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Draft Indian Standard

MINERAL WOOL THERMAL INSULATION MATERIALS - METHOD OF TEST

(Third Revision of IS 3144)

(ICS 91.100.60)

**Thermal Insulation Sectional Committee,
CHD 27**

Last Date for Comments: 16 Feb 2023

FOREWORD

(Formal clauses shall be added later)

Mineral wool, is a generic term for the products known as rock wool, slag wool or glass wool. The basic structure of each of these types of materials is similar. These are fluffy, light-weight mass of fine intermingled vitreous mineral fibres composed of complex silicates. Mineral wool is used principally as a thermal insulation material. It is also extensively used for sound absorption and as a filter medium.

The purpose of this standard is, therefore, to provide details of standard methods of tests required for the assessment of properties which are likely to be of immense value and relevance to applicators and users.

This standard was first published in 1965. The first revision in 1981 was taken up to incorporate additional tests for alkalinity, organic matter and chloride content. The committee, then, had also decided to modify the test methods for determination of apparent density under specified load, incombustibility, fibre diameter and water vapour permeance.

In the second revision in 1992, the test method for determination of apparent density was modified; reference to thermal conductivity was incorporated; and test for maximum recommended temperature, determination of shot content, determination of settling due to vibration and jolting, determination of fibre diameter were also modified.

This revision has been taken up in order to bring out the standard in the latest style and format of the Indian Standards. The relevant clauses have been added and the references have been updated. The test method for determination of shot content has been modified. Amendment No. 1, 2, and 3 issued to the previous version of this standard have also been incorporated in this revision.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

Draft Indian Standard

MINERAL WOOL THERMAL INSULATION MATERIALS - METHOD OF TEST

(Third Revision)

1 SCOPE

This standard prescribes the methods of test for mineral wool thermal insulation materials.

2 REFERENCES

The Indian standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Indian standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No</i>	<i>Title</i>
196 : 1966	Atmospheric conditions for testing (<i>revised</i>)
250 : 1964	Specification for potassium bichromate, technical and analytical reagent (<i>revised</i>)
265 : 2021	Hydrochloric acid - Specification (<i>Fourth Revision</i>)
309 : 2005	Compressed oxygen gas - Specification (<i>Fourth Revision</i>)
323 : 2009	Rectified spirit for industrial use - Specification (<i>Second Revision</i>)
460 (Part 1) : 2020	Test Sieves — Specification Part 1 Wire Cloth Test Sieves (<i>Fourth Revision</i>)
718 : 1977	Specification for carbon tetra-chloride (<i>second revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>Third revision</i>)
1476 (Part 1) : 2000	Performance of household refrigerating appliance - Refrigerators with or without low temperature compartment: Part 1 energy consumption and performance (<i>Third Revision</i>)
2263 : 1979	Methods of preparation of indicator solutions for volumetric analysis (<i>first revision</i>)
2316 : 1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (<i>Second Revision</i>)
2711 : 1979	Specification for direct reading pH meters (<i>second revision</i>)
2835 : 1987	Specification for flat transparent sheet glass (<i>third revision</i>)
3069 : 2020	Glossary of Terms, Symbols, and Units Relating to Thermal Insulation Materials (<i>Second Revision</i>)
3346 : 1980	Method for the determination of thermal conductivity of thermal insulation materials (two slab guarded hot plate method) (<i>first revision</i>)
9489 : 1980	Method of test for thermal conductivity of thermal insulation materials by means of heat flow meter
9490 : 2018	Method for determination of thermal conductivity of thermal insulation materials (Water Calorimeter Method) (<i>First Revision</i>)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 3069 shall apply.

4 SAMPLING

Representative samples of the material shall be drawn as prescribed in the relevant material specifications.

5 QUALITY OF REAGENTS

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

6 DETERMINATION OF LENGTH AND WIDTH

6.0 Principle

Length and breadth are measured with a steel rule.

6.1 Materials with Clearly Defined Edges

Procedure- Lay the material on a plane surface and measure its length and breadth by laying a steel scale across the surface of the insulating material parallel to one edge and at right angles to the adjacent edge. Measurements shall be taken at three positions on one face for both length and breadth, namely, about 15 mm inwards from each edge and across the centre. Turn the insulation over and repeat the measurements on the opposite face. Read the measurements to the nearest millimetre. Record the mean of the six measurements as length and breadth of the material.

6.2 Materials with Indeterminate Edges

Materials which are compressed or tightly rolled during transit shall be unpacked and unrolled on to a flat surface and allowed to expand freely for at least 4 hours before measurement.

Procedure- Measure the full size of the material as accurately as possible consistent with the nature of the edges in accordance with the procedure prescribed in **6.1**. A T-square erected perpendicular to the plane surface and in reasonable contact with the edge of the material may be of assistance in determining the point of reading on the scale. The mean of six measurements shall be recorded as length and breadth of the material.

6.3 Materials in the Form of Long Rolls

In this case, additional measurements of width shall be made and turning the material over shall be omitted.

Procedure- Cut the test samples under a template and check the bottom face against the template for accuracy of square cut edges. When cutting round the template, care shall be taken to avoid any extrusion of the material from the edges due to excessive pressure on the template. Measure the length and breadth on the template as prescribed in **6.1** but on one face only and these measurements shall be considered to be those of the specimen.

7 DETERMINATION OF THICKNESS

7.0 Principle

The thickness of the material is measured by pushing the needle of the thickness-measuring instrument into the material and then measuring the length of the needle with a steel scale.

7.1 Apparatus

7.1.1 Thickness Measuring Instrument

It consists of a 3 mm diameter, 200 mm long mild steel rod pointed to a sharp conical end. The length of the conical portion shall be 20 mm. This rod is fitted with a metallic disc, 75 mm in diameter and 50 g in mass, which is provided with means of clamping it to the rod.

7.1.2 *Steel Scale* — of suitable length having an accuracy of 1 mm.

7.1.3 *Glass Sheet or Mild Steel Sheet* — Plane, 3 mm thick and at least 60 × 60 cm in size.

7.2 Test Specimens

The test specimens shall be square, of the size of the full width of the material.

7.3 Procedure

7.3.1 Cut test specimen from the material and place it on a glass sheet or a mild steel sheet. Allow it to expand for 4 hours without disturbing the confining medium, if any. Consider the test specimen divided into nine squares of approximately equal area and measure the thickness at the centre of each square as given in 7.3.2.

7.3.2 Push the thickness-measuring instrument into the specimen perpendicular to the plane of the material surface, until its point touches but does not penetrate the underlying surface. Lower the disc gently down the rod until it rests under its own mass at constant level on top of the material, and is not pushed into the surface. Clamp the disc in this position and remove the measuring instrument. Measure the penetrated exposed length of the rod to 1 mm with a steel scale.

NOTE — A suitable deduction shall be made for the thickness of any knot, tie, washer or facing in the material.

7.4 Report

The mean of the nine determinations shall be taken as the representative value of the thickness of the test sample material.

8 DETERMINATION OF RESISTANCE TO COMPRESSION

8.0 Principle

Resistance to compression is determined by finding the dimension in thickness on the application of a specified pressure. Separate methods are followed for pressure up to or exceeding 6.9 kPa (0.07 kg/cm²).

8.1 Number of Tests

Carry out the determination on three test specimens cut from the same test sample material.

8.2 Conditioning

It is desirable that the test specimens be conditioned in an atmosphere of $27 \pm 2^\circ\text{C}$ and 65 ± 5 percent relative humidity (*see* IS 196) to constant mass.

NOTE — Variation due to the conditioning procedure are not normally significant, but where the test conditions are different from the above, these should be stated.

8.3 Test Specimens

8.3.1 The test specimens shall be square with sides at least 10 cm and not less than twice the thickness of the specimen. Each specimen should be cut in such a manner as to preserve as many of the original surfaces as possible and only one specimen shall be cut from a single sample. If the outer surfaces of the sample are appreciably harder than the inner portion, for example, when the sample has outer harder finish, the hard surface shall be retained in the specimen but not more than two such hard surfaces shall be included in any one of the test specimens, for example, by the use of multiple layers, unless a note to this effect is included in the report. The surface to which load is applied shall be substantially plane and parallel. Wherever practicable, this may be achieved by grinding the surfaces of the specimen, provided such treatment does not weaken or damage the essential surfaces.

NOTE — It may not be possible to obtain specimens suitable for this test from pipe sections in which the outer surfaces are appreciably harder than the inner portion.

8.3.2 The test specimen shall be tested at the nominal thickness specified by the manufacturer. If the nominal thickness specified by the manufacturer is less than 25 mm, two or more layers shall be superimposed to produce nominal thickness of 25 mm or greater. If, as a result of the resilient nature of the material, the thickness as supplied by the manufacturer is significantly greater than the nominal thickness, a preliminary load may be applied prior to application of the test load so that the thickness at the commencement of the test is equal to the nominal thickness or to an integral multiple thereof. The surface to which the load is to be applied shall be substantially plane and parallel and as far as practicable only original surfaces shall be in contact with the platens.

8.3.3 The area under test shall be assumed to be the average of the areas of the top and the bottom faces of the specimen or specimens when determined at the nominal thickness specified by the manufacturer. The apparent density in kg/m^3 at the nominal thickness and at thickness after application of the specified load shall also be determined.

8.4 Test at Pressure not Exceeding 6.9 kPa (0.07 kg/cm²)

8.4.1 Apparatus

A flat horizontal surface, a flat metal plate (platen) and appropriate weights. The platen shall be square with sides at least 5 cm greater than those of the specimen to be tested.

8.4.2 Procedure

Place the specimen centrally in the platen. If the specimen has any hard outer surface, it shall be placed parallel to the plane of the platen. Place the load as specified in the material specification carefully on the platen over the centre of the specimen and maintain it for 5 minutes before the

reduction in thickness is ascertained. The test load, additional to that required to produce the nominal thickness shall be such that the required pressure is applied within the tolerance of ± 1 percent. The reduction in thickness shall be derived from the mean of the four measurements taken near the vertical edges of the specimen.

8.5 Test at Pressures Greater than 6.9 kPa (0.07 kg/cm²)

8.5.1 Apparatus

A hydraulic or mechanical compression testing machine of appropriate size, compression rate and sensitivity. The platen shall be of such dimensions as to completely cover the specimen during the test and to enable measurements to be made. The platen shall be mounted on a self-aligning bearing to ensure axial loading.

8.5.2 Procedure

Place the specimen centrally in the platen. Compress the specimen between plane and parallel surfaces provided by the platen and the bearing block. The rate of compression shall be not more than 1.25 mm/min. Apply the minimum pressure to the specimen for which data are required and measure the thickness after the pressure has been maintained for 5 minutes. Increase the pressure in convenient steps until the maximum desired pressure is reached or until the sample fails. The reduction in thickness shall be derived from the mean of four measurements taken near the vertical edges of the specimens.

