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Refractory materials — Determination of thermal conductivity —

Part 2: Hot-wire method (parallel)

*Matériaux réfractaires — Détermination de la conductivité thermique —
Partie 2: Méthode du fil chaud (parallèle)*



Reference number
ISO 8894-2 : 1990 (E)

Foreword

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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.

International Standard ISO 8894-2 was prepared by Technical Committee ISO/TC 33, *Refractories*, Sub-Committee SC 2, *Methods of testing*.

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

- *Part 1: Hot-wire method (cross-array)*
- *Part 2: Hot-wire method (parallel)*

Annexes A and B of this part of ISO 8894 are for information only.

Refractory materials — Determination of thermal conductivity —

Part 2 : Hot-wire method (parallel)

1 Scope

1.1 This part of ISO 8894 specifies a hot-wire method for the determination of the thermal conductivity of refractory products and materials.

1.2 The method is applicable at temperatures up to and including 1 250 °C and to materials whose thermal conductivity is less than 25 W/(m·K). Electrically conducting materials are excluded.

1.3 Subject to the limits in 1.2 the method is applicable to powdered or granular materials (see 7.2).

NOTES

1 The thermal conductivity of bonded bricks and of prepared unshaped (monolithic) refractories may be affected by the appreciable amount of water that is retained after hardening or setting and is released on firing. These materials may 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 should be agreed between the parties concerned.

2 In general it is difficult to make measurements on anisotropic materials, particularly those containing fibres, and the use of this method for such materials should also be agreed between the parties concerned.

1.4 The determination of thermal conductivity by the hot-wire method (cross-array) is the subject of ISO 8894-1.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8894. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8894 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5022 : 1979, *Shaped refractory products — Sampling and acceptance testing*.

ISO 8894-1 : 1987, *Refractory materials — Determination of thermal conductivity — Part 1: Hot-wire method (cross-array)*.

3 Definitions

For the purposes of this part of ISO 8894, the following definitions apply.

3.1 thermal conductivity, λ : Density of heat flow rate divided by temperature gradient.

The unit of thermal conductivity is the watt per metre-kelvin.

3.2 thermal diffusivity, α : Thermal conductivity divided by heat capacity per unit volume.

The unit of thermal diffusivity is the metre squared per second.

3.3 power, P : Product of current and potential difference.

The unit of power is the watt (volt-amperes).

4 Principle

The hot-wire method (parallel) is a dynamic measuring procedure based on the measurement of the temperature increase at a certain location and at a specified distance from a linear heat source embedded between two test pieces.

The test pieces are 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 embedded in the test piece and carries an electrical current of known power that is constant in time and along the length of the test piece.

A thermocouple is fitted at a specified distance from the hot wire, the thermocouple leads running parallel to the wire (see figure 1). The increase in temperature as a function of time,

measured from the moment the heating current is switched on, is a measure of the thermal conductivity of the material of which the test pieces are made.

5 Apparatus

5.1 Furnace, electrically heated, capable of taking one or more test assemblies (see 6.2) up to a maximum temperature of 1 250 °C. The temperature at any two points in the region occupied by the test pieces shall not differ by more than 10 °C. The temperature measured on the outside of the test assembly during a test (of duration about 15 min) shall not vary by more than $\pm 0,5$ °C, and shall be known with an accuracy of ± 5 °C.

5.2 Hot wire, preferably of platinum or platinum/rhodium, about 200 mm in length and not exceeding 0,5 mm in diameter, the actual length being known to within $\pm 0,5$ mm. One end of the wire is attached to the lead for supply of the heating current. This may also be a continuation of the wire itself, and shall in any case be of the same diameter as the wire when within the assembly. The other end is attached to a lead for measurement of voltage drop, which shall be of diameter not greater than that of the hot wire when within the assembly. Leads outside the assembly shall consist of two or more tightly twisted 0,5 mm diameter wires. External to the furnace the current lead connections shall be made with heavy-gauge cable (20 A/2,5 mm²).

NOTE — A hot wire made of base metal is also permitted, in which event the lead wires shall be made of the same material and shall conform in other respects to the requirements of this sub-clause.

5.3 Power supply to the hot wire, stabilized a.c., not varying in power by more than 2 % during the period of measurement. A supply to the hot wire of at least 80 W is required (equivalent to 250 W/m for a 200 mm long wire). A constant power supply, if available, is to be preferred.

