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Second edition
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Refractory materials — Determination of thermal conductivity —

Part 1: Hot-wire methods (cross-array and resistance thermometer)

Matériaux réfractaires — Détermination de la conductivité thermique —

Partie 1: Méthodes du fil chaud («croisillon» et «thermomètre à résistance»)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8894-1 was prepared by Technical Committee ISO/TC 33, *Refractories*.

This second edition cancels and replaces the first edition (ISO 8894-1:1987), which has been revised to include a hot-wire “resistance thermometer” method, as well as the hot-wire “cross-array” method and to harmonize the text with that of EN 993-14:1998, *Methods of testing dense shaped refractory products — Part 14: Determination of thermal conductivity by the hot-wire (cross-array) method*, prepared by CEN/TC 187.

ISO 8894 consists of the following parts, under the general title *Refractory materials — Determination of thermal conductivity*:

— *Part 1: Hot-wire methods (cross-array and resistance thermometer)*

— *Part 2: Hot-wire method (parallel)*

Refractory materials — Determination of thermal conductivity —

Part 1: Hot-wire methods (cross-array and resistance thermometer)

1 Scope

This part of ISO 8894 describes the hot-wire methods ("cross-array" and "resistance thermometer") for the determination of the thermal conductivity of non-carbonaceous, dielectric refractory products and materials.

This methods are applicable to dense and insulating refractories (shaped products, refractory castables, plastic refractories, ramming mixes, powdered or granular materials) with thermal conductivity values less than 1,5 W/m·K ("cross-array") and less than 15 W/m·K ("resistance thermometer") and thermal diffusivity values less than 5×10^{-6} m^2/s.

Thermal conductivity values can be determined at a room temperature up to 1 250 °C. The maximum temperature (1 250 °C) can be reduced by the maximum service limit temperature of the refractory, or by the temperature at which the refractory is no longer dielectric.

NOTE 1 In general, it is difficult to make accurate measurements on anisotropic materials and the use of this method for such materials can be agreed between the parties concerned.

NOTE 2 The thermal conductivity of products with a hydraulic or chemical bond can be affected by the appreciable amount of water that is retained after hardening or setting and is released on firing. These materials might therefore require pre-treatment; the nature and extent of such pre-treatment and the period for which the test piece is held at the measurement temperature as a preliminary to carrying out the test, are details that are outside the scope of this part of ISO 8894 and are agreed between the parties concerned.

NOTE 3 The measurement of thermal conductivity is not sufficiently uncomplicated for an engineer to expect to achieve correct results without having particular work experience and if the work is based exclusively on this standard. Sufficient experience of measuring temperatures and laboratory skills are imperative.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
thermal conductivity
λ
density of heat flow rate divided by the temperature gradient

NOTE Thermal conductivity is expressed in watts per metre kelvin (W/m·K).

2.2
thermal diffusivity
a
thermal conductivity divided by the bulk density times the specific heat capacity

NOTE 1 $a = \lambda / \rho \cdot c_p$

where:

- λ is the thermal conductivity;
- ρ is the bulk density;
- c_p is the specific heat capacity at constant pressure per weight.

NOTE 2 Thermal diffusivity is expressed in units of square metres per second (m^2s^{-1}).

2.3
power
P
rate of energy transfer

NOTE Power is expressed in watts (W).

3 Principle

Both the hot-wire “cross-array” and “resistance thermometer” methods are dynamic measuring procedures based on the determination of the temperature increase against time of a linear heat source (hot wire) embedded between two test pieces which make up the test assembly.

The test assembly is heated in a furnace to a specified temperature and maintained at that temperature. Further local heating is provided by a linear electrical conductor (the hot wire) that is symmetrically embedded in the test assembly and carries an electrical current of known power that is constant in time and along the length of the test pieces.

The increase in temperature as a function of time follows a logarithmic law, and is measured and recorded from the moment the local heating current is switched on. The thermal conductivity of the test pieces is calculated using the rate of temperature increase and the power input.

For the “cross-array“ method, the temperature increase is measured using a thermocouple that is welded to the hot wire at its centre. The thermocouple leads are perpendicular to the hot wire.