8.6 Calculation

$$\text{Reduction in thickness, percent} = \frac{a - b}{a} \times 100$$

where

a = initial thickness of the specimen, and

b = thickness under specified load or pressure.

8.7 Report

The mean of the three results shall be reported as the percentage reduction in thickness at given loads. The statement of results shall include the details of conditioning, initial thickness of the specimen and thickness after specified load and apparent density at the nominal thickness and the applied test pressure.

9 DETERMINATION OF BULK DENSITY

9.0 Principle

The bulk density of the material is determined by weighing the test specimen whose dimensions are already measured. The bulk density of loose fill material is, however, not uniquely defined as it will depend on the degree of compression while filling a container of known dimensions. As such for loose fill materials, bulk density should be determined under specified load as specified in **10.2**.

9.1 Number of Tests

Carry out three determinations on the test specimen cut from the same test sample material.

9.2 Test Specimens

The test specimens shall be of the same size as used for thickness determination (*see* 7.2 and 7.3.1).

9.3 Procedure

Measure its length and width by the method prescribed in 6. Determine its gross mass to an accuracy of ± 0.5 percent of the sample mass.

9.4 Calculation

Calculate the mass of the confining medium, if any, along with its accessories and subtract the same from the gross mass to get the net mass of the specimen. Calculate the volume of the specimen from its width, length and nominal thickness. Determine the bulk density of the sample by dividing the net mass of the specimen by its calculated volume and express the results in kg/m^3 .

9.5 Report

The mean of the three determinations shall be taken as the representative value of the bulk density of the test sample material.

10 DETERMINATION OF BULK DENSITY UNDER SPECIFIED LOAD

10.0 Two methods are prescribed for determination of bulk density under specified load.

10.1 Bulk Density Under Specified Load for Felted Materials

10.1.1 Principle

Bulk density under specified load is determined by weighing the test specimen whose dimensions are measured after application of specified load.

10.1.2 Number of Tests

Carry out the determination on three test specimens cut from the same test sample material.

10.1.3 Procedure

Remove the confining medium, if any, from both sides of the test specimen by cutting (not pulling) stitches, if any. Determine its thickness on application of specified load in accordance with the procedure prescribed in 8. Measure the length and width of the specimen by the method prescribed in 6 and determine its mass to the nearest gram.

10.1.4 Calculation

Calculate the volume of the specimen from its length, width and thickness. Determine the density of the material by dividing the mass of the specimen by the calculated volume and express the results in kg/m^3 .

10.1.5 Report

The mean of the determinations on three specimens shall be taken as the representative value of the bulk density under specified load of the test sample material.

10.2 Bulk Density Under Specified Load for Loose Materials

10.2.1 Apparatus

10.2.1.1 Cylindrical vessel

A metallic cylindrical vessel (A) having a cross-sectional area of 100 cm² (112.8 mm dia) (see Fig. 1) and having a uniform wall thickness of 3 mm.

10.2.1.2 Plunger

A metallic plunger (B) having two pans of uni-form wall thickness, 3 mm and cross-sectional area of 98.5 cm² (112.0 mm dia) each fixed at the ends of a 305 mm long, 20 mm outer diameter, pipe/rod (see Fig. 1).

10.2.1.3 Lifting device

A lifting device (C) consisting of a pulley.

10.2.1.4 Metre scale

10.2.2 Procedure

Take 180 to 200 g of the loose insulation material. Fluff it out and place it in horizontal layers to fill the cylindrical vessel. Place the plunger gently on the surface of the loose wool by means of lifting device. Add mass of 0.2, 0.4, 0.7, up to 10 kg on the pan of the plunger and measure the height of the loose wool with a metre scale each time. Take the mean of four readings recorded in diagonally opposite directions. Wait for exactly 1 minute (measure with a stop-watch) after adding the mass and then measure the height of the loose wool. Calculate the bulk densities and loads and draw a graph with bulk density as ordinate and loads as abscissa. From the graph, read the values of bulk densities for 0.01, 0.02, etc, kg/cm² loads.

10.2.3 Calculation

$$10.2.3.1 \text{ Bulk density (in kg/m}^3 \text{)} = \frac{M \times 100}{h}$$

where,

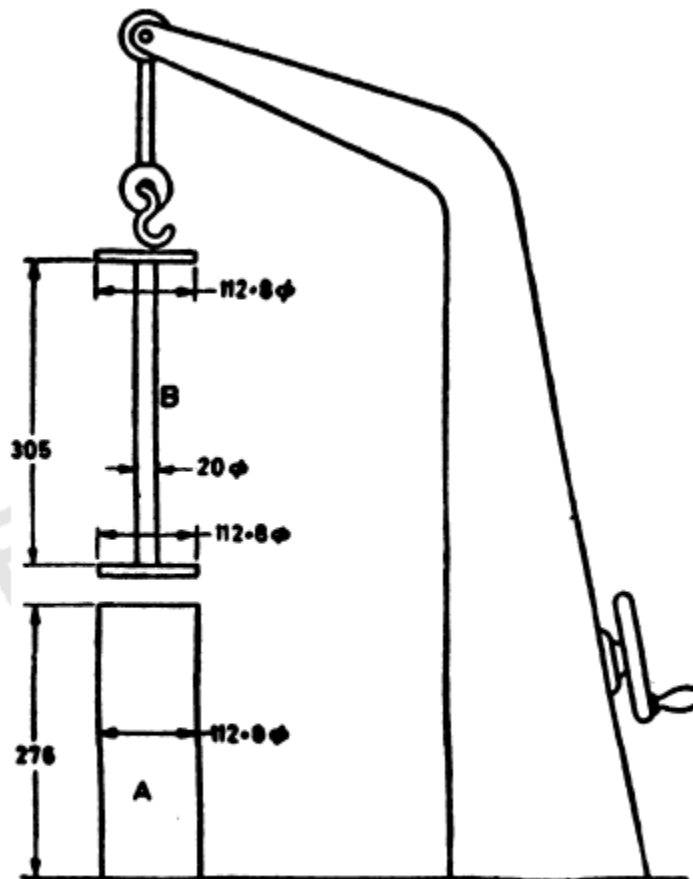
M = mass, in g, of loose wool, and

h = height, in mm, of the loose wool after adding each weight.

$$10.2.3.2 \text{ Pressure or load (in kg/cm}^2 \text{)} = \frac{K}{100}$$

where

K = mass in kg of the plunger + mass in kg added.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR APPARENT DENSITY
UNDER SPECIFIED LOAD

11 TEST FOR THERMAL CONDUCTIVITY

11.1 The method of determining thermal conductivity shall be normally as given in IS 3346.

However, for routine testing, secondary appropriate methods of determination of thermal conductivity such as described in IS 9489 and IS 9490 may be used. In case of disputes, IS 3346 shall apply.

11.2 Further, for conformance purposes, it would be sufficient to have representative product type tests for thermal conductivity using the two-slab guarded hot-plate method (*see* IS 3346) on appropriate specimens corresponding to product and density of the material supplied by a plant, one per twelve months, or as agreed to between the supplier and the purchaser or whenever significant product change occurs, to ensure that conformity with the requirements of the specification is maintained. The composition may be indicated by fibre diameter, shot content, binder content, etc.

12 TEST FOR MAXIMUM RECOMMENDED TEMPERATURE

12.0 Principle

The test is to determine the changes in thickness, cracking, delamination, incidence of fusion or flaming and also of internal self-heating, and if necessary, changes in resistance to compression and dimensions, after heating of the test specimens for the specified period, at the maximum recommended temperature of use. For conformance purposes, it would be sufficient to have representative product type testing and may not necessitate the fulfilment of the sample number requirements, of conformity control prescribed in the relevant material specifications.

12.1 Number of Tests

The tests shall be carried out on two test specimens cut at random from different portions of the material.

12.2 Conditioning

The test specimens shall be dried to constant mass either in a desiccator or in a ventilated oven.

12.3 Apparatus

12.3.1 The test apparatus shall consist of either:

- a) a hot plate not less than 300 mm × 300 mm for test materials of thickness not exceeding 100 mm and a hot plate not less than 450 × 450 mm for test materials of thickness exceeding 100 mm, and a larger plate for test materials exceeding thickness of 300 mm. The hot plate should be positioned horizontally and may be heated by electricity or gas; or
- b) a pipe mandrel of 75 mm outer diameter and about 1.25 m long equipped for internal heating by gas or electric current, and should be positioned horizontally.

At least five thermocouples shall be mounted on the surface of the heated plate or pipe. One thermocouple shall be fitted inside the test specimen centrally at mid-height to determine internal self-heating.

12.3.2 In the absence of a special apparatus for pipe section, this test may be conducted with an equivalent flat slab of same bulk density and fibre structure as that of the pipe section.

12.4 Test Specimens

The test specimens shall be of the specified thickness and of sufficient area so as to cover the hot plate or pipe mandrel.

12.5 Procedure

12.5.1 Determine accurately the mass of the dried specimen and measure its length, width and thickness in accordance with the procedures given in 6 and 7. Place the test specimen on the plate or fix it on the pipe mandrel. The edges of the test specimen should be surrounded by the same material of identical thickness having a minimum width of 75 mm so as to prevent edge heat losses. Heat the plate or pipe to the test temperature which shall be the same as that claimed by the manufacturer as the maximum temperature for use of the material under test. Pre-heat the hot plate to the test temperature insulated with a dummy specimen. When the test temperature has been reached and stabilized within ±2 percent, the dummy specimen should be quickly replaced by the

test specimen. Maintain the temperature within 5 percent or $\pm 10^{\circ}\text{C}$, whichever is less, until the temperature at the mid height of the test specimen reaches the maximum value and do not vary by more than 2 percent of the hot face temperature in one hour or for 16 hours whichever is larger. At the completion of the above test period, remove the specimen and keep it on a steel plate allowing it to cool to the room temperature. Weigh it to an accuracy of ± 0.5 percent of the sample mass. Determine its length, width and thickness and note its physical state.

12.5.2 The specimen shall be subjected to resistance to compression test prescribed in **8**, if required.

12.6 Report

The report shall include:

- a) the type, designation and grade of material;
- b) the bulk density of material at the start of test;
- c) average thickness before and after testing;
- d) the temperature of the hot face during test;
- e) details of any physical change in the material, for example, cracking, fusion, delamination, etc;
- f) the maximum temperature reached at the insulation mid height during the test (exceeding hot plate temperature, indicating unacceptable self-internal heating);
- g) the percentage change in length, width and mass; and
- h) The details of the results of the compression test on the specimen before and after heating, if required.

13 DETERMINATION OF SHOT CONTENT

13.0 Principle

The shots are solid particles of fusible rock, slag or glass which have not been elongated during manufacture of mineral wool fibres from the fused mass and are heavier than the mineral wool fibres. The lighter mineral wool fibres are carried away by a stream of water leaving behind the heavier particles (shots) in the elutriator.

