

British Standard

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Methods of test for

Inorganic thermal insulating materials

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Refrigeration Heating and Air Conditioning Standards Policy Committee (RHE/-) to Technical Committee RHE/9, upon which the following bodies were represented:

British Ceramic Research Ltd.
 British Gas plc
 Chartered Institution of Building Services Engineers
 Combustion Engineering Association
 Cranfield Institute of Technology
 Department of Health and Social Security
 Department of the Environment (Building Research Establishment)
 Department of Trade and Industry (National Engineering Laboratory)
 Department of Trade and Industry (National Physical Laboratory)
 Electricity Supply Industry in England and Wales
 Engineering Equipment and Materials Users' Association
 Eurisol (UK Mineral Wool Association)
 Gypsum Products Development Association
 Phenolic Foam Manufacturers' Association
 Refrigeration Industry Board
 Royal Institute of British Architects
 Thermal Insulation Manufacturers' and Suppliers' Association (TIMSA)
 Thermal Insulations Contractors' Association
 Water Tube Boiler Group, Power Plant Contractors' Association

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

British Rubber Manufacturers' Association
 Calcium Silicate Brick Association Limited
 Institute of Refrigeration
 Institution of Mechanical Engineers
 Yarsley Technical Centre Ltd

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Foreword

This revision of BS 2972 was prepared under the direction of the Refrigeration, Heating and Air Conditioning Standards Policy Committee. It supersedes BS 2972:1975, which is withdrawn.

All the test methods of the 1975 edition have been reviewed. A few remain in their original form, with some minor amendments. The sections on odour, and emissivity of metal foil (sections 13 and 15 in the 1975 edition) have been deleted. The new section 8 gives a test for interlaminar strength. The new section 22 gives a method of test for pH value of a water extract.

Significant modifications have been made to the following sections:

Section 2. Dimensions. Measurement of the deviation from squareness of a board has been added.

Section 3. Bulk density. This now includes a method of test for blown materials.

Section 6. Heat stability. This has been re-written to clarify and improve the test equipment and method.

Section 9. Vibration settlement. Requirements for testing the settlement of blown materials have been added to the high frequency/small amplitude test.

The test for jolting at low frequency/high amplitude has been deleted because it was rarely, if ever, used.

The test for stability under heat and vibration has been deleted pending the development of a more suitable method.

Section 12. Water absorption. A new test method has been added for resistance to capillary absorption.

Section 14. Coarse shot content. A new dry method has been introduced in place of the former wet method, which used a special glass elutriator.

Section 19. Fibre diameter. The method has been revised to facilitate the identification and measurement of fibre diameters.

Section 21. Trace quantities of water-soluble chlorides. The potentiometric titration method, introduced by amendment in 1982, has been deleted and replaced by a polarographic titration method.

It is very important that claims for technical quality of materials should be related to well established methods of test which are based on sound fundamental principles, and the purpose of this standard is to provide details of basic tests for the assessment of a range of properties which are likely to be of value to a potential user. Some of these tests may not be suitable for quick routine control checking; essentially, they should be regarded as the basis for reference purposes in the event of dispute or of doubt concerning the accuracy of such more rapid methods as may be used as a matter of convenience. It is strongly recommended that, where any dispute arises, the methods of this standard only be used.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 56, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Section 1. General

1 Scope

This British Standard specifies methods of test for inorganic thermal insulating materials, including preformed, plastic composition, flexible and loose-fill materials.

The following methods of test are given.

- Section 2. Dimensions
- Section 3. Bulk density
- Section 4. Thermal properties
- Section 5. Assessment of fire hazard
- Section 6. Heat stability
- Section 7. Resistance to compression
- Section 8. Interlaminar strength
- Section 9. Vibration settlement
- Section 10. Covering capacity
- Section 11. Moisture content
- Section 12. Water absorption
- Section 13. Water vapour permeance
- Section 14. Coarse shot content
- Section 15. Oil content
- Section 16. Organic matter
- Section 17. Air permeability
- Section 18. Flexural strength
- Section 19. Fibre diameter
- Section 20. Electrical conductivity of aqueous extract
- Section 21. Determination of trace quantities of water-soluble chlorides
- Section 22. pH value of water extract

NOTE The titles of publications referred to in this standard are listed on the inside back cover.

2 Definitions

For the purposes of this British Standard the definitions given in BS 874 and BS 3533 apply together with the following.

2.1

wet covering capacity

the wet covering capacity of a plastic composition material is defined as the area covered to a thickness of 25 mm by unit mass of dry composition when the material is mixed with the quantity of water recommended by the manufacturer and applied in accordance with the method given in section 10 (usually expressed as m²/1 000 kg)

2.2

sample

a portion of material or group of units, taken from a large mass or aggregation, which is used to give information as to the quality of the larger quantity. The sample may be "random" or "representative"

generally, samples of preformed and flexible insulation will, of necessity, be random samples while those of insulating aggregates and powders will be representative ones

2.3

random sample

a sample taken without bias, i.e. so that every item has an equal chance of inclusion

2.4

representative sample

a sample in the selection of which planned action is taken to ensure that a specified proportion of its contents is drawn from a different subportion, e.g. case or package, of the whole aggregation. The sampling from within each subportion may, however, be random

2.5

specimen

a suitably sized portion of a sample which is used for a specific test

2.6

coarse shot

non-fibrous material retained on a 250 µm nominal aperture test sieve complying with BS 410

3 Sampling

3.1 General

Although there are well established procedures for the selection of samples for quality control purposes where the units are of similar type, size and shape, such as insulating bricks, it is not practicable to apply such methods to many consignments of insulating materials owing to the very wide variety of types and shapes which may be involved. It is therefore necessary to adapt the method of sampling to the type of product in question.

Sampling becomes difficult when the consignment consists of relatively small quantities of insulating materials of different types and physical properties, and it may be necessary to restrict the size of the samples in such cases to avoid the destructive testing of too large a proportion of each subportion of the consignment.

As a general rule, it is desirable to select the samples from material at the manufacturer's works, but this procedure may not always be appropriate owing to the possibility that the properties of the material can be modified prior to or during application at site.

Plastic composition materials are particularly vulnerable to such modification, if only through the method of application, so that tests at the source of supply may not be a reliable guide to the behaviour of the material during service on the plant.

3.2 Selection of samples

Samples shall be taken only from unbroken packages or from undamaged units and, as far as possible, they are to be representative of each material in the consignment.

Where the consignment comprises more than one type of insulating material and/or more than one thickness or density, each type, each thickness and each density shall be treated as a separate consignment for the purposes of sampling, unless other arrangement is made between the purchaser and the vendor.

Samples of materials consisting of relatively small and similar units, e.g. insulating bricks, shall be obtained by random selection, preferably while the consignment is being unloaded, as it is not practicable to ensure correct sampling from a stock-pile of the units.

Samples of granular, powdered and plastic composition materials shall be prepared by withdrawing material from each container selected for sampling: the material shall be taken after emptying and thoroughly mixing the contents of the container. All the samples of one type (preferably not less than 2 kg total) shall be thoroughly mixed together and gathered into a conical heap which shall then be divided into quarters by vertical cuts. Any two diametrically opposite quarters shall be discarded and the two remaining quarters mixed together and quartered as before. The process of mixing, quartering, discarding and remixing shall be repeated until a sample of size requisite for the tests remains.

When samples are to be drawn from material which has already been applied to a portion of plant, e.g. for bulk density determination on plastic composition or loose-fill insulating materials, care shall be taken to ensure that the portion of material selected is representative of all the material on the plant.

3.3 Size of sample

The sample will vary in size according to the number of tests that are to be carried out, but it should contain a sufficient number of units to permit random selection of specimens for each test.

Although, statistically, the minimum size of sample required is not dependent on the size of the batch being sampled, it is usually convenient to base a sampling schedule to some extent on the size of the consignment. Subject to there being a sufficient number of specimens or bulk of material selected for the tests required, recommendations for sampling are detailed in Table 1.

3.4 Specimens

Specimens which are cut from larger units shall be taken from an inner area of the unit, avoiding the outer edges as far as possible. Unless otherwise specified both original faces of the unit shall be retained in each specimen and not more than one specimen for a given test shall be taken from each unit of the sample.

Table 2 is a guide to the number and size of specimens required for the tests which are described in this standard. Each test is normally carried out three times, and the individual results quoted.

Table 1 — Sample sizes and rates of additional sampling

Material	Minimum size of sample	Rate of additional sampling
Small similar types of units, e.g. insulating bricks and small blocks	10 units	1 unit per 3 000 units above first 30 000 units
Plastic composition, loose-fill granular and powdered materials	2 kg ^a	0.5 kg per 1 000 kg above first 5 000 kg
Other materials	5 units	1 unit per 200 units

^a Taken from a minimum of five containers, where possible.

Table 2 — Size and number of specimens

Section	Test	Material	Size of specimen	Number of specimens
3	Bulk density	Preformed slabs	0.05 m ²	3
		Preformed sections	250 mm long	3
		Plastic composition	0.1 m ² before application	3
			0.013 m ² after application	3
		Flexible	0.1 m ²	3
		Loose-fill	Over 850 µm, 0.007 m ³	3
Under 850 µm, 0.002 m ³	3			
	Loose-fill: packing test	0.03 m ³	3	
	blown material	0.02 m ³	3	
4	Thermal conductivity		As required by BS 874	
5	Fire hazard tests to BS 476 Self heating hazard: hot plate test		As required by BS 476	
			Square with sides 300 mm up to 100 mm thick	2
		Square with sides 450 mm over 100 mm thick	2	
	Self heating hazard: hot bolt test		Square with sides 300 mm and thickness 150 mm	4
6	Heat stability		Square with sides 100 mm min.	5
7	Resistance to compression		Square with sides 100 mm min. and not less than twice the thickness of the specimen	5
8	Interlaminar strength		Square with sides 200 mm	5
9	Vibration settlement		900 mm × 600 mm piece or 0.2 m ³ loose-fill	3
10	Covering capacity	Plastic composition	0.004 m ³	3
11	Moisture content		5 g or sufficient to permit determination of 0.5 %	3 (at each required humidity level)
12	Water absorption		150 mm ² × 25 mm (immersion test)	6 or 9
			150 mm ² × 50 mm (capillary test)	6
13	Water vapour permeance		See BS 3177	3
14	Coarse shot content		10 g	1
15	Oil content		15 g or multiple thereof	3
16	Organic matter		10 g	3
17	Air permeability	Loose-fill	Approximately 0.004 m ³	3
18	Flexural strength	Preformed	300 mm × 150 mm (not less than 75 mm)	5
19	Fibre diameter		10 g	3 (min.)
20	Electrical conductivity of aqueous extract		5 g	3
21	Trace quantities of water-soluble chlorides		5 g or 20 g according to method	6
22	pH value		2 g	2

Section 2. Dimensions

4 General

Measurements of length, breadth, thickness, squareness and volume are required to check the dimensions of insulating materials supplied both before and after application and also for certain of the test methods specified in this standard.

These dimensions shall be determined as far as possible by the methods specified in this section but where these methods are impracticable, owing to size and nature of the insulating material and other difficulties, other methods may be agreed between the manufacturer and the purchaser.

This section does not attempt to lay down tolerances of manufacture of application; the tolerances quoted are essentially those of measurement only.

5 Measurement before application

5.1 Length and breadth of flat surfaces

5.1.1 Apparatus

5.1.1.1 *Steel rule*, graduated in millimetres.

5.1.1.2 *Tee square*

5.1.1.3 *Template* for cutting test sample, the size of which is dependent on the test for which the sample is to be used.

5.1.1.4 *Container* of constant cross section throughout its height, to determine the volume of loose-fill materials, the dimensions of which being dependent on the test for which the sample is to be used (see 12.3).

5.1.2 *Procedure for materials with clearly defined edges*. For rectangular surfaces, lay the material on a flat plane and measure length and breadth by laying a steel rule across the surface parallel to one edge and at right angles to an adjacent edge. Take measurements at three positions on one face for both length and breadth, namely at about 10 mm inwards from each edge and across the centre. Turn the material over and repeat the measurements for the opposite face, in all cases taking measurements to the nearest millimetre.

Record the mean of the six representative measurements as the length and breadth of the material.

For surfaces which are not rectangular, measurements shall be taken as appropriate.

Cut test samples required for other tests in this standard to the specified rectangular size, with square edges, preferably using a template.

5.1.3 *Procedure for materials with indeterminate edges*. Unpack and unroll materials which are compressed or tightly rolled during transit and allow them to expand freely for 4 h before measuring.

Measure full size material as specified in 5.1.2 as accurately as possible consistent with the nature of the edges. A tee 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 rule. Record the mean of the six respective measurements as the length and breadth of the material.

In the case of long rolls of material, additional measurements of width may be called for and turning the material may be omitted.

Cut test samples under a template and check the bottom face against the template for accuracy of square cut edges. When cutting round the template take care to avoid any extrusion of the material from the edges due to excessive pressure on the template.

Take measurements of length and breadth of the template as in 5.1.2, but on one face only, and consider these measurements to be those of the sample.

5.1.4 *Procedure for loose-fill materials*.

Measurements of material as delivered are not relevant. In the case of test samples, the length and breadth of the material correspond to the internal dimensions of the container used in the test. Measure the internal length and breadth of the container and record the measurements taken. Measure the position where the insulating material is levelled off and record the depth of material in the container.

5.2 Thickness of flat surfaces

5.2.1 *Methods of measurement*. Four methods are available for use in determining thickness. These are detailed in 5.2.3 to 5.2.6 and their applicability with respect to different types of material is given in Table 3.

NOTE These methods may be varied by agreement between the user and the supplier in such details as the mass of the plate in 5.2.3 or the load on the plate in 5.2.6.

5.2.2 Apparatus

5.2.2.1 *Steel rule*, graduated in millimetres.

5.2.2.2 *Thickness gauge*, as shown in Figure 1 (for use with the method given in 5.2.3). The apparatus comprises three parts: a probe, a collar and a 200 mm × 200 mm plate. The mass of the plate and the collar together shall be such as to exert a pressure of 30 N/m², i.e. 122 ± 1 g.

NOTE This can be achieved by making the plate from 3 mm acrylic sheet and the collar of aluminium, as detailed in Figure 1. The plate may be made from other materials provided that it is rigid and presents a flat, smooth, continuous surface to the insulation.

5.2.2.3 *Straightedge* (for use with methods given in 5.2.4 and 5.2.6).

5.2.2.4 *Callipers* (for use with the methods given in 5.2.5 and 5.2.6).

Table 3 — Methods of thickness measurement

BS number	Type of material	Method to be used	Notes
BS 3958-1	Magnesia: flat slabs curved surfaces	5.2.4 or 5.2.5 5.2.5	
BS 3958-2	Calcium silicate: flat slabs curved surfaces	5.2.4 or 5.2.5 5.2.5	
BS 3958-3	Metal mesh faced man-made mineral fibre mattresses	5.2.6	Facing materials shall not be disturbed. Sample size to be the same as plate
BS 3958-4	Bonded preformed man-made mineral fibre pipe sections	5.2.3	
BS 3958-5	Bonded man-made mineral fibre slabs	5.2.3, 5.2.4 or 5.2.5	
BS 5803-1	Man-made mineral fibre mats Compressible sheet materials	5.2.3 5.2.3	Material shall be allowed to expand freely for 4 h after unpacking or unrolling. Test area shall be not less than 400 mm × 400 mm
	Enclosed loose-fill materials	5.2.6	Enclosing material shall not be disturbed
	All materials after application in even layers, including unenclosed loose-fill materials	5.2.3	

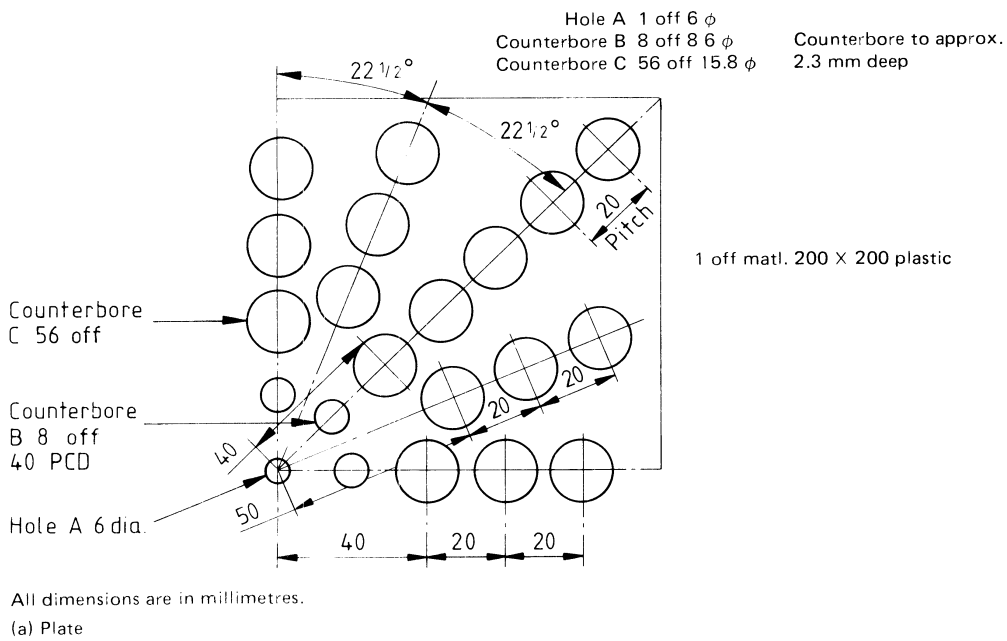
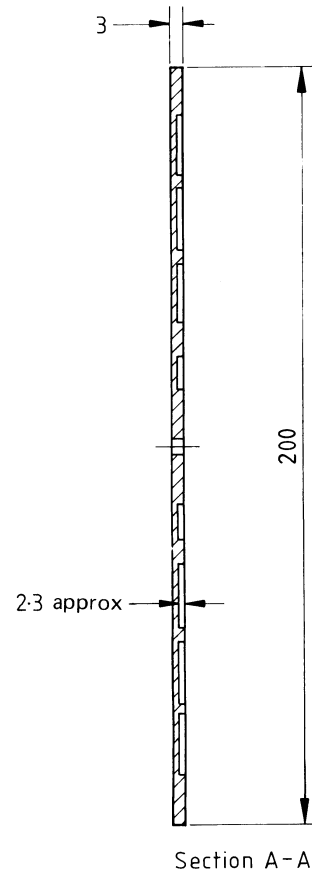
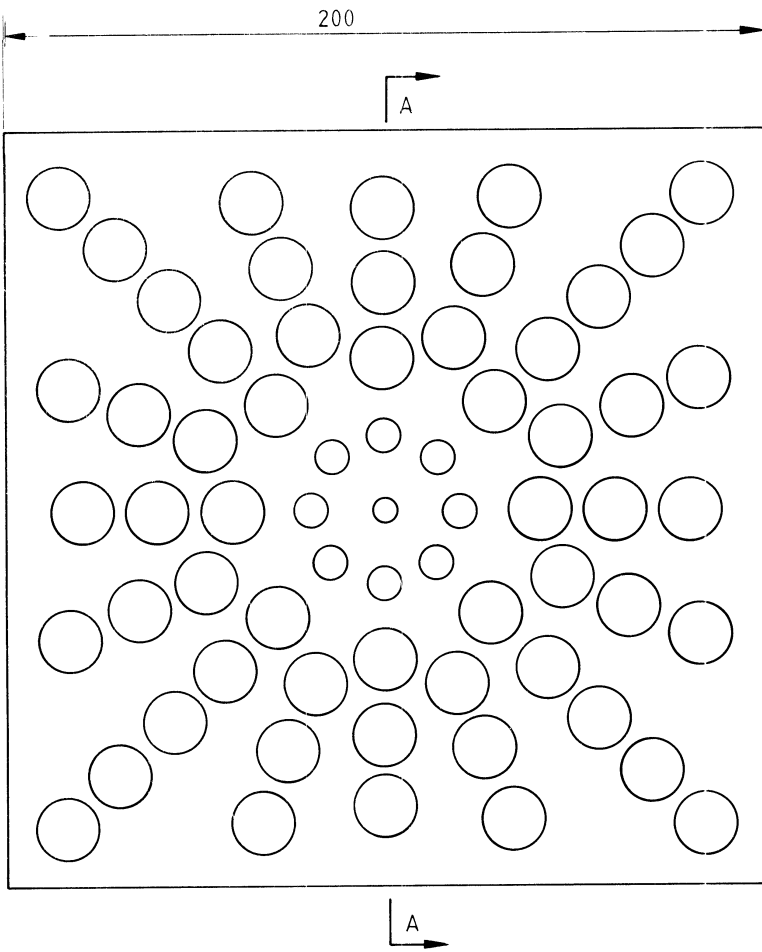
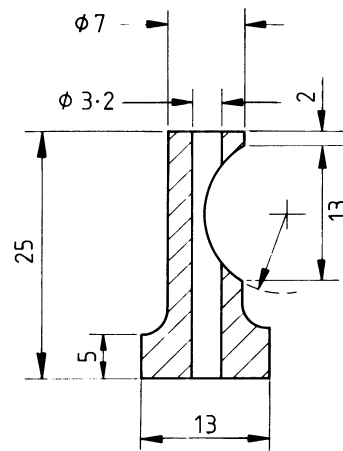
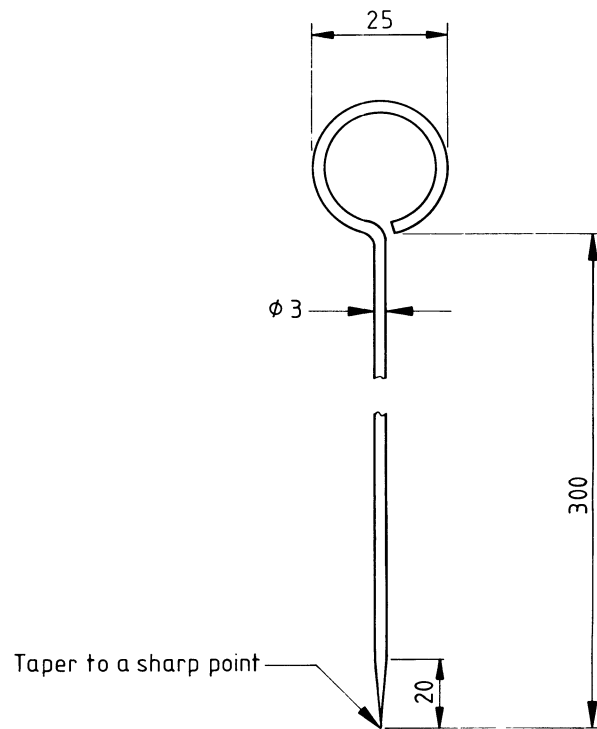