5.4 Differential platinum/platinum-rhodium thermocouple (Type R or S), formed from a measurement thermocouple and a reference thermocouple connected in opposition (see figure 1). The leads of the measurement thermocouple shall run parallel to the hot wire at a distance of 15 mm $\pm$ 1 mm (see figure 2). 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 1). The diameter of the thermocouple wires shall be the same as that of the hot wire and the thermocouple wires 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.

NOTES

1 Base metal thermocouples are also permitted for use at temperatures below 1 000 °C.

2 An insulating layer between the cover and the upper test piece is allowed.

5.5 Digital multimeter, 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 $\pm 0,5$ %.

NOTE — An instrument of class 0,2 or better (see IEC 51-2 : 1984, *Direct acting indicating analogue electrical measuring instruments and their accessories — Part 2: Special requirements for ammeters and voltmeters*) is suitable.

5.6 Data acquisition system.

A temperature-time registration device with a sensitivity of at least 2 μ V/cm or 0,05 μ V/Digit, with a time resolution better than 0,5 s, and a temperature measurement to 0,05 K.

5.7 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 6, so that the test assembly shall consist of two sections as specified in 6.2. 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 3).

6 Test pieces

6.1 Sampling

The number of items of the material to be tested shall be determined in accordance with ISO 5022 or another standard sampling plan.

6.2 Dimensions

Each test assembly shall consist of two identical test pieces, not less than 200 mm $\times$ 100 mm $\times$ 50 mm in size.

NOTE — It is recommended that the size of each test piece be 230 mm $\times$ 114 mm $\times$ 64 mm or 230 mm $\times$ 114 mm $\times$ 76 mm. Standard-size bricks may then be used as the pieces forming the test assembly, subject to the requirements of 6.3.

6.3 Surface flatness

The surfaces of the two test pieces forming the test assembly which are in contact with each other shall, if necessary, be ground so that the deviation from flatness between two points not less than 100 mm apart is not more than 0,2 mm.

6.4 Groove in dense materials

In dense materials, a groove to accommodate the hot wire and the thermocouple shall be machined in either both the contact faces or in the lower face only of the test assembly (see figure 4). The width and depth of the groove shall permit the arrangement shown in figure 4 to be achieved, where required.

NOTE — Grooves in both faces will be necessary for materials of higher conductivity, e.g. greater than 5 W/(m·K).

7 Procedure

7.1 Arrange the test assembly ready for testing. Place the hot wire (5.2) and differential thermocouple (5.4) between the two test pieces, with the hot wire along the centreline of the brick

faces in contact with each other and cement them into the grooves where appropriate, using a cement made from finely ground test material mixed with a small amount of a suitable binder (e.g. 2 % dextrin and water). Ensure that the wires are cemented evenly, to allow equal heat transfer to the two test pieces, as shown in figure 4.

7.2 If the test is being performed on powdered or granular material, fill the bottom container (5.7) with the test material up to its top, and place on it the hot wire and differential thermocouple as shown in figure 1. Place the top container (5.7) on the bottom one and fill with the test material. Cover the test assembly with a slab of the same material as that of the containers. Determine the apparent bulk density of the test material in the poured, untamped state.

NOTE — The container may be filled by vibration or by pressing to give a specific bulk density, where a figure has been agreed upon.

7.3 Place the test assembly in the furnace (5.1), resting each assembly (to ensure uniform heating) on three supports of a material similar to that being tested and having dimensions of 125 mm × 10 mm × 20 mm. The supports shall rest on a 125 mm × 10 mm face, and be placed parallel to the 114 mm × 76 mm (or 100 mm × 50 mm) faces of the test assembly about 20 mm in from these faces.

7.4 Connect the test assembly to the measuring apparatus (5.5). With the hot-wire circuit open, raise the temperature of the furnace, at not more than 10 K/min, to the first test temperature required.

NOTE — Heating rates should be low enough to ensure that there is no risk of thermal shock damage.

7.5 Set the power input to a value that (from preliminary tests) is known to produce, for a chosen recorder sensitivity, an instrument deflection of at least 60 %, and preferably about 80 %, of full-scale deflection.

