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Indian Standard

METHOD FOR THE DETERMINATION OF THERMAL CONDUCTIVITY OF THERMAL INSULATION MATERIALS (TWO SLAB, GUARDED HOT-PLATE METHOD)

(*First Revision*)

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(First Revision)

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Indian Standard

METHOD FOR THE DETERMINATION OF THERMAL CONDUCTIVITY OF THERMAL INSULATION MATERIALS (TWO SLAB, GUARDED HOT-PLATE METHOD)

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 May 1980, after the draft finalized by the Thermal Insulation Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 One of the important properties of a thermal insulation material from the performance point of view is its thermal conductivity. Numerous methods of determining this property have been used and very often, the results obtained by different methods are not the same. The method, which has been recognized by scientists and engineers in most of the countries as the most dependable and reproducible, is the one known as the 'two-slab, guarded hot-plate method'. It is suitable for materials which can be laid flat between two parallel plates and can be adapted for loose-fill materials which can be filled between such plates.

0.3 Since much of the details of the apparatus and procedures will depend on the available resources and scientific background of the operators, it is possible for a standard like this to lay down only the general design principles of apparatus, methods for checking its performance and essential precautions and procedures which have been found in practice to yield reproducible and correct results. The ultimate objective of this standard is to obtain the value of thermal conductivity accurate to within $\pm$ 5 percent.

0.4 This standard was first published in 1966, deriving assistance from ASTM C 177-1945 and ASTM C 177-1963 'Test for thermal conductivity of materials by means of the guarded hot plate' issued by the American Society for Testing and Materials. Further, the earlier version of this standard included a specific design of the apparatus in the light of equipment already fabricated and used in the country. The present situation is that ASTM C 177-1963 has been revised again in 1971 and 1976. To keep pace with these developments and keeping in view the growing requirements of high class insulating materials, it has been considered essential to revise this standard. While doing so, it has not been found practical to base this revision on ASTM C 177-1976. However, this revision is, by and large, based on ASTM C 177-1971 'Standard method of test for thermal conductivity of material by means of guarded hot plate'.

0.4.1 In this revision, only essential design features and performance requirements of the apparatus have been specified. The temperature range of operation has also been extended so that the cold face temperature may be as low as 77 K and the hot face temperature up to 1 350 K. Any apparatus capable of measurements at intermediate temperature in the range 77 to 1 350 K and satisfying all other relevant conditions of the standard shall be treated as complying with the standard.

0.5 When a new or modified apparatus is constructed, tests shall be made on at least two sets of materials of known thermal stability which have been calibrated. Tests shall be made for each specimen at two mean temperatures typical of the operating range. All tests shall be conducted within 90 days of calibration, where possible. Any differences in results should be carefully studied to determine why they arise and what is the method for removing them. Appropriate action should be taken. It shall be ensured that the thermal flux is normal to the surface plates. Tests shall be made to determine the minimum temperature difference required between the hot and the cold plates to give a constant observed value of thermal conductivity and to determine the variation of the observed value of thermal conductivity with temperature difference across the central and the guard heater. Only after a successful comparison the apparatus should be considered satisfactory in performance. No further checking is necessary, though it is desirable to have periodic checks.

0.6 SI Units have been used in this standard. But in India various units are being used at present for reporting the values of thermal conductivity. Considering this, the Sectional Committee decided to give the factors for converting watt per metre kelvin to some of the important units used in the country at present and *vice-versa*. These conversion factors are given in Appendix A.

0.7 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the general procedures for determining the thermal conductivity of dry specimens of thermal insulating materials, building and other materials provided that:

a) the materials are homogeneous and sufficiently uniform with regard to their aggregates and pores;

b) the thermal conductance of the specimen does not exceed 60 W/m^{2}K; and

c) the two specimens used for test are identical in thickness and density within 2 percent and conform to **5.1**.

2. GENERAL

2.1 Terminology — For the purpose of this standard, the definitions given in IS : 3069-1965* shall apply.

2.2 Two different types of guarded hot plate apparatus have been prescribed in this standard. They are similar in principle but differ in construction to warrant separate descriptions for each in regard to design. The low-temperature guarded hot plate, which has metal surface plates and a definite guard gap (*see* **3.3**), is generally used for measurements at mean temperatures such that the temperature of the cold surface may be as low as 77 K or that of the hot surface as high at 550 K. The high-temperature guarded hot plate, which may or may not have metal surface plates and may or may not have a definite guard gap, is ordinarily used for measurements where the hot plate temperature is greater than 550 K and less than 1 350 K. It is made of a cast, or otherwise formed, electrically insulating (at the highest temperature of operation) refractory material. Metal surface plates may or may not be used although they are recommended to ensure a more uniform temperature distribution on the surfaces of the plate. All measurements made with specimen hot surface temperature below 550 K shall be carried out using a guarded hot plate having metal surface plates and a definite guard gap. In all other respects, the method is the same for both types of apparatus. It is intended, in presenting these descriptions, to indicate the essential elements and details which experience has shown to be necessary or important for reliable measurements by this method.

2.3 This standard does not prescribe details of construction and procedure to cover all contingencies that might offer difficulties to a person without technical knowledge concerning the theory of heat flow, temperature

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*Glossary of terms, symbols and units relating to thermal insulation materials.

measurement, and general testing practices. Nevertheless, standardization of the method does not reduce the need for such technical knowledge. It is also recognized that it would be undesirable to restrict in any way further development of improved or new methods or procedures by research workers.