Figure 1 — Thickness gauge (continued)



NOTE. Radius for thumb-hold should extend to centre line of hole.



All dimensions are in millimetres.
(b) Collar and probe

Figure 1 — Thickness gauge (concluded)

5.2.2.5 Weighted plate (for use with the method given in 5.2.6).

5.2.3 30 N/m² method. Place the sample (not less than 400 mm × 400 mm) on a plane, hard surface and lay the plate centrally on the upper surface of the insulating material. Insert the probe with the sliding collar through the hole in the centre of the plate and push through the insulating material, normal to the principle surface, until its point touches but does not penetrate the underlying plane surface. Hold the collar firmly to the probe by pressure of the thumb in the position where it just touches the upper surface of the plate and, after withdrawal, measure the distance between the point of the probe and the underside of the collar to the nearest millimetre by means of a suitable rule. Record the thickness of the material as this measurement minus the thickness of the plate.

5.2.4 Straightedge method. Place the sample on a plane surface and lay a straightedge across the face of the material in three positions parallel to one edge of the material and at distances from the edge of one-sixth, half and five-sixths of the length of the adjacent edge. Determine the thickness by measuring with a steel rule the distance between the plane surface and the straightedge at both edges of the slab. Repeat measurements for the three positions of the straightedge at right angles to the initial set. Read measurements to the nearest millimetre and record the thickness as the mean of the 12 measurements.

5.2.5 Calliper method. Rule the sample of material into nine rectangles of equal area. Apply callipers at the centre of each area, taking care not to penetrate the surfaces. Take measurements to the nearest millimetre and record the thickness as the mean of nine measurements.

5.2.6 Weighted plate method. Place the specimen on a plane surface and subject it to a pressure of 725 N/m². A flat, stiff plate measuring at least 200 mm × 200 mm may be used for this. The specimen size is to be the same as the plate size. Determine the combined thickness of the specimen and plate by the use of a straightedge (see 5.2.4). Determine the thickness of the plate by callipers (see 5.2.5) and record the thickness as the difference between these two measurements.

NOTE A plate measuring 200 mm × 200 mm and weighing or loaded to weigh 2.96 kg exerts a pressure of 725 N/m². The methods appropriate to the different types of material are listed in Table 3.

When the thickness of an insulating material after application in an uneven layer (e.g. plastic compositions “finger-marked”) is to be determined and it cannot be measured by any of the methods described in 5.2 the methods described in 6.2.2 and 6.5.2 shall be used.

5.3 Volume of a flat shape

Calculate and record the volume from the significant length, breadth and thickness measurements obtained as in 5.1 and 5.2.

5.4 Deviation from square of a board

5.4.1 General. Methods are given for the determination of the deviation from square for the thickness, width and length of boards that are nominally square.

5.4.2 Apparatus

5.4.2.1 Steel square, with limbs of at least 500 mm in length.

5.4.2.2 Steel tape or ruler, graduated in millimetres.

5.4.3 Specimen. The specimen under test shall be a full board laid on a flat surface.

5.4.4 Thickness deviation. Place steel square with limbs longer than the board thickness on the flat surface against one end of the board as shown in Figure 2(a). Using a steel tape or ruler, take measurements to the nearest millimetre around the edge corners of the board.

The deviation from square is the distance between the edge corner of the board and the edge of the steel square at the point of greatest deviation (*A*).

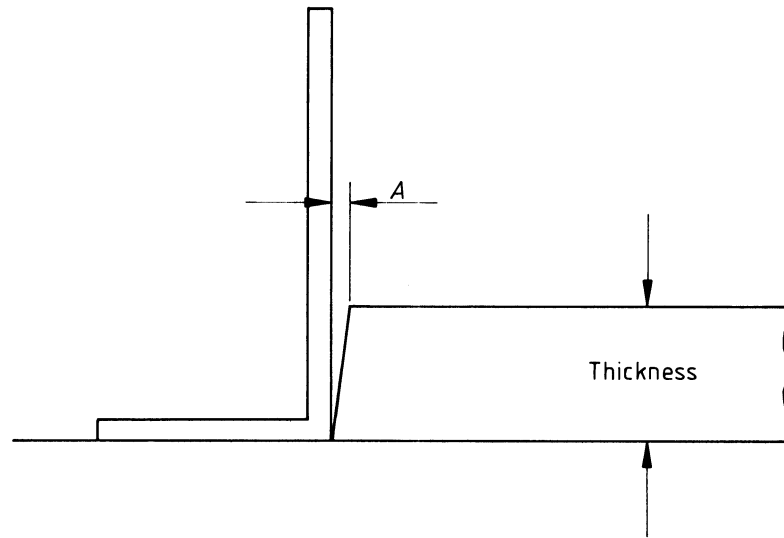
Report the deviation in millimetres.

5.4.5 Width or length deviation. Place a steel square along one of the parallel sides of the insulation with the right angle of the square aligned against the adjoining edge as in Figure 2(b). Using a steel tape or ruler measure the distance (*C*) between the edge of the board and the edge of the square at each corner and determine the greatest deviation (*C*). *B* is the distance over which the deviation is measured [see Figure 2(b)].

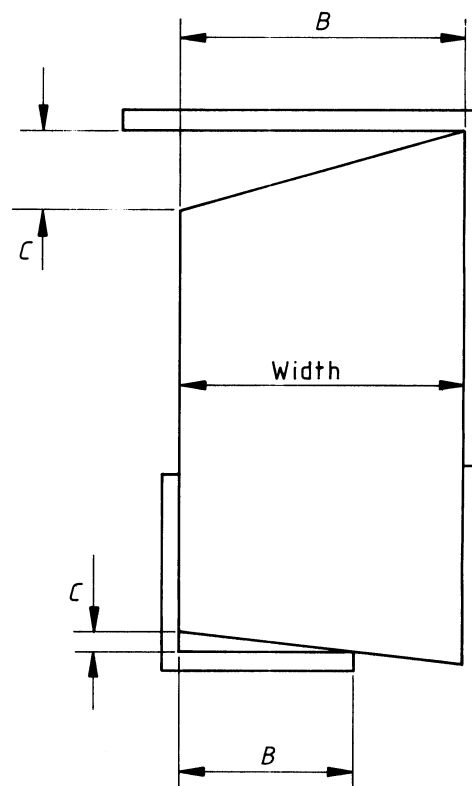
Calculate and report the deviation from square as millimetres gap per 100 mm of width or length.

5.5 Length and circumference of curved shapes

5.5.1 General. This sub-clause applies to materials of regular curvature in one direction only, this curvature being built into the material so that before application it conforms without change of shape to the surface it is intended to fit, e.g. circular, semi-circular and segmental pipe sections, radiused and bevelled lags. It does not apply to flexible shapes which when not applied will lie flat, or to special shapes such as hemispheres, domes or dished shapes etc.



(a) Thickness deviation



(b) Width or length deviation

Figure 2 — Determination of deviation from square of a board

5.5.2 Length. Take length measurements along the straight dimension by laying a steel rule along this direction at four positions equally spaced around the outside circumference in the case of completely circular sections, and at two positions for each semi-circular or segmental section or lag, or similar. Repeat the measurements for the inner surface, i.e. inside the bore of the section or on the other face of the lag. Read measurements to the nearest millimetre and record the length as the mean of the measurements taken.

5.5.2 Circumference or breadth. Measure the circumference, part circumference or breadth by laying a flexible steel rule in close contact with the surface. Take measurements at three positions on the external face and three positions on the internal face, namely, at about 10 mm inwards from each end and in the centre. In the case of completely circular sections the internal centre measurement may be omitted. Read measurements to the nearest millimetre. Record the mean of the external measurements as the breadth or circumference over the external curved surface and the mean of the internal measurements as the breadth or circumference over the internal curved surface.

5.6 Thickness of a curved shape

Thickness should preferably be measured by means of a probe and collar, (see 5.2.2.2 and Figure 1). Fit the material over a hard surface of radius equal to or less than the internal radius of the material and push the probe radially through the insulation from the external surface until the point is in contact with the hard surface lining held in close contact with the internal surface of the insulation. Measure the thickness from the probe setting as in 5.2.3.

Divide the material along its length into three equal distances and take thickness measurements to the nearest millimetre at the centre of each division at three positions equidistant around the circumference or part circumference.

Record the thickness as the mean of the measurements taken.

Thickness shall not, unless required otherwise, include the thickness of any external covering.

Alternatively, take thickness measurements by means of suitable callipers at positions outlined above. Where measurements at the centre are not possible due to the length of the calliper arms other positions may be chosen.

5.7 Volume of a curved shape

Calculate and record the volume as the product of the mean length, the mean of the breadth of the internal and external curved surfaces, and the thickness, as measured in 5.5 and 5.6.

6 Measurement after application

6.1 Length and breadth on flat surfaces

Measurement of length and breadth after application is not normally required in situ (except as indicated below). If specimens can be removed, the length and breadth can be measured by any of the methods outlined in 5.1.

The process of removal may result in a break-up of the specimen, preventing length and breadth measurement after removal. In this case, mark off the applied material into an area not less than 0.1 m² before removal, the developed area being as far as possible rectangular and of sides as far as possible dimensionally similar. Measure the length and breadth between the markings by means of a steel rule as in 5.1.2, but on one face (i.e. exposed insulation face) only. Record the length and breadth as the mean of these three respective measurements.

If it is not found possible to remove the marked area accurately trim the sides until an approximately rectangular hole is obtained and the length and breadth of the hole measured as above.

6.2 Thickness on flat surfaces

6.2.1 Even layers. Where the surface is sufficiently uniform, thickness shall be measured by means of a probe and collar as specified in 5.2.2.2. Insert the probe from the outer face only and record the thickness as the mean of the nine measurements.

6.2.2 Uneven layers. When the applied insulating material has an uneven surface, its thickness cannot be satisfactorily measured by the methods already described. The most usual example of this is when plastic composition is left "finger-marked" ready for the addition of a layer of some different material. In such cases determine the average thickness by measuring in situ the length and breadth of a marked off area of minimum 0.1 m², then carefully remove all the material in this area and weigh the material removed. The bulk density of the material is determined as in section 3 and the average thickness calculated from the following equation:

$$L = \frac{1000 m}{\rho A}$$

where

- L is the average insulation thickness (in mm);
- m is the mass of insulation removed (in kg);
- ρ is the bulk density of insulation (in kg/m³);
- A is the area of insulation removed (in m²).

6.3 Volume applied on flat surfaces

The volume shall be recorded as the product of the mean length, breadth and thickness as measured in 6.1 and 6.2 above.

6.4 Length and circumference on curved surfaces

Measurement of length and circumference after application is not normally required in situ (except for plastic composition). Test specimens can be removed and length and circumference measured by the methods outlined in 5.5.

For materials of uneven surface an area shall be marked off in situ and measured as in 6.1.

6.5 Thickness on curved surfaces

6.5.1 Even layers. Where the surface is sufficiently uniform, thickness shall be measured by means of a probe and collar as detailed in 5.2.2.2.

6.5.2 Uneven layers. For materials of uneven surface, the method given in 6.2.2 shall be adopted. The formula to be used for evaluation of thickness will depend on the degree of curvature and the thickness.

If the external circumference of the insulation in relation to the nominal thickness exceeds the values shown in column 1, Table 4, the surface may be regarded as flat and the formula in 6.2.2 used.

If the circumference is less than the value given in column 1 of Table 4, but exceeds the values in column 2, the appropriate equation is:

$$L = L_1 + \frac{\pi L_1^2}{C_2 - 2\pi L_1}$$

where

L is the average insulation thickness (in mm);

L_1 is $\frac{1000 m}{\rho A}$ (see 6.2.2);

C_2 is the circumference over insulation (in mm).

If the circumference is less than the values given in column 2, the insulation shall be completely removed from a 250 mm straight length of pipe (in this case ignoring the minimum 0.1 m² area earlier specified) and the following equation used:

$$L = \sqrt{\frac{(C_1^2 + (16 \times 10^6 \pi m/\rho)) - C_1}{2\pi}}$$

where C_1 is the external circumference of the pipe (in mm).

For curved surfaces in those cases where it is not possible to measure the internal circumference of the material, it shall be permissible to estimate this dimension from knowledge of the dimensions of the apparatus on which the insulation is applied.

6.6 Volume applied on curved surfaces

The volume shall be recorded as the product of the mean length, the mean of the breadth of the internal and external curved surfaces, and the thickness.

7 Weighing

Weighing shall be done on scales of sufficient capacity and sensitivity to weigh the test specimens to an accuracy of $\pm 0.5\%$. Unless specified otherwise, the result shall be recorded in kilograms.

Table 4 — Calculation of insulation thickness

Nominal thickness	External circumference of insulation	
	Column 1	Column 2
mm	m	m
up to 25	1.2	0.3
over 25 up to and including 50	5	0.75
over 50 up to and including 75	12	1.2
over 75 up to and including 100	20	1.7
over 100 up to and including 125	32	2.1
over 125 up to and including 150	45	2.7

Section 3. Bulk density

8 General

Methods are given for determining the bulk density of:

- a) preformed materials;
- b) plastic composition materials;
- c) flexible materials;
- d) loose-fill materials;
- e) blown materials.

Some insulating materials may be provided in composite form with a surface finish or protective covering which is substantially different in composition from that of the insulating material itself. Thus, it is common practice for flexible materials to be enclosed in galvanized wire netting, expanded metal, cloth etc., while flexible or preformed insulating materials may have a surface finish of man-made mineral fibre tissue, board or sheet material to facilitate handling or to provide an abrasion resisting surface. Normally, the contribution of this surface sheet to the thermal characteristics of the insulating material is negligible. Its mass shall therefore be omitted from bulk density figures referring to the insulating material.

Alternatively it may be desirable to know the total mass of the complete assembly of insulating material together with the surface finish; this shall be given in terms of mass per square metre or, for pipe insulation, mass per linear metre, as a supplementary figure to the bulk density of the insulating material alone and such a figure shall be accompanied by the corresponding total thickness of the composite insulation.

The test for bulk density shall be carried out on the material as ready for application in the case of preformed and flexible materials. It is recommended that the packing density of loose-fill materials be determined after application and bulk density of blown materials determined after the material has been blown through a suitable blowing unit. In the case of plastic composition materials, the test shall be carried out on the material after mixing the water, applying either to a test plate or to a portion of the plant, and drying. The amount of water used for mixing shall be noted as a percentage of the weight of the material.

Where required by the purchaser or supplier, the moisture content at the time of test shall be stated (see section 11).

In every case, three specimens shall be tested and the result expressed as the average of three tests.

The test for bulk density involves two types of measurement:

- a) measurement of volume, as determined by direct measurement of length, width and thickness;
- b) determination of mass.

9 Preformed materials

9.1 Specimens

Flat specimens should be not less than 0.05 m² in area or, failing this, the largest size available. Pipe sections shall be not less than 250 mm in length.

9.2 Determination of dimensions

Each sample shall be measured by the methods specified in section 2.

9.3 Calculation of results

The bulk density (ρ) in kg/m³ shall be calculated from the equation.

- a) For flat specimens

$$\rho = \frac{m}{abL}$$

where

- m is the mass of test specimen (in kg);
- a is the length of specimen (in m);
- b is the width of specimen (in m);
- L is the thickness of specimen (in m).

- b) For pipe sections (complete cylinder):

$$\rho = \frac{2m}{\pi aL(d_1 + d_2)}$$

where

- m is the mass of specimen (in kg);
- a is the length of specimen (in m);
- L is the thickness = $\frac{1}{2}(d_2 - d_1)$, (in m);
- d_1 is the internal diameter (in m);
- d_2 is the external diameter (in m).

9.4 Expression of results

The results of the test shall be expressed in kg/m³. If required, the moisture content shall be given as a percentage of the dry mass.

10 Plastic composition materials

10.1 Determination for manufacturer's declared value

For the purposes of determining bulk density in connection with the manufacturer's declared value, the specimens shall be prepared as given in section 10. The bulk density shall be determined substantially as given in clause 9, the linear dimensions of the dried sample being measured as in section 2, and the result shall be given to the nearest 10 kg/m³.

10.2 Determination after application

For the purposes of determining the bulk density of material which has been applied to portions of a plant and dried in situ, one of the following methods shall be adopted.

- a) Where the material is conveniently removable in sufficiently large specimens, remove a piece and determine its mass and volume after smoothing such faces as may be necessary to permit measurement of volume. The size of the specimen shall be not less than 0.01 m².
- b) Where the material cannot be removed in pieces which are sufficiently large for measurement of volume, determine the linear dimensions in situ.

