the same notations as in [5.7.1.1](#) plus C_{pt} as the specific heat capacity of the test specimen, one obtains for each temperature:

— for the empty crucible:

$$KS_c = (C_c + C_o)\beta \quad (17)$$

— for crucible plus test specimen:

$$KS_{c+t} = (m_t C_{pt} + C_c + C_o)\beta \quad (18)$$

The specific heat capacity of the test specimen for each temperature, T , is then obtained from:

$$C_{pt} = \frac{K(S_{c+t} - S_c)}{m_t \cdot \beta} \quad (19)$$

where

β is the constant heating rate in ($\text{K} \cdot \text{s}^{-1}$);

K is the calibration factor at a temperature, T ;

S_c, S_{c+t} are the output signals of the calorimeter in volts (V).

5.7.2 Method using an SRM

5.7.2.1 Stepwise heating method

See [Figure 3](#).

The use of a computer with adapted software greatly simplifies data acquisition and treatment, and is recommended.

For the considered temperature interval, the shaded areas, A , are the integrals of the output signal, s , with respect to time.

For the baseline run with an empty crucible, one obtains:

$$\bar{K}A_c = (C_c + C_o)(T_2 - T_1) \quad (20)$$

— for crucible plus test piece:

$$\bar{K}A_{c+t} = (m_t \bar{C}_{pt} + C_c + C_o)(T_2 - T_1) \quad (21)$$

— for crucible plus reference piece:

$$\bar{K}A_{c+r} = (m_r \bar{C}_{pr} + C_c + C_o)(T_2 - T_1) \quad (22)$$

where

\bar{K} is the mean calorimeter calibration factor in watts per volt ($W \cdot V^{-1}$);

C_o is the heat capacity of the calorimeter in joules per kilogram and per kelvin ($J \cdot kg^{-1} \cdot K^{-1}$);

C_c is the heat capacity of the crucible in joules per kilogram and per kelvin ($J \cdot kg^{-1} \cdot K^{-1}$);

\bar{C}_{pt} is the mean specific heat capacity of the test piece in joules per kilogram and per kelvin ($J \cdot kg^{-1} \cdot K^{-1}$);

\bar{C}_{pr} is the mean specific heat capacity of the reference piece in joules per kilogram and per kelvin ($J \cdot kg^{-1} \cdot K^{-1}$);

m_t is the mass of the test specimen in kilograms (kg);

m_r is the mass of the standard reference material in kilograms (kg).

The mean specific heat capacity of the test piece is then obtained from:

$$\bar{C}_{pt}(T_1, T_2) = \bar{C}_{pr} \frac{m_r (A_{c+t} - A_c)}{m_t (A_{c+r} - A_c)} \quad (23)$$

5.7.2.2 Continuous heating method

See [Figure 4](#).

Using the same notations as in [5.7.2.1](#) plus C_{pt} as the specific heat capacity of the test specimen and C_{pr} as the specific heat capacity of the standard reference material, one obtains for each temperature:

— for the empty crucible:

$$KS_c = (C_c + C_o)\beta \quad (24)$$

— for crucible plus test specimen:

$$KS_{c+t} = (m_t C_{pt} + C_c + C_o) \beta \quad (25)$$

— for crucible plus standard reference material:

$$KS_{c+r} = (m_r C_{pr} + C_c + C_o) \beta \quad (26)$$

The specific heat capacity of the test specimen for each temperature, T , is then obtained from:

$$\overline{C}_{pt} = \overline{C}_{pr} \frac{m_r (S_{c+t} - S_c)}{m_t (S_{c+r} - S_c)} \quad (27)$$

where

β is the constant heating rate in (K_s^{-1})

K is the calibration factor at a temperature, T

S, S_{c+t}, S_{c+r} are the output signals of the calorimeter in volts (V).