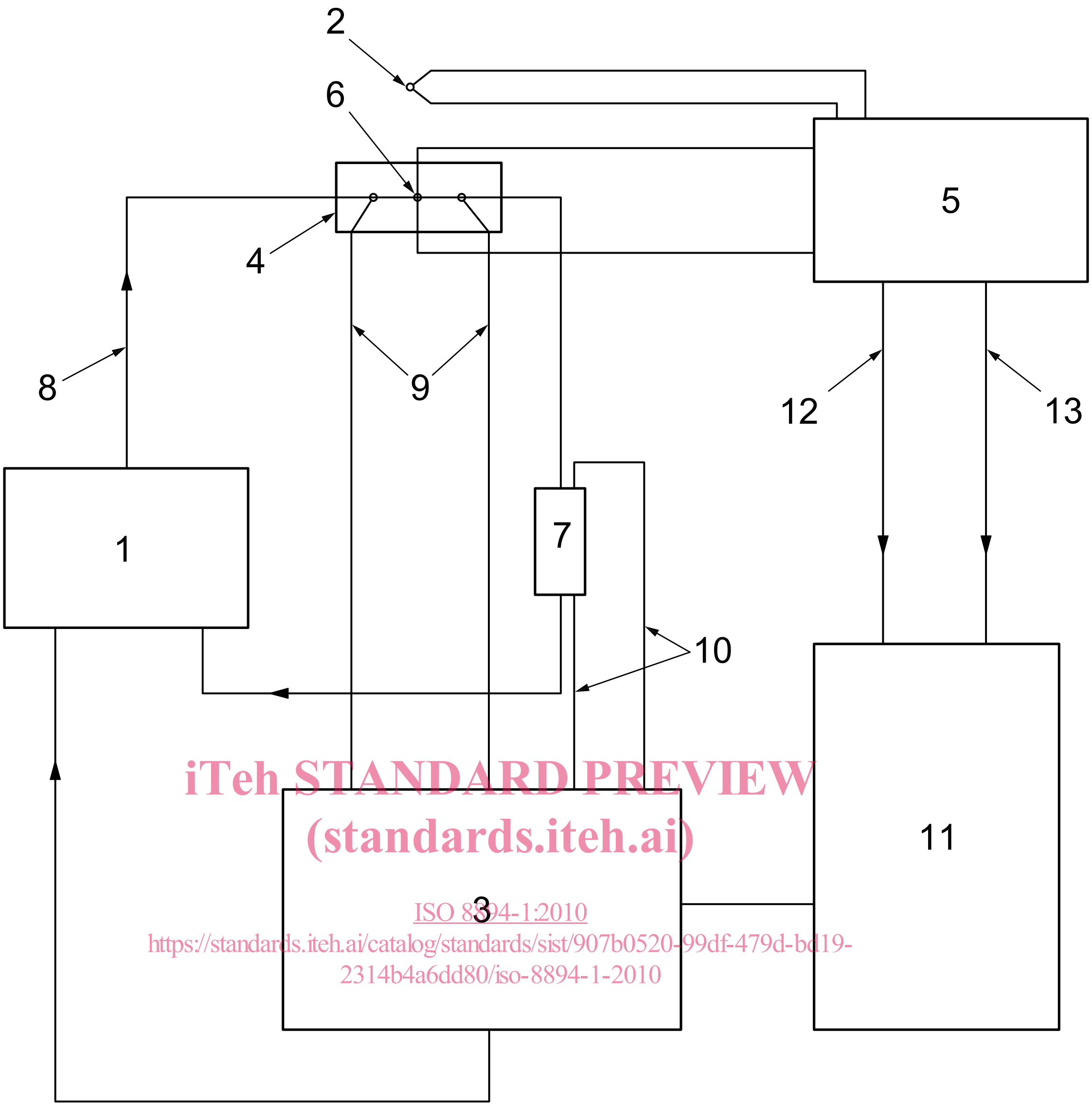
For the “resistance thermometer“ method, the temperature increase is measured by using the hot wire itself as both heat source and temperature sensor. An integral temperature measurement of the hot wire is carried out over the length between the voltage taps. The change in resistance of this part of the hot wire is determined. From these data, its temperature increase is calculated. The mathematical procedure is described in Annex A.

4 Apparatus

NOTE A block diagram of a suggested test apparatus for the “cross-array” method is shown in Figure 1 and for the “resistance thermometer” method in Figure 2.

4.1 Furnace, electrically heated, capable of taking one or more test assemblies (see 5.1) up to the required maximum test temperature. The temperature at any two points in the region occupied by the test pieces shall not differ by more than 10 K. The temperature measured on the outside of the test assembly during a test (of duration about 15 min) shall not vary by more than ±0,5 K, and shall be known with an accuracy of ±10 K.