2.4 This method may be adapted for loose-fill materials provided the size of the largest aggregate or void does not exceed one-tenth of the distance between a cooling plate surface and the surface of the heater assembly.

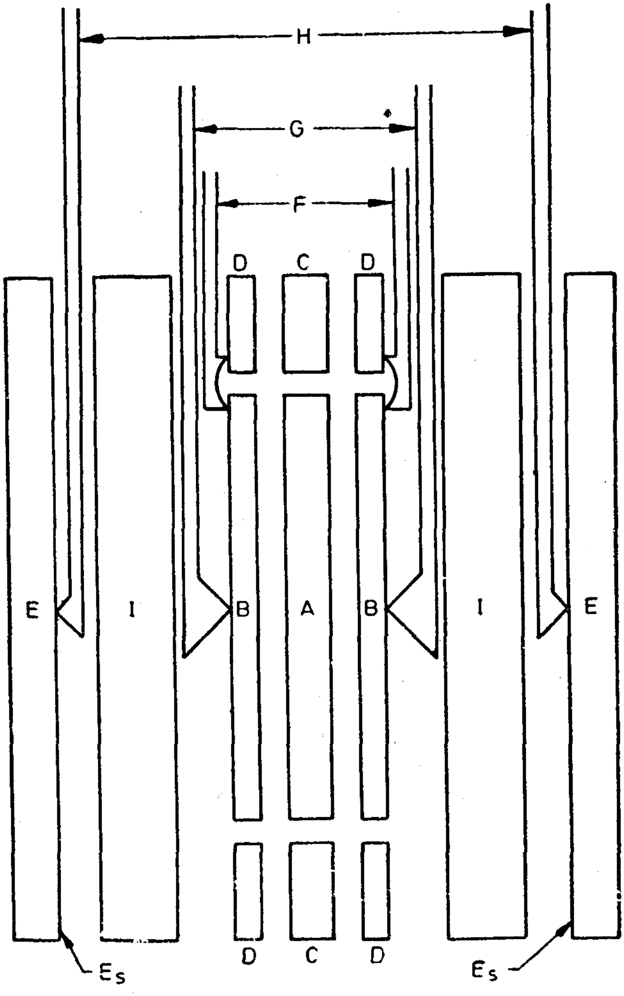
2.5 The thermal conductivity values obtained would apply only to the particular samples tested.

3. LOW-TEMPERATURE HOT PLATE

3.1 The general features of the metal-surfaced guarded hot plate are shown schematically in Fig. 1. The plate may be square, or round. The heating units consist of a central or metering section and a guard section. The central section consists of a central heater and central surface plates. The guard section consists of one or more guard heaters and the guard surface plates. The surface plates are made of noncorroding metal of high thermal conductivity. The working surfaces of the heating unit and cooling plates should be smoothly finished to conform to a true plane as closely as possible, and should be checked periodically. The maximum departure of a surface from a plane shall not exceed 0·25 mm/m. The planeness of the surface can be checked with a steel straight-edge held against the surface and viewed at grazing incidence with a light behind the straight-edge. Departures as small as 0·025 mm are readily visible, and larger departures can be measured using shim-stock or thin paper.

3.2 In the design of the guarded hot plate, the materials used in its construction should be considered with respect to their performance at the temperature at which the plate will be operated. The electrical design of the heater and the design of the cooling plate should also be considered to assure adequate capacity and suitable characteristics for the intended use. In all cases, design and construct the guarded hot plate so that in operation the two faces of the central section, and of the guard section, are substantially at the same uniform temperature, and that the heating units do not warp or depart from planeness at the operating temperature.

3.3 Heating units shall have a definite separation or gap not greater than 3 mm between the central surface plates and the guard surface plates. The area of the gap in the plane of the surface plate shall be not more than 6 percent of the metering section area on that side. The separation between the heater windings of the central section and the continuous guard section shall not exceed 20 mm, and this separation is allowable



Heating Unit
A — Metering area heater
B — Metering area surface plates
C — Guard heater
D — Guard surface plate
E — Cooling units
Es — Cooling unit surface plates
F — Differential thermocouples
G — Heating unit surface thermocouples
H — Cooling unit surface thermocouples
I — Test specimens

Metering Section of Heating Unit

Guard Section of Heating Unit

FIG. 1 GENERAL FEATURES OF THE METAL-SURFACE HOT PLATE APPARATUS only if the spacing bars on either side of the separation are of a high-conductivity material such as copper, in order to distribute heat to the surface plates. In all other cases, the heater winding separation shall not exceed 3 mm. The dimensions of the test area shall be established by measurements to the centres of the separations that surround this area. The surfaces of all plates shall be painted or otherwise treated to have a total hemispherical emittance greater than 0·8 at operating temperatures.

NOTE — Total hemispherical emittance greater than 0·8 at operating temperatures may be obtained by spraying or lightly brushing lamp black in methylated spirit to a thickness just enough to cover the original surface when viewed at grazing incidence, or some other suitable surface treatment may be used.

3.4 The guarded hot plate shall be provided with a suitable means of detecting temperature imbalance between the areas of the central and guard surface plates contiguous to the separation between them. The temperature-sensing elements shall be distributed to register adequately the temperature balance existing along the length of the central section periphery. The temperature-sensing elements may be read either individually to indicate any temperature difference that may exist, or they may be connected to be read differentially to indicate such temperature difference directly. Thermocouples are generally used for this purpose, with connections arranged so that they are read as a differentially connected thermopile. The detection system shall be sufficiently sensitive to ensure that variation in conductivity due to gap temperature imbalance is restricted to not more than 0·5 percent. The maximum temperature difference between the central and the guard surface plates shall be within 0·05 K. For testing at the lower temperatures, particular caution shall be used in designing for adequate sensitivity of the thermopile measurement and control system.