A guide to the choice of power input for a range of thermal conductivities and for a range of recorder sensitivities is given

in table 1. The power levels are based on a recorder deflection of $0,8 \times$ full-scale deflection for a given maximum duration of the test ($t_{\max}$), and table 1 also shows the required accuracy for the measurement of time (accuracy t).

NOTE — The appropriate level of power input to the hot wire will differ from equipment to equipment and needs to be evaluated in preliminary tests, but may eventually be based on experience.

7.6 When the furnace reaches the test temperature, verify that the temperature in the region occupied by the test assembly is uniform and constant. The differential thermocouple (5.4) shall not show a variation of more than 0,05 °C over a period of 10 min immediately prior to the test.

7.7 When the conditions of 7.6 are met, close the heating circuit and make a record of the output of the differential thermocouple with time. Mark the exact moment the power input to the hot wire was made. If not using an automatically controlled power supply, measure and record the voltage drop across the hot wire and the current in it immediately after switching on the heating circuit and again at intervals during the test period.

7.8 After an appropriate heating time (see table 1), disconnect the heating circuit and discontinue recording the output of the differential thermocouple.

7.9 Allow time for the hot wire and test assembly to reach temperature equilibrium. Verify the uniformity and constancy of the temperature as specified in 7.6. Repeat the procedures of 7.7 and 7.8, so obtaining a further measurement of the rate of rise of temperature of the hot wire under the same conditions.

7.10 Raise the temperature of the furnace to the next higher test temperature at not more than 10 K/min. Carry out again the procedure described in 7.5 to 7.9.

7.11 Repeat the procedure of 7.10 until at least two measurements have been obtained at each of the required test temperatures.

Table 1 — Recommended choice of scales and of power level
(based on a deflection of $0,8 \times$ full-scale)

Thermal conductivity, λ W/(m·K)	Maximum duration of test $t_{\max}$ s	Accuracy of measurement of t s	Recommended power level (W/m)			
			0 to 20 μ V scale	0 to 50 μ V scale	0 to 100 μ V scale	0 to 200 μ V scale
0,1	2 500	4,0			7,5	15
0,4	1 260	2,0		15	30	60
1,0	900	2,0	15	40	75	150
2,0	450	1,0	30	75	150	—
4,0	350	1,0	60	150	300	—
8,0	190	0,4	120	300	—	—
16	100	0,2	240	—	—	—
25	65	0,2	375	—	—	—

NOTE — The figures in table 1 are based on the use of type "S" thermocouples (see 5.4), and should be adjusted if a type "R" thermocouple is used.

8 Expression of results

Calculate the thermal conductivity, λ , of the material, in watts per metre kelvin, at each test temperature from the equation

$$\lambda = \frac{V \cdot I}{4\pi l} \times \frac{-\text{Ei}\left(\frac{-r^2}{4at}\right)}{\Delta\theta(t)}$$

where

I is the heating current, in amperes;

V is the voltage, in volts;

l is the length, in metres, of the hot wire between the voltage taps P and Q in figure 2;

$\Delta\theta(t)$ is the temperature difference, in kelvins, between the measurement and reference thermocouples at time t ;

t is the period of time, in seconds, between switching on and switching off the heating circuit;

r is the separation, in metres, of the hot wire and the measurement thermocouple;

a is the thermal diffusivity, in square metres per second;

$-\text{Ei}\left(\frac{-r^2}{4at}\right)$ is an exponential integral of the form

$$\int_x^{\infty} \frac{e^{-u}}{u} du.$$

After determining $\frac{\Delta\theta(2t)}{\Delta\theta(t)}$, the expression

$$-\text{Ei}\left(\frac{-r^2}{4at}\right) \text{ is calculated from table 2.}$$

The values of λ which can be considered accurate are those which correspond to values of $\frac{\Delta\theta(2t)}{\Delta\theta(t)}$ between 1,5 and 2,4.

9 Test report

The test report shall include the following information:

- the testing establishment;
- the date of the test;
- reference to this part of ISO 8894;
- the material tested (manufacturer, product, type, batch number, etc.);
- any pre-treatment given to the test material (see note 1 to clause 1);
- in the case of powders or granular materials, the apparent bulk density in the poured, untamped state (see 7.2);
- the furnace atmosphere;
- the test temperature or temperatures and, for each of them, the individual and mean values of thermal conductivity.