13.1 Number of Tests

Carry out the determination for three samples cut and random from different portions of the material.

13.2 Apparatus

13.2.1 An elutriator made of glass on the general principles as shown in Fig. 2 shall be used.

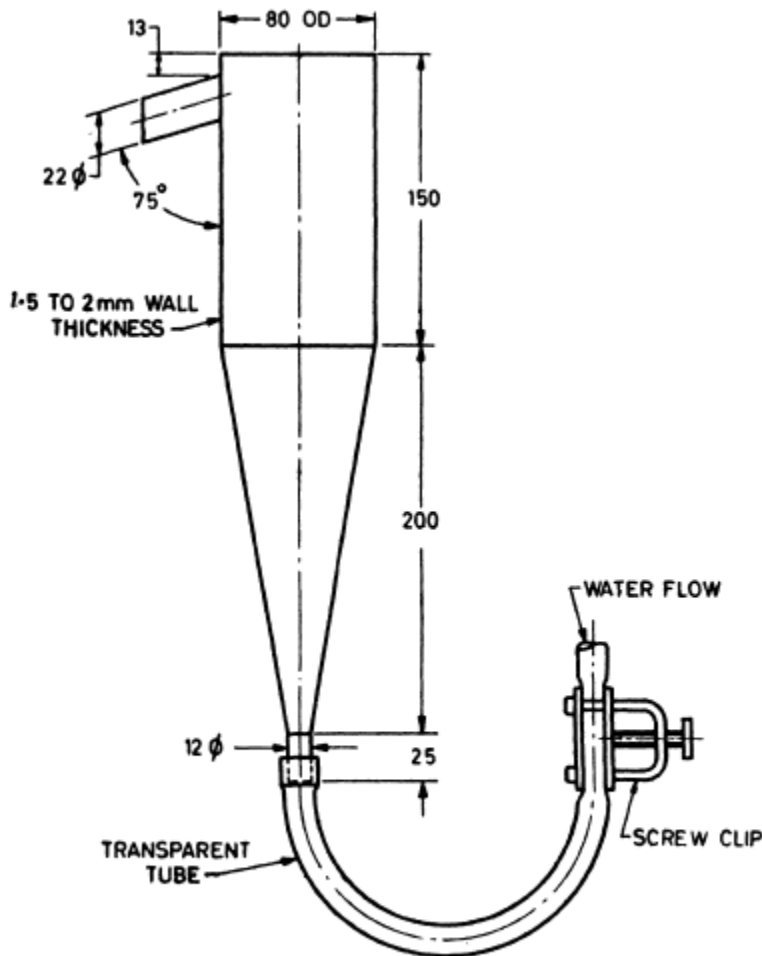
13.2.2 Muffle furnace (Temperature controlled)

13.2.3 Laboratory balance (accurate to $\pm 1\text{mg}$)

13.2.4 Sieves- Three sieves conforming to IS 460 (Part 1), 150 micron IS sieves, 250 micron IS sieves and 500 micron IS sieves

13.3 Procedure

13.3.1 Remove the confining medium, if any, from the three test specimens. Cut approximately 10 g of the material from each of the three specimens and mix these into one composite sample, while taking all the precautions that none of the shots fall during this process, and weigh accurately to the nearest milligram. Cut the sample into thin slices using a knife. Heat it in a furnace maintained at $550^\circ \pm 10^\circ\text{C}$ for 1 hour for removal of organic binder. If the material shows a tendency to fuse or melt at this temperature, a lower temperature shall be used. Place the material in a beaker, add water and a few drops of wetting agent and disperse the fibres thoroughly in water by stirring.



All dimensions in millimetres.

FIG. 2 ELUTRIATOR FOR THE DETERMINATION OF SHOT CONTENT

13.3.2 Transfer the material into a glass elutriator. Regulate the water flow (*see* Note) through the bottom inlet of the elutriator at the rate of approximately one liter per minute. A glass stirring rod shall be used to prevent the aggregation of fibres in the elutriator and the solid material carried over at the top outlet shall be collected on a 150 micron IS Sieve [conforming to IS 460 (Part 1)]. As elutriation proceeds, increase the water flow up to a maximum value of approximately three liters per minute. Examine the fibres collected from the outlet of the elutriator to make sure that it is free from shots. If it is not, either it shall be returned for re-separation or the test recommended with a fresh sample. When the water is clear of fibres, close the screw clip and allow the shots to settle in the bottom of the elutriator tube. Remove the tube from the elutriator and transfer completely the shots on to 150 micron IS Sieve. Cover the shots collected on the sieve with water and agitate thoroughly by using a spatula to ensure that undersize material is not retained. Transfer the material retained on the sieve to a small beaker. Dry it in an oven maintained at $105 \pm 2^\circ\text{C}$ for 2 hours. Cool in a desiccator and weigh. Repeat this operation till a constant mass is obtained to the nearest milligram.

NOTE — A detergent may be added to water and/or hot water may be used to accelerate the process, if necessary.

13.3.3 Transfer the dried material from the beaker to an assembly of 250 micron IS Sieve on top and 150 micron IS Sieve at the bottom both conforming to IS 460 (Part 1). Sieving operation is then done by pressing the shots against the top sieve surface using the flat face of a suitable rubber stopper. Continue the operation until all the finer shots have passed through the top sieve. Transfer the shot particles retained on 250 micron IS Sieve and on 150 micron IS Sieve separately to weighing dishes using bristles brush and weigh each accurately to the nearest milligram.

13.3.4 Transfer the shot particles retained on 250 micron IS sieve as obtained in **13.3.3** to an assembly of 500 micron IS Sieve on top and 250 micron IS Sieve at the bottom both conforming to IS 460 (Part 1). Sieving operation is then done by pressing the shots against the top sieve surface using the flat face of a suitable rubber stopper. Continue the operation until all the finer shots have passed through the top sieve. Transfer the shot particles retained on 500 micron IS Sieve and on 250 micron IS Sieve separately to weighing dishes using bristles brush and weigh each accurately to the nearest milligram.

13.4 Calculation

a) Shot content retained on

$$500 \text{ micron sieve, percent by mass} = \frac{M_1}{M} \times 100$$

b) Shot content retained on

$$250 \text{ micron sieve, percent by mass} = \frac{M_2}{M} \times 100$$

c) Shot content retained on

$$150 \text{ micron sieve, percent by mass} = \frac{M_3}{M} \times 100$$

where

M = mass in g of the material taken initially for the test after removing the confining medium, if any;

M_1 = mass in g of the shots retained on 500 micron sieve;

M_2 = mass in g of the shots retained on 250 micron sieve as per 13.3.4; and

M_3 = mass in g of the shots retained on 150 micron sieve.

13.5 Report

This shall be taken as the representative value of the shot content present in the test sample material.

14 DETERMINATION OF MOISTURE CONTENT ON AS RECEIVED BASIS

14.0 Principle

The moisture content is determined by heating the sample at $105 \pm 2^\circ\text{C}$ and finding the loss in mass between initial and final mass of the sample.

14.1 Number of Tests

Carry out the determination of three specimens cut from different portions of the material.

14.2 Apparatus

The drying unit shall be a thermostat fitted drying oven with a suitable vent for escape of moisture. The material shall be kept on a chicken gauze, protected from direct radiations of the heater and kept in such a way that no part of the material is above 110°C .

14.3 Procedure

Cut a sample weighing not less than 25 g from the material, as received and weigh accurately. Dry this material in an oven maintained at a temperature of $105 \pm 2^\circ\text{C}$ till a constant mass is obtained.

14.4 Calculation

$$\text{Moisture content percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where,

M_1 = mass in g of the material taken for test, and

M_2 = mass in g of the material after heating in the oven.

14.5 Report

The mean of results on the three specimens shall be taken as the representative value of the moisture content on as received basis. If moisture content is less than 0.5 percent by mass, it shall be reported as nil.

15 DETERMINATION OF MOISTURE ABSORPTION

15.0 Principle

The moisture absorption is determined by suspending the sample in a humidity chamber and finding the increase in mass between the initial and final mass of the sample.

NOTE — For conformance purposes, it would be sufficient to have representative product type testing and may not necessitate the fulfilment of the sample number requirement of the conformity control prescribed in the relevant material specification.

15.1 Number of Tests

Carry out the determination on three specimens cut from different portions of the material.

15.2 Apparatus

15.2.1 *Drying Unit* — See 14.2.

15.2.2 *Humidity Chamber* — Thermostatically controlled.

15.3 Procedure

Cut a sample of at least 20 × 20 cm from the material and weigh accurately. Dry this material to a constant mass in an oven maintained at 105 ± 2°C. Suspend the specimen in a humidity chamber maintained at a temperature of 50 ± 2°C and 90 ± 3 percent relative humidity for 96 hours. The specimen shall be protected from the condensate dripping from the humidity chamber ceiling by a slanting false roof immediately above the specimen. Remove the specimen from the humidity chamber and reweigh immediately.

15.4 Calculation

Moisture absorption

$$\text{percent by mass} = \frac{M_2 - M_1}{M_1} \times 100$$

where,

M_2 = mass in g of the material after keeping it in the humidity chamber; and

M_1 = mass in g of the material taken for the test.

15.5 Report

The mean of three determinations shall be taken as the representative value of moisture absorption of the test sample of the material.

16 INCOMBUSTIBILITY TEST

16.0 Principle

The test specimen is lowered into a suitable furnace maintained at 750°C and the escaping gases are tested with a lighted pilot gas flame for the tendency to burn. The object of this test is to find out the incombustibility of the material and of the evolved gases.

16.1 Number of Tests

Carry out the determination of three test specimens cut from different portions of the material.