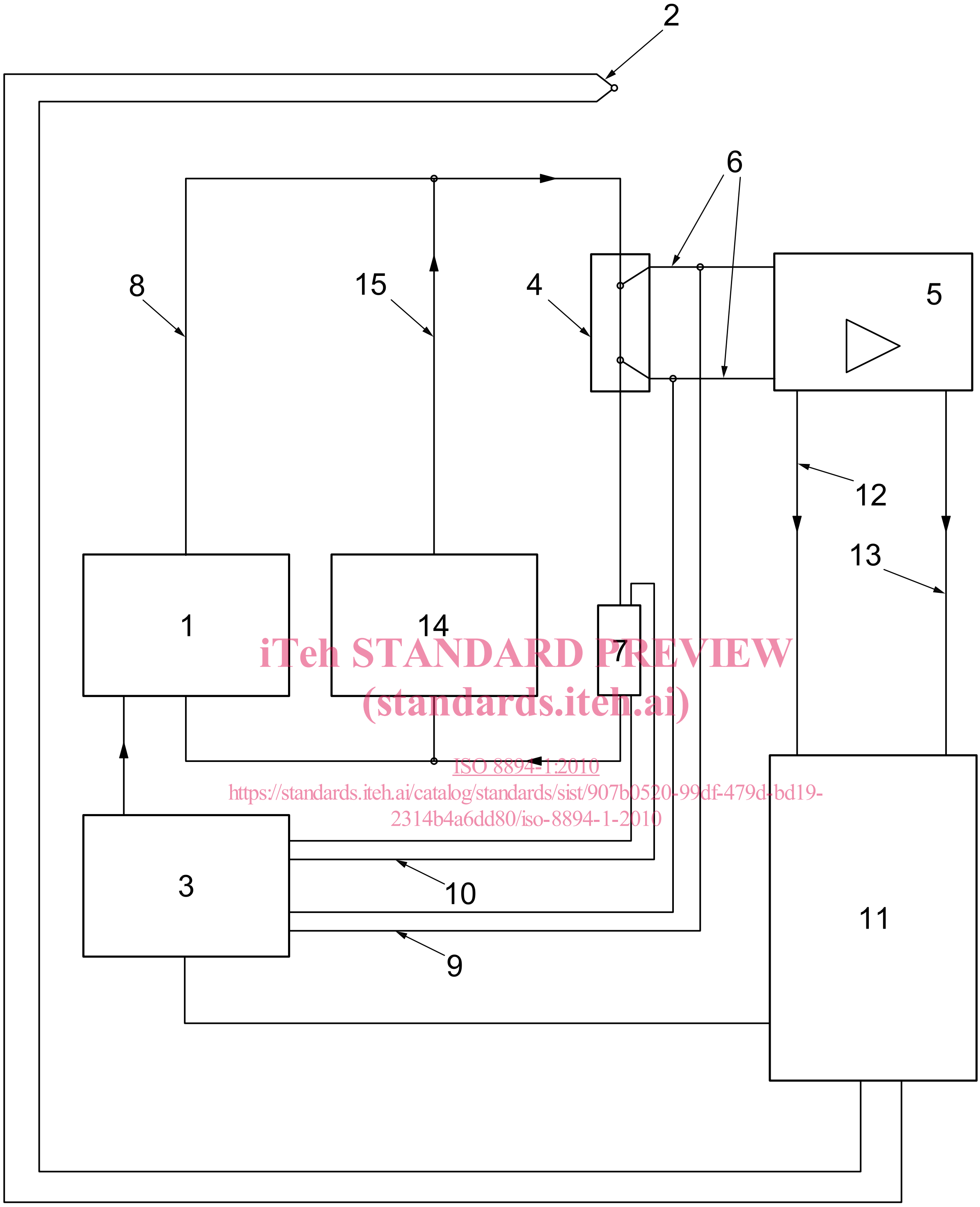
4.2 Hot wire, preferably of platinum or platinum-rhodium, with a minimum length equivalent to that of the test piece and a diameter not more than 0,5 mm. Both ends of the hot wire are attached to the power supply (4.4). Leads outside the assembly shall consist of two or more tightly twisted wires of 0,5 mm diameter. The current lead connections external to the furnace shall be made of heavy gauge cable.