Table 2 — $-\text{Ei}\left(\frac{-r^2}{4at}\right)$ as a function of $\frac{\Delta\theta(2t)}{\Delta\theta(t)}$

$\frac{\Delta\theta(2t)}{\Delta\theta(t)}$	0	1	2	3	4	5	6	7	8	9
1,1	6,928 7	6,296 6	5,768 9	5,321 3	4,936 6	4,602 1	4,308 5	4,048 3	3,816 2	3,607 7
1,2	3,419 2	3,248 0	3,091 8	2,948 5	2,816 6	2,694 9	2,582 0	2,477 2	2,379 5	2,288 3
1,3	2,202 8	2,122 7	2,047 3	1,976 4	1,909 4	1,846 1	1,786 3	1,729 5	1,675 7	1,624 5
1,4	1,575 8	1,529 5	1,485 2	1,443 1	1,402 8	1,364 2	1,327 4	1,292 0	1,258 2	1,225 7
1,5	1,194 5	1,164 6	1,135 8	1,108 1	1,081 4	1,055 7	1,031 0	1,007 1	0,984 1	0,961 9
1,6	0,940 5	0,919 7	0,899 7	0,880 3	0,861 6	0,843 4	0,825 9	0,808 9	0,792 4	0,776 4
1,7	0,760 9	0,745 9	0,731 3	0,717 1	0,703 4	0,690 0	0,677 0	0,664 4	0,652 1	0,640 2
1,8	0,628 6	0,617 3	0,606 3	0,595 6	0,585 2	0,575 0	0,565 2	0,555 5	0,546 1	0,537 0
1,9	0,528 0	0,519 3	0,510 8	0,502 5	0,494 4	0,486 5	0,478 8	0,471 2	0,463 9	0,456 7
2,0	0,449 6	0,442 8	0,436 0	0,429 5	0,423 0	0,416 8	0,410 6	0,404 6	0,398 7	0,392 9
2,1	0,387 3	0,381 8	0,376 4	0,371 1	0,365 9	0,360 8	0,355 8	0,351 0	0,346 2	0,341 5
2,2	0,336 9	0,332 4	0,328 0	0,323 7	0,319 4	0,315 2	0,311 2	0,307 2	0,303 2	0,299 4
2,3	0,295 6	0,291 9	0,288 2	0,284 6	0,281 1	0,277 6	0,274 2	0,270 9	0,267 6	0,264 4
2,4	0,261 3	0,258 2	0,255 1	0,252 1	0,249 1	0,246 2	0,243 4	0,240 6	0,237 8	0,235 1
2,5	0,232 5	0,229 8	0,227 3	0,224 7	0,222 2	0,219 8	0,217 4	0,215 0	0,212 6	0,210 3
2,6	0,208 1	0,205 8	0,203 6	0,201 5	0,199 3	0,197 2	0,195 2	0,193 1	0,191 1	0,189 2
2,7	0,187 2	0,185 3	0,183 4	0,181 6	0,179 7	0,177 9	0,176 1	0,174 4	0,172 7	0,171 0
2,8	0,169 3	0,167 6	0,166 0	0,164 4	0,162 8	0,161 2	0,159 7	0,158 2	0,156 7	0,155 2
2,9	0,153 7	0,152 3	0,150 9	0,149 5	0,148 1	0,146 7	0,145 4	0,144 1	0,142 7	0,141 4
3,0	0,140 2	0,138 9	0,137 7	0,136 4	0,135 2	0,134 0	0,132 9	0,131 7	0,130 5	0,129 4
3,1	0,128 3	0,127 2	0,126 1	0,125 0	0,123 9	0,122 9	0,121 8	0,120 8	0,119 8	0,118 8
3,2	0,117 8	0,116 8	0,115 8	0,114 9	0,113 9	0,113 0	0,112 1	0,111 2	0,110 3	0,109 4
3,3	0,108 5	0,107 6	0,106 8	0,105 9	0,105 1	0,104 3	0,103 4	0,102 6	0,101 8	0,101 0
3,4	0,100 2	0,099 5	0,098 7	0,097 9	0,097 2	0,096 4	0,095 7	0,095 0	0,094 3	0,093 6