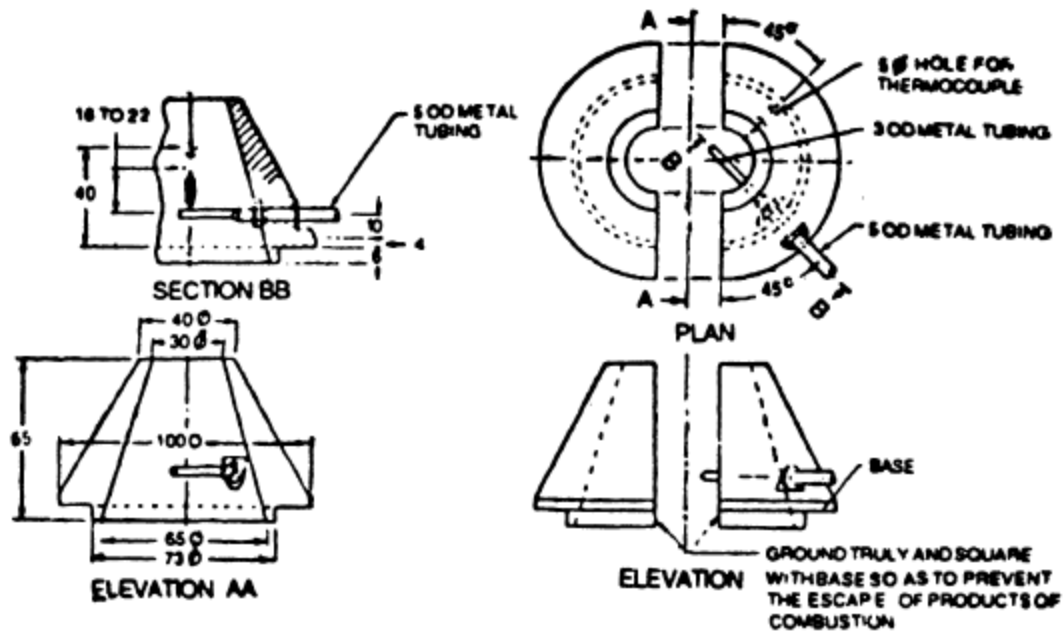
16.2 Test Specimens

The test specimens shall be $50 \times 40 \times 40$ mm in size. Specimens of materials which are normally less than 40 mm thick shall be made of sufficient layers to achieve the final thickness as near as possible but not exceeding 40 mm.

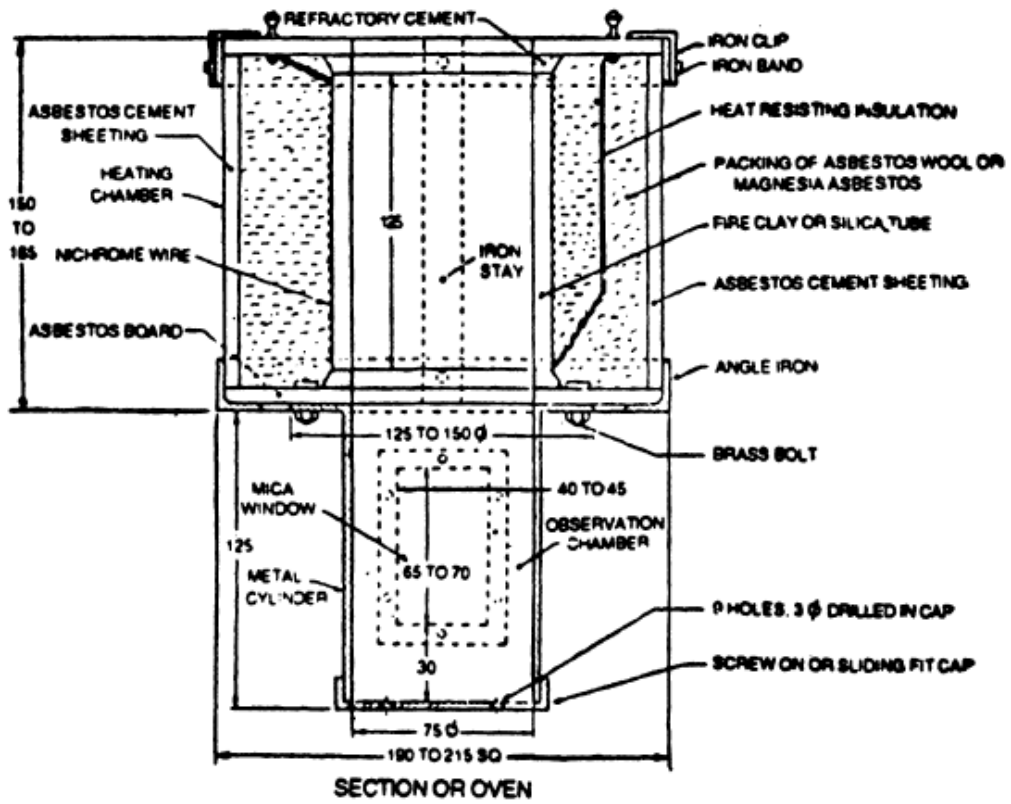
16.3 Apparatus

The specimen shall be tested in the heating tube of an apparatus of the general type, shown in Fig. 3, the pilot flame being located 20 mm above the upper end of the specimen. A support for the specimen shall be provided in the heating tube and this may suitably consist of a light stirrup of nichrome wire, supported by a length of nichrome wire passing over the pilot flame tube. The support shall be such that the specimen is fixed centrally in the heating tube. The apparatus shall be heated by passing a suitably regulated electric current through a nichrome resistance wire surrounding the heating tube.

NOTE — The apparatus may be insulated further on the outside, if necessary, for uniformity of temperature.

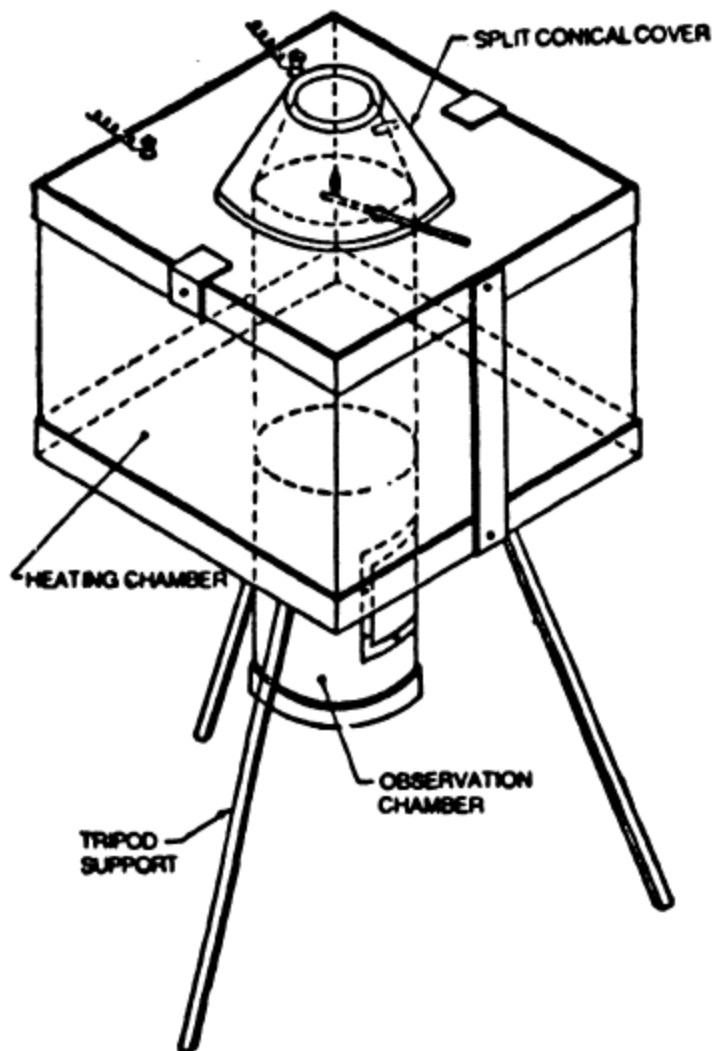


3A DETAILS OF CONICAL COVER
 (TO BE OF REFRACTORY MATERIAL)



SECTION OF OVEN
3B DETAILS OF TEST OVEN
All dimensions in millimetres.

FIG. 3 APPARATUS FOR INCOMBUSTIBILITY TEST — *Continued*



3C SKETCH OF ASSEMBLED APPARATUS
FIG. 3. APPARATUS FOR INCOMBUSTIBILITY TEST

16.4 Measurement of Temperature of Tube

Temperature of the tube shall be taken as shown by the thermocouple situated at the level of the centre of the specimen and equidistant from the inner surface of the tube and the specimen. The wires of which the thermocouple is made shall be not larger than 1.25 mm and not smaller than 0.462 mm in diameter and shall be bare for a length of 25 mm from the junction.

16.5 Procedure

16.5.1 Dry the test specimens in an oven maintained at $103 \pm 2^\circ\text{C}$ for 1 hour. Repeat this operation till the constant mass is obtained.

16.5.2 Raise the temperature of the tube to a constant temperature of $750 \pm 10^\circ\text{C}$ before insertion of the specimen. Insert the specimen centrally in the tube with its long axis vertical. The maximum time taken for inserting the specimen and bringing it in position shall be about 30 s. If the specimen is likely to disintegrate or melt during the test, it shall be supported in wire gauze or in a thin metal sheet box having a base of 40 mm square and height of 50 mm. During this period, a conical cover at the top shall limit the opening to approximately 6.5 cm^2 . Allow the specimen to remain there for a period of 15 minutes. During this period note the rise or fall of the temperature of the thermocouple. The gas coming through the opening shall be tested for combustibility by the lighted pilot flame. Then remove the test specimen from the tube, cool it to room temperature and weigh again to an accuracy of nearest milligram.

NOTE — If the material is combustible, the furnace temperature shall start rising and shall fall after sometime when the combustion is complete.

16.5.3 For the purpose of this standard, the material shall be considered incombustible, if during the test period none of three specimens of the sample flames or produces vapours which are ignited by pilot flame or causes the temperature of the furnace to be raised by 50°C or more above 750°C .

NOTE — Initial flame, for not more than 15 second, when the material is inserted in the apparatus, may be ignored.

16.6 Calculation

$$\text{Loss in mass, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where,

M_1 = total mass in g of all the test specimens before test; and

M_2 = total mass in g of all the test specimens after test.

16.7 Report

16.7.1 If any one of the three test specimens does not satisfy the test prescribed in **16.5.3**, the material shall be declared as combustible.

16.7.2 If all the test specimens satisfy this test, the material shall be considered incombustible. In this case, report the loss in mass as calculated in **16.6**.

16.7.3 The report should also contain observation made during the test; initial, maximum and final furnace temperature and any physical changes occurred.

17 DETERMINATION OF RESISTANCE TO MICRO-ORGANISMS

17.0 Applicability

This test is only relevant when the insulation is used at temperatures below 40°C and where it is expected to come in contact with food products. This is also relevant for applications in buildings and enclosures for use as human habitation.

17.1 Principle

The material is tested for mould and bacterial growth with *Aspergillus niger* and *Bacillus subtilis* culture respectively.

17.2 Preparation of the Sample

The sample shall be sterilized in a hot air-oven at 160°C for minimum one hour to get rid of any aerial contamination. Any initial microbial loading on the sample may give misleading results.

17.3 Apparatus and Materials

17.3.1 *Incubator* — accuracy $\pm 1^\circ\text{C}$, Temperature 25-35°C.

17.3.2 *Air-Oven* — capable of maintaining temperature at $160 \pm 2^\circ\text{C}$.

17.3.3 *Autoclave* — for sterilization of media.

17.3.4 *Analytical Balance* — accuracy ± 0.1 mg.

17.3.5 *Stereoscopic Microscope* — magnification $\times 50$.

17.3.6 *Petri Dish* (Glass or Plastic, Disposable).