3.5 The cooling units shall have surface dimensions at least as large as those of the heating unit including the guard heater. They shall consist of metal plates maintained at a uniform temperature lower than that of the heating unit, either by a constant-temperature fluid, or by electrical heating, or by thermal insulation of uniform conductance applied on the outermost surface, as appropriate for the cooling unit temperature desired.

3.6 For measuring the surface temperature of the central section of the heating unit, each of the central surface plates shall be provided with, preferably, permanently installed thermocouples set in grooves or just under the working surface. The number of such thermocouples on each side shall be not less than $5\sqrt{A}$, where A is the area in square metres of one side of the central surface plate. There shall be the same number of thermocouples similarly installed at corresponding positions in the facing cold plate. If the hot and cold plate thermocouples on each side are to be connected differentially, which is usual, they shall be electrically insulated from the plates.

3.7 Means shall be provided for imposing a reproducible constant pressure of the plates against the specimens to promote good thermal contact. A steady force thrusting the cold plates toward each other may be imposed by means of a calibrated compressed spring, or a system of levers and dead weights, or an equivalent method. It is unlikely that a pressure greater than 2·5 kPa ($2·5 \times 10^2$ kgf/m^2) on the specimens would be required; for easily-compressible specimens, small stops interposed between the corners or edges of the cold plates, or some other positive means, may be used to limit the compression of the specimen.

3.8 Means shall also be provided for measuring the effective thickness of the specimen to within 0·5 percent. Because of the changes in specimen thickness possible as a result of temperature, or compression by the plates, it is recommended that specimen thickness be measured in the apparatus, at the existing test temperature and compression conditions, when possible. Gauging points, or measuring studs, at the outer four corners of the cold plates or along the axis perpendicular to the plates at their centres, will serve for these measurements. The effective combined specimen thickness shall be determined by the difference in the micrometered distance, or average distance, between the gauging points when the specimen is in place in the apparatus, and when is not in place, and the same force is used to press the cold plates towards each other.

3.9 The best method of determining the temperature drop in the specimen depends upon its characteristics, and in some instances the choice of method is left to the judgement of the operator. For non-rigid specimens with flat, uniform surfaces that conform well to the flat working surfaces of the plates, the temperature drop in specimens of thermal conductance less than 10 W/m^2K shall be taken as that indicated by the thermocouples permanently set in the hot and cold surface plates, and the thickness of the specimen shall be taken as the mean distance between the working surface of the hot and cold plates. For non-rigid specimens of conductance greater than 10 W/m^2K, the operator's judgement should rule in accordance with the circumstances. Rigid specimens to be tested shall have surfaces both flat and parallel to within 0·25 mm/m. One method of testing rigid specimens is to install them in the apparatus with a thin sheet of suitable homogeneous material interposed between the specimen and each plate surface. This thin sheet should have a high thermal conductance relative to that of the insulating material being tested. The method of determining the conductance of a rigid specimen is to interpose the thin layer of material between the specimen and plates as indicated above, and to determine the temperature drop across the rigid specimen by means of separate thermocouples mounted flush with, or interior to the surface of the rigid specimen. This method of measuring the specimen temperature drop may be subject to uncertainties difficult to evaluate, among them being the effects of (a) distortion of heat flow lines in the immediate vicinity of the thermocouple, due to its presence; (b) imprecision in ascertaining the exact position of the effective thermocouple junctions; and (c) local inhomogeneities in the surface of the specimen at the thermocouple junctions such as pores, voids or inclusions. The number of separate thermocouples used on each side of the specimen shall be not less than $10\sqrt{A}$, where A is the area in square metres of one side of the central surface plate. If separate thermocouples are used, the effective thickness of the specimen shall be taken as the average distance, perpendicular to the face of the specimen, between the centres of the thermocouples on the two sides.

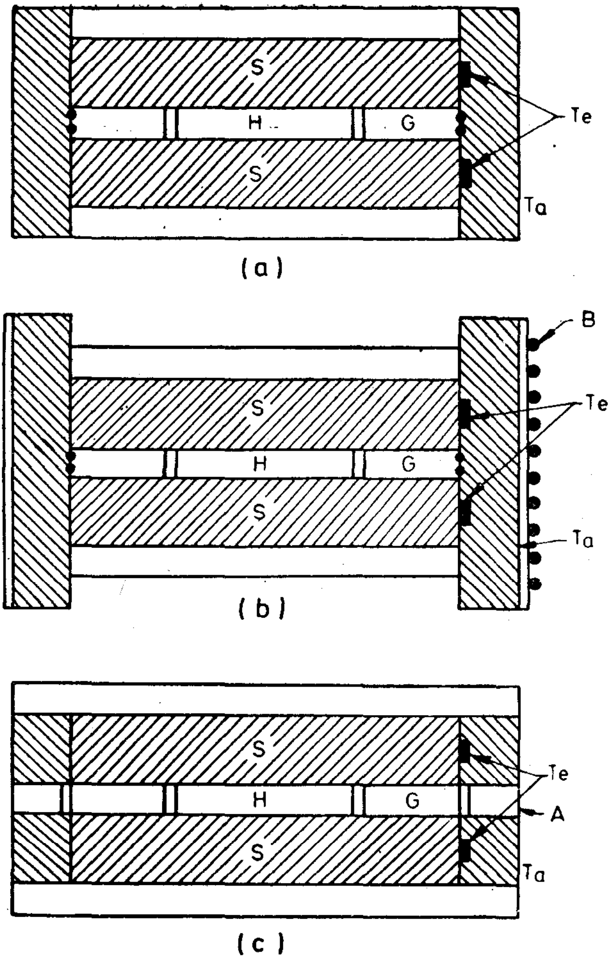
3.10 Thermocouples mounted in the surfaces of the plates shall be made of wire not thicker than 0·57 mm in diameter (24 SWG); specimen surface thermocouples shall be made of wire not thicker than 0·29 mm in diameter (32 SWG), and preferably of 0·19 mm in diameter (36 SWG). The thermocouples which are used to measure the temperatures of the hot and cold faces of the specimen shall be fabricated from calibrated thermocouple wire. Thermocouples used to measure temperatures in the range 77 to 170 K shall have a standard limit of error of $\pm$ 1 percent.