First, smooth at least 0.05 m² of the outer surface of the material on the plant (or 250 mm lengths in the case of pipes) as necessary to remove any irregularities such as finger marks. Measure the thickness of insulating material either by means of a probe or, in the case of cylindrical surfaces, by circumferential measurement. Remove the insulating material entirely from the surface of the plant, taking care to avoid loss, and weigh. Determine the appropriate volume by measuring the area of the surface of the plant from which the insulation has been removed, in relation to the previously determined thickness of insulating material.

10.3 Expression of results

The results of the test shall be expressed in kg/m³. If required, the moisture content shall be given as a percentage of the dry mass.

11 Flexible materials

11.1 Specimens

The specimens shall be not less than 0.1 m².

11.2 Determination of dimensions

Each sample shall be measured by the methods specified in section 2.

11.3 Calculation of results

The bulk density shall be calculated in accordance with the appropriate formula as given in 9.3 and, unless measurements of thickness have been made, shall be expressed as the bulk density at the nominal thickness. The nominal thickness shall be that quoted by the manufacturer as relating to the particular material supplied and this figure shall be used for dimension *L* in the appropriate formula given in 9.3.

11.4 Expression of results

The results of the test shall be expressed in kg/m³ (based on the nominal thickness, where applicable). If required, the moisture content shall be given as a percentage of the dry mass.

12 Loose fill materials

12.1 General

For the purposes of this standard, loose-fill insulating materials are divided into two classes: powdered or granular, and fibrous. It should be recognized, however, that this division is not well defined, since the different types of material overlap and can in fact be used when mixed together. Further difficulty arises from the fact that nearly all loose-fill materials may be subjected, intentionally or otherwise, to compression or settlement in use. The practical performance of insulation is more directly linked with the bulk density of the loose-fill material after application, but for some purposes it may be necessary to determine the bulk density before application or to prepare a specimen having some specified bulk density.

Furthermore, the bulk density of many powdered and granular materials is not a uniquely defined property, but will depend on the history of the material. A test for determining the bulk density under specified conditions is given in 12.3 but it should be recognized that the value given by this test will not necessarily represent the bulk density which may be obtained in any particular application.

For methods of measuring bulk density after vibration, see section 9.

12.2 Measurements of bulk density of powdered, granular and fibrous loose-fill materials after application

No satisfactory test exists for measuring the bulk density of loose-fill materials after application. The following observations may give some guidance to suitable methods of approach to the problem.

The average bulk density may be deduced from the total volume of a section of insulation and the mass of insulating material that has been put into it. This may be misleading since it takes no account of possible variations in the degree of compression throughout the section. It is therefore desirable to test at a number of points in a given section of insulation. If this is done after the installation has been completed, as is preferable, it is generally necessary to remove part of the containing structure, such as a panel, so that the insulation from a measurable volume can be removed and weighed.

The procedure for removing the insulation, and the accuracy with which the volume of space from which it is removed can be determined, depend upon the nature of the material and upon the site. A coherent powder can fairly easily be divided from the surrounding material and removed without disturbing the latter. Fibrous materials are not so easily separated and therefore reasonably large amounts should be removed so as to reduce the ratio of edge to area. The controlled removal of freely flowing granules from a vertical cavity is likely to be difficult and their subsequent replacement may be impracticable.

If access can be gained to a horizontal space, however, it may be possible to press into the material a light sheet metal frame from within which the granules can be removed. Variations of the above methods should be devised to suit the particular material and site.

If it is impracticable to test after the insulation is complete, the less desirable course may be adopted of removing samples of material as the work proceeds. The general principles are much the same as those outlined in the preceding paragraph.

12.3 Bulk density of powdered and granular materials before application

12.3.1 Indication of bulk density. An indication of the bulk density of the material may be obtained by using one of the procedures given in 12.3.2, 12.3.3 or 12.3.5.

12.3.2 Material passing an 850 μm nominal aperture BS 410 test sieve

12.3.2.1 Apparatus

12.3.2.1.1 Measuring cylinder of 25 mL capacity.

12.3.2.1.2 Apparatus which can raise the cylinder through a distance of 5 mm by means of a cam (see Figure 3).

12.3.2.1.3 A means of weighing the sample.

12.3.2.2 Procedure. Mount the measuring cylinder on the table of the apparatus, fill to the 25 mL mark, drop ten times at 5 s intervals and determine the level of the powder. Weigh the powder.

12.3.3 Other materials

12.3.3.1 Apparatus

12.3.3.1.1 Box, 200 mm \times 200 mm \times 250 mm high.

12.3.3.1.2 Straightedge

12.3.3.1.3 Means of weighing the sample.

12.3.3.2 Procedure. Fill the box by allowing the material to fall evenly and loosely into it from a height of 150 mm above the upper edge. When the container is full, smooth the surface off with a straightedge and drop the box ten times from a distance of 25 mm onto a hard surface. Determine the level of the upper surface of the material below the edge. Weigh the contents of the box.

12.3.4 Expression of results. The results of the tests carried out in 12.3.2 and 12.3.3 shall be expressed in kg/m^3 . If required, the moisture content shall be given as a percentage of the dry mass.

12.3.5 Determination of the loose bulk density and the bulk density

12.3.5.1 General. The object of the test given in 12.3.5.3 is to determine the loose bulk density of the insulating material and also the bulk density after a specified regime of compaction. It is applicable to powders and granular materials.

12.3.5.2 Apparatus

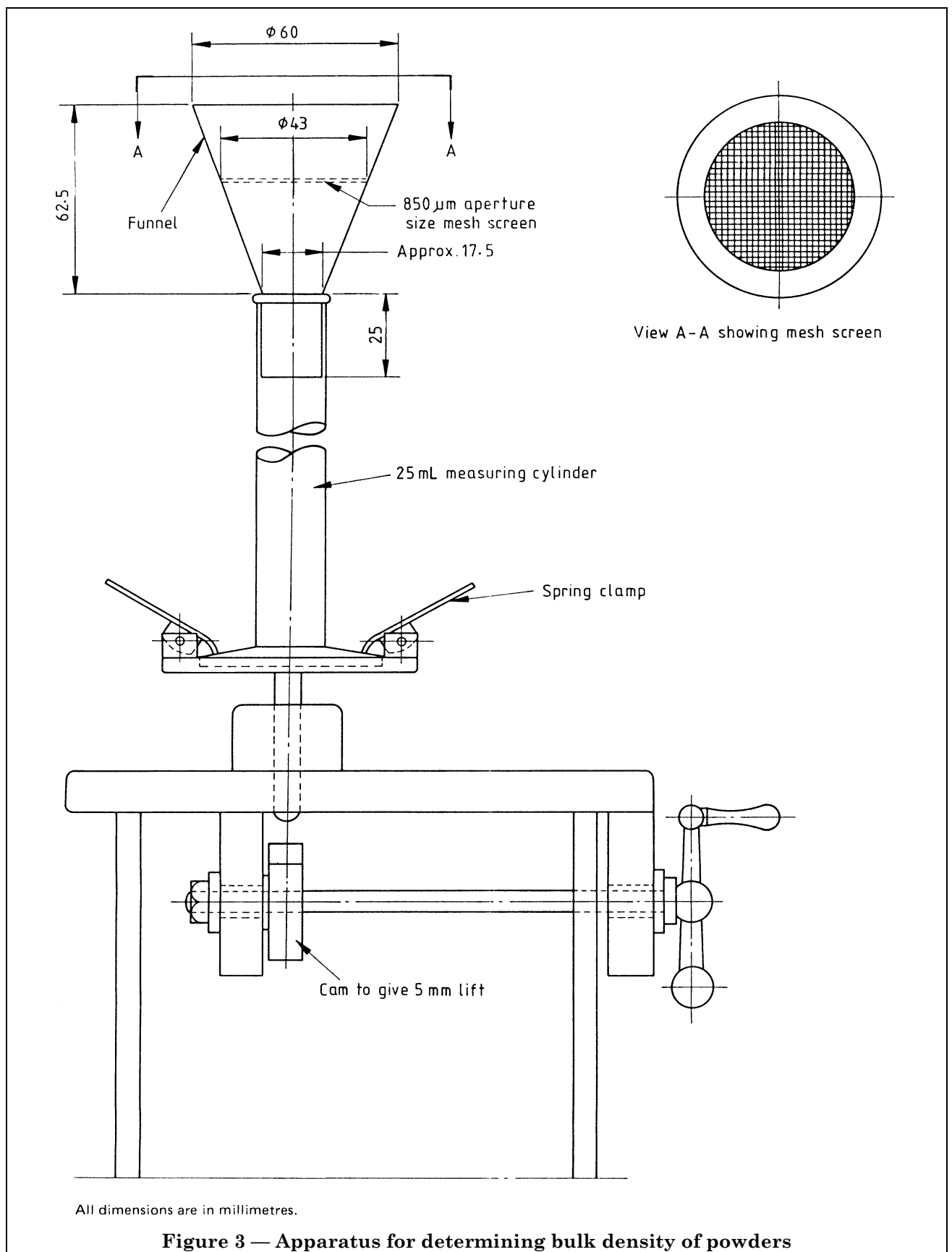
12.3.5.2.1 500 mL graduated plastics measuring cylinder of known mass.

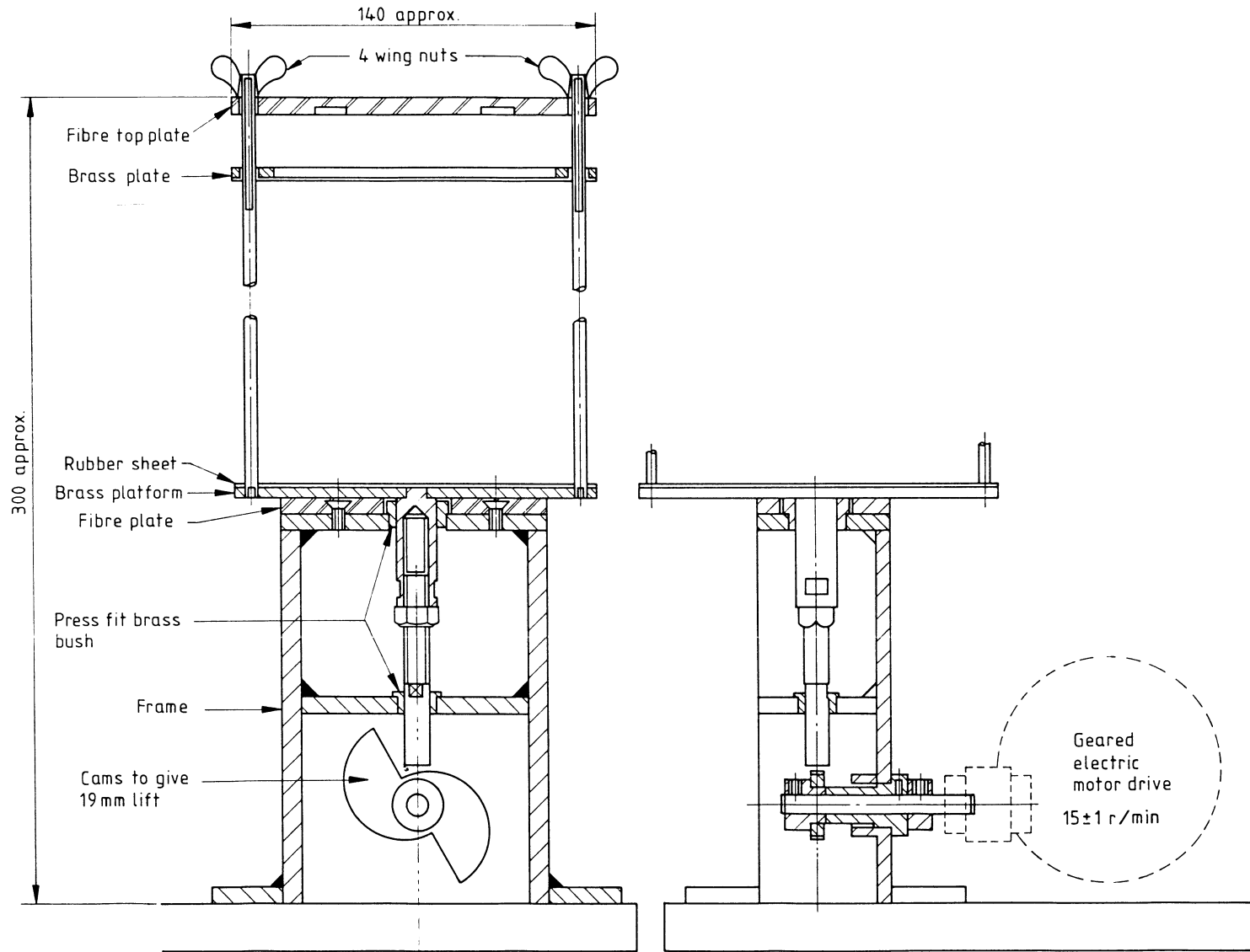
12.3.5.2.2 The apparatus shown in Figure 4. This is provided with a platform which can be raised 19 mm by a motorized cam and then allowed to fall onto a 6.3 mm thick pad of flexible closed pore expanded PVC (not shown in Figure 4). The cam is driven at 15 ± 1 r/min by a geared induction motor.

12.3.5.2.3 Means of weighing the sample.

12.3.5.3 Procedure. Using a scoop, transfer material from a coned sample, tipped from its packed or other container, into a cylinder held at approximately 45° to the vertical which is slowly rotated. Fill the cylinder to the 500 mL mark, weigh the cylinder and its contents and determine the mass of the specimen, m (in kg) by deducting the weight of the cylinder.

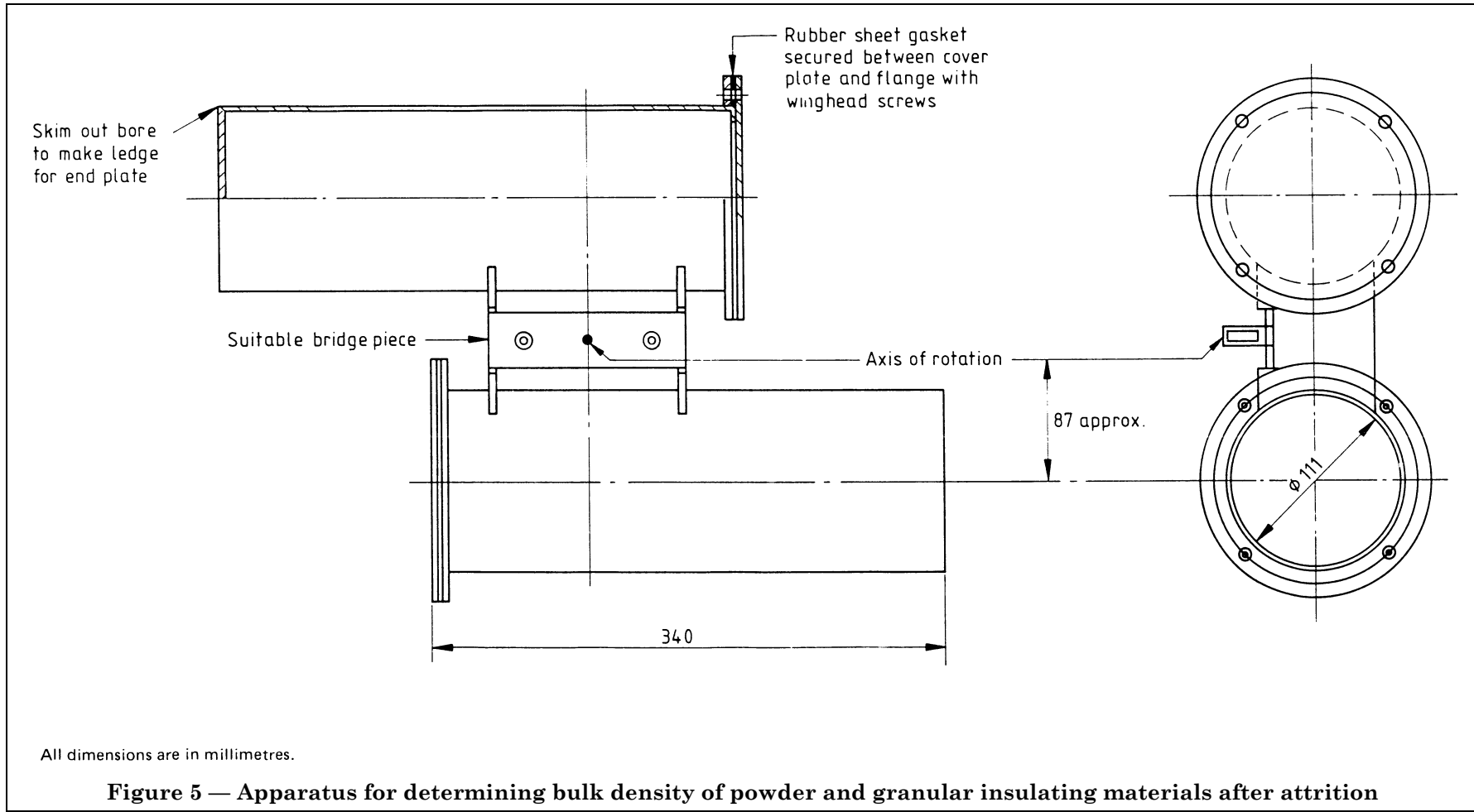
Place the cylinder and contents onto the platform of the apparatus, close the cover plate by means of the wing nuts and run the motor of the apparatus for a specified time, (e.g. for 20 min or until no further settling appears), after which read the new (compacted) volume, V (in litres) of the specimen from the cylinder graduations.





All dimensions are in millimetres.

Figure 4 — Alternative apparatus for determining bulk density of powders



12.3.5.4 Expression of results. The loose bulk density shall be given by $2m$ kg/L.

The compacted bulk density shall be given by m/V kg/L.

The mean of two determinations shall be quoted in each case.

12.4 Test for bulk density of powder and granular insulating materials after attrition

12.4.1 Object. The object of the test is to determine the change in bulk density produced by a specified regime of oscillatory movement of the material. It is particularly applicable to insulation materials composed of expanded minerals.

12.4.2 Apparatus

12.4.2.1 Apparatus as shown in Figure 5, consisting of two cylindrical vessels 111 mm i.d. \times 340 mm long closed at both ends and mounted on a common shaft rotatable at 30 r/min by an induction motor.

12.4.2.2 3 L graduated glass vessel with flat base which can be mounted on the bulk density apparatus of the previous test, shown in Figure 4.

12.4.3 Procedure

12.4.3.1 Preparation of specimen. Prepare the specimen by putting such a quantity of insulating material into the 3 L glass vessel that after compaction on the apparatus of the previous test (see Figure 4) for 40 s the volume is 2 760 mL.

Prepare two such specimens, one being transferred to each of the cylinders which shall thus be filled to 85 % of its capacity at the compacted bulk density. Close the cylinders.

12.4.3.2 Testing. Rotate the cylinders continuously for 48 h after which return the contents of each separately to the 3 L glass vessel, compacted for 40 s and measure the new volume V .

12.4.4 Expression of results. The result shall be expressed as the percentage reduction in the volume, i.e.:

$$100 \times \frac{2760 - V}{2760}$$

quoted as the mean of that for the two cylinders.

12.5 Packing test for loose-fill materials

The density of loose-fill materials is dependent upon the compression applied during packing and the procedure described in 12.3 is not always appropriate, particularly for fibrous or cellular materials. When it is desired to determine if a sample of the material can be packed at a preselected bulk density, a convenient method is as follows.

A mass of material shall be determined from the equation:

$$m = \rho V$$

where

m is the mass of material (in kg);

ρ is the preselected bulk density (in kg/m³);

V is the volume of the container (in m³).

The material shall be uniformly packed by hand in convenient increments into a box of known dimensions, e.g. a 300 mm cube. The investigation will indicate whether or not the loose-fill material can or cannot be packed at the preselected bulk density.