17.3.7 *Test Organism* — *Aspergillus Niger* and *Bacillus Subtilis*.

17.3.8 *Glasswares* — Volumetric flask – 1 000 ml; Pipette : 1 ml, 5 ml, 10 ml; Test tube — 25 cm long, 3 cm diameter; Erlenmeyer flask: 250 ml 500 ml and 1 000 ml.

17.3.9 *Microbial Solutions*

17.3.9.1 *Ethanol water mixture* (70 : 30)

17.3.9.2 *O-Phenylphenol*

Dissolve 1 g *O-Phenylphenol* in 50 ml of 90 percent ethanol, make up to one litre with water and adjust pH to 3.5 by adding lactic acid dropwise.

17.3.10 *Soyabean Casein Digest Agar (SCDA)*

17.3.11 *Potato Dextrose Agar (PDA)*

NOTE — SCDA & PDA shall be prepared from dehydrated readymade powders as per the manufacturer's instruction.

17.3.12 *Sterile Saline*

Dissolve 0.85 percent NaCl in distilled water and autoclave at 121°C for 15 min.

17.4 Detection of Mould Growth

17.4.1 *Preparation of Inoculum*

Grow *Aspergillus niger* on potato dextrose agar for 5 - 7 days at 22 - 25°C. Harvest spores and transfer into sterile saline. Store stock spore suspension at 2-8°C and use within a week. Dilute stock spore suspension to prepare an inoculum of 10^8 spores per ml, at the time of use.

17.4.2 Procedure

Slip a long piece of material under test into a 25 cm long, 3 cm diameter, glass test tube containing such quantity of culture inoculum that nearly half of the sample is in the inoculum suspension and the rest above it. Incubate at 22 -25°C for 120 hours in a vertical position. Then remove the sample from the tube and check under a Steriomicroscope to detect any mycelial growth of *Aspergillus niger*. Both the portions dipped in the liquid as well as above it shall be examined for any mould growth.

17.5 Detection of Bacterial Growth

17.5.1 Preparation of Inoculum

Grow *Bacillus subtilis* on soyabean casein agar at 32 - 35°C for 120 hours. Harvest the growth into sterile saline. Store stock culture suspension in refrigerator and use within a week. At the time of use, dilute the stock to prepare an inoculum of 10^8 spores per ml.

17.5.2 Procedure

Slip a long piece of the material under test into a 25 cm long, 3 cm diameter glass test tube containing such a quantity of culture suspension that nearly half of the sample is in the solution and rest above it. Inoculate for 32 - 35°C for 120 hours. Then check the bacterial load of culture suspension. If cell concentration is less than or equal to that of control, it is indicated that sample is resistant to bacterial growth.

NOTE — A part of inoculum, which is not exposed to the mineral wool sample, serves as control.

18 ODOUR EMISSION TEST

18.0 Applicability

This test is only relevant to insulation application in domestic refrigerators including cold-storages, and in buildings and enclosures for use as human habitation.

18.1 Principle

This test is carried out by placing butter on a piece of the material in a refrigerator for 24 hours and then examining it for any change in odour against a blank.

18.2 Test Specimens

Three test specimens of the material of given thickness shall be cut to a size approximately 16 cm wide and 25 cm long.

18.3 Apparatus and Materials

18.3.1 Refrigerator — see IS 1476.

18.3.2 Pans

Six porcelain enamelled refrigerator pans having dimensions approximately 15 cm wide, 25 cm long and 10 cm deep, with snug fitting lids.

18.3.3 *Glass Squares*

Six 50 mm square pieces of glass — (*see* IS 2835).

18.3.4 *Butter* — Unsalted, sweet cream butter, less than 72 hours old, cut into pieces of 25 mm square and 5 mm thick.

NOTE — *Care of the Apparatus* — All equipment shall be kept odour-free. Before each test, the pans, the glass squares, and the knife with which the butter is cut shall be sterilized. The refrigerator shall be free from any odour and in good operating condition at all times.

18.4 Procedure

Place the six pieces of butter on the six sterilized glass squares. Place these pieces of glass with butter on top of each of the insulation specimens and keep each assembly in a separate enamelled pan. Place the other three pieces of butter in separate enamelled pans without insulation specimens to serve as blank during the test. Place the lids and put all the pans in a refrigerator. Operate the refrigerator at normal temperature (about 8°C) for 24 hours and remove the pans from the refrigerator. Remove the lids and note any odour emitted from the pans containing an insulation specimen while comparing it against the blank.

18.5 Report

Report the observations of the odour test after comparing with the blank as follows:

- a) Number of specimens with no apparent difference in odour,
- b) Number of specimens with slight odour, and
- c) Number of specimens with strong or objectionable odour.

19 DETERMINATION OF SULPHUR

19.0 Principle

Sulphuric acid is added to the sample and the hydrogen sulphide evolved is absorbed in cadmium acetate solution. The precipitated cadmium sulphide is decomposed with hydrochloric acid, and a known volume of iodine solution is added and the excess is titrated against standard sodium thiosulphate solution.

19.1 Number of Tests

Carry out the determination in duplicate.

19.2 Apparatus

19.2.1 *Gas Absorption Flasks*

Three round bottom flasks of 50 ml capacity connected in series, each containing about 20 ml of cadmium acetate solution, having standard ground glass joints.

19.2.2 *Flask*

250 ml capacity fitted with a dropping funnel, having standard ground glass joints.

19.3 Reagents

19.3.1 Sulphuric Acid Solution — approximately 2 N.

19.3.2 Cadmium Acetate Solution — approximately 2 N.

19.3.3 Concentrated Hydrochloric Acid — see IS 265.

19.3.4 Standard Iodine Solution — 0.1 N. See **49** of IS 2316.

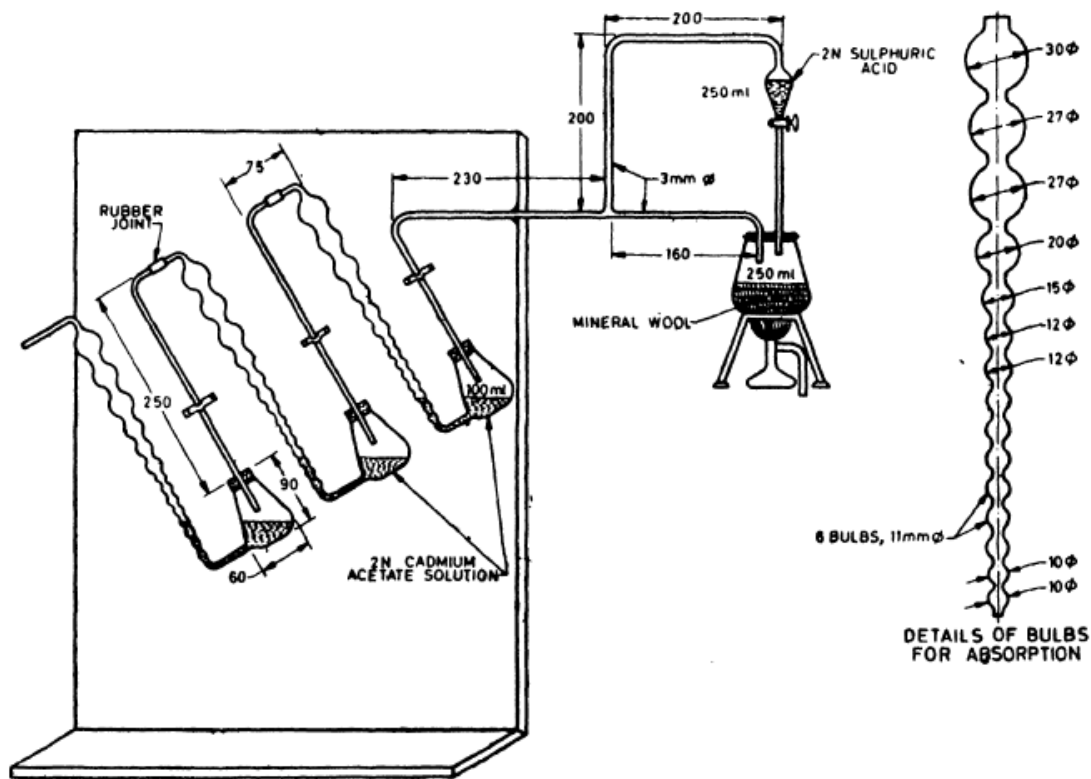
19.3.5 Standard Sodium Thiosulphate Solution — 0.1 N. See **55** of IS 2316.

19.3.6 Starch Solution

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml distilled water at room temperature and slowly pour it with stirring into 1 litre of boiling water. Boil for 3 minutes. Allow to cool and decant off the supernatant clear liquid (see IS 2263).

19.4 Procedure

19.4.1 Weigh accurately about 5 g of the material in a 250 ml flask, and assemble the apparatus as shown in Fig. 4. Add 10 ml of sulphuric acid solution through a dropping funnel and heat the flask over a low flame for 1 to 2 hours so that all the hydrogen sulphide evolved is absorbed in the cadmium acetate solution.



All dimensions in millimetres.

FIG. 4 APPARATUS FOR SULPHUR DETERMINATION

19.4.2 Disconnect the three gas absorption flasks and add 5 ml of hydrochloric acid followed by exactly 20 ml of iodine solution to each. Shake the flask and collect the solution from all the flasks in a 250 ml iodine flask. Rinse twice all absorption flasks with distilled water and add this to the 250 ml conical flask. Titrate the solution against the standard sodium thiosulphate solution using starch solution as an indicator.

19.4.3 Carry out a blank determination with 60 ml of iodine solution in iodine flask.

19.5 Calculation

$$\text{Sulphur content, percent by mass} = \frac{0.16 (V_2 - V_1)}{M}$$

where,

V_2 = volume, in ml, of standard sodium thiosulphate solution used for the blank titration;

V_1 = volume, in ml, of standard sodium thiosulphate solution used for the titration of the test solution; and

M = mass, in g, of the material taken for the test.

19.6 Report

The mean of two determinations shall be taken as the representative value of the sulphur content present in the material.

20 DETERMINATION OF OIL

20.0 Applicability

This test is only relevant to insulation applications where liquid or gaseous elemental oxygen is expected to come in direct contact with the insulation material.

20.1 Principle

The oil present in the sample is extracted with carbon tetrachloride.

20.2 Number of Tests

Carry out the determination in duplicate.