3.11 A voltage measuring system having a sensitivity of $\pm$ 1 μV or better and an accuracy of $\pm$ 0·1 percent or better shall be used for measurement of all thermocouple and thermopile emfs.

3.12 Heat losses from the outer edges of the guard section and the specimens shall be restricted by edge insulation, by governing the surrounding ambient temperature, by an additional outer guard, or by a combination of these methods. Three possible configurations that could be used to restrict edge heat losses or gains are shown in Fig. 2. A useful method of determining whether or not sufficient edge guarding insulation is present is to measure the average temperature T_e at the edge of the specimen (this may be done using a thermocouple soldered or peened to a thin metal strip centered on the edge of one of the specimens). Under these conditions, the satisfactory condition shall be:

$$[\,\Delta T_1/\Delta T\,] \leqslant 0{\cdot}1$$

where ΔT_1 is the difference between T_e and T_m (T_m is the mean temperature of the specimens) and ΔT is the temperature difference across the specimens. In the first two cases shown in Fig. 2, if no guard



A — Second guard heater
B — Outer cylindrical guard
S — Sample
H — Heater
G — Guard
T_e — Tedge — Average temperature at the edge of the specimen
T_a — Temperature of the outer surface of the edge insulation

FIG. 2 POSSIBLE CONFIGURATIONS TO RESTRICT EDGE HEAT LOSS AND GAIN ring perimeter heater is used, the required minimum thermal resistance of the edge insulation, on the basis that the total edge heat loss shall not exceed one-fifth of the heat flow through the two specimens, is given by the following equation:

$$R = 5x/S\lambda\,[(4x + 2y)(T_m - T_a)/\Delta T + y]\,\frac{m^2K}{W}$$

where

x = thickness of each specimen in m;

y = thickness of the heating unit in m;

S = length of the side (or diameter) of the guard section in m;

λ = thermal conductivity of the specimens in W/mK;

T_m = mean temperature of the specimens in K;

T_a = temperature of the outer surface of the edge insulation in K; and

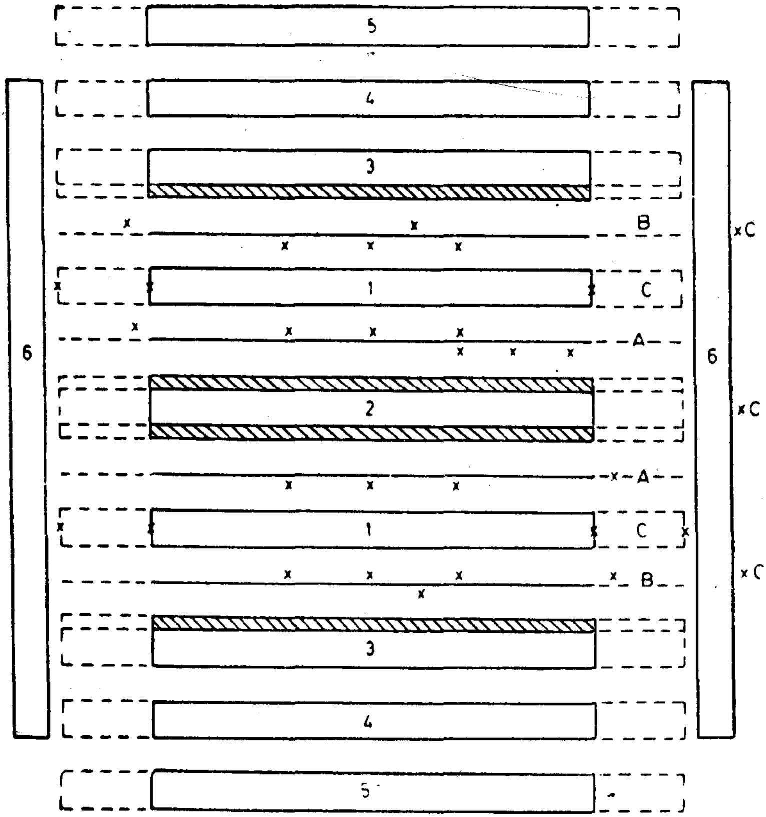
ΔT = temperature difference across the specimens in K.

Since it is desirable that the net heat transfer from the outer edges of the specimens should be kept nearly equal to zero, ($T_m - T_a$) should be kept small.

3.13 A cabinet or enclosure surrounding the guarded hot plate, and equipped for maintaining the desired interior air temperature and dew point shall be used in tests conducted at mean temperatures differing substantially from the laboratory air temperature.