13 Blown materials

13.1 General

The installation of blown insulation requires a knowledge of the bulk density of the material after passing through a blowing machine. A test procedure is described which is suitable for use with a commercial insulation blowing machine.

13.2 Apparatus

13.2.1 Commercial blowing machine

13.2.2 Open-topped collection box constructed from 10 mm thick plywood with internal measurements of 600 mm \times 300 mm \times 100 mm deep. The top edges of the box shall be smooth and in the same plane.

13.2.3 Measuring device, constructed as shown in Figure 6.

13.3 Procedure

Carry out the procedure as follows.

- Make positioning marks on the top of the collection box such that the rule carried by the measuring device may be located over the principal axis of the box at six points:
 - 50, 150, 250, 350, 450 and 550 mm (measured from one end).
- Weigh this box to the nearest 10 g and place it on a level floor surface.
- Position the measuring device at each of the six locations pre-marked on the top of the collection box, with the inside edge of its feet aligned with the inside faces of the box.
- Take readings by pushing the rule gently downwards until its end contacts the floor of the collection box with the rule vertical.

e) With the blowing nozzle of the commercial unit held horizontally and approximately 1 m above the floor, blow the fibre into the box to a depth marginally above 100 mm and such that a sensibly smooth and level surface is obtained. Carefully remove any material resting on the top edges of the box.

f) Weigh the filled collection box to the nearest 10 g.

g) Take readings with the measuring device positioned as in Figure 7(c) by pushing the rule gently downwards until its end contacts the surface of the insulation with the rule vertical.

13.4 Expression of results

Determine the mass of insulation material used to fill the box (in kilograms) from the difference of the masses obtained in b) and f). Determine the initial thickness of material blown into the box as the arithmetic mean, to the nearest millimetre, of the difference between the pairs of readings taken at each of the six locations in d) and g).

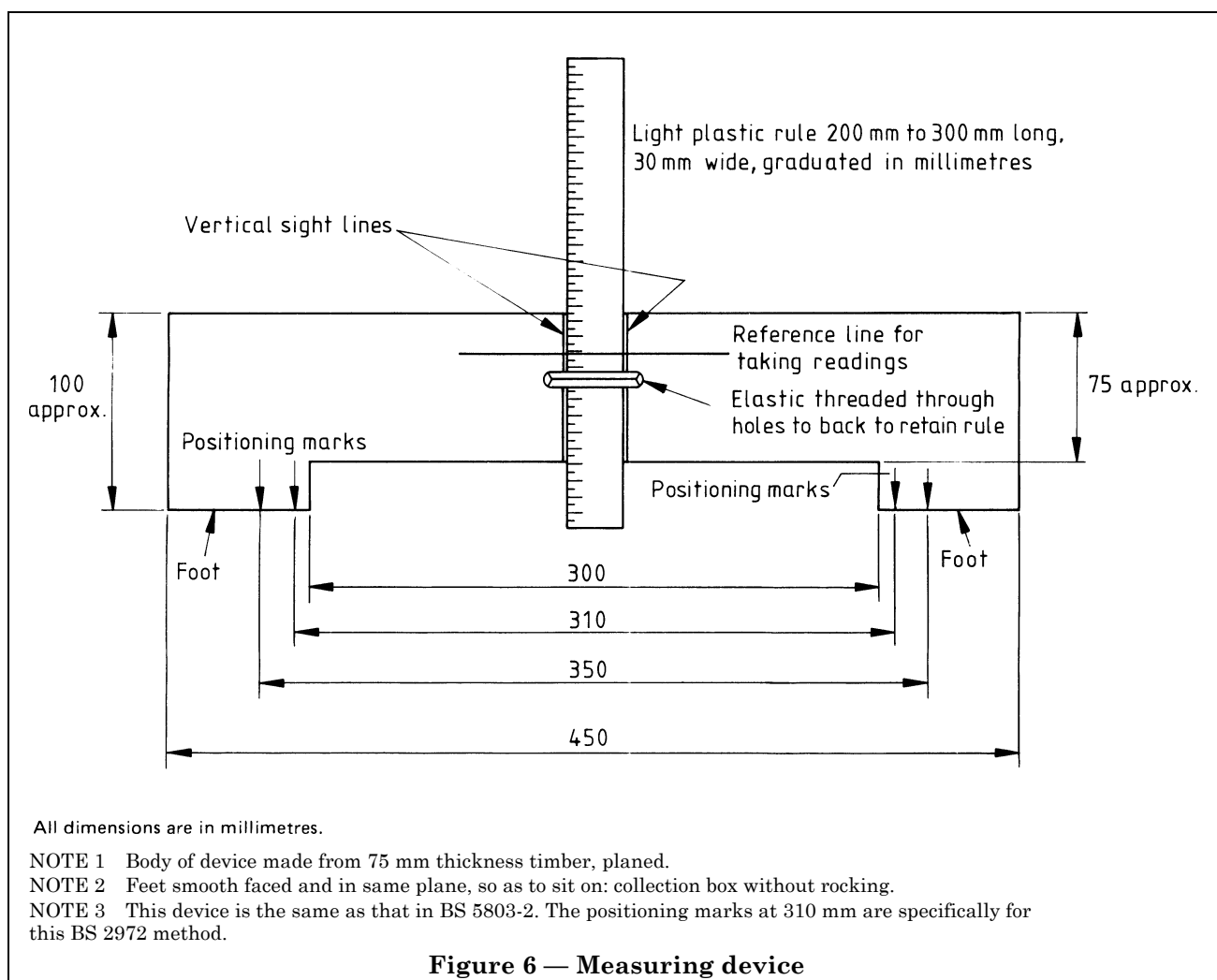
Calculate the density ρ of the insulation (in kg/m^3) used to fill the collection box using the following equation:

$$\rho = \frac{m}{0.00018L}$$

where

m is the mass of the insulation (in kg);

L is the thickness (in mm).



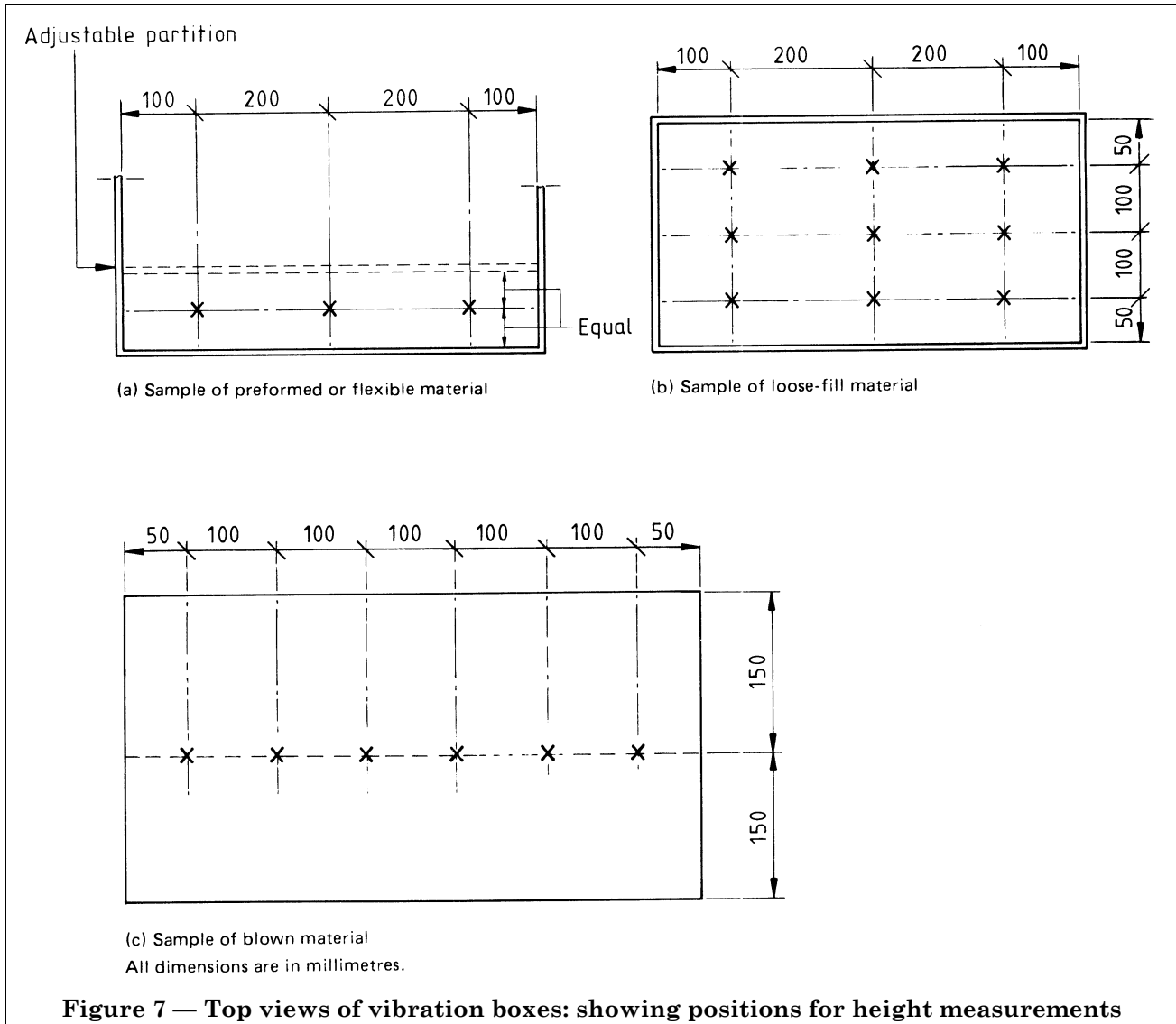


Figure 7 — Top views of vibration boxes: showing positions for height measurements

Section 4. Thermal properties

14 Thermal conductivity

The appropriate method of determining thermal conductivity given in BS 874 shall be used.

15 Specific heat capacity

At present, there is no suitable British Standard method for measuring the mean specific heat capacity of thermal insulating materials used at ambient temperatures.

Method 610 of BS 1902-6 is applicable to ceramic fibres and some other fibrous insulating materials used at elevated temperatures. The method given in ASTM-C351-82 is also suitable for materials used in hot conditions.

Results should be expressed in J/(kg.K).

16 Thermal diffusivity

The physical property which is important in considering variable heat flow is the thermal diffusivity. Whereas the conductivity measures the steady rate of heat flow, the thermal diffusivity measures the rate at which a temperature change is propagated through a medium and has the equation:

$$\alpha = \frac{\lambda}{\rho c}$$

where

α is the thermal diffusivity (in m²/s);

λ is the thermal conductivity [in W/(m.K)];

ρ is the bulk density (in kg/m³);

c is the specific heat capacity [in J/kg.K].

NOTE Thermal diffusivity can be calculated from the conductivity, density and specific heat capacity as shown by the above expression or, alternatively, it can be determined experimentally. There are several experimental methods available for the direct determination of thermal diffusivity but at present there is no standard method for measuring this quantity in the sense that the guarded hot plate apparatus is regarded as standard for the determination of thermal conductivity.

Section 5. Assessment of fire hazard

17 General

When an assessment of fire hazard is required, the appropriate tests described in BS 476 shall be used.

WARNING. A thermal insulation user should satisfy himself that the insulating materials he adopts will have an adequate degree of fire safety under the proposed conditions of service.

18 Assessment of self heating hazard

18.1 General

Some insulation materials which contain organic matter, e.g. as bonding material, although suitable for the service temperatures anticipated, may, in fact, constitute a fire risk under certain conditions, e.g. through the incidence of welding sparks, or through an internal self heating phenomenon.

Evidence of this internal self heating may be a transient rise in temperature above the theoretical value within the insulation. If excessive, this rise may present a hazard. Manufacturers normally state the limiting temperature of use and the limiting thickness (if one applies) that may be used at that temperature. The purpose of the tests in 18.2 and 18.3 is to ensure that the degree of self heating does not exceed safe limits. They differ from each other as follows.

The test described in 18.2 applies to the thickness, bulk density and the maximum hot-face temperature that may be attained during intended use. Accidental overheating from external sources, e.g. welding sparks, is excluded from the test.

The test described in 18.3 intentionally utilizes excessive over-heating to well beyond the recommended maximum service temperature of a material to instigate any self heating which may subsequently occur.

These tests are intended to refer to homogeneous insulating materials excluding added facings.

If there is a danger of the emission of noxious gases, the test should be carried out under conditions of adequate ventilation.

18.2 Self heating test: hot plate test

18.2.1 Apparatus

18.2.1.1 Hot plate of minimum size 300 mm × 300 mm if materials of total thickness not exceeding 100 mm are to be tested, or of minimum size 450 mm × 450 mm if the test thickness exceeds 100 mm. For thicknesses greater than 300 mm consideration should be given to a larger plate.

In all cases the hot plate shall be positioned horizontally and be capable of being controlled to within ± 2 % of the predetermined temperature.

18.2.1.2 Ambient compensated continuous temperature recorder suitable for use with fine wire thermocouples and having a range of up to 750 °C.

18.2.1.3 Chromellalumel thermocouples of maximum diameter 0.35 mm and of welded hot junction construction shall be used throughout.

18.2.2 Procedure. Raise the temperature of the hot plate, fitted with a thermocouple centrally on its surface, to the required test temperature whilst covered with a pack of insulation of the thickness and bulk density required for the actual test.

Meanwhile prepare the actual test pack, this being of the same surface dimensions as the hot plate and of the full thickness and bulk density ultimately to be used. Not less than three thermocouples shall be positioned within a radius of 25 mm from the centre of the pack in a horizontal plane. This is aided if the pack can be split open along a mid-plane.

When the hot plate has reached a steady state within ± 2 % of the required test temperature, quickly remove the insulation on the hot plate and replace with the prepared test pack.

The temperatures are recorded as follows:

a) until those of the mid-position thermocouples have passed or reached their maximum value and do not vary by more than 2 % of the hot face temperature in 1 h when the recorded temperature shall be taken as steady;

or

b) for 16 h;

whichever is the longer.

NOTE It is recommended that duplicate test packs be tested. Each individual result, together with the average of these results, should be quoted in the report.

18.2.3 Test criteria. The material shall be deemed to be satisfactory at the temperature, bulk density and thickness used in the test if the following conditions are satisfied:

- there shall be no sign of flame or glow from the stack during the test;
- there shall be no visible collapse or fusing of the interior when the stack is dismantled;
- at no time during the test shall the average mid-position temperature exceed the hot surface temperature.

In the event of self-heating (see 18.1) the record of the mid-position thermocouples will show a peak, and then will decline, tending towards a steady value. Criterion c) above shall apply to the mid-point temperature at all times during the test.

18.2.4 Test report. The test report shall include the following data:

- a) the type, designation and grade of the material tested;
- b) the bulk density of the material at the start of the test;
- c) the applied thickness of insulation;
- d) the temperature of the hot face during the test;
- e) the maximum temperature reached at the mid-point during the test.

18.3 Self heating test: hot bolt test

18.3.1 Sample preparation. Two specimens of the material, each 300 mm × 300 mm × 150 mm, shall be prepared so that when placed one on top of the other they form a sample or test piece consisting of a 300 mm cube. Each specimen may be formed of several layers and the sample shall be representative of the material as a whole.

18.3.2 Bolt specification. The "bolt" shall be a steel cylindrical block 36 mm in length and 32 mm in diameter, and weighing approximately 225 g.

If the material to be tested is rigid or semi-rigid, hollows to fit the bolt shall be cut out from the centre of each specimen so that when the bolt is placed in the centre of the sample, good contact shall be retained between the two adjacent interfaces of the specimens.

18.3.3 Temperature measurement. Three thermocouples shall be positioned on the top surface of the bottom specimen. These shall be positioned at 25 mm, 50 mm and 75 mm from the edge of the bolt. A thermocouple shall be securely attached to the bolt. The thermocouples shall be chromel/alumel with elements of approximately 0.5 mm diameter. It may be found most convenient to use thermocouple wire 1 m long and short lengths of ceramic beading to allow greater flexibility.

18.3.4 Procedure. Uniformly heat the bolt to more than 900 °C. Observe the temperature of the bolt as it cools in air free of draughts.

Insert the bolt between the two specimens as its temperature reaches 900 °C. Place a steel plate (3 mm thick and 300 mm²) on top of the sample so that its weight ensures good contact between the two specimens. Take readings of all the thermocouples at intervals of not more than 5 min for the first 2 h and 10 min thereafter. Continue the test until all thermocouples have cooled to 200 °C or less after passing through a maximum temperature, or until it is indicated that the material has failed to pass the test.

NOTE It is recommended that duplicate samples be tested. Each individual result, together with the average of these results, should be quoted in the report.

18.3.5 Test criteria. In order to pass the test the material shall satisfy the following conditions:

- a) there shall be no indication of flame or glow on the exterior of the cube during the test;
- b) the bolt temperature at any reading shall be not greater than at any previous readings, but readings during the first 5 min shall be ignored;
- c) none of the three thermocouples at the sample interface shall indicate a temperature of more than 10 °C above the temperature of the bolt at that time.

NOTE With fibrous insulating material of low bulk density, or with material which may tend to soften at the temperature involved, it may be found that the bolt sinks downwards into the sample. Note should be taken of any such displacement when the results of the test are assessed.

Section 6. Heat stability

19 General

This test is suitable for determining the heat stability of solid insulants, i.e. magnesia and calcium silicate preformed insulation. It is designed to determine changes in dimensions, mass and compressive strength following the heating of test specimens for 24 h at the desired temperature.

NOTE In the lower temperature ranges, the ageing properties of certain materials can assume greater importance than heat resistance as defined by conditions of test given in this section. BS 903-A19 specifies an ageing test conducted in an air oven for 168 h at 70 ± 1 °C.

20 Apparatus

A furnace or oven having a cavity size sufficient to accommodate at least two test specimens measuring 100 mm × 100 mm × 50 mm and two shielding specimens of similar size spaced so as to allow a clearance of at least 10 mm on all surfaces of every test specimen. The temperature of the furnace shall be controlled to within ± 1 % of the desired temperature.

21 Preparation of test specimen

Test specimens measuring 100 mm × 100 mm × 50 mm maximum thickness shall be cut from a sample leaving at least one of the original principal faces of the insulation. The length, width and thickness of each specimen shall be measured accurately using a calliper gauge which can be read to 0.1 mm and they shall be dried in a ventilated oven at 105 °C to 110 °C to constant mass.

22 Procedure

22.1 Heating of specimens

Place the specimens in the furnace at a temperature not exceeding 100 °C. Arrange them face to face resting vertically on their edges on at least three ceramic supporting bars and separated from each other by a distance of at least 10 mm. The supporting bars shall be of such a size that the specimens are at least 10 mm above the base of the furnace cavity with a clearance of at least 10 mm above them.

Place shielding blocks of calcium silicate insulation with similar dimensions to the test specimens in the furnace at each end of the group of specimens as protection against direct radiation from the furnace cavity walls.

Raise the furnace temperature to the desired temperature, the average rise in temperature shall not exceed 250 °C/h.

Heat the specimens continuously for 24 h at the desired temperature.

At the end of the period of heating, allow the specimens to cool to room temperature in a desiccator.

NOTE Where there is a danger of degradation due to thermal shock, the specimens should be allowed to cool initially in the furnace.

22.2 Inspection and testing of specimens after heating

Inspect and test the specimens as follows.

- a) The specimens shall be weighed and examined visually for cracking and distortion immediately after removal from the desiccator.
- b) The dimensions shall then be redetermined without delay in accordance with the procedure given in clause 21.
- c) The specimens shall be subjected to a compression test either immediately after measuring or later provided that they are stored in a desiccator. The tests shall be conducted as given in section 7 of this standard except that the conditioning procedure shall be omitted.

NOTE It is a requirement of this section that the resistance to compression of unheated material should also be determined. It is essential that the specimens for both tests be cut from the same samples.