20.3 Apparatus

20.3.1 Soxhlet Extraction Apparatus

20.4 Reagent

20.4.1 Carbon Tetrachloride — see IS 718.

20.5 Procedure

20.5.1 Dry the extraction thimble for 2 hours at $105 \pm 2^\circ\text{C}$ and cool it in a desiccator and weigh. Place 5 to 10 g of the material in the extraction thimble and weigh again. Place it in the siphon of the extraction apparatus. Fit the condenser, siphon tube and flask together and add 100 ml of carbon tetrachloride. Regulate the heating so that the siphoning takes place at the rate of 2 to 3 times per hour. Continue the extraction for 2 hours. At the end of this time, remove the flask and filter the contents of the flask into a dish or into a smaller flask which has been weighed to the nearest milligram. Wash the flask from which the contents have been poured with 20 ml of carbon tetrachloride and add the washings to the extract. Evaporate the carbon tetrachloride by first keeping it over a low flame and then over a sand bath maintained at about 70°C . Place the dish or flask containing the residue in a well-ventilated oven at $70 \pm 2^\circ\text{C}$ for 3 hours. Cool in a desiccator and weigh. Repeat the process of heating and cooling until the mass is constant to within 3 milligrams.

20.5.2 Evaporate 120 ml of carbon tetrachloride as prescribed in **20.4.1** to determine the amount of oil present, if any, in carbon tetrachloride initially.

20.6 Calculation

Oil content,

$$\text{percent by mass} = \frac{100 \times (M_1 - M_2)}{M}$$

where,

M_1 = mass in g of the residue obtained after extraction,

M_2 = mass in g of the residue obtained after evaporating 120 ml of carbon tetrachloride, and;

M = mass in g of the material taken for the test.

20.7 Report

The mean of two determinations shall be taken as the representative value of the oil content present in the material.

21 DETERMINATION OF CARBON (TOTAL)

21.0 General

This test is only relevant to insulation applications where liquid or gaseous elemental oxygen is expected to come in direct contact with the insulation material. Two methods have been prescribed, namely, the volumetric method and direct method. However, in case of dispute, the direct method shall be the referee method.

21.1 Volumetric Method

21.1.0 Principle

The carbon dioxide evolved by oxidation of the materials is absorbed in barium hydroxide solution and the resulting solution is titrated against standard hydrochloric acid solution.

21.1.1 Number of Tests

Carry out the determination in duplicate.

21.1.2 Apparatus

21.1.2.1 Porcelain or silica boat

21.1.2.2 Combustion tube

Open at both ends and suitable to accommodate porcelain or silica boat.

21.1.2.3 Electric furnace

To accommodate the combustion tube and to give temperature up to at least 900°C.

21.1.2.4 Absorption assembly

Three flasks connected in series, each containing 20 ml of 0.1 N barium hydroxide solution.

21.1.3 Reagents

21.1.3.1 Potassium bichromate — see IS 250.

21.1.3.2 Copper oxide — in the form of a wire.

21.1.3.3 Lead chromate — granulated and fused.

21.1.3.4 Oxygen gas — see IS 309.

21.1.3.5 Standard barium hydroxide — 0.1 N. Prepared by dissolving barium hydroxide in distilled water in an atmosphere of nitrogen.

21.1.3.6 *Standard hydrochloric acid solution* — 0.1 N.

21.1.3.7 *Phenolphthalein indicator* — 0.1 g in 100 ml of 60 percent rectified spirit (*see* IS 323).

21.1.4 *Procedure*

21.1.4.1 Weigh accurately about 1.5 g of the material in a porcelain or silica boat and mix thoroughly with 10 g of potassium bichromate. Place this boat inside the combustion tube in which 15 cm column of lead chromate and 20 cm column of copper oxide are placed towards the exit end. Insert a stopper carrying a thermocouple for measuring temperature and a tube for the admission of oxygen into the inlet of the combustion tube. The thermocouple shall be so adjusted that it is in contact with the boat.

21.1.4.2 Pass oxygen gas through the combustion tube at the rate of 15 ml/min and maintain the same rate of flow during the experiment. Place the combustion tube in the electric furnace and connect its outlet to the absorption assembly containing barium hydroxide solution. Heat the furnace to 800-850°C and maintain this temperature for 1 hour. Allow the carbon dioxide evolved to get absorbed in the flasks containing standard barium hydroxide solution.

21.1.4.3 Collect the contents of the absorption assembly in a conical flask and titrate against standard hydrochloric acid using phenolphthalein as an indicator while taking the usual precautions against absorption of atmospheric carbon dioxide.

21.1.4.4 Carry out the blank titration with 60 ml of 0.1 N barium hydroxide solution.

21.1.5 *Calculation*

Carbon content,

$$\text{percent by mass} = \frac{0.06 (V_2 - V_1)}{M}$$

where,

V_2 = volume, in ml, of standard hydrochloric acid solution consumed in the blank titration,

V_1 = volume, in ml, of standard hydrochloric acid solution consumed in the titration of test solution, and

M = mass, in g, of the material taken for the test.

21.1.6 *Report*

The mean of two determinations shall be taken as the representative value of carbon content present in the material.

21.2 *Direct Method*

21.2.1 *Principle*

Direct determination of carbon can be carried out using carbon determination apparatus as shown in Fig. 5.

21.2.2 *Procedure*

The evolved gases by oxidation of the material are passed through a cooling condenser and collected in a burette, directly calibrated in terms of carbon percentage (19). After collection is complete, the evolved gases are led to the absorption vessel (21) containing strong solution of KOH by the use of the three-way valve (18) to absorb all the CO₂ in the gas. The reduction in the volume registered in the calibrated burette due to this carbon dioxide absorption is measured and the percentage of carbon content in the sample is determined by reading of the calibrated burette using the suitable conversion chart supplied with the instrument.

22 DETERMINATION OF SETTLING DUE TO VIBRATION AND JOLTING

22.0 Application

The jolting test is not relevant to stationary applications of the materials.

22.1 Principle

Methods are given for evaluating the settlement of thermal insulation materials under the two conditions given below:

- a) vibration at high frequency with small amplitude, and
- b) jolting at low frequency with large amplitude.

These have been used mainly for assessing the behaviour of mineral wool and granular products for transport service and may not be necessary for other purposes.

For conformance purposes, it would be sufficient to have representative product type testing and may not necessitate the fulfilment of the sample number requirements of conformity control prescribed in the relevant material specifications.

22.2 Vibration at High Frequency and Small Amplitude

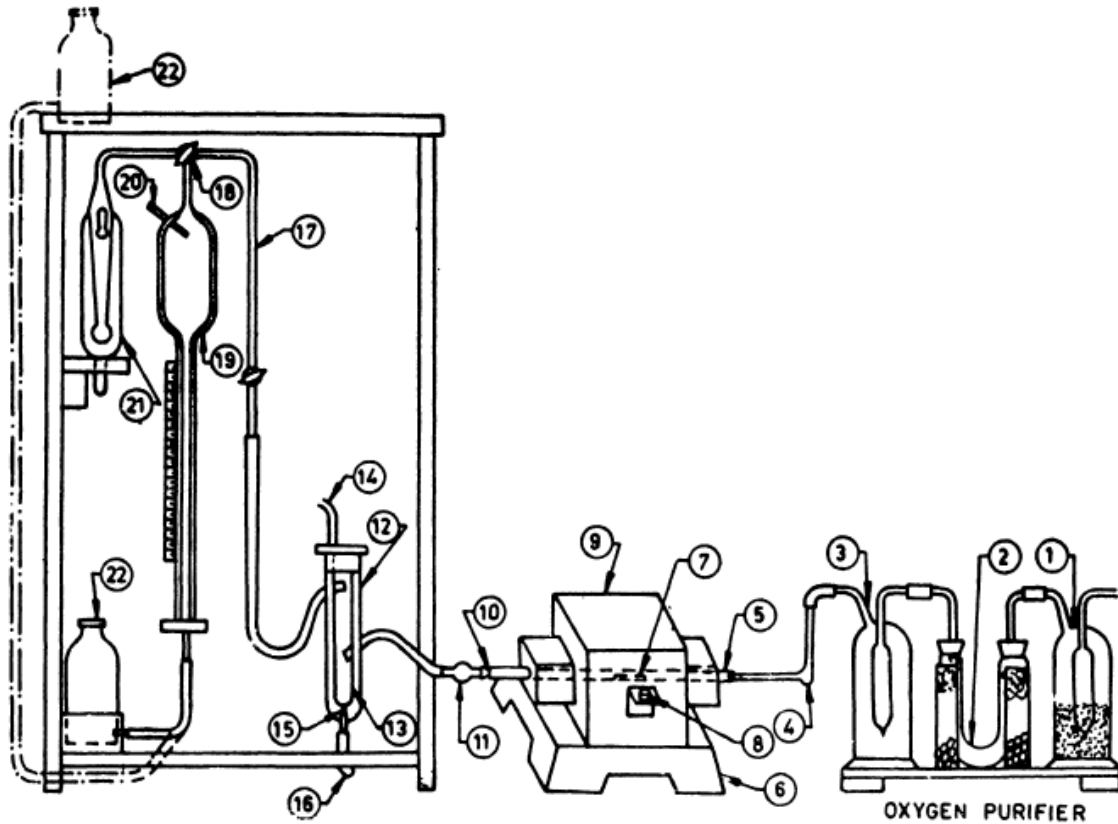
22.2.1 Apparatus

A wooden box with smooth interior surfaces, having internal dimensions of 900 mm height, 600 mm length, and 300 mm width, provided with a removable lid, shall be secured within a steel framework fixed to a steel base plate, which may be rigidly mounted on unsprung wheels to render the apparatus mobile, or it may be properly grouted or fixed on the floor. The box may be conveniently adapted for testing one or more specimens less than 300 mm thick by providing detachable vertical wooden partitions, 900 mm high by 600 mm long and 12 mm thick at required spacings having suitable fixing arrangements such as proper clamps, to provide uniform constant pressure on the vertical partition, pressing the test sample. Provision is made for vibrating the base plate in a vertical plane at a frequency of about 24 Hz. Figure 6 illustrates the typical form of the apparatus, with which an amplitude of approximately 1 mm about the mean position is obtained. A vertical strip of transparent plastic material may conveniently be inserted in one face of the box so that the progress of the test may be observed visually.