4. HIGH-TEMPERATURE HOT PLATE

4.1 The general features of the high-temperature guarded hot plate are shown schematically in Fig. 3. The plate may have round or square configuration. If a square plate configuration is used, separate independently controlled guard heaters shall be provided to allow for the additiona heat losses which occur at the corner regions. The heating units consist of (a) a central or metering section; (b) a guard section, that is, either a double guard heater, with the outer guard section having a width equal to or greater than one half of the inner primary guard width, or a primary guard heater with an outer cylindrical guard extending over the length of the composite sample stack; (c) additional corner heaters (for square configuration only); and (d) cold surface heaters. If metal surface plates are used, they should be of a suitable non-corroding metal of as high a thermal conductivity as possible. Physical separation of the central and inner guard areas is to be preferred if associated problems of the alignment and flatness of the plates are minimized by suitable design. All heaters should be capable of being adjusted to any desired temperature level



1 — Test sample (2 pieces)
2 — Metering area heater (with optional metal surface plates)
3 — Cold surface heater (with optional metal surface plates)
4 — Insulation slab
5 — Liquid cooled heat sink
6 — Outer cylindrical guard
A — Hot surface temperature measurement and control system
B — Cold surface temperature measurement system
C — Edge temperature measurement and control system
——— Schematic arrangement for guard plus cylindrical guard configuration
- - - - Schematic arrangement for double guard configuration

FIG. 3 GENERAL FEATURES OF THE HIGH TEMPERATURE HOT PLATE APPARATUS within the limits specified in **2.2**. The cooling units normally consist of two liquid-cooled heat sinks with an adequate layer of insulation between them and the adjacent cold surface heater. An additional outer peripheral liquid-cooled shroud is recommended (*see* **4.11**). The working surface of the heating units and cooling plates shall be finished smoothly to conform to a true plane as closely as possible and should be checked regularly. The maximum departure of the surface from a plane shall not exceed 0·25 mm/m. The refractory material plates should have a thermal expansion not greater than 1 percent of the linear surface dimension of the hot plate. This expansion shall be computed from the difference between the length measurements taken at maximum use temperature and at room temperature.

4.2 In the design of the high-temperature guarded hot plate, due consideration shall be given to obtaining satisfactory performance at the temperatures at which the plate will be operated. The electrical design of the heaters and the design of the cooling plates shall be considered to assure adequate capacity and suitable characteristics for the intended use. The refractory plate composition should have adequate electrical resistance at the maximum use temperature to prevent possible power exchange between adjacent heating circuits embedded within the refractory material. The maximum permissible power exchange shall be 0·5 percent of the test area power consumption at the particular test temperature. In all cases great care should be taken to ensure that there will be no compatibility problems between the test specimens and the materials used in the construction of the plates for the temperature and environment conditions of a specific measurement. In all cases, the guarded hot plate shall be designed and constructed so that in operation the two faces of the central section, and of the guard section, are substantially at the same uniform temperature, and that the heating units do not warp or depart from planeness at the operating temperatures. The surfaces facing both sides of the test specimens should be treated so that they have a total hemispherical emittance at the operating temperatures of not less than 0·7 and preferably much higher.

4.3 In a high-temperature plate having a gap between the central and guard areas, the gap shall not exceed 2 mm. The gap between the central and guard areas shall be filled with a thermally and chemically compatible high-temperature insulation to avoid radiative heat transfer across the gap. The effective metering area of the high-temperature plate shall be determined by the positions of the potential taps used to evaluate the power input to the metering area winding. For a double-spiral (bifilar) winding with the spacing between wires equal to b, and with the potential taps for the metering section in effect at points on the wires at the ends of a diameter $2a$ (or for a single-spiral winding of spacing b with the potential taps in effect at the centre and at a radius a), the effective metering area is equal to:

$$\pi a^2 [1 + (1/12) (b/a)^2]$$

4.4 The guarded hot plate shall be provided with a suitable means for detecting temperature imbalance between the central and guard sections of the plate. The temperature-sensing elements shall be distributed to register adequately the temperature balance existing between the outer edge of the metering area and the inner edge of the guard section. Thermocouple junctions used for detecting temperature imbalance shall be located at the edge of the metering area on the same radius, and distant from the edge of the metering area by not more than one-quarter of the guard width. The temperature-sensing elements may be read either individually to indicate any temperature difference that may exist, or they may be connected to read differentially to indicate such temperature difference directly. The detection system shall be sufficiently sensitive to assure that variation in conductivity due to temperature imbalance between the central and guard sections shall be restricted to not more than 0·5 percent. Thermocouples should be fixed on the edge of each test specimen at the centre position (*see* **3.12**).

4.5 The cold-surface heaters shall have surface dimensions at least as large as those of the combined central and guard sections of the hot plate. They shall consist normally of a flat single heater and refractory formers with or without metal surface plates, maintained at a uniform temperature lower than that of the main hot plate.

4.6 Permanently installed thermocouples to be used in determining the temperature difference across the specimen shall be set flush with the working surface and shall number not less than $5\sqrt{A}$ on each working surface, where A is the area in square metres of the metering area of the hot plate on one side. However, permanently installed thermocouples are not mandatory if the temperature difference across the specimen is to be determined by means of separate thermocouples (*see* **3.9**).

4.7 Means shall be provided (a) for imposing a reproducible constant load upon the system to promote good thermal contact (*see* **3.7**), and (b) for measuring the effective thickness of the specimen to within 0·5 percent (*see* **3.8**). Thickness measurement *in-situ* at the temperature of test is necessary or should be reliably calculated if an accurate thermal conductivity value is to be obtained. Furthermore, due care should be taken to measure the thickness before and after the test has been completed in order to check for irreversible changes.