23 Expression of results

Results shall be reported as follows.

- a) Average percentage change of length, width, thickness, and volume of the specimens.
- b) Percentage change (positive or negative) between the average dry mass of the specimens at the commencement of the test and the corresponding average dry mass after the test.
- c) Details of compression resistance as determined in section 7 of this standard on unheated material and the corresponding details of compression after heating.
- d) Any visible signs of cracking or distortion of the specimens both before and after heating.

Section 7. Resistance to compression

24 Apparatus

- a) A flat horizontal surface, a flat metal plate (platen) and appropriate weights. The platen should be square with sides at least 30 mm greater than those of the specimen to be tested.
- b) A hydraulic or mechanical compression testing machine of the appropriate size, compression rate and sensitivity. The platen shall be of such dimensions as to cover completely the specimen during the test and to enable thickness measurements to be taken. The platen should either be mounted on a self-aligning bearing to ensure axial loading or else the test machine should be of such design that the load is axial.

NOTE The apparatus as described in a) is more suitable for low loads (up to 7 kN/m²) but the apparatus as described in b) has been found to be suitable for the full range of compression testing.

25 Specimens

25.1 Sampling

The appropriate number of specimens shall be taken as laid down in clause 3.

25.2 Preparation

Each specimen shall be cut out in such a manner as to preserve as many of the original surfaces as possible; one specimen only shall be cut from a single sample. Damaged edges of a sample shall not be included in the test specimen.

The surfaces to which the load is to be applied shall be substantially plane and parallel and, as far as is practicable, original surfaces only shall be in contact with the platens.

If the outer surfaces are appreciably harder than the inner portion, e.g. when the sample has a hard outer finish, the hard surfaces shall be retained, but no more than two such hard surfaces are to be included in any one test specimen, e.g. by the use of multiple layers, unless a note to this effect is included in the report. Surfaces with a hard outer finish shall lie parallel to the platens.

25.3 Dimensions

The test specimen shall be square with sides of not less than 100 mm and not less than twice the thickness of the specimen. In the case of low density insulation the compression may be affected by the sample size and it is advisable to use specimens with sides of not less than 300 mm.

25.4 Determination of area

The area under test shall be assumed to be the average of the areas of the top and bottom faces of the specimen or specimens when determined at the nominal thickness specified by the manufacturer.

25.5 Thickness

The test thickness of the specimen shall be measured under a pressure that will depend upon the bulk density of the sample, as follows.

Table 5 — Thickness test under pressure

Sample bulk density at nominal thickness	Initial pressure
kg/m ³	kN/m ²
0 up to and including 35	0.030
Over 35 up to and including 100	0.1
Greater than 100	0.25

Metal mesh faced mineral wool mats and mattresses shall be measured at an initial pressure of 725 N/m² (see 5.2.6).

25.6 Bulk density

The bulk density of kg/m³, at the nominal thickness, shall be determined as in section 3.

25.7 Conditioning

The test specimen shall be conditioned in an atmosphere at 20 ± 2 °C and 65 % r.h. (see note to 39.2) to constant mass unless otherwise agreed.

26 Procedure

26.1 Specimen

The upper and lower faces of the specimen shall be flat and parallel and the specimen placed centrally in relation to the platen.

26.2 Initial load

Apply the initial pressure as indicated in 25.5 to the test sample within a tolerance of ± 1 %. Measure the thickness. This is the starting thickness for the test.

26.3 Application of load

Use either of the two forms of apparatus described in clause 24.

a) *Method 1.* Carefully place the load on the platen over the centre of the test specimen and maintain for at least 5 min before the reduction in thickness is ascertained.

b) *Method 2.* If an automatic recording of thickness is being made the rate of loading should not exceed 1 mm/min. If the thickness is not being recorded automatically the rate of loading may be increased and maintained for 5 min at each step load before measurement of thickness.

This procedure should be used until either the desired pressure or compression is reached, or until the sample fails.

26.4 Measurement of reduction in thickness

The reduction in thickness shall be derived from the mean of four measurements taken near to the vertical edges of the specimen or, if an automatic recording of thickness is being made the thickness should be noted from the record at convenient load intervals.

The statement of results shall include details of the conditioning which the specimen has received, the bulk density (see 25.6) and the nominal thickness.

If failure of the specimen is observed during the test, the loading at which failure occurred shall be recorded.

27 Expression of results

The percentage reduction in thickness after application of the load shall be derived from the following expression which may be represented graphically as a function of loading:

$$\frac{A - B}{A} \times 100$$

where

A is the initial thickness of the specimen (see Table 5);

B is the thickness under load.

Section 8. Interlaminar strength

28 General

NOTE This test is designed to give information on thermal insulation materials in slab or board form of damage caused by external stresses, e.g. such as those caused by winds upon faced and bonded roofing insulation boards.

28.1 Apparatus

28.1.1 *Five pairs of flat rigid plates* at least 200 mm × 200 mm, with suitable means on one face for fixing horizontally to the testing machine crossheads.

28.1.2 *Suitable tensile testing device* either hydraulic or mechanical of the appropriate size and sensitivity, with crosshead speed of 5 mm/min, or similar.

28.1.3 *Suitable adhesive* for bonding the samples to the plates, such as asphalt or wax.

28.2 Specimens

28.2.1 *Sampling*. The appropriate number of specimens shall be taken as laid down in Table 2.

28.2.2 *Preparation*. One specimen shall be cut from a single sample but no specimen shall be taken from within 50 mm of the edge. Each test specimen shall be carefully bonded between two plates as shown in Figure 8. Only sufficient pressure to ensure complete surface contact shall be applied. The surfaces to which the load is to be applied shall be substantially plane and parallel and, as far as is practicable, original surfaces only shall be bonded to the plates.

If the outer surfaces are appreciably harder than the inner portion, e.g. when the sample has a hard outer finish, the hard surfaces shall be retained. Surfaces with a hard outer finish shall lie parallel to the plates.

28.2.3 *Dimensions*. The test specimen shall be square with sides of not less than 200 mm × 200 mm.

28.2.4 *Determination of area*. The area under test shall be assumed to be the average of the top and bottom faces of the specimen or specimens when determined at the nominal thickness specified by the manufacturer.

28.2.5 *Thickness*. The test thickness of the specimen shall be measured as the distance between the bonded surfaces of the plates. Measurements shall be made on each edge.

28.2.6 *Bulk density*. The bulk density in kg/m³, at the nominal thickness, shall be determined as in section 3.

28.2.7 *Conditioning*. The test specimen shall be conditioned in an atmosphere at 20 ± 2 °C and 65 % r.h. (see note to 39.2) to constant mass unless otherwise agreed.

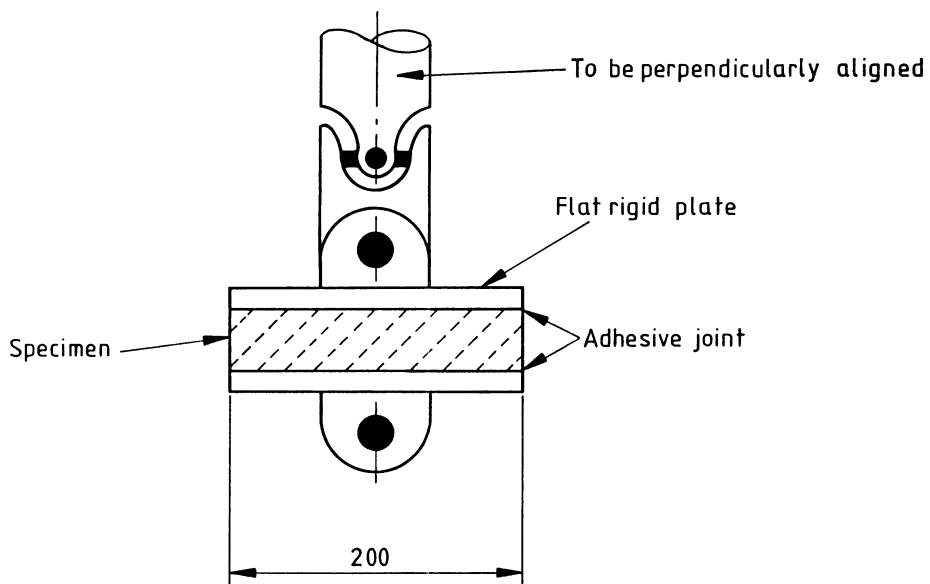
28.3 Procedure

28.3.1 *Specimen*. Attach the specimen plates to the tensile machine crossheads ensuring that the specimen is centrally located with the plates supported horizontally.

28.3.2 *Application of load*. Apply the load at the crosshead speed, indicated in 28.1.2 and determine the load required to fracture the specimen. Observe how the material failed or the facing membrane delaminated. Discard samples where the specimen failed in the adhesive layer.

28.4 Expression of results

The maximum load in N/m² causing failure of the specimen and the area at which failure occurred shall be reported. The thickness of the specimen and its bulk density shall also be included in the report.



Dimension is in millimetres.

Figure 8 — Specimen mounting for interlaminar strength test

Section 9. Vibration settlement

29 Vibration at high frequency and small amplitude

NOTE This test has been used mainly for assessing the behaviour of mineral fibre and granular products for transport service and may not be acceptable for other purposes.

29.1 Apparatus

29.1.1 A wooden box with smooth interior surfaces, having internal dimensions of 600 mm long × 300 mm wide and normally 900 mm high, unless specified otherwise in the BS relevant to the material being tested. The box is provided with a removable lid, and secured within a steel framework fixed to a steel baseplate, which may be rigidly mounted on unsprung wheels to render the apparatus mobile. The box may be conveniently adapted for testing one or more specimens of less than 300 mm thickness by providing detachable vertical partitions, 900 mm high × 600 mm long, at suitable spacings. Provision is made for vibrating the baseplate in a vertical plane at a frequency of about 24 cycles per second.

Figure 9 illustrates the standard form of apparatus¹⁾, with which an amplitude of approximately 1 mm about the mean position is obtained. A vertical strip of transparent plastics material may conveniently be inserted in one face of the box so that the progress of the test may be observed visually.

29.2 Preformed and flexible materials

29.2.1 *Specimens.* The following specimens are required.

a) *Specimens having substantially plane parallel surfaces and which are to be tested with the width or length placed vertically.*

A test specimen 900 mm long × 600 mm wide shall be placed in the box and if the manufacturer's declared thickness is less than the corresponding width of the box, a partition should be adjusted to hold the specimen securely at that thickness.

b) *Specimens which are to be tested with the width and length placed horizontally.*

A test specimen 600 mm long × 300 mm wide shall be laid in the box which may be reduced to a height more convenient for the thickness of sample to be tested.

29.2.2 *Procedure.* Calculate the mean height of the specimen from measurements made at three points as shown in Figure 7(a).

Place the lid on the box and vibrate the rig continuously for 16 h.

Calculate the mean height of the specimen as above.

29.3 Loose fill materials

29.3.1 *Specimen.* Apply the specimen to the box at a thickness appropriate to the thickness of usage and at the density specified in accordance with the procedure described in 12.5.

29.3.2 *Procedure.* Calculate the mean height of the specimen from measurements made at the positions indicated in Figure 7(b).

Place the lid on the box and vibrate the rig continuously for 16 h.

Calculate the mean height of the specimen as above.

29.4 Blown materials

29.4.1 *Specimen.* Apply the specimen to the box at a thickness appropriate to the thickness of usage and at the density specified in accordance with the procedure described in 13.3.

29.4.2 *Procedure.* Transfer the filled box without delay to the vibration apparatus. Ensure that no jarring of the box occurs during the transfer or when clamping it into the apparatus.

Vibrate the rig continuously for 2 h.

Calculate the mean height of the specimen as in 13.3.

30 Expression of results

The settlement of the material under test shall be expressed as a percentage of the initial height of the specimen in the box, using the equation:

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

where

a is the height before testing (in mm);

b is the height after testing (in mm).

The specimens of preformed and flexible materials should also be inspected after completion of the test and a report made on the presence of loose dust or other evidences of breakdown.

¹⁾ Working drawings of this apparatus are contained in PD 6467.

31 Description of a gauge for measuring settlement of fibrous materials

The gauge consists of a timber beam about 600 mm long and 75 mm × 75 mm section which may be placed across the 300 mm width of the test box. Sliding vertically and freely through a hole drilled in the middle of the beam is a measuring rod consisting of a 15 mm diameter hardwood dowel, having at its lower end a 22 mm diameter mild steel disc, 3 mm thick. The dowel is graduated in millimetres.

To measure settlement involves gently lowering the measuring rod so that it rests under its own weight upon the surface of the material in the positions described in **29.2.2**, **29.3.2** or **29.4.2** as applicable.

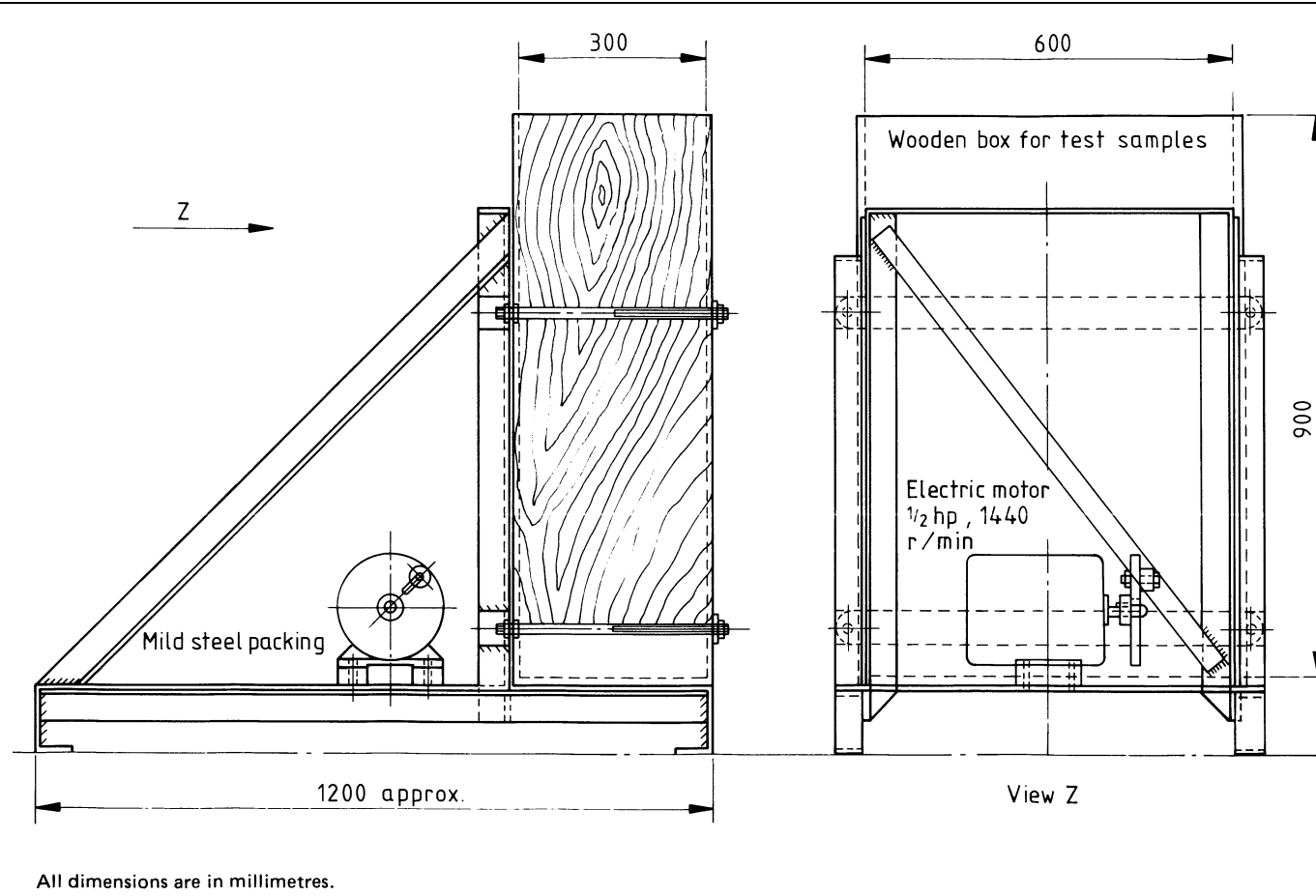


Figure 9 — Vibration tester

Section 10. Covering capacity

32 General

The test for covering capacity involves the preparation of a sample slab of a plastic composition material under standardized conditions from a known mass of dry material and determining the dimensions.

NOTE A similar technique may be used for finishing materials which are applied in the wet state except that a mould depth of 13 mm may be used where this is appropriate.

33 Apparatus

33.1 Rigid mould, with one end open, for which convenient internal dimensions are 450 mm × 300 mm × 50 mm deep. A piece of wood of dimensions approximately 50 mm × 25 mm × 297 mm shall be a sliding fit within the mould walls to close the open end.

The base of the mould may be of metal plate or of other non-absorbent smooth, flat material. The walls may be of angle section steel or of wood firmly secured to the baseplate by tap bolts or screws.

NOTE The provision of wing nuts for securing the walls to the baseplate is of assistance for each removal of the specimen.

33.2 Trowel

33.3 Steel rule

33.4 Depth gauge or probe

34 Procedure

34.1 Preparation

34.1.1 Preparation of the mix. Weigh the sample of plastic composition material as supplied and place in a bucket or bowl. Mix water with the material in accordance with the recommendations of the manufacturer. Take care not to over-work the mix.

It may be necessary to carry out a preliminary test to determine the mass of material to be taken. In the case of hydraulic setting cements the mass of the final dried block is greater than the mass of the dry material used in the mix.

34.1.2 Preparation of the mould. The inside of the mould shall be lubricated or lined with polyethylene film to prevent the plastic composition material sticking or drying and to facilitate removal of the dried specimen.

34.2 Application of the mix

The entire sample of wet mix shall be trowelled into the mould. The mix shall be levelled off to the top of the mould by means of a straightedge, and shall be drawn towards the open end of the mould. The loose end-piece of the mould shall be adjusted to give a rectangular shape of the specimen.

When the mould is filled the surface of the mix shall be lightly trowelled over to give a smooth finish. Care shall be taken that undue pressure is not employed.

34.3 Drying of specimen

The specimen shall be dried in the mould to constant mass at a temperature not exceeding 110 °C. For this purpose, the mould may be placed in a well ventilated oven or, alternatively, over a hot plate.

If the manufacturer so recommends, specimens shall be allowed to stand at room temperature for a specified initial setting period before drying at elevated temperature.

34.4 Removal of specimen from mould

After drying to constant mass, the specimen shall be removed from the mould and its dimensions determined.

The length and breadth shall be measured in at least three places. The thickness shall be measured by means of a depth gauge or suitable callipers in not less than nine different places, preferably at the intersection of lines of quadrisection.

35 Expression of results

Results shall be expressed as follows.

- a) The wet covering capacity is expressed as

$$C_w = \frac{dbl}{m} \times 4 \times 10^{-5}$$

- b) The dry covering capacity is expressed as

$$C_d = \frac{d'b'l'}{m} \times 4 \times 10^{-5}$$

where

C_w is the wet covering capacity per 1 000 kg at 25 mm thickness [in (m²/1 000 kg)/25 mm];

C_d is the dry covering capacity per 1 000 kg at 25 mm thickness [in (m²/1 000 kg)/25 mm];

d is the depth of mould (in mm);

b is the breadth of mould (in mm);

l is the length of mould filled by sample (in mm);

m is the mass of sample taken (in kg);

d' is the average thickness of dried sample (in mm);

b' is the average breadth of dried sample (in mm);

l' is the average length of dried sample (in mm).

- c) When required by the purchaser or supplier, the moisture content of the material at the time of starting the test shall be stated.