22.2.2 Procedure

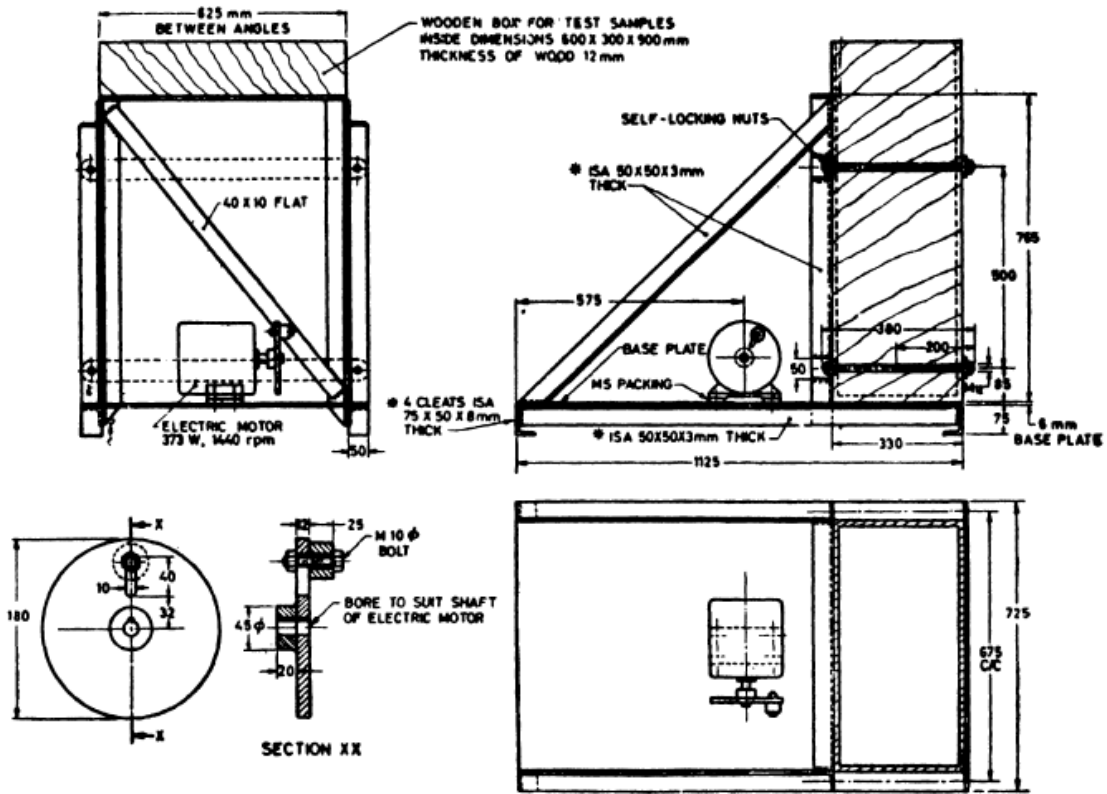
Place the test specimen in the box and adjust the partition to hold the specimen securely at the manufacturer's defined thickness. Determine the mean height of the upper surface of the specimen

in the box from three measurements made at points shown in Fig. 7. Fix the lid properly on the box, vibrate the box continuously for 16 h and then determine the mean height of the specimen after testing as described above.



NO.	NAME OF THE PART
1	GAS BUBBLER FOR KON
2	U-TUBE FOR CaCl ₂
3	GAS BUBBLER FOR H ₂ SO ₄
4	OBSERVATION TUBE
5	COMBUSTION TUBE
6	FURNACE
7	COMBUSTION BOAT
8	THERMOCOUPLE
9	ELECTRIC HEAT SHOCK PROOF INSULATOR
10	RUBBER CORK
11	GLASS FILTER
12	SPIRAL CONDENSER
13	WATER JACKET
14	WATER INLET TUBE
15	DRAIN STOP COCK
16	RUBBER DRAIN TUBE
17	2-WAY WITH U-BEND STOP COCK
18	3-WAY STOP COCK
19	CARBON BURETTE 4.5 PERCENT OR 1.5 PERCENT
20	THERMOMETER
21	ABSORPTION VESSEL
22	LEVELLING BOTTLE

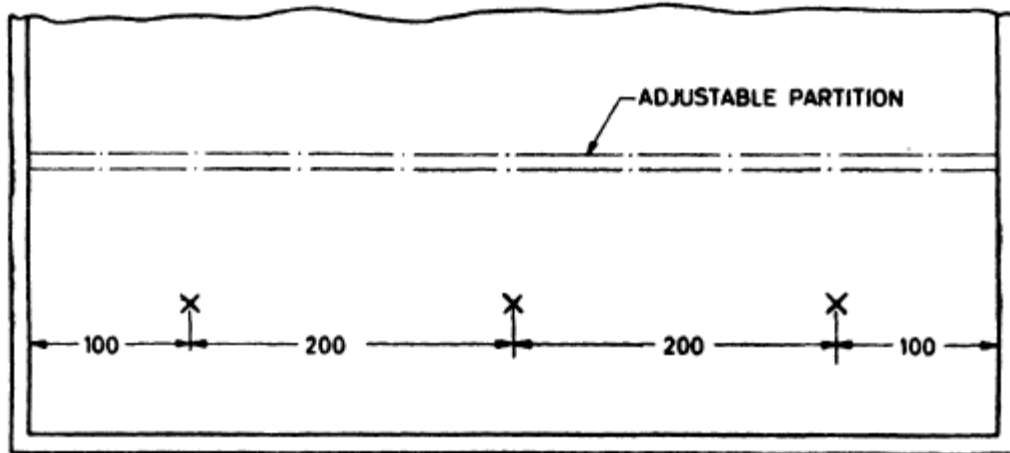
FIG. 5 CARBON DETERMINATION APPARATUS (WITHOUT CONTROL UNIT)



*IS 808 Dimensions for hot-rolled sections: (Part V) - 1976 Equal leg angles, and (Part VI) - 1976 Unequal leg angles.

All dimensions in millimetres.

FIG. 6 VIBRATION TESTER



All dimensions in millimetres.

FIG. 7 TOP VIEW OF VIBRATION BOX, POSITIONS FOR HEIGHT MEASUREMENTS

NOTE — One method of measuring the amplitude of vibration is by attaching a vibration scale, similar to that illustrated in Fig. 8, to the base plate near the centre and observing a point at which the sloping line appears to intersect the horizontal line when the machine is in operation. The scale reading at this point indicates twice the amplitude of vibration.

22.3 Jolting at Low Frequency and High Amplitude

22.3.1 Apparatus

A wooden box as described in 22.2.1 shall be secured within a steel framework, as shown in Fig. 9. Provision is made for raising the framework through a height of 25 mm and dropping vertically onto renewable steel blocks with a frequency of 1 Hz. Four guide pins are provided to assist in insuring vertical travel in both directions.

22.3.2 Procedure

Place the specimen of material in the box and determine the mean height before testing as prescribed in 22.2.2. Place the lid properly on the box, which shall then be jolted continuously for 16 h. Determine the mean height of the specimen after testing as prescribed in 22.2.2.

22.4 Report

22.4.1 Report the settlement of the material under test as a percentage of the initial height of the specimen in the box as follows:

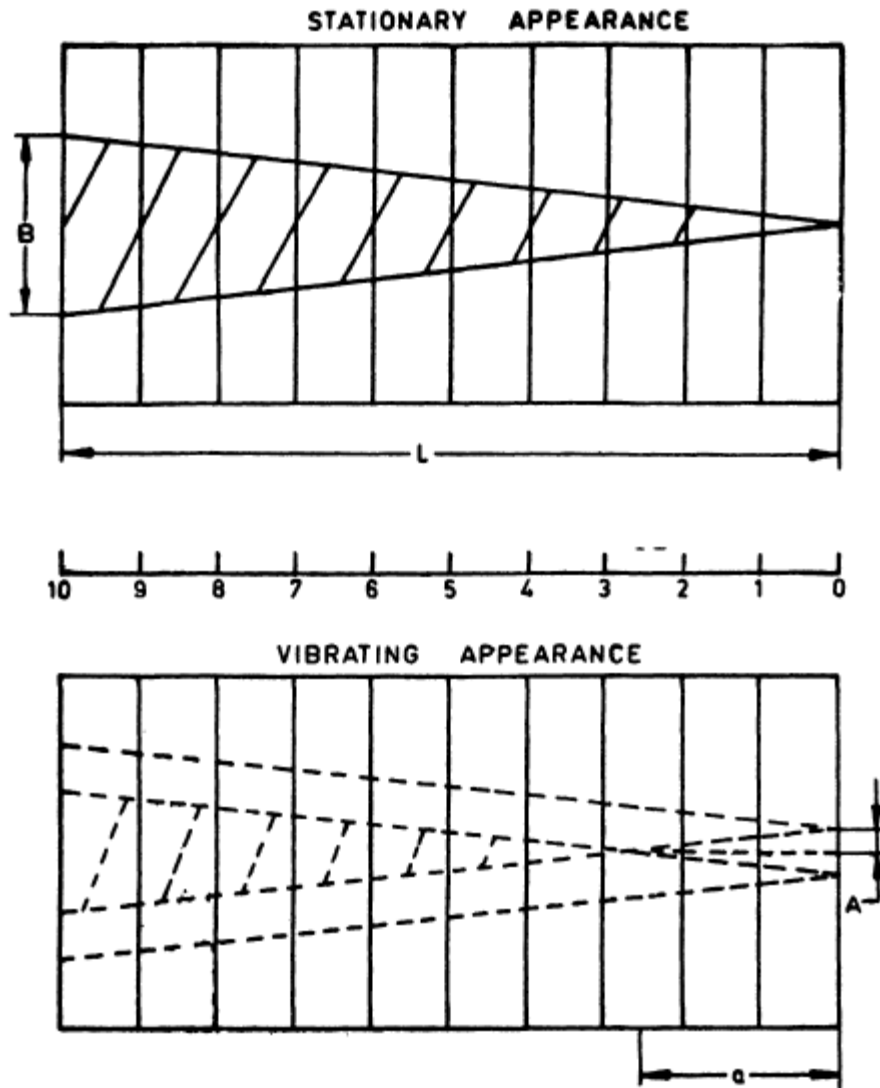
$$\text{Settlement, percent} = \frac{a - b}{a} \times 100$$

where,

a = mean height before testing, and

b = mean height after testing.