4.8 The best method of determining the temperature drop across the specimen depends on the circumstances and is, therefore, left to the best judgement of the operator. One method often used is to attach separate thermocouples on a sheet of asbestos paper or other suitable material, and to interpose the sheet between the specimen and the adjacent working surface of the apparatus, with the thermocouples in contact with the specimen. For rigid and hard specimens, which should be flat to within 0·25 mm/m, it may be important to set the separate thermocouples in tight grooves in the faces of the specimens. The number of separate thermocouples used, at each face of the specimen, shall be not less than $10\sqrt{A}$, where A is the metering area in square metres of one side of the hot plate. If separate thermocouples are used, the effective thickness of the specimen shall be taken as the average distance, perpendicular to the face of the specimen, between the centres of the thermocouples on the two sides. Another method, feasible if a suitable resilient sheet material is available for the test temperatures in question, is to use the composite sandwich (sheet/specimen/sheet) technique described in **3.9**, in which permanently installed thermocouples in the plates are used. This method automatically compensates for any effective or virtual thermal resistance between the positions where the permanently installed plate thermocouples are located and the plate working surface. Such resistance may be appreciable in the case of a high-temperature plate.

4.9 Thermocouples mounted in the surfaces of the plates shall be of wire not thicker than 0·64 mm in diameter (23 SWG); and specimen surface thermocouples shall be made of wire not larger than 0·46 mm in diameter (26 SWG). The thermocouples that are used to measure the temperatures of the hot and cold faces of the specimen shall be fabricated from calibrated thermocouple-wire.

4.10 A voltage-measuring system having a sensitivity of $\pm$ 1 μV or better and an accuracy of $\pm$ 0·1 percent or better shall be used for measurements of all thermocouple and thermopile emfs.

4.11 To reduce heat losses from the outer edges of the composite test section, the assembly shall be surrounded by a coaxial cylindrical container of suitable material of internal diameter at least twice the diameter of the stack assembly. The interspaces and surrounds shall be filled with a suitable insulation within distance of somewhat greater than the axial length between the outermost surfaces of the heat sinks. Extreme care should be taken to ensure that no voids, gas pockets, or other extraneous sources of high temperature radiative heat transfer can occur at or near the test section.

5. TEST SPECIMENS

5.1 Prepare the specimens from each sample. They shall be as nearly identical as possible, of such size as to completely cover the heating unit surfaces, and shall be of sufficient thickness to give a true average representation of the material to be tested. The relationship between the thickness of the test specimen used and the dimensions of the guarded hot plate shall be as given in Table 1.

TABLE 1 THICKNESS OF SPECIMEN AND DIMENSIONS OF CENTRAL AND GUARD HEATER

THICKNESS OF EACH SPECIMEN, *Max*	LINEAR DIMENSION OF THE TWO HEATERS*, *Min*	
	Central Heater	Guard Heater Width
(1)	(2)	(3)
mm	mm	mm
33	100	50
50	150	75
75	225	113
100	300	150

*Diameter in case of circular heater assemblies and edge in case of square heater assemblies.

5.2 Insulating materials may be broadly classified as blocks, fluffy materials and loose-fill materials.

5.2.1 A block may be considered soft or hard for this purpose on the basis whether the thermocouple wire can dig into it or not when clamped in the thermal conductivity apparatus. Insulating and finishing cements and moulding compositions are made into blocks before such a test is carried out, the idea being to test the material in the condition in which it will be actually used.

5.2.2 Fluffy materials are of two types — they may be in a loose form or in the form of a continuum. Grass, fodder and wool are some examples of the former while blankets, matts, and semirigid preparations of such materials belong to the latter class.

5.2.3 Loose-fill materials, on the other hand, may be in the form of aggregates, granules or fines. Fines may be powders or dust depending on their grain size.

5.3 In testing all forms of homogeneous materials, the surface of the test specimens shall be made as plane as possible, by sandpapering, face-cutting in a lathe, grinding, or otherwise, so that intimate contact between the specimens and the plates or interposed sheets is effected. For rigid materials, the faces of the specimens shall be made flat to within 0·25 *mm/m* and parallel, within the total plate area, to within 1 percent of the sample thickness.

5.4 When testing homogeneous solid or blanket-type materials, prepare the specimens in accordance with **5.1** and **5.3**. Determine their mass before and after they have been dried to constant mass in a ventilated oven at a temperature from 375 to 395 K. If the material may be adversely affected by heating to 375 K, dry it in a desiccator at a temperature of approximately 330 K. From these masses calculate the percentage moisture as-received. Promptly after drying and weighing, place the specimens in the apparatus taking care to prevent loss of material or moisture gain. Determine the as-tested thickness and volume by measurements made preferably at the end of the test under conditions of test-temperature equilibrium, and from these data and the dry mass, calculate the as-tested density. If it is feasible to do so with good thermal contact between the plates of the apparatus and the specimens, blanket-type material should be tested at approximately the thickness and density determined in accordance with **6** of IS : 3144-1965*.

5.5 When testing loose-fill materials, take a representative portion slightly greater than the amount needed for the test from the sample. Weigh this material before and after it has been dried to constant mass in a ventilated oven at a temperature from 375 to 395 K. If the material may be adversely affected by heating to 375 K, dry it in a desiccator at 330 K. From these masses, calculate the percentage moisture as-received. Then weigh out an amount of the dried material such that it will produce two specimens of the desired as-tested density using either Method A or B, given in Appendix B. As the volume of the specimen space is known, the required mass can be determined. During placement, take care to prevent loss of the weighed material, or significant moisture gain. When Method A is used, or Method B with covers of insignificant thermal resistance, the specimen surface temperatures shall be taken as equal to those of the surface of the hot and cold plates.