6 Test report

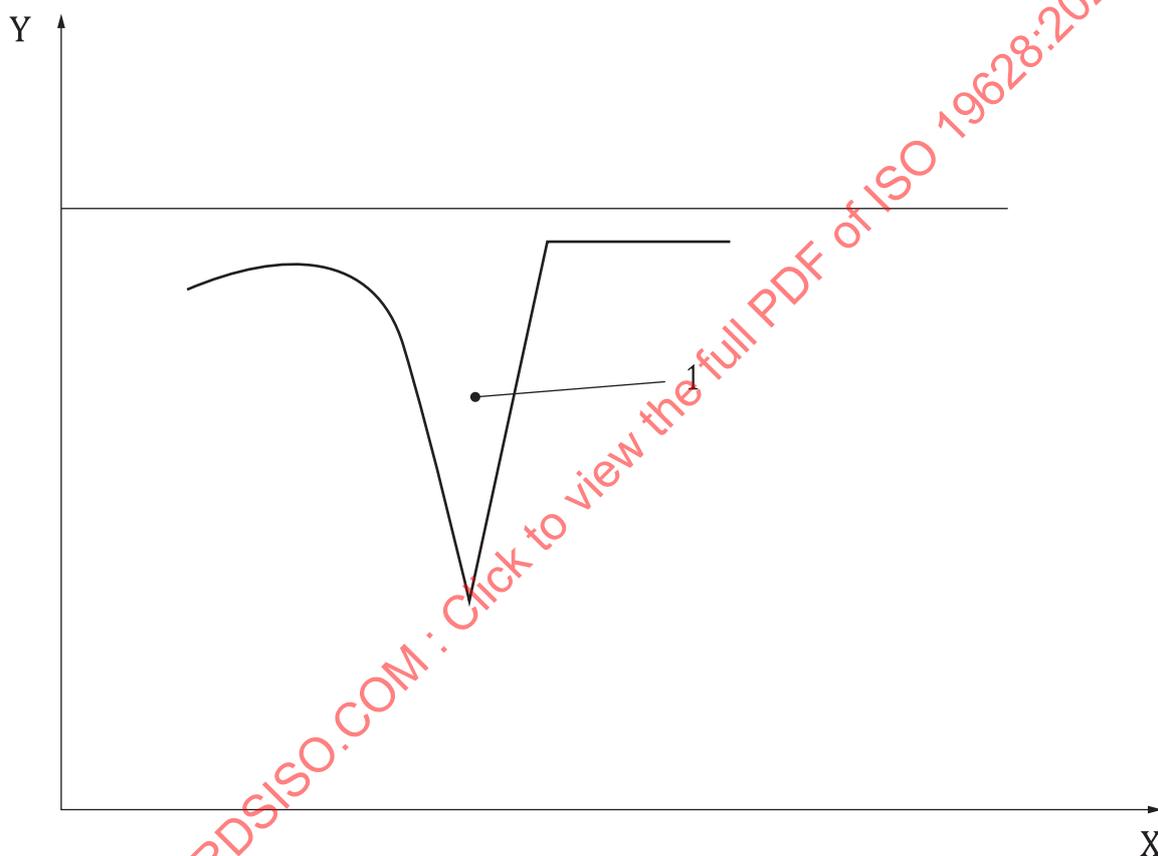
The test report shall contain at least the following information:

- a) name of the testing establishment;
- b) date of the test; a unique identification of the report and of each page, the customer's and signatory's names and addresses;
- c) reference to this document, i.e. "Determined in accordance with ISO 19628:2024";
- d) type of method of measurement used;
- e) brief details of the equipment used, or any procedures adopted, including necessary deviations;
- f) calibration procedures if applicable;
- g) material type, manufacturing code, batch number;
- h) methods of production of test specimens from supplied material (if appropriate);
- i) relevant test parameters (as required for each test);
- j) results of individual tests on required number of test specimens;
- k) mean results and standard deviation (if required);
- l) comments about the test or the test results;
- m) value of specific heat capacity at each required temperature, or value of the mean specific heat capacity between two temperatures calculated on at least three test specimens;
- n) any unusual features observed.

Annex A (informative)

Drop calorimetry – determination of the calibration factor using standard reference material

A test specimen made from a standard reference material (mass m_r) is dropped from the furnace at a temperature T_2 into the calorimeter at temperature T_1 in “conventional” drop calorimetry or from a temperature T_1 into the calorimeter at temperature T_2 in “inverse” drop calorimetry. [Figure A.1](#) shows an example of a heat dissipation curve on a reference material.



Key

X time

Y output signal

1 A_r in $\mu\text{V} \cdot \text{s}$

Figure A.1 — Heat dissipation curve as a function of time

The calibration factor K of the calorimeter at temperature T expressed in watt/volts is equal to:

$$K = \frac{Q_r}{A_r} \tag{A.1}$$

where

A_r is the area under the peak in volt seconds;

Q_r is the amount of heat required for the reference test specimen to heat from temperature T_1 to temperature T_2 .

The calibration coefficient K varies with temperature and shall be measured for each temperature.

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Annex B (informative)

Standard reference material

The most convenient standard reference material for this method is the α -alumina or sapphire, certified by the National Institute of Science and Technology of USA (NIST) under reference SRM 720 up to 2 250 K.

This standard reference material (SRM) is intended for use in calibrating or checking calorimeters used to measure either enthalpy or heat capacity within the range of 10 to 2 250 K.

The material furnished is synthetic sapphire cylinders, cut from centreless-ground rods grown by the Vernieul process and obtained from the Union Carbide Corporation.