Key

1 hot-wire power supply; a.c. source 1 kHz
2 reference thermocouple Tr
3 hot-wire power control unit
4 test assembly
5 cold junction of thermocouples
6 measurement thermocouple Ti
7 shunt
8 heating circuit
9 voltage taps
10 current measurement
11 data acquisition system and computer
12 absolute signal (Ti)
13 difference signal (Ti – Tr)

Figure 1 — Block diagram of apparatus for “cross-array” method



Key

1 hot-wire power supply; a.c. 1 kHz
2 thermocouple
3 hot-wire power control unit
4 test assembly
5 amplifier
6 voltage taps
7 shunt
8 heating circuit
9 a.c. voltage measurement
10 a.c. current measurement
11 data acquisition system and computer
12 absolute signal R
13 difference signal ΔR
14 d.c.source 100 mA
15 resistance measurement circuit

Figure 2 — Block diagram of apparatus for “resistance thermometer” method

4.3 Voltage taps, made of the same material as the hot wire. The welded connections to the hot wire should be located in the test piece with a distance of about 200 mm, known to the nearest ±0,5 mm. The wires shall be of a diameter not greater than that of the hot wire. Both ends of the voltage taps are attached to the hot-wire power control unit (4.6).

4.4 Power supply, to the hot wire (4.2).

For the electrical heating of the hot wire during a single measurement (6.7) an adequate power supply is required.

4.4.1 For the “cross-array” method, the power supply shall be stabilized a.c. or d.c., but preferably a.c., and shall not vary in power by more than 2 % during the period of measurement. It shall be variable between 1 W/m and 20 W/m. This is equivalent to 0,2 W to 4 W between the voltage taps for a distance of 200 mm (see 6.5, Note).

4.4.2 For the “resistance thermometer” method, the power supply shall be stabilized a.c., and shall not vary in power by more than 2 % during the period of measurement. It shall be variable between 1 W/m and 125 W/m. This is equivalent to 0,2 W and 25 W between the voltage taps for a distance of 200 mm (see 6.5, Note).

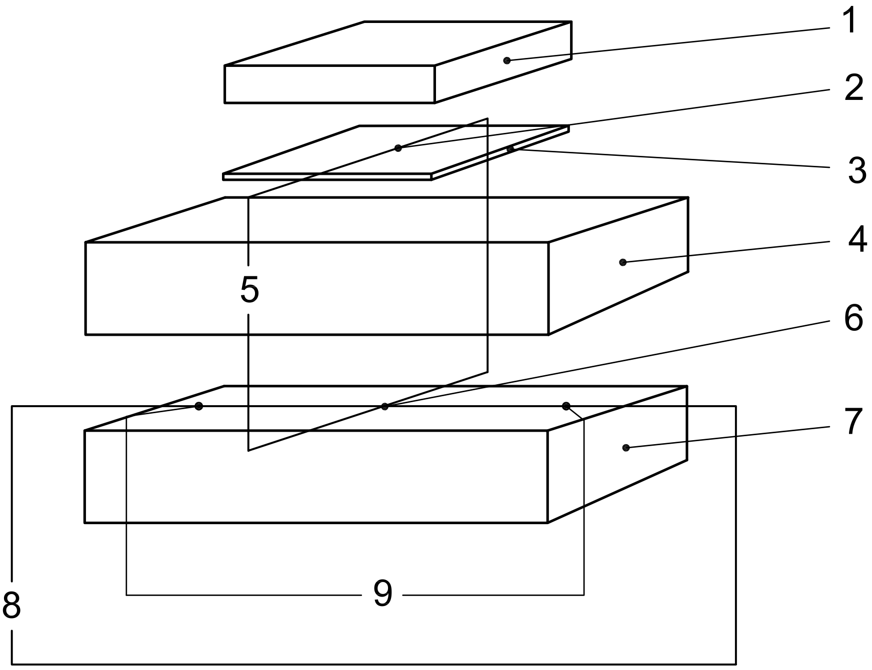
4.5 Equipment for the measurement of the temperature increase of the hot wire. The following arrangements for the “cross-array” and “resistance thermometer” methods shall be applied.

4.5.1 “Cross-array” method. For the “cross-array” method, use a differential platinum/platinum-rhodium thermocouple (Type S: platinum 10 % rhodium/platinum thermocouple or Type R: platinum 13 % rhodium/platinum thermocouple) formed from a measurement thermocouple (Ti) which is welded to the hot wire at its centre and a reference thermocouple (Tr) connected in opposition outside the furnace (see Figure 1). The leads of the measurement thermocouple shall run perpendicular to the hot wire. The output of the reference thermocouple shall be kept stable by placing it between the top outer face of the upper test piece and a cover of the same material as the test piece (see Figure 3). The maximum diameter of the measurement thermocouple wires shall not be greater than the diameter of the hot wire (to minimize loss of heat at the measuring point by conduction) and the wires of both thermocouples shall be long enough to extend outside the furnace where connections to the measuring apparatus shall be made by wire of a different type. The external connections of the thermocouple shall be isothermal. The measurement thermocouple (Ti) shall be used to indicate the temperature of the test assembly.