3,5	0,092 8	0,092 2	0,091 5	0,090 8	0,090 1	0,089 5	0,088 8	0,088 1	0,087 5	0,086 9
3,6	0,086 2	0,085 6	0,085 0	0,084 4	0,083 8	0,083 2	0,082 6	0,082 0	0,081 4	0,080 8
3,7	0,080 3	0,079 7	0,079 1	0,078 6	0,078 0	0,077 5	0,077 0	0,076 4	0,075 9	0,075 4
3,8	0,074 9	0,074 4	0,073 9	0,073 4	0,072 9	0,072 4	0,071 9	0,071 4	0,070 9	0,070 5
3,9	0,070 0	0,069 5	0,069 1	0,068 6	0,068 2	0,067 7	0,067 3	0,066 9	0,066 4	0,066 0
4,0	0,065 6	0,065 2	0,064 7	0,064 3	0,063 9	0,063 5	0,063 1	0,062 7	0,062 3	0,061 9
4,1	0,061 5	0,061 2	0,060 8	0,060 4	0,060 0	0,059 7	0,059 3	0,058 9	0,058 6	0,058 2
4,2	0,057 9	0,057 5	0,057 2	0,056 8	0,056 5	0,056 1	0,055 8	0,055 5	0,055 1	0,054 8
4,3	0,054 5	0,054 2	0,053 8	0,053 5	0,053 2	0,052 9	0,052 6	0,052 3	0,052 0	0,051 7
4,4	0,051 4	0,051 1	0,050 8	0,050 5	0,050 2	0,049 9	0,049 6	0,049 4	0,049 1	0,048 8
4,5	0,048 5	0,048 2	0,048 0	0,047 7	0,047 5	0,047 2	0,046 9	0,046 7	0,046 4	0,046 2
4,6	0,045 9	0,045 6	0,045 4	0,045 2	0,044 9	0,044 7	0,044 4	0,044 2	0,043 9	0,043 7
4,7	0,043 5	0,043 2	0,043 0	0,042 8	0,042 5	0,042 3	0,042 1	0,041 9	0,041 7	0,041 4
4,8	0,041 2	0,041 0	0,040 8	0,040 6	0,040 4	0,040 2	0,040 0	0,039 8	0,039 6	0,039 3
4,9	0,039 1	0,038 9	0,038 7	0,038 6	0,038 4	0,038 2	0,038 0	0,037 8	0,037 6	0,037 4
5,0	0,037 2	0,037 0	0,036 8	0,036 7	0,036 5	0,036 3	0,036 1	0,035 9	0,035 8	0,035 6
5,1	0,035 4	0,035 2	0,035 1	0,034 9	0,034 7	0,034 6	0,034 4	0,034 2	0,034 1	0,033 9
5,2	0,033 7	0,033 6	0,033 4	0,033 3	0,033 1	0,032 9	0,032 8	0,032 6	0,032 5	0,032 3
5,3	0,032 2	0,032 0	0,031 9	0,031 7	0,031 6	0,031 4	0,031 3	0,031 1	0,031 0	0,030 9
5,4	0,030 7	0,030 6	0,030 4	0,030 3	0,030 2	0,030 0	0,029 9	0,029 7	0,029 6	0,029 5
5,5	0,029 3	0,029 2	0,029 1	0,029 0	0,028 8	0,028 7	0,028 6	0,028 4	0,028 3	0,028 2
5,6	0,028 1	0,027 9	0,027 8	0,027 7	0,027 6	0,027 5	0,027 3	0,027 2	0,027 1	0,027 0
5,7	0,026 9	0,026 8	0,026 6	0,026 5	0,026 4	0,026 3	0,026 2	0,026 1	0,026 0	0,025 8
5,8	0,025 7	0,025 6	0,025 5	0,025 4	0,025 3	0,025 2	0,025 1	0,025 0	0,024 9	0,024 8
5,9	0,024 7	0,024 6	0,024 5	0,024 4	0,024 3	0,024 2	0,024 1	0,024 0	0,023 9	0,023 8
6,0	0,023 7									