NOTE Because of the lubrication of the mould surfaces, the shrinkage will be greater in this test than that encountered in actual service conditions, where adhesion will be obtained between the wet plastic composition material and the surface to which it is applied.

Section 11. Moisture content

36 General

Methods are specified for the determination of the moisture content of preformed, flexible and loose fill insulating materials in the condition as received and after conditioning in atmospheres of low, medium and high humidity.

37 Specimens

37.1 Number of specimens

The appropriate number of specimens shall be taken as laid down in clause 3.

37.2 Size of specimens

The specimens shall be of suitable size to allow determination of the moisture content to within 0.5 % based on the dry mass of the specimen. Each specimen shall weigh not less than 5 g.

38 Preparation of test specimens

The determination of the moisture content shall be conducted on specimens in a condition given by either or both of the following:

- a) as received;
- b) conditioned to constant mass at 20 °C under one or more of the following conditions: low humidity, medium humidity and high humidity.

39 Procedure

39.1 Moisture content as received

Weigh the specimens as received and subsequently dry to constant mass. Determine the dry mass of the specimen.

For materials having a recommended upper temperature limit of less than 120 °C, dry the specimen in a desiccator. Where the upper temperature limit is 120 °C or over, dry the specimen in a ventilated oven at 105 °C to 110 °C.

39.2 Moisture content after conditioning at low humidity

Condition the dried specimens (see 39.1) to constant mass in an atmosphere of 35 ± 5 % r.h. and at 20 ± 2 °C (see note). Determine the mass of the specimens before and after conditioning.

NOTE If a controlled humidity chamber is not available the required humidity conditions may be obtained approximately by using an airtight vessel containing saturated solutions of salts. Convenient salts with their corresponding relative humidity at 20 ± 2 °C are given below.

Sodium carbonate	90 %
Ammonium nitrate	65 %
Magnesium chloride	35 %

Suitable means of circulating the air in the vessel should be provided. (See NATIONAL PHYSICAL LABORATORY, Notes on applied science, No. 4, Measurement of humidity²⁾)

39.3 Moisture content after conditioning at medium humidity

Condition the dried specimens (see 39.1) to constant mass in an atmosphere of 65 ± 5 % r.h. and at 20 ± 2 °C (see note to 39.2). Determine the mass of the specimens before and after conditioning.

39.4 Moisture content after conditioning at high humidity

Condition the dried specimen (see 39.1) to constant mass in an atmosphere of 90 ± 5 % r.h. and at 20 ± 2 °C (see note to 39.2). Determine the mass of the specimens before and after conditioning.

40 Expression of results

The results shall be given as the moisture content expressed as a percentage by mass based on the dry mass of the specimen or, alternatively, in terms of kilograms of moisture per cubic metre. If:

- m_1 is the mass of specimen as received (in kg);
- m_2 is the mass of specimen after drying to constant mass (in kg);
- m_3 is the mass of specimen after conditioning at the required humidity (in kg);
- a_1 is the moisture content as received (in % by mass);
- b_1 is the moisture content after conditioning (in % by mass);
- a_2 is the moisture content as received (in kg/m³);
- b_2 is the moisture content after conditioning (in kg/m³);

then:

$$a_1 = \frac{m_1 - m_2}{m_2} \times 100,$$

$$a_2 = \frac{m_1 - m_2}{m_1} \times \text{bulk density as received},$$

$$b_1 = \frac{m_3 - m_2}{m_2} \times 100$$

and

$$b_2 = \frac{m_3 - m_2}{m_1} \times \text{bulk density as received}.$$

The temperature and relative humidity to which the sample was exposed shall be stated.

²⁾ Available through the British Library Lending Division, Boston Spa, Wetherby, W. Yorks LS23 7BA.

Section 12. Water absorption

41 General

Methods are specified for testing preformed, flexible and loose-fill materials under conditions of partial immersion and total immersion in water, for resistance to capillarity of man-made mineral fibre batts and slabs, and for determining changes in mass and volume.

42 Specimens

42.1 Number and size

The appropriate number of specimens shall be taken as laid down in Table 2. Their size shall be 150 mm square and preferably not less than 25 mm thick. For the test for capillarity, the thickness shall be not less than 50 mm.

42.2 Preparation of test specimens

Specimens shall be dried as in 39.1.

Plastic composition materials shall be compounded with water, made into block form and dried before being subjected to the test.

Loose, fibrous, granular and powdered materials shall be carefully and evenly packed to a specified bulk density into an open topped, perforated sheet-metal or gauze container, measuring 150 mm × 150 mm × preferably 25 mm internally, fitted with a lid of the same material.

43 Procedure

43.1 Partial immersion

Suspend the specimen, prepared as in 42.2, vertically with one 150 mm × 25 mm side immersed 6 mm below the surface of tap water for 48 h. Maintain the water at constant level by means of a slow constant feed and fixed overflow pipe.

At the end of the period of immersion, suspend the specimen by one corner and allow to drain for 5 min.

When applicable, wipe off the excess surface water with a dry cloth or remove by the careful application of blotting paper. Weigh the specimen immediately.

43.2 Total immersion

Completely immerse the specimen, prepared as in 42.2, in tap water for 2 h³⁾. Immerse the specimen horizontally, with the upper surface approximately 25 mm below the surface of the water.

At the end of the period of immersion, suspend the specimen by one corner and allow to drain for 5 min.

When applicable, wipe off the excess surface water using a dry cloth or by the careful application of blotting paper. Weigh the specimen immediately and measure the length, width and thickness. Note the mechanical condition of the specimen.

When specially required, this test may be supplemented by a test carried out in a similar manner but using boiling water. When this is done, special reference to the condition shall be made in the report.

43.3 Resistance to capillarity

Place the specimens vertically on edge in tap water 6 mm to 10 mm deep containing 0.05 g/L of fluorescence indicator.

NOTE It is convenient to use a water bath with an overflow drain fixed at the required depth; it may be necessary to top up the bath with more fluorescence solution after the specimens have been positioned to replace displaced water, and at intervals during the test period.

Leave the samples standing in the fluorescent solution for 24 h.

Record to the nearest 1 mm, the depth of solution, d_0 , in which the specimens are immersed.

At the end of the 24 h period, remove the specimens and examine each in ultraviolet light.

Record, to the nearest 1 mm, the maximum extent of fluorescence on each specimen, d_1 . Calculate the mean value of the six observations.

43.4 Drying after test

If desired, the specimens may subsequently be dried to constant mass as specified in 39.1 and any change in dimensions or condition reported.

44 Expression of results

The results of the tests shall be expressed as follows.

- a) The increase in mass expressed in kg of water per m² of immersed horizontal surface (partial immersion), or per m³ of dry material (total immersion). For the capillarity test record the extent of capillary absorption for each specimen, $d_1 - d_0$, in mm and the mean extent of capillary absorption for the six specimens in mm.
- b) The bulk density of the specimen before testing.
- c) The condition of the specimen, particularly any signs of deterioration.

Additionally, for the total immersion test, any change in volume (increase or decrease) shall be expressed as a percentage of the dry volume.

³⁾ For materials used in low temperature insulation, the time of immersion shall be six weeks at 15 °C to 20 °C.

Section 13. Water vapour permeance

45 Procedure

Where an assessment of water vapour permeance is required the method of BS 3177 should be used.

BS 3177 specifies two test conditions, i.e. temperate and tropical and the results are expressed as g/m^2 per 24 h at the test condition.

The test conditions and thickness of the test sample shall be stated.

When a thick specimen has to be tested and the apparatus of BS 3177 is unsuitable then reference should be made to method 8 of BS 4370-2 where a beaker test is specified (method for water vapour permeability of expanded polystyrene).

46 Conversion of units

If it is desired to express the results normalized to a standard atmosphere this can be calculated from a knowledge of the vapour pressure at each of the test conditions.

Temperate 25 °C and 75 % r.h. = 0.0234 atmospheres

Tropical 38 °C and 90 % r.h. = 0.0588 atmospheres

The standard atmosphere used for the purposes of this test is 1013.2 mbar⁴⁾ corresponding to a pressure of 760 mmHg of density 13.5951, gravitational acceleration being taken as the standard acceleration, g_n (9.806 65 m/s^2). The vapour pressure of anhydrous calcium chloride is taken as zero.

Table 6 enables the permeance to be expressed in other units. Where the results are converted to a standard atmosphere, or any of the units shown in Table 6, the original test conditions shall be stated because the conversion factors to different units are only applicable to the same test conditions.

⁴⁾ 1 mbar = 0.1 kN/m^2 = 0.1 kPa.

Table 6 — Water vapour permeance conversion factors

	g/(s · MN)	g/(cm ² · s · mbar)	g/(m ² · 24 h · mmHg)	lb/(ft ² · h · atm) (see note 2)	gr/(ft ² · h · mbar) (see note 4)	gr/(ft ² · h · inHg) = 1 perm	Temperate g/(m ² · 24 h)	Tropical g/(m ² · 24 h)
g/(s · MN)	1	1 × 10 ⁻⁸	1.152 × 10	7.471 × 10 ⁻²	5.161 × 10 ⁻¹	1.749 × 10	2.052 × 10 ²	5.149 × 10 ²
g/(cm ² · s · mbar)	1 × 10 ⁸	1	1.152 × 10 ⁹	7.471 × 10 ⁶	5.161 × 10 ⁷	1.749 × 10 ⁹	2.052 × 10 ¹⁰	5.149 × 10 ¹⁰
g/(m ² · 24 h · mmHg)	8.681 × 10 ⁻²	8.681 × 10 ⁻¹⁰	1	6.486 × 10 ⁻³	4.481 × 10 ⁻²	1.517	1.782 × 10	4.472 × 10
lb/(ft ² · h · atm) (see note 2)	1.339 × 10	1.339 × 10 ⁻⁷	1.542 × 10 ²	1	6.909	2.339 × 10 ²	2.747 × 10 ³	6.896 × 10 ³
gr/(ft ² · h · mbar) (see note 4)	1.937	1.937 × 10 ⁻⁸	2.233 × 10	1.447 × 10 ⁻¹	1	3.388 × 10	3.975 × 10 ²	9.980 × 10 ²
gr/(ft ² · h · inHg) = 1 perm	5.719 × 10 ⁻²	5.719 × 10 ⁻¹⁰	6.590 × 10 ⁻¹	4.275 × 10 ⁻³	2.951 × 10 ⁻²	1	1.174 × 10	2.948 × 10
Temperate g/(m ² · 24 h)	4.874 × 10 ⁻³	4.874 × 10 ⁻¹¹	5.613 × 10 ⁻²	3.641 × 10 ⁻⁴	2.515 × 10 ⁻³	8.514 × 10 ⁻²	1	See note 3
Tropical g/(m ² · 24 h)	1.942 × 10 ⁻³	1.942 × 10 ⁻¹¹	2.236 × 10 ⁻²	1.450 × 10 ⁻⁴	1.002 × 10 ⁻³	3.392 × 10 ⁻²	See note 3	1
<p>NOTE 1 To convert units in the first column to the units shown in the heading, multiply by the factor given at the intersection of the appropriate row and column.</p> <p>NOTE 2 This was the term used by the building industry.</p> <p>NOTE 3 No conversions from temperate to tropical are shown for the reasons given in clause 46.</p> <p>NOTE 4 The symbol “gr” refers to grains.</p>								

Section 14. Coarse shot content

47 General

The method for the determination of the coarse shot content of fibrous insulation material consists in separating the shot by vibrating the fibres through a sieve and leaving the coarse shot on top of the mesh.

48 Apparatus

48.1 *Laboratory balance* (accurate to ± 1 mg).

48.2 *Electric muffle furnace* (temperature controlled).

48.3 *Silica or other suitable refractory basin* (90 mm diameter \times 35 mm deep approximately).

48.4 *Sieve*, 200 mm diameter, complying with BS 410: 250 μm aperture, receiver and lid.

48.5 *Mechanical sieve shaker*

48.6 *Three ceramic cylinders*, 19 mm diameter \times 19 mm long, (see note 1 to clause 49).

48.7 *Aluminium foil dish* or other suitable container, approximately 70 mm in diameter.

49 Procedure

Weigh the refractory basin (48.3). Cut a specimen of approximately 10 g from the insulation, preferably from the full thickness, and place in the basin, then weigh the basin and contents.

Place the basin in the furnace at the required temperature and leave for the specified time (see note 3). Remove the basin from the furnace and place on a heat resistant surface to cool for approximately 30 min, then reweigh the basin and contents.

Transfer the specimen to the sieve and place the ceramic cylinders (48.6) in the sieve. Fit the sieve to the receiver and place the lid on the sieve. Place the sieve assembly on the shaker and allow to shake for 30 min. Inspect the material in the sieve; continue shaking if necessary.

NOTE 1 Difficult material may benefit from hand teasing of remaining lumps. Small amounts should be brushed through using a bristle sieve brush.

Weigh the foil dish (48.7), then transfer the separated shot carefully from the sieve to the foil dish.

Reweigh the foil dish and shot.

NOTE 2 Suitable ceramic cylinders are available from Royal Worcester Industrial Ceramics Ltd. under the trade name Regalox Cylpebs.

NOTE 3 Heating mineral wools to a predetermined high temperature aids the sieving process:

a) rock wools should be heated at a temperature of 600 °C for 1 h.

b) glass wools should be heated at a temperature of 500 °C for at least 16 h.

50 Expression of results

Results shall be expressed in the form:

$$\text{Percentage shot} = \frac{\text{mass of shot}}{\text{mass of specimen}} \times 100$$

* Corrected if necessary for loss on ignition.

Section 15. Oil content

51 General

Though applied to the determination of oil in mineral wool, the result as here obtained may include other soluble components, for example, bitumen and certain resins.

If this is so and the oil alone is required, further tests will be necessary.

52 Apparatus

52.1 Soxhlet extraction apparatus

52.2 Sintered alumina thimble

NOTE Thimbles 80 mm high \times 30 mm diameter and of coarse porosity (20 μ m to 30 μ m) are suitable.

Before use, all traces of organic matter due to contamination of the thimble are removed by ignition in a muffle furnace at 600 °C to constant mass.

53 Procedure

Weigh at least 15 g to the nearest 0.1 mg of dried sample into the tared dry thimble ensuring that it is well tamped down (see note 1). Extract with carbon tetrachloride or other suitable solvent (see note 2). Remove the thimble from the soxhlet extractor, drive off the excess solvent in a suitable vented oven, cool in a desiccator and reweigh.

NOTE 1 A large sample is to be preferred in order to obtain improved accuracy in the extraction mass difference. It should be tamped down in order to prevent fine fibres being lost by flotation.

NOTE 2 The number of extraction cycles necessary will depend on the oil content of the wool. Normally six to ten times are sufficient.

54 Expression of results

Obtain the mass of oil by difference and express as a percentage of the original mass of the sample.

Section 16. Organic matter

55 General

Three methods are given; method 1 for moisture and total organic matter; method 2 for low concentrations of carbon in inorganic insulating materials without combined water and/or carbonates; method 3 for determining the concentration of carbon in inorganic insulating materials containing combined water and/or carbonates.

Where doubt exists concerning the value of the content of organic matter, it will be advisable to apply method 1 initially with one sample, followed by method 2 or 3 with a fresh sample, since if method 2 or 3 were to be used initially with excess carbonaceous material, a violent reaction could occur.

56 Method 1. Determination of organic matter

By this method, both moisture and total organic material are normally determined. If, however, thermosetting resins in an uncured or partially cured state are believed to be present, moisture cannot be reliably estimated since in removing it the resin will crosslink, giving rise to a change in mass. Hence, in such cases the sample should be fully cured at an appropriate temperature and time, before any weighing is carried out.

Apart from the foregoing case, any volatile organic materials will be represented in the result as "moisture".

56.1 Apparatus

56.1.1 *Silica dish* or other suitable container.

56.1.2 *Coring tube*, about 10 mm diameter (if used).

56.2 Sampling

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

56.3 Procedure

Weigh not less than 10 g of the sample to ± 0.01 g into a tared dry dish. Dry in a ventilated oven at $105\text{ }^{\circ}\text{C}$ to $110\text{ }^{\circ}\text{C}$ for 40 min, cool in a desiccator and reweigh.

Ignite the sample in a muffle furnace at $550 \pm 50\text{ }^{\circ}\text{C}$ for 10 min. Partially cool, for example on a heat-resistant mat, and then transfer to a desiccator. When cool, reweigh the sample.

56.4 Expression of result

The mass of moisture shall be obtained by difference and shall be expressed as a percentage of the original sample mass.

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

57 Method 2. Determination of the concentration of carbon in inorganic insulation materials without combined water and/or carbonates

The object of this method is to determine the concentration of carbon in inorganic insulating materials. This concentration is calculated from the mass of carbon dioxide produced when a known mass of insulating material is ignited in oxygen at $500 \pm 50\text{ }^{\circ}\text{C}$. The method is applicable only to insulating materials with low concentrations (e.g. less than 1 %) of carbon.

57.1 Apparatus

NOTE The apparatus required is shown in Figure 10.

57.1.1 *Flowmeter* (A).

57.1.2 *Bottle containing soda lime* between two layers of anhydrous magnesium perchlorate to remove moisture and carbon dioxide from the oxygen supply (B).

57.1.3 *Electric furnace with combustion tube* having water cooled ends and a glass inspection inlet. A plug of glass wool in the outlet retains solid particles (C).

57.1.4 *Bottle containing manganese dioxide* to remove sulphur dioxide formed during combustion of sulphur compounds. The inlet and outlet connections are ground glass fittings. They are held in place and a gas tight joint formed by covering the surface with picien wax, not grease (D).

57.1.5 *A tube containing anhydrous magnesium perchlorate* retained between plugs of glass wool to absorb water produced in the ignition (E).

57.1.6 *Bottle containing soda asbestos* with a layer of anhydrous magnesium perchlorate, to absorb the carbon dioxide. It is fitted with a one way stopcock outlet, and is connected to drying tube E by means of a rubber joint, the glass ends being pressed as closely together as possible (F).

57.1.7 *Unglazed porcelain boats*

57.1.8 *Balance* capable of weighing the sample and bottle (F) to 0.1 mg.

57.1.9 *Commercial oxygen* in accordance with BS 4364 to be supplied from a high pressure cylinder through a suitable regulator.

57.2 Procedure

Bring the furnace to the operating temperature and, before the first test, pass oxygen through the apparatus at a rate of 15 L/h for 20 min. Remove bottle F, weigh and reconnect. Repeat until the mass of the bottle is constant to 0.2 mg.

Place at least 0.5 g of the test material in a weighed combustion boat, weigh again and insert into the hot part of the combustion tube. Pass oxygen through at a rate of 15 L/h for 12 min. Remove bottle F, reweigh and reconnect, leaving the apparatus ready for the next test.

57.3 Results

Results shall be expressed as follows.

$$\text{a) Carbon, \%} = \frac{(m_2 - m_1) \times 12/44 \times 100}{S}$$

where

S is the mass of sample;

m_1 is the initial mass of bottle F;

m_2 is the final mass of bottle F.

b) If it is desired to express the carbon content in terms of the equivalent hydrocarbon, the following equation may be used on the assumption that carbon forms 85 % by mass of equivalent hydrocarbon.

$$\begin{aligned} \text{Hydrocarbon, \%} &= \frac{\text{carbon \%} \times 100}{85} \\ &= 1.176 \times \text{carbon \%} \\ &= 32.07 \times \frac{(m_2 - m_1)}{S} \end{aligned}$$

58 Method 3. Determination of the concentration of carbon in inorganic insulating materials containing combined water and/or carbonates

58.1 General

The object of this method is to determine the concentration of carbon in inorganic insulating materials which are known to contain combined water and/or carbonates. This concentration is calculated from the difference between the mass of carbon dioxide produced when a known mass of insulating material is ignited in oxygen at $1\,000 \pm 50$ °C (stage A) and the mass of carbon dioxide produced when an equivalent mass of insulating material is boiled in acid solution (stage B).