NOTE 1 An insulating layer can be inserted between the reference thermocouple and the upper test piece.

NOTE 2 For hot wire, voltage taps and thermocouples made of base metal can be used at temperatures below 1 000 °C.

NOTE 3 The reference thermocouple (Tr) can be replaced if a data acquisition system with an adequate resolution is used.

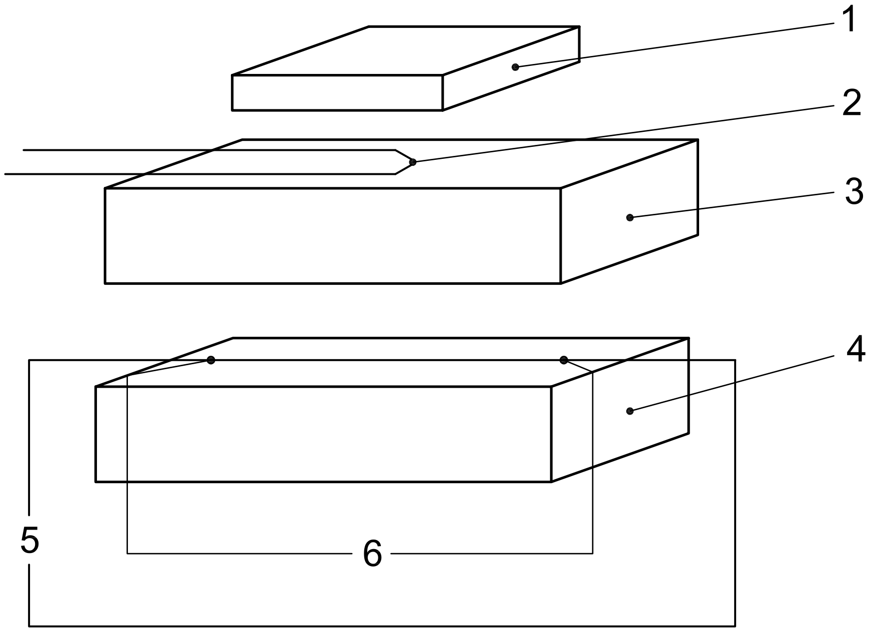


Key

1 cover
2 reference thermocouple Tr
3 optional insulating layer
4 test piece
5 differential measurement circuit
6 measurement thermocouple Ti
7 test piece
8 heating circuit
9 voltage taps

Figure 3 — Location of heating circuit and measurement circuit (differential thermocouple circuit) for "cross-array" method

4.5.2 "Resistance thermometer" method. For the "resistance thermometer" method, to measure the change in resistance of the hot wire, a low (e.g.100 mA) constant direct current (d.c.) is superimposed on the heating current (a.c.). The change of the d.c. voltage drop between the voltage taps is a measure for the change in temperature of the hot wire (see Figure 2). To indicate the temperature of the test assembly a separate thermocouple [e.g. the reference thermocouple (Tr)] shall be used (see 4.5.1). The arrangement of the hot wire and thermocouple is shown in Figure 4. As the hot-wire resistance during the measurement only changes within the range of parts per million (ppm) compared to its absolute resistance, a data acquisition system with an adequate resolution shall be used.



Key

1 cover
2 thermocouple
3 test piece
4 test piece
5 hot wire
6 voltage taps

Figure 4 — Location of heating circuit and thermocouple for the "resistance thermometer" method

4.6 Hot-wire power control unit, or similar equipment, which may be combined with the constant power supply (4.4), used for measuring the current in the hot wire and the voltage drop across it, and capable of measuring both to an accuracy of at least ±0,5 %.

4.7 Data acquisition system, consisting of a temperature-time registration device with a sensitivity of at least 2 µV/cm or 0,05 µV/digit, or a temperature measurement of 0,01 K or better and with a time resolution better than 0,5 s.

4.8 Containers, for use if the test is performed on powdered or granular material, having internal dimensions equal to those of the solid test assembly specified in Clause 5, so that the test assembly shall consist of two test pieces as specified in 5.1. The bottom container shall have four sides and a base, and the top container shall have four sides only, plus a detachable cover (see Figure 5).

Containers should be of a material that will not react with the test piece at the test temperature and should not be electrically conducting.