6. PROCEDURE

6.1 Temperature Difference and Gradient — For any test, adjust the temperature difference between the hot and cold surfaces of the specimens to not less than 5 K and not more than 100 K and preferably of the order of 20 to 50 K.

*Methods of test for mineral wool thermal insulation materials.

6.2 Ambient Conditions — When thermal conductivity values are desired for the situation where the specimen is immersed in air (or some other gas), adjust the atmosphere surrounding the guarded hot plate during a test to a dew-point temperature 5 K or more lower than the cold-plate temperature. For operation at cryogenic temperature this shall require purging the system with dry gas prior to cooling the apparatus. Between 77 K and 230 K, use dry nitrogen gas, rather than air as the atmosphere and contain the apparatus in a sealed system. At cold-plate temperatures below 125 K, care shall be taken to adjust the nitrogen pressure so as to avoid condensation. When thermal conductivity values are desired for the situation where the specimen is *in vacuo*, evacuate the system prior to cooling.

6.3 Heat Flow Measurement — Supply the heating element of the central heater with electrical energy for heating it, providing for the measurement of the average rate of heat generation therein to an accuracy of not less than 0·5 percent. Automatic regulation of the input power is desirable; in any case, fluctuations or changes in input power shall not cause the temperature of the hot plate surfaces to fluctuate, or to change in 1 hour of a test period, by more than 0·5 percent of the temperature difference between the hot and cold plates. Measure the input power in such a way that it is possible to determine the average power input during a test period. If the power input is of fluctuating kind, make an integrated energy measurement. Adjust and maintain the power input to the guard section, preferably by automatic control, so as to effect the degree of temperature balance between the central and guard heater sections that is required for conformity to **3.4** or **4.4**. For measurements at high temperatures, adjust the power to the outer guard or cylindrical guard heater, preferably by automatic control so as to effect, for the test specimen, the degree of temperature balance between the centre of the outer edge and the mean temperature necessary to conditions given in **3.12**.

6.4 Cold Surface Control — Adjust the cooling units or cold surface heaters so that the temperature drops through the two specimens do not differ by more than 1 percent. The temperatures of the cooling plates or cold surface heaters, in the case of high-temperature plate, during a test period shall not fluctuate or change, in 1 hour, by more than 0·5 percent of the temperature difference between the hot and cold surfaces of the specimen.

6.5 Temperature Difference Measurement — Determine the temperature difference across the specimens, the hot- and cold-plate temperature, the centre-to-guard temperature balance, and the electrical power input to the central section.

6.6 Equilibrium Time and Measurement Interval — In order to attain the thermal conductivity value, it is essential to allow sufficient time for the apparatus and specimens to attain thermal equilibrium. The time required will depend on the specific apparatus, the control system and its operation, the test temperatures, and on the thermal diffusivity and thickness of the specimens. The observations listed in **6.5** should be made at intervals of not less than 30 minutes, until four successive sets of observations give thermal conductivity values differing by not more than 1 percent. In low temperature measurements on good insulators having low thermal diffusivities, the internal temperatures of the specimens may require a very long time to attain thermal equilibrium so that it is possible to have four consecutive 30 minute tests which yield thermal conductivity values within 1 percent of each other and still not have steady-state conditions. Sufficient time shall be allowed for the internal temperatures of the insulation to stabilize.

6.7 Operation in Vacuum — Particular care is indicated if a guarded hot plate is used for measurements under vacuum conditions. If a hard vacuum is desired, materials should be carefully selected to avoid excessive outgassing. Under vacuum conditions, especially at lower temperatures, serious errors may arise if due care is not taken in installing heater and thermocouple leads so as to minimize extraneous heat flows and temperature measurement errors. Vacuum operation may greatly increase the time required for the apparatus and specimen to reach thermal equilibrium (due to outgassing of the apparatus and specimens and to the lower thermal diffusivity of the specimens).

7. CALCULATIONS

7.1 Calculate the density of the dry specimen as tested, D, the as-received moisture content of the material, M; and the moisture regain of the specimen during test, R_W or R_V, as follows:

$$D = M_2/V$$
$$M = [(M_1 - M_2)/M_2] \times 100$$
$$R_W = [(M_4 - M_3)/M_3] \times 100$$
$$R_V = [(M_4 - M_3)/1\,000\,V] \times 100$$

where

D = density of the dry material as tested in kg/m^3;

M = moisture content of the material as-received, dry mass percent;

R_W = moisture regain of material during test, dry mass percent;

R_V = moisture regain of material during test, dry volume percent;

M_1 = mass of material in as-received condition in kg;
M_2 = mass of material after drying in kg;
M_3 = mass of dry material in specimens in kg;
M_4 = mass of material in specimens immediately after test in kg; and
V = volume occupied by material in specimens during test in m^3.

7.2 Calculate the thermal conductivity of the specimens using steady state data as follows:

$$\lambda = \frac{i\,v\,l}{2A\,(T_h - T_c)}\ \text{W/mK}$$

where

= current in amperes through the central heater;
v = potential drop in volts;
l = thickness of specimen during test in m;
A = specimen area enclosed by the boundary running midway through the air gap between the central and the guard heater in m^2;
T_h = hot face temperature in K; and
T_c = cold face temperature in K.