The enthalpy and heat-capacity data have been derived from high-temperature enthalpy and low-temperature heat-capacity measurements. These data are presented in both tabular ([Table B.1](#)) and equation format.

The enthalpy values are accurate to $\pm 0,1$ % from 70 to 1 173 K and the heat-capacity values have an accuracy ranging from $\pm 0,1$ % at 70 K to $\pm 0,3$ % at 1 200 K. Below 70 K, the inaccuracy in heat-capacity and enthalpy values increase gradually to ± 10 % at 10 K, because, with decreasing temperature, the heat capacity of sapphire diminishes at a much faster rate than does that of the specimen container (mainly copper). The precision of the heat-capacity measurement between 100 and 380 K is estimated to be $\pm 0,02$ %. The precision of the enthalpy measurement from 273,15 to 1 173 K is estimated to be 0,02 %. For the temperature range 1 173 to 2 250 K, the precision of the enthalpy measurement is estimated to be $\pm 0,03$ %, and the accuracy of the measured enthalpy is estimated to be $\pm 0,2$ %, to a large extent reflecting the uncertainty in temperature measurements at these high temperatures. Above 1 700 K, a detectable weight loss was observed in an open container due to evaporation of material.

Table B.1 — Relative enthalpy and heat capacity^b

Temp ^a K	$H_T - H_{0K}$ J·mol ⁻¹	C_p J·mol ⁻¹ ·K ⁻¹
10	0,023	0,009 1
15	0,115	0,030 7
20	0,364	0,073 2
25	0,898	0,146
30	1,905	0,265
35	3,646	0,443
40	6,460	0,697
45	10,77	1,046
50	17,11	1,507
60	38,18	2,793
70	74,68	4,592
80	131,7	6,901
90	214,2	9,679
100	326,6	12,855
110	472,4	16,347
120	654,3	20,07
^a	Temperatures expressed on IPTS-68 scale.	
^b	Molecular weight = 101,961 3 in atomic mass unit (amu).	

Table B.1 (continued)

Temp ^a K	$H_T - H_{0K}$ J·mol ⁻¹	C_p J·mol ⁻¹ ·K ⁻¹
130	874,3	23,95
140	1 133,7	27,93
150	1 433,1	31,95
160	1 772,7	35,95
170	2 152,0	39,90
180	2 570,3	43,75
190	3 026,7	47,50
200	3 519,9	51,12
210	4 048,7	54,61
220	4 611,6	57,95
230	5 207,1	61,14
240	5 833,9	64,18
250	6 490,3	67,08
260	7 175,0	69,82
270	7 886,3	72,12
273,15	8 115,6	73,21
280	8 622,8	74,87
290	9 383,2	77,20
298,15	10 020	79,01
300	10 166	79,41
310	10 971	81,51
320	11 796	83,49
330	12 641	85,37
340	13 503	87,16
350	14 383	88,84
360	15 280	90,45
370	16 192	91,97
380	17 119	93,41
390	18 060	94,78
400	19 014	96,08
410	19 982	97,32
420	20 961	98,50
430	21 951	99,62
440	22 953	100,69
450	23 965	101,71
460	24 987	162,68
470	26 018	103,60
480	27 059	104,48
490	28 108	105,33
500	29 165	106,13
510	30 230	106,90
^a Temperatures expressed on IPTS-68 scale. ^b Molecular weight = 101,961 3 in atomic mass unit (amu).		

Table B.1 (continued)

Temp ^a K	H _T -H _{0K} J·mol ⁻¹	C _p J·mol ⁻¹ ·K ⁻¹
520	31 303	107,64
530	32 383	108,35
540	33 470	109,02
550	34 563	109,67
560	35 663	110,29
570	36 769	110,89
580	37 881	111,46
590	38 998	112,02
600	40 121	112,55
610	41 249	113,06
620	42 382	113,55
630	43 520	114,02
640	44 663	114,48
650	45 810	114,92
660	46 961	115,35
670	48 117	115,76
680	49 276	116,16
690	50 440	116,55
700	51 607	116,92
720	53 953	117,64
740	56 313	118,32
760	58 685	118,96
780	61 071	119,56
800	63 468	120,14
820	65 876	120,69
840	68 295	121,21
860	70 724	121,71
880	73 163	122,20
900	75 612	122,66
920	78 070	123,11
940	80 536	123,55
960	83 011	123,97
980	85 495	124,37
1 000	87 986	124,77
1 020	90 486	125,16
1 040	92 992	125,53
1 060	95 507	125,90
1 080	98 028	126,26
1 100	100 560	126,61
1 120	103 090	126,95
1 140	105 640	127,29
^a Temperatures expressed on IPTS-68 scale. ^b Molecular weight = 101,961 3 in atomic mass unit (amu).		