NOTES

1) Table 2 has been made up of statements in the literature (see annex B, [1], [2] and [3]).

2) The expressions $\frac{\Delta\theta(2t)}{\Delta\theta(t)}$ and $-\text{Ei}\left(\frac{-r^2}{4at}\right)$ are quoted in the literature as $\frac{-\text{Ei}\left(-\frac{x}{2}\right)}{-\text{Ei}(-x)}$ and $-\text{Ei}(-x)$ respectively.

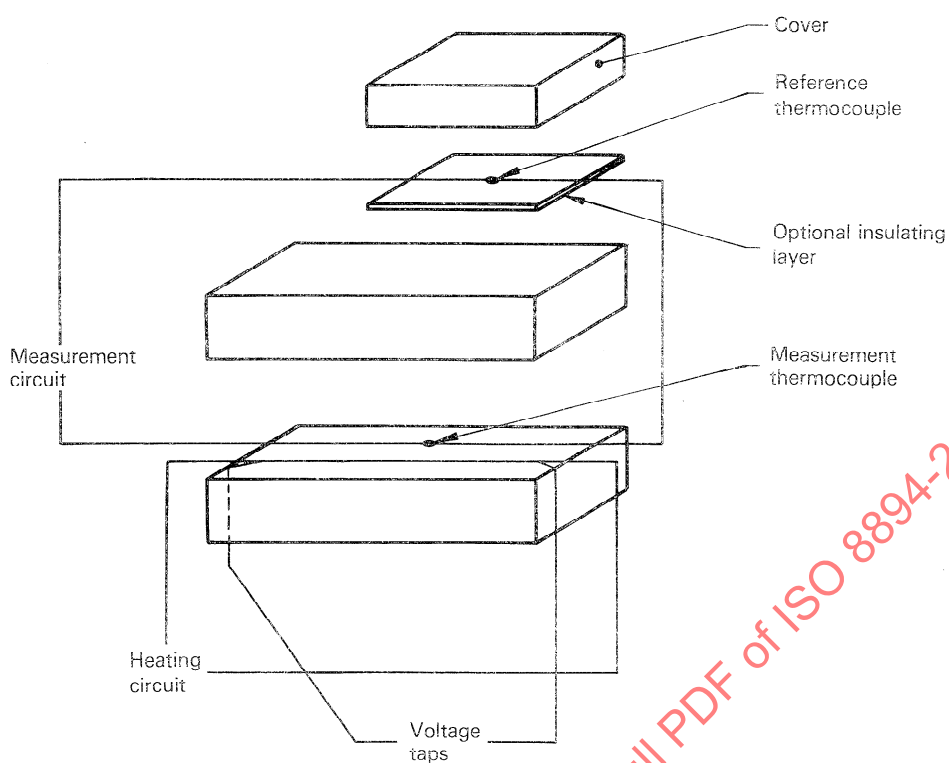


Figure 1 — Diagram showing location of heating circuit and measurement circuit (differential thermocouple circuit)