7.2.1 In case the thickness of the two specimens is not exactly the same and, consequently, their hot and cold face temperatures are not identical, the thermal conductivity, λ of the specimens shall be determined by the following formula:

$$\lambda = \frac{i\,v}{A\left\{\dfrac{T_{h1} - T_{c1}}{l_1} + \dfrac{T_{h2} - T_{c2}}{l_2}\right\}}\ \text{W/mK}$$

where

l_1 and l_2 are thicknesses of the two specimens respectively in m;
T_{h1} and T_{h2} are hot face temperatures of the two specimens respectively in K;
T_{c1} and T_{c2} are the cold face temperatures of the two specimens respectively in K; and
other symbols have the same meaning as in **7.2**.

7.2.1.1 The value of λ computed in this way is generally referred to the mean temperature $\frac{1}{2}(T_h + T_c)$, although strictly speaking, it can refer only to the respective hot face and cold face temperatures. The essential condition of this computation is that the lines of thermal flux should remain straight and normal to the hot and cold faces while passing through the specimen.

8. REPORT

8.1 The report of the results of each test shall include the following:

a) Name and other identification of the material;

b) Thickness of the specimen tested (mean for two specimens);

c) Mass after drying of the two specimens used for the test (total mass);

d) Method and temperature of drying;

e) Density, before test just after placing the samples in the apparatus, of the two specimens used for the test (mean for two specimens);

f) Moisture, as received, in the two specimens used for the test (mean for two specimens);

g) Moisture regain, during test, of the two specimens used for the test (mean for two specimens);

h) Hot face temperature (mean for two hot faces);

j) Cold face temperature (mean for two cold faces);

k) Mean temperature of the test (arithmetic mean of the hot and cold face temperature);

m) Heat input per unit area (average of the two specimens);

n) Thermal conductivity;

p) Orientation of plane of specimen (horizontal);

q) Special remarks, if any. Here mention should be made of:

1) Whether the specimens adjacent to the central heater were isolated by air gap from those next to guard heater;
2) Whether some metallic portions, or coatings of insulation, were removed during test;
3) Whether any tests on cold face isothermals were carried out;
4) Any other special point tha tmay be relevant from the scientific or application point of view; and
5) The vacuum reading or type and pressure of purge gas surrounding the specimen.

8.2 For tests made using sheet material interposed between the specimen and the plate surfaces, give information as to the nature, thickness and thermal conductance of the sheet material. If separate thermocouples were used to determine the temperature drops in the specimens, also give information as to the size of the thermocouples, the method of affixing them to the specimen, and the measured distance between their centres.

8.2.1 Where appropriate to the condition and temperature of the test, indicate the approximate resistance of the edge insulation and the ambient temperature surrounding the plate during the test, and also the duration of the measurement portion of the test.

8.2.2 Include a graphical representation of the results in the report when pertinent. This shall consist of a graph of each value of thermal conductivity obtained versus the corresponding test mean temperature, plotted as ordinates and abscissae, respectively.

APPENDIX A

(*Clause* 0.6)

CONVERSION FACTORS OF THERMAL CONDUCTIVITY

	Multiply by	*For Reverse Conversion Multiply by*
To convert 1 W/mK into Btu in/ft²h deg F	6·933	0·144 2
To convert 1 W/mK into Btu/ft h deg F	0·577 79	1·730 73
To convert 1 W/mK into cal/cm s K	0·002 388	418·7
To convert 1 W/mK into kcal/m hK	0·859 85	1·163

APPENDIX B

(*Clause* 5.5)

METHODS FOR PREPARING TEST SPECIMENS OF LOOSE MATERIALS

B-1. Method A — Set up the guarded hot plate with the required spacings between the heating unit and the cold plates. Place low-conductivity material that is suitable for confining the sample around or between the outer edges of the guard section and the cold plates in such a manner that it forms two boxes, one on either side of the heating unit, and each open at the top. Divide the weighed, dried material mentioned in **5.5** into eight equal portions; four for each specimen. Place each portion in turn in each of the two specimen spaces, each portion being vibrated, packed, or tamped in position until it occupies its appropriate one quarter volume of the space, care being taken to produce specimens of uniform density.

B-2. Method B — Use two shallow boxes of thin-walled low-conductivity material, having outside dimensions the same as those of the heating unit. The box edges shall be of such width as to make the depth of the box equal to the thickness of the specimen to be tested. Make covers for the open faces of the boxes, using (a) thin sheet plastic material not more than 0·05 mm thick or (b) blotting or asbestos paper or other suitable uniform sheet material; these to be glued or otherwise fastened to the edges of the boxes. If the covers have significant thermal resistance, the methods of determining the net specimen conductance presented in **3.9** for rigid specimen may be used. Divide the weighed, dried material mentioned in **5.5** into two equal portions, one for each specimen. With one cover in plane, and with the boxes lying horizontally on a flat surface, place one portion in each box, care being taken to produce two specimens of equal and uniform density throughout. Then apply the remaining cover to make closed specimens that can be put into position in the guarded hot plate. Compressible materials are fluffed when placed so that the covers bulge slightly and make good contact with the plates of the apparatus at the desired density. For loose material not readily compressed or altered in density, Method A is preferred to avoid gaps at the top of the specimens after they have been placed in the apparatus. For some materials moisture gain, or material loss, during preparation of the specimens may necessitate redrying the completed specimen in an oven and reweighing before test. In this case determine the mass of the dried box and covers after the test to compute the as-tested density of the material.

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