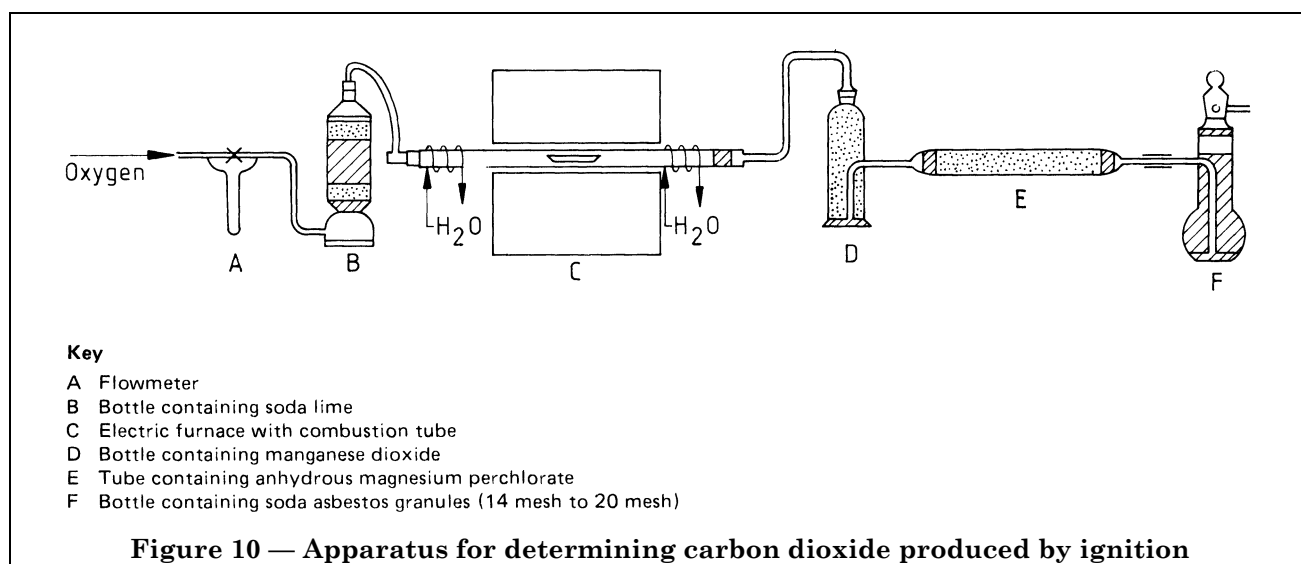
58.2 Stage A

58.2.1 Apparatus. The apparatus used is shown in Figure 10 and consists of the items detailed in 57.1.

58.2.2 Procedure. Weigh not less than 5 g of the sample to ± 0.01 g into a tared dry dish. Dry in a ventilated oven at 105 °C to 110 °C for 2 h, cool in a desiccator and reweigh.

Bring the furnace to the operating temperature and, before the first test, pass oxygen through the apparatus at a rate of 15 L/h for 20 min. Remove bottle F, weigh and reconnect. Repeat until the mass of the bottle is constant to 0.2 mg.

Place at least 0.5 g of the dried test material in a weighed combustion boat, weigh again and insert into the hot part of the combustion tube. Pass oxygen through at a rate of 15 L/h for 20 min. Remove bottle F, reweigh and reconnect, leaving the apparatus ready for the next test.



58.2.3 Results. Results shall be expressed as follows. Carbon and carbonates (expressed as carbon).

$$\% = \frac{(m_2 - m_1) \times 12/44 \times 100}{S}$$

where

S is the mass of sample;

m_1 is the initial mass of bottle F;

m_2 is the final mass of bottle F.

58.3 Stage B

58.3.1 Apparatus

NOTE The apparatus required is shown in Figure 11.

58.3.1.1 U-tube containing soda asbestos granules (14 mesh to 20 mesh) and anhydrous magnesium perchlorate to remove CO₂ from the air entering the apparatus (A).

58.3.1.2 Reaction flask (B).

58.3.1.3 5 % copper sulphate to remove H₂S (C).

58.3.1.4 Empty tube (D).

58.3.1.5 Flake calcium chloride to remove water (E and F).

58.3.1.6 Anhydrous magnesium perchlorate to complete the drying of the gas (G and H).

58.3.1.7 Absorption tubes containing soda asbestos granules (14 mesh to 20 mesh) and anhydrous magnesium perchlorate (I and J).

58.3.1.8 U-tube as in a) above and serving the same purpose if air or moisture should travel back from the pump (K).

58.3.1.9 Safety trap immediately before the filter pump (L).

Use is made of a Davies condenser of 140 mm nominal effective length and joint size either B. 19/26 or B. 24/29⁵⁾ to condense most of the water and hydrochloric acid (if this acid is used).

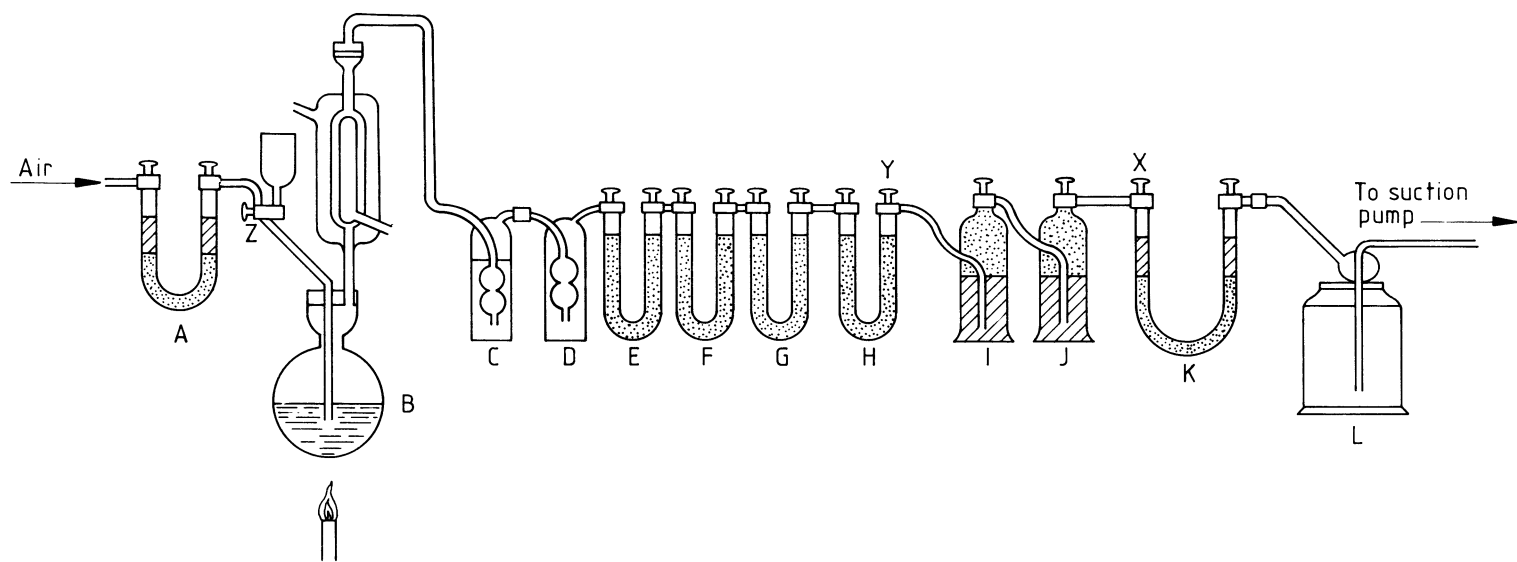
The absorption tubes for CO₂ are of the Midvale Stetser-Norton pattern, and shall be carefully packed. A thin layer of cotton-wool is placed at the bottom of the tube to prevent the end of the inlet-tube from becoming blocked with soda asbestos granules (14 mesh to 20 mesh). The tube is then filled with soda asbestos granules (14 mesh to 20 mesh) up to approximately the point where the inlet-tube passes through the wall of the body of the absorption tube. Care shall be taken to ensure that "channelling" does not take place between the glass and the granules; this may be eliminated by tapping lightly with the finger-nail while packing. Next is placed a thin layer of cotton wool, followed by anhydrous magnesium perchlorate to within 6 mm to 3 mm of the neck, and the anhydrous magnesium perchlorate is covered with a final layer of dried cotton wool.

The second absorption tube is to act as a guard, and to indicate when the first absorption tube is beginning to fail. Both tubes are to be weighed in the course of a determination.

Ensure that all taps and joints are gas tight, applying petroleum jelly where necessary.

58.3.2 Procedure. Transfer at least 0.5 g of the dried sample, accurately weighed, to the flask and cover with water. (It is convenient to use an amount of sample, if possible, which will evolve about 0.2 g CO₂.) Insert the stopper carrying the dropping-funnel and condenser, and connect the latter with C, D, E, F, G and H, but at present *omit* the CO₂ absorption tubes, connect H across to K by inserting an empty U-tube. Connect A to the 2-way tap of the dropping-funnel and pass air that is free from CO₂ through the system for 5 min to 10 min (or until it is judged that all CO₂ has been removed from the apparatus). Close the stopcocks X, Y and Z in that order, remove the empty U-tube between H and K, and insert the weighed absorption tubes I and J.

⁵⁾ See BS 5922.



Key

- A U-tube containing soda asbestos granules (14 mesh to 20 mesh) and anhydrous magnesium perchlorate
- B Reaction flask
- C 5 % copper sulphate
- D Empty tube
- E and F Flake calcium chloride
- G and H Anhydrous magnesium perchlorate
- I and J Absorption tubes
- K U-tube (as A)
- L Safety trap

Figure 11 — Apparatus for determining carbon dioxide produced by boiling in acid

Prepare a quantity of concentrated phosphoric acid, or dilute hydrochloric acid (1 : 1), approximately equal to twice the amount theoretically necessary, turn the tap water on for the condenser, running this steadily, and nearly fill the dropping-funnel with the acid; check again to see that incoming CO₂-free air is connected and instantly available.

Now turn the stopcock in the dropping-funnel so that acid runs down into the reaction flask B. This should be done *slowly* to prevent violent evolution of gas; also the dropping-funnel should not be allowed to empty completely, to avoid entraining air containing CO₂ into the apparatus.

Immediately open the stopcock Y and then without loss of time gradually open the stopcock X, and see that the gas is bubbling steadily and consistently through the train. (The greatest speed should be not more than two to three bubbles per second.)

Continue with the addition of acid if necessary with careful regulation, and then when all the acid has been used (except for a drop left to seal the dropping-funnel), turn the stopcock Z so that CO₂-free air is feeding steadily through the train.

Light the burner under the reaction flask and *slowly* heat the reactants, keeping careful observation and control over the rate of flow of the CO₂ through the train.

Eventually, the system will come to a balanced condition with the solution boiling gently in the flask.

When it is judged that all CO₂ has been boiled out of the solution in the flask, simultaneously decrease the heat and increase the suction with a view to keeping the gas flow rate reasonably steady until the flame is removed altogether and the flask and contents are cooling down. Continue with the air flow until it is judged that all the CO₂ has been swept over to the absorption tubes containing the soda asbestos granules (14 mesh to 20 mesh). Now close the stopcocks X, Y and Z in that order, and open the leading part of the system to atmosphere by disconnecting the flask from its bung. Remove the suction line.

Transfer the absorption tubes to the balance case for 10 min and reweigh.

58.3.3 Results. Results shall be expressed as follows.

a) Carbonates, % (expressed as carbon)

$$= \left\{ \frac{(m_2 - m_1) + (m_4 - m_3)}{S} \right\} \times 12/44 \times 100$$

where

S is the mass of sample (in g);

*m*₁ is the initial mass of absorption tube (I) (in g);

*m*₂ is the final mass of absorption tube (I) (in g);

*m*₃ is the initial mass of absorption tube (J) (in g);

*m*₄ is the final mass of absorption tube (J) (in g).

The difference between the result obtained for stage A and that for stage B represents the "organic carbon" content of the sample.

b) If it is desired to express the carbon content in terms of the equivalent hydrocarbon, the following equation may be used on the assumption that carbon forms 85 % by mass of the equivalent hydrocarbon.

$$\begin{aligned} \text{Hydrocarbon, \%} &= \frac{\text{carbon \%} \times 100}{85} \\ &= 1.176 \times \text{carbon \%} \end{aligned}$$

Section 17. Air permeability

59 General

A test for air permeability is applied in order to give an indication of the relative air flow resistance to convection currents that are likely to occur within the body of an insulant when it is applied to large vertical areas, for example, in the cold box of an air separation plant. The main object of the test is to compare the behaviour of materials for use at very low temperatures, down to 90 K.

60 Apparatus

The apparatus (see Figure 12) consists of a tube of 60 mm internal diameter and 1.2 m long, with pressure tapplings 50 mm from each end. One end is permanently closed and fitted with an air inlet tube, while the other can be sealed by a cap fitted with an air outlet tube. This tube is connected to a suitable flowmeter⁶⁾, which vents to atmosphere. The tube and pressure tapplings may conveniently be made of glass, in which case visual examination of the packed insulant is possible and the uniformity of the packing can be checked. The tube may be marked off in a number of equal lengths to assist in filling it throughout to the prescribed packing density. The end closures may take the form of closely fitting rubber bungs. The pressure tapplings are connected one to each side of a differential manometer capable of measuring differential pressures between 0.1 and 5 mmH₂O. An inclined manometer filled with ethyl alcohol is suitable for this.

61 Procedure

Pack the tube carefully with the calculated mass of the insulating material, inserting small quantities at a time to ensure uniform packing density. If the tube is marked off in equal lengths, each section should be filled with a separately weighed quantity. Connect an air supply and measure the pressure drop for a number of flow rates, the normal range being from 10 mL/min to 1 000 mL/min, corresponding to linear velocities of about 0.2 m/h to 20 m/h, calculated for the empty tube.

62 Expression of results

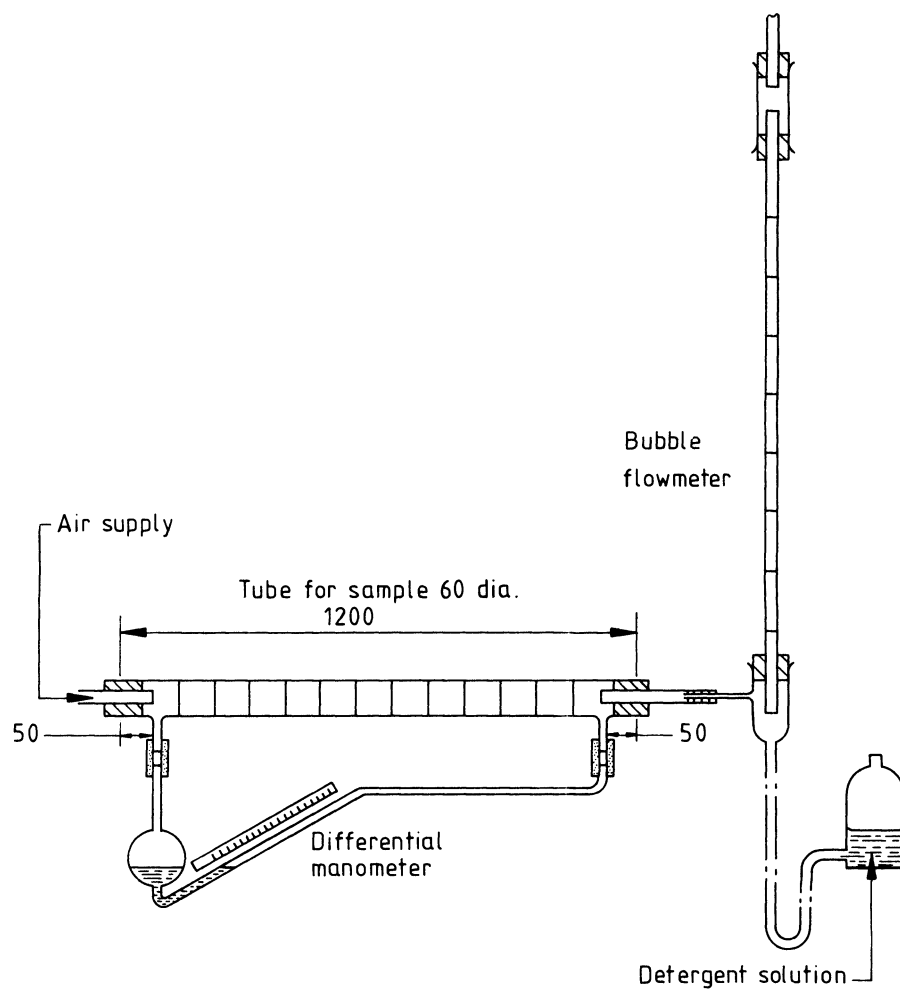
The results shall be expressed as the flow resistance, i.e. the ratio of pressure gradient d_p/L to the linear velocity Q/A , and the linear velocity at which the measurement is made is stated. Any consistent units may be used:

$$\text{Flow resistance} = \frac{d_p A}{L Q}$$

where

- L is the length of tube between pressure tapplings (in mm);
- A is the cross-sectional area of tube (in mm²);
- d_p is the pressure differential (in N/mm²);
- Q is the volumetric flow rate (in mm³/s).

⁶⁾ Separate flowmeters may be required to cover the range 10 mL/min to 1 000 mL/min with sufficient accuracy.



All dimensions are in millimetres.

Figure 12 — Apparatus for measuring air permeability of insulating materials

Section 18. Flexural strength

63 General

This test is designed to give information on the resistance of a thermal insulating material in slab or sheet form to damage from handling and to fracture under its own or an applied weight. Some British Standard specifications for materials already contain details of flexural strength tests differing slightly from that described here and these are not superseded.

64 Apparatus

64.1 Testing machine. Any form of standard testing machine capable of applying and measuring the required load to an accuracy of $\pm 2\%$.

64.2 Bearing edges. Two cylindrical bearing edges of diameter 32 ± 6 mm and length at least 150 mm, rigidly fixed at a distance apart of 250 mm between centres.

NOTE It is convenient if at least one of the bearing edges is adjustable in position but can be rigidly clamped at the required distance(s).

65 Specimens

65.1 Sampling

Sampling shall be carried out in accordance with clause 3.

65.2 Preparation

The two principal faces of each test specimen shall be approximately plane and parallel, at least one face being that of the original sample. Any method of preparation which will not weaken or strengthen the structure of the specimen may be employed.

65.3 Dimensions

Each test specimen shall be approximately 300 mm long and 150 mm wide except that narrower specimens may be used provided that the width is not less than 75 mm.

NOTE With some materials it is advisable to test in two directions at right angles to each other.

65.4 Thickness

Where practicable, test specimens shall be of the same thickness as the original sample, otherwise a thickness of 38 mm shall be used.

65.5 Conditioning

Materials shall be conditioned as follows.

a) *Dry materials.* The test specimens shall be dried in a ventilated oven at $105\text{ }^{\circ}\text{C}$ to $110\text{ }^{\circ}\text{C}$ for 24 h, unless the material has a recommended upper temperature limit below $120\text{ }^{\circ}\text{C}$, in which case drying shall be carried out in a suitably charged desiccator at $50\text{ }^{\circ}\text{C}$ to $60\text{ }^{\circ}\text{C}$.

b) *Material in other conditions.* If results are required on material in other conditions, the procedures of section 11 or 12 should be adopted to ensure that a standard specimen is obtained.

66 Procedure

66.1 Location of specimen

Place the test specimen symmetrically against the bearing edges.