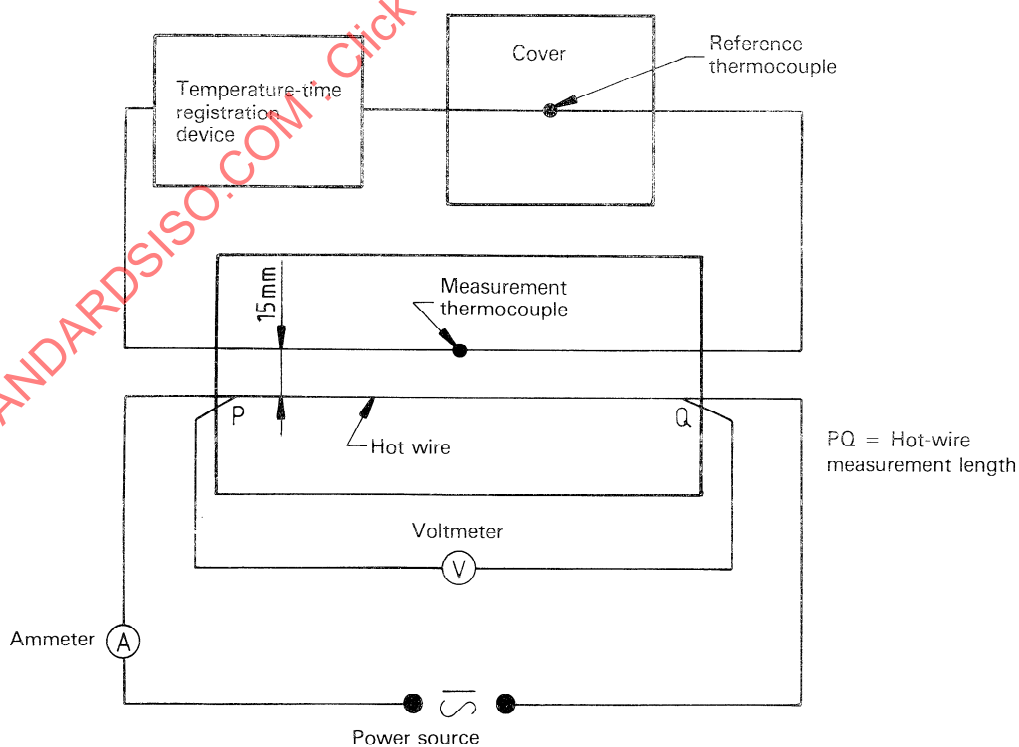


Figure 2 — Diagram showing measurement arrangement

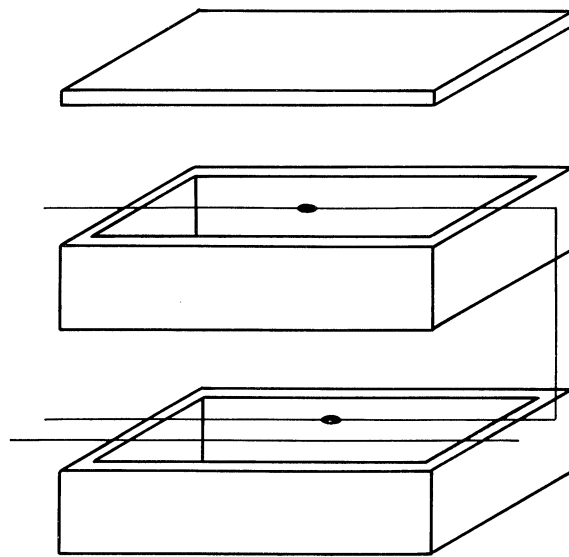


Figure 3 — Container with hot wire and thermocouple laid on it

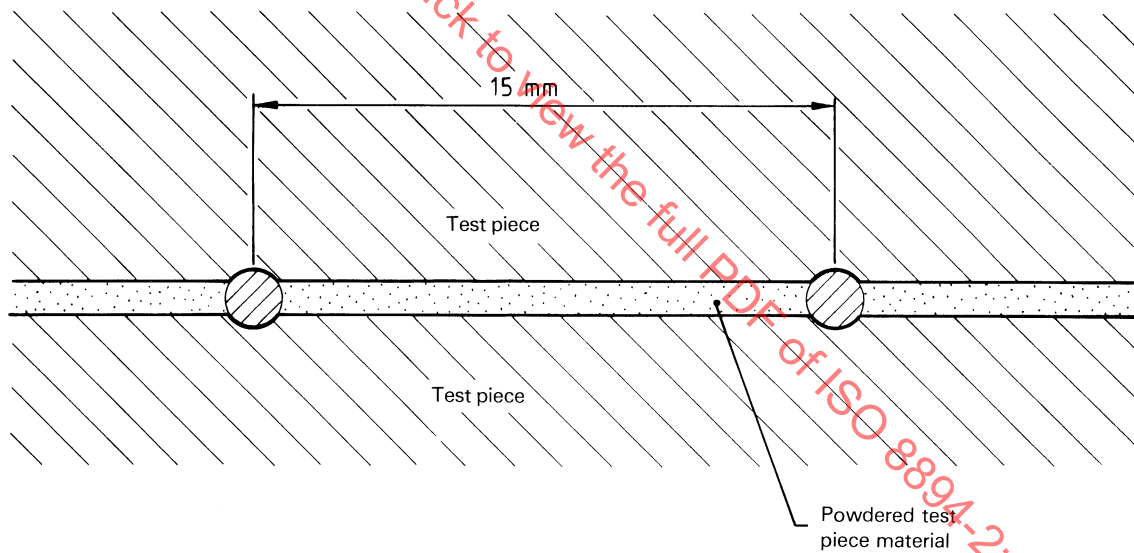


Figure 4 — Symmetrical embedding of hot wire and thermocouple in test pieces (where required)