22.4.2 Examine the specimen after completion of the test and report any breakdown in the test specimen also.



A — ACTUAL AMPLITUDE
 a — APPARENT AMPLITUDE
 $\frac{a}{10} \times B = 2A$ OR $\frac{a}{L} = \frac{2A}{B}$

FIG. 8 VIBRATION SCALE

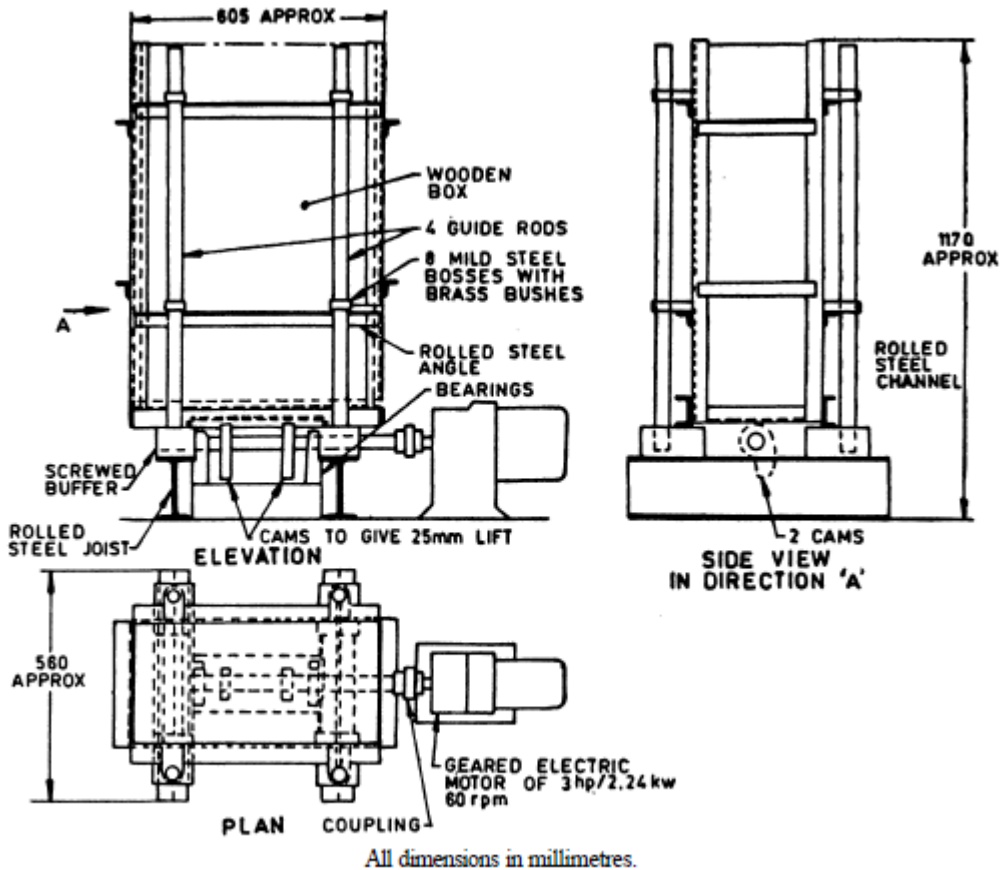


FIG. 9 JOLTING MACHINE

23 DETERMINATION OF WATER VAPOUR PERMEANCE

23.0 General

Water vapour permeance of the material is determined on a specimen 50 mm thick. The test shall be carried out at a temperature of $38^{\circ} \pm 0.5^{\circ}\text{C}$ with relative humidity of 90 ± 2 percent on one side of the specimen and less than 2 percent on the other side.

For conformance purposes, it would be sufficient to have representative product type testing and may not necessitate the fulfilment of the sample number requirements of conformity control prescribed in the relevant material specifications.

23.1 Test Specimen

The test specimen shall be a cylinder 50 ± 1 mm in height. The diameter shall be such that the specimen can be pushed into the beaker used for test without undue compression on the sides of the specimen. For test specimen, less than 50 mm thick plying up without the use of adhesive is

permissible. It is cut from the sample by a sharp-edged, thin, hollow cylindrical steel cutter. The flat faces shall be lightly sanded unless they have been freshly cut.

23.2 Number of Test Specimens

Carry out the determination on three specimen cut at random from different portions of the sample.

23.3 Apparatus

23.3.1 250 ml Glass Beaker — of approximately 65 mm internal diameter.

23.3.2 Humidity Cabinet

Maintained at $38 \pm 0.5^\circ\text{C}$ and 90 ± 2 percent relative humidity.

23.4 Reagents

23.4.1 Sealing Wax

The wax compound used for sealing shall be stable and non-absorbent to water vapour and shall not be brittle at room temperature. It shall strongly adhere to the beaker and the specimen. A suitable composition is a mixture of 90 parts of microcrystalline wax, such as paraffin wax, and 10 parts of a suitable nonreactive plasticizer, for example, polyethylene film scrap, shredding, etc. The melting point of the sealing wax shall be below 75°C .

23.4.2 Anhydrous Calcium Chloride

23.5 Procedure

Fill the beaker with anhydrous calcium chloride to about 6 mm below the expected position of the underside of the specimen. The mass of the desiccant shall be at least ten times the total expected take-up of water during the test. Insert the specimen in the beaker so that the flat surface of the test specimen, remaining exposed to the atmosphere, is in level with the top of the beaker rim. Seal the test specimen in position by running the melted sealing wax around the inner rim of the beaker (*see* Fig. 10), care being taken not to contaminate the exposed flat surface of the specimen. The temperature of the wax shall not be high enough to soften the specimen. Weigh accurately each assembly to 0.001 g and place it in humidity cabinet. Weigh successively at intervals of 24 hours and plot cumulative mass increase against time until at least three points (excluding the point of origin) lie on a straight line.

NOTE — The beaker shall be kept out of the humidity cabinet for the same length of time and under the same conditions at each successive weighing.

23.6 Calculation

23.6.1 Water vapour permeance (WVP) shall be calculated from the slope of the straight line drawn as nearly as possible through those points on the graph which represent a substantially constant rate of gain.

23.6.2 Water vapour permeance, in g/m^2 , 24 h = $\frac{240 M}{AT}$

where

M = increase in mass, in milligrams;

A = area of the test specimen, in cm^2 ; and

T = time for increase in mass, in hours.

23.7 Report

The mean of three determinations shall be reported as the water vapour permeance of the test sample material.

24 DETERMINATION OF FIBRE DIAMETER

24.0 Principle

It is carried out by projection, on a screen, of the mineral wool fibres and measuring the diameter by means of a graduated scale. The purpose of this test is to determine the mean fibre diameter of any fibrous product.

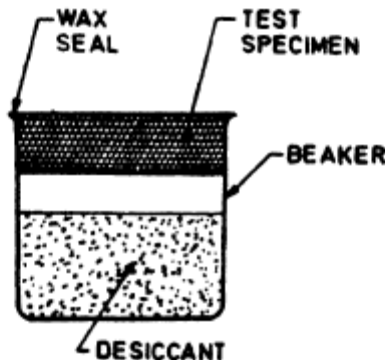


FIG. 10 ARRANGEMENT OF SPECIMEN FOR WATER VAPOUR TRANSMISSION TEST

24.1 Apparatus

24.1.1 A suitable projection microscope having linear magnification of $\times 1\,000$ or at least of $\times 400$ for fibres up to 20 microns or at least of $\times 250$ for fibres above 20 microns. It should have, a micrometer eye piece or a graduated screen rotatable through 360°C with pointer or any other suitable measuring devices.

24.1.2 Microscope slides and mounting medium comprising equal volumes of glycerine and distilled water.

24.2 Sample

Take handful of fibres from different parts of the test specimen and thoroughly mix them up to make a representative test sample. The sample should be free from foreign matter. Take 5 g from the sample for testing. It may be unbonded or it may contain resin binder.

24.3 Procedure

24.3.1 If the sample is bonded, the binder shall be burnt off in a furnace at the appropriate temperature. Mount a small number of fibres on a slide by floating in the glycerine/water medium.

Spread out well so that each fibre is separated from each other, and place a cover disc on the top. 25 readings shall be taken from each slide. Three such test slides may be made for observation.

24.3.2 Mount the slide at the microscope stage and roughly focus. Move the stage in any one direction until no more fibres cross the field of vision. Then move the stage into opposite direction until the first fibre appears at the centre of screen. Focus the image exactly. Read out diameter of the fibre in micrometer to the nearest micron.

24.3.3 Move the stage until second fibre reach at the centre of the focus. Refocus image and read out the diameter. Continue until 25 readings have been taken, if necessary the stage may be moved vertically and the observations continued.

24.4 Report

The mean fibre diameter shall be calculated using the statistical technique of grouped frequencies:

- a) Establish the number of fibres having an identical diameter x . For example f_2 fibres with x_2 diameter.
- b) Multiply each diameter x by the number of fibres f found with this diameter.
- c) Establish a sum of these products and divide by total number of fibres to give the mean fibre diameter:

$$\bar{X} = \frac{\sum fx}{\sum f}$$

25 TEST FOR ALKALINITY

25.1 Apparatus

25.1.1 pH Meter

Use a standard pH meter conforming to IS 2711.

25.2 Procedure

From the bulk sample draw approximately 5 g of mineral wool. Then weigh 2 g, shake well for 10 minutes with 100 ml distilled water (see IS 1070) at room temperature. Allow 5 minutes time and then measure the pH of the mixture, using a standard pH meter. Repeat the test on a farther 2 g of sample and record the mean pH value.

26 DETERMINATION OF CHLORIDE CONTENT

26.1 Reagents

26.1.1 Standard Silver Nitrate Solution

Prepare 0.014 1 N solution by dissolving 2.396 g silver nitrate in distilled water and dilute to 1 litre. Standardize the solution against 0.014 1 N sodium chloride solution (0.824 1 g NaCl

dissolved in water and diluted to 1000 ml). One millilitre of standard silver nitrate solution is equivalent to 0.500 mg chloride.

26.1.2 Potassium Chromate

Prepared by dissolving 50 g of potassium chromate in distilled water. Add silver nitrate solution till a definite red precipitate is formed. Allow to stand overnight and filter. Dilute the filtrate to 1 litre with distilled water.

26.2 Procedure

26.2.1 Take 30 g of mineral fibre in a 600 ml beaker. Add 300 ml chloride-free distilled water. Cover the beaker and place it in a steam bath overnight. Add fresh water for evaporation losses. Filter through Whatman No. 42 or equivalent filter paper.

26.2.2 Take 100 ml of the above extract and adjust the pH in the range 7 to 10 by adding 0.1 N sulphuric acid or 0.1 N sodium hydroxide solution. Add 1 ml of potassium chromate indicator solution. Titrate with standard silver nitrate solution to a pink-yellow end point. Run a blank also.

NOTE — Chloride contamination should be avoided at all stages during test.

26.3 Calculation

Chlorides (as Cl),

$$\text{percent by mass} = \frac{(V_1 - V_2) \times 0.05}{M}$$

where

V_1 = volume, in ml, of standard silver nitrate solution used in the titration with test sample;

V_2 = Volume in ml of standard silver nitrate solution used in the blank; and

M = mass, in g, of the test sample.

27 DETERMINATION OF ORGANIC MATTER

27.1 Apparatus

27.1.1 Silica Dish

27.1.2 Coring Tube — about 10 mm diameter.

27.2 Sampling

Representative material shall be used, obtained if necessary by corings through the whole thickness of a mat or slab at representative points. Other methods of cutting the sample may be used.

27.3 Procedure

Not less than 10 g of the sample shall be weighed to ± 0.01 g into a tared dry dish. It shall be dried in a ventilated oven at 105° to 110°C for 40 min, cooled in the desiccator and reweighed. The sample shall be ignited in a muffle furnace at $550 \pm 50^\circ\text{C}$ for 10 min. It shall be partially cooled, for example, on an asbestos mat, and then transferred to a desiccator. When cooled, the sample shall be reweighed.

27.4 Expression of Result

The mass of moisture shall be obtained by difference during drying and shall be expressed as a percentage of the original sample mass. The total organic material shall also be obtained by difference during ignition and shall be expressed as percentage of the dry sample mass.