



**SLOVENSKI STANDARD**  
**oSIST prEN ISO 19628:2024**  
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**Fina keramika (sodobna keramika, sodobna tehnična keramika) - Termofizikalne lastnosti keramičnih kompozitov - Ugotavljanje specifične toplotne kapacitete (ISO/DIS 19628:2024)**

Fine ceramics (advanced ceramics, advanced technical ceramics) - Thermophysical properties of ceramic composites - Determination of specific heat capacity (ISO/DIS 19628:2024)

Hochleistungskeramik - Thermophysikalische Eigenschaften von keramischen Verbundwerkstoffen - Bestimmung der spezifischen Wärmekapazität (ISO/DIS 19628:2024)

Céramiques techniques - Propriétés thermophysiques des composites céramiques - Détermination de la capacité thermique massique (ISO/DIS 19628:2024)

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81.060.30      Sodobna keramika      Advanced ceramics

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# DRAFT International Standard

## ISO/DIS 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*

ICS: 81.060.30

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### Foreword

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

## 1 Scope

This document describes 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.

They are applicable from ambient temperature up to a maximum temperature, depending on the method: 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: Reference tables*

## 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 terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

### 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  $m$ .

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### 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.



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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<sup>[2]</sup> 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<sup>[3]</sup>.

**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

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](#).

#### 4.6.1 General

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<sup>[4,5]</sup>.

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.

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### 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.

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### 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  $(110 \pm 5) ^\circ\text{C}$  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;