66.2 Test load

Apply the load across the geometric centre line of the test specimen, by means of a third bearing edge, parallel to and of the same dimensions as the fixed bearing edges, moving uniformly at not more than 25 mm/min or at a rate of loading of not more than 2 kN/min.

66.3 Measurement

Continue loading until definite failure occurs and record the applied load in newtons.

NOTE Arrangements may be made for recording, intermittently or continuously, the deformation of the specimen under applied load and representing the test results graphically.

67 Expression of results

The flexural strength shall be calculated as follows:

$$R = 3 \times 10^3 W/l^2 b L^2$$

where

R is the flexural strength (in kN/m^2);

W is the applied load (in N);

l is the distance between supports (in mm);

b is the width of specimen (in mm);

L is the thickness of specimen (in mm).

The speed of the test machine or the rate of loading shall be reported.

Section 19. Fibre diameter

68 General

The purpose of this test is to determine the mean fibre diameter of fibrous insulation products by optical means.

69 Apparatus

69.1 *Optical microscope* having a magnification of $\times 1\ 000$ and measuring graticule.

69.2 *Ultrasonic bath*

69.3 *Mounting medium*

69.4 *Microscope slides and covers*

70 Specimen preparation

The bulk sample of the material shall be representative of the product, and bonded products shall be of the full original thickness. A specimen shall be taken weighing approximately 10 g, and shall be cut through the full thickness of the material at several places over the complete area of the sample. A cork borer is a useful tool for cutting a specimen from bonded products. Specimens of loose-fill totalling 10 g shall be taken from several places in bag or bale of this type of product.

Any original binder in the specimen shall be removed by ignition in a furnace.

NOTE Rock wools should be heated at 600 °C for 1 h. Glass wools should be heated at 500 °C for at least 16 h.

The length of the fibres shall be reduced so that for most of the fibres the whole reduced length is capable of being seen within the field of view of the microscope.

One way of achieving this is by using a metallurgical press capable of applying a pressure of 30 MPa to a 25 mm diameter mould assembly. As much unbonded material as possible is placed in the mould and a pressure of 30 MPa is applied for a period of 10 s. After crushing carefully, the material is removed from the mould.

Whichever method of length reduction is employed, the shortened fibre shall be placed in a clean dry sample bottle fitted with a lid and large enough to hold approximately 12 g of fibre. If necessary the length reduction process shall be repeated until 6 g of fibre have been collected. The sample bottle shall be shaken vigorously to mix the fibres.

71 Slide preparation

80 mL of mounting medium shall be placed in a 125 mL beaker and approximately 0.1 g of the fibre added. The beaker shall be placed in an ultrasonic bath and the mounting medium stirred vigorously with a glass rod for 1 min. While the mounting medium/fibre suspension is still in motion, a drop shall be removed by means of the glass rod and deposited on a glass microscope slide.

A cover slip shall be placed over the drop and the drop allowed to spread for 1 min before a little pressure is applied to the centre of the cover slip.

NOTE 1 A mounting medium of refractive index at least 0.03 less than that of the fibre is recommended. For most purposes a mixture of equal volumes of analytical grade glycerol and distilled water will suffice.

NOTE 2 It is imperative that the sample bottle containing the fibre is shaken vigorously before the 0.01 g specimen is taken, and that the drop be taken from the suspension whilst it is still in motion so that the fibres do not have time to settle.

72 Measurement procedure

Measure the diameters of the shortened fibres at a magnification of $1\ 000 \pm 100$ using an eyepiece graticule. An example of such a device is the "Walton-Beckett" graticule.

Alternatively the images may be projected optically onto a screen or electronically onto a TV monitor, and a rule graduated in millimetres used for the measurement. An eyepiece graticule provides an area, smaller than the total field of view, in which measurements are made. Such a defined area, smaller than the total field, which is projected on a screen, shall be approximately two thirds of the edge length or diameter (see Figure 13).

Place the slide on the microscope stage and position the stage so that the first area to be viewed is in position 1 (see Figure 14). Measure the diameters of all fibres in the defined area of the field, adjusting the focus to scan through the thickness of the mounted specimen on the slide.

When all the fibres in the field of position 1 have been measured, move the stage to position 2. Space position 2 five field diameters from position 1 to avoid the possibility of double counting the same fibre. When all the fibres in position 2 have been measured move the slide systematically until 200 fibre diameters have been recorded. The measurement rules are as follows.

- a) A particle is regarded as a fibre in this test if its length to diameter ratio is 2 : 1 or greater. Particles with a length to diameter ratio of less than 2 : 1 are regarded as debris and ignored.
- b) All fibres with both ends within the defined area are measured.
- c) Fibres with neither end within the defined area are not measured.
- d) Fibres with one end only within the defined area are measured provided that the fibre crosses the bottom or right hand side of a rectangular defined area or crosses the right hand half of the circumference of a circular area. Fibres crossing other parts of the boundary are not measured.

NOTE These rules are illustrated in Figure 13.

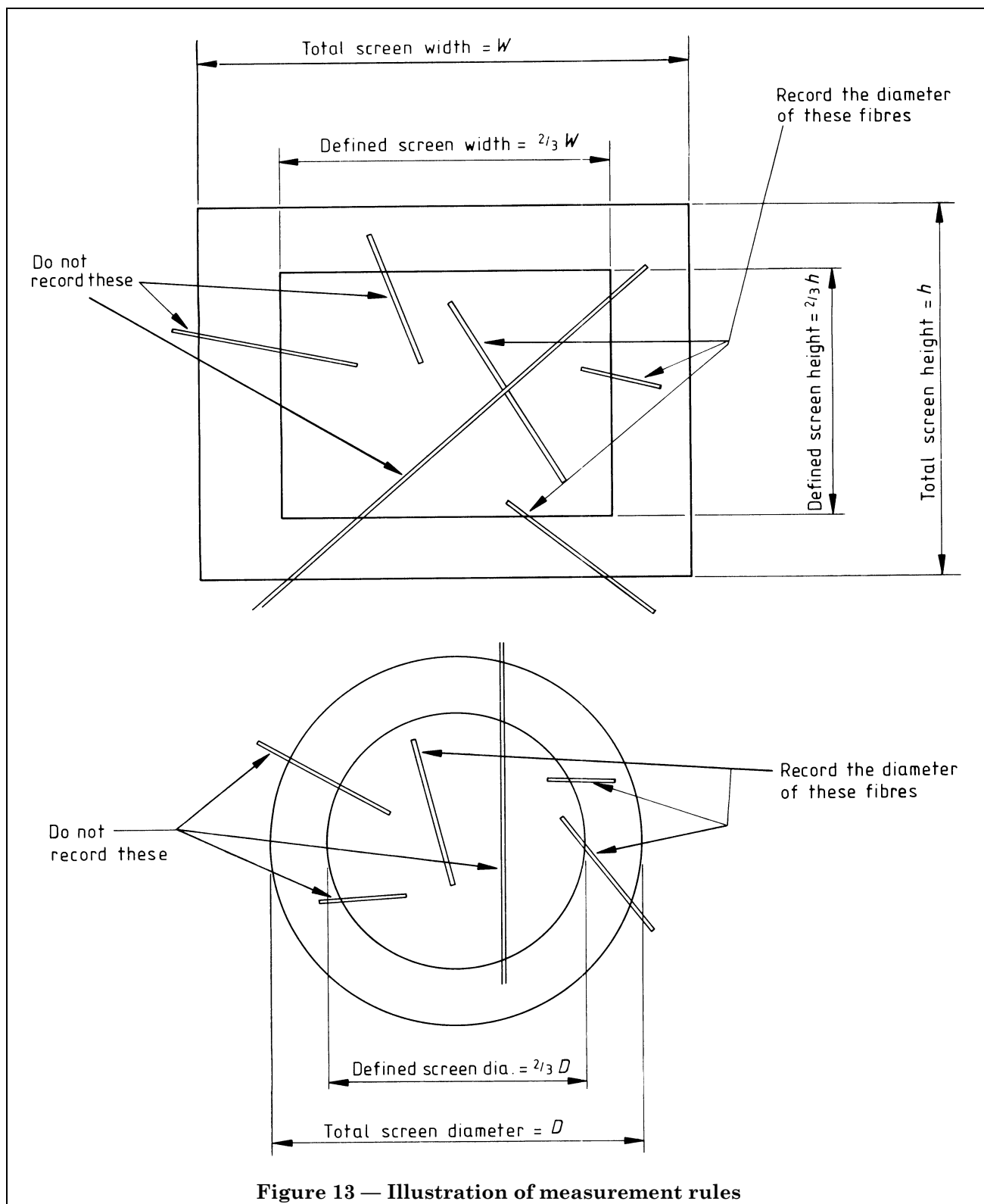


Figure 13 — Illustration of measurement rules

Record the diameters of fibres in 1 micrometre intervals in the following classes: 0 to 2 μm , 2 μm to 3 μm , 3 μm to 4 μm , etc.

73 Expression of results

The mean fibre diameter (\bar{x}) is calculated from the formula:

$$\bar{x} = \frac{\sum f_x}{N}$$

where

x is the central diameter of a given class;

f is the number of fibres in that class;

N is the total number of fibres measured.

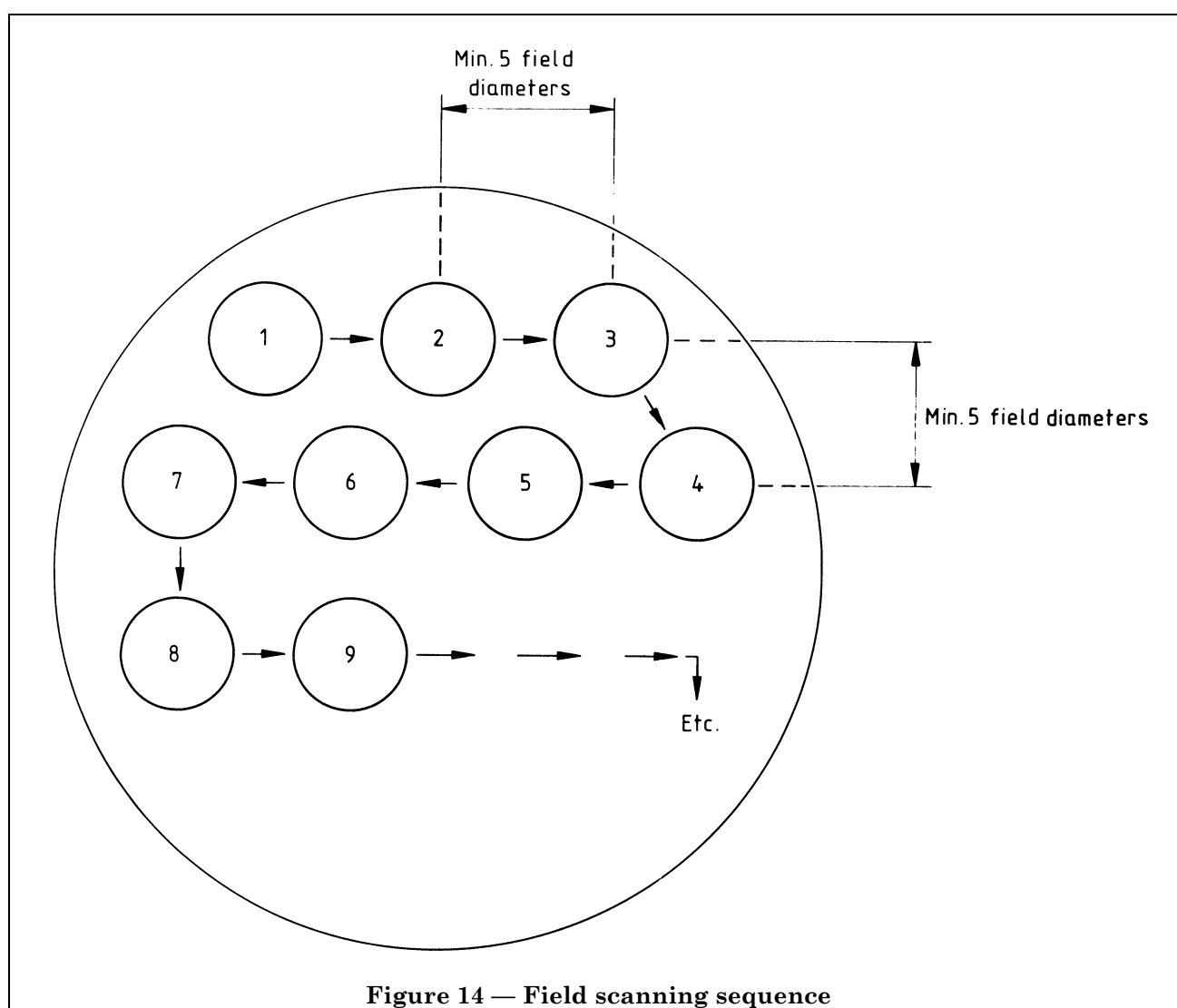


Figure 14 — Field scanning sequence

Section 20. Electrical conductivity of aqueous extract

74 General

In this determination, a single method of preparation of an aqueous extract is described, with precautions to ensure as nearly complete extraction of the water-soluble impurity as possible.

Low-conductivity water is used.

The conductivity of the extract is expressed in microsiemens/m ($\mu\text{S}/\text{m}$).

75 Apparatus

75.1 Glassware, of chemically resistant glass, each item fitted with the same size of ground glass joints, e.g. B.24 size to BS 572. Apparatus complying with British Standards shall be used where appropriate.

The following items are required.

- a) *Flat-bottomed flasks* of capacity approximately twice the volume of extract to be prepared, with a glass stopper incorporating a stopcock. The flasks used for the preparation of extracts shall not be used for any other purpose.
- b) *Efficient water-cooled condensers*.

75.2 Conductivity cell, with a cell constant of about 0.1 cm^{-1} is suitable (electrodes 10 mm diameter, 1 mm to 2 mm apart). The electrodes shall be of blackened platinum.

Each cell shall be calibrated at $20 \text{ }^\circ\text{C}$ with N/100 potassium chloride solution which has a conductivity of $127\,800 \mu\text{S}/\text{m}$ at this temperature.

When not in use the electrodes shall be immersed in distilled water.

75.3 Conductivity meter, consisting of a Wheatstone bridge network of resistors, fed from an alternating source of frequency not less than 1 000 Hz, with audible or visual means of assessing bridge balance. One arm of the network is formed by the cell containing the liquid under test.

76 Conditioning atmosphere

The sample shall be kept for not less than 24 h in an atmosphere of relative humidity $65 \pm 2 \%$ and temperature $20 \pm 2 \text{ }^\circ\text{C}$ (see note to **39.2**).

77 Sample

A sample shall be taken that is representative of the bulk and sufficient to provide all the test specimens that are required.

The sample under test shall be cut into pieces of such size that all parts readily wet out, taking care to handle the material as little as possible.

78 Procedure

78.1 Preparation of extract by hot water extraction

Using low-conductivity water, thoroughly steam out the flasks immediately before use. When the low-conductivity water is refluxed in the flask for 1 h and cooled, its conductivity, measured as described in **78.2**, shall not exceed $200 \mu\text{S}/\text{m}$.

Condition the sample in the standard atmosphere for at least 24 h. Take from the conditioned sample a specimen of convenient mass and transfer it to the flask.

Add to the flask $50 \pm 0.2 \text{ mL}$ water per gram of specimen.

NOTE Some government specifications may require a different water/specimen ratio.

Connect the flask to a reflux condenser, bring rapidly to the boil and continue to boil the liquor gently for 60 min. Disconnect and remove the flask while the liquor is still boiling and close it immediately with the glass stopper fitted with a stopcock. Do not filter or make up to volume, but cool rapidly to $20 \pm 2 \text{ }^\circ\text{C}$. Do not remove or open the tap until ready to make the conductivity measurements.

Reject any extract unless there is a partial vacuum inside the flask immediately before it is opened.

78.2 Measurement of conductivity

After preparing an extract as described in **78.1**, remove the stopper from the flask and transfer some of the extract to the conductivity cell. Wash the electrodes with two or three changes of extract, restopping the flask as soon as possible.

Measure electrical resistance with the meter. Record the temperature of the extract and calculate the conductivity from the formula in **78.3**.

78.3 Expression of results

Calculate conductivity from the following formula.

$$\begin{aligned} & \text{Conductivity at } 20 \text{ }^\circ\text{C} \\ &= \frac{10^8 K}{R [1 + 0.02 (t - 20)]} \mu\text{S}/\text{m} \end{aligned}$$

where

K is the cell constant (in cm^{-1});

R is the measured resistance (in Ω);

t is the temperature of extract (in $^\circ\text{C}$).

78.4 Conductivity of repeat extractions

The extract prepared as described in **78.1** shall be re-extracted a second and a third time; the conductivity of both these extractions shall also be measured as in **78.2**.

The results of each of the three measurements will then provide an indication of the efficiency of extraction; if a large discrepancy still exists between the results of the second and third extractions, it will be necessary to proceed to even further extractions until successive conductivity measurements are sensibly constant.

Section 21. Trace quantities of water-soluble chlorides

79 General

The purpose of this test is to determine trace quantities of water-soluble chlorides in thermal insulating materials. It involves extraction with de-ionized water and the measurement of chloride concentration in the aqueous extract. Two alternative methods of measurement are described.

- a) **Method 1:** A colorimetric method substantially in accordance with BS 2690-6, but with some modifications in the reagents.
- b) **Method 2.** A polarographic titration method with amperometric indication.

80 Method 1

80.1 Apparatus for extraction

80.1.1 *Standard soxhlet extractor* as described in BS 2071, with a flask and condenser of the requisite size.

80.1.2 *Thimble* complying with one of the following requirements⁷⁾:

- a) glass with a sintered base (grade 3 to 4);
- b) wholly sintered glass (grade 3 to 4);
- c) cellulose⁸⁾.

80.2 Extraction procedure

Ensure that all apparatus is free from chloride and provide a supply of de-ionized water as described in BS 2690-6.

Accurately weigh about 5 g of the sample of insulating material into the extraction thimble.

Extract until no further chloride can sensibly be detected, using approximately 200 mL of de-ionized water. (An extraction time of about 5 h is normally sufficient.)

Transfer the extract to a standard 250 mL one-mark volumetric flask (see BS 1792) and make up to the mark with de-ionized water.

80.3 Colorimetric estimation procedure

80.3.1 *Apparatus.* A visual range spectrophotometer is required.

80.3.2 *Reagents* (analytical grades). The following reagents are required.

- a) Sodium chloride.
- b) 0.25 M ferric ammonium sulphate solution. (121 g ferric ammonium sulphate in 270 mL of concentrated nitric acid of relative density 1.42). Dilute to 500 mL with de-ionized water.
- c) Saturated mercuric thiocyanate in 95 % ethanol.

NOTE Mercuric thiocyanate is highly toxic and may be absorbed through the skin.

80.3.3 *Procedure A. Preparation of calibration curve.* Weigh accurately 0.0412 g of dried sodium chloride, transfer to a 1 L one-mark volumetric flask with de-ionized water, dissolve and dilute to the graduation mark.

Using a 10 mL burette, add 0 (blank sample), 2, 4, 6, 8 and 10 mL of the stock chloride solution to six one-mark volumetric flasks to 50 mL capacity.

Add 4 mL of the 0.25 M solution of ferric ammonium sulphate to each of the six samples, then add 4 mL of the saturated solution of mercuric thiocyanate to each of the six samples. Dilute to the graduation mark with de-ionized water, and allow to stand for 15 ± 1 min.

Measure the absorption of each of the ferric thiocyanate complex solutions against the blank sample at the peak absorption wavelength (approximately 460 nm, but the exact wavelength shall be checked for each spectrophotometer).

Prepare a calibration curve for absorption versus milligrams of chloride. This calibration covers the range zero to 0.05 %.

80.3.4 *Procedure B. Determination of chloride.* By means of a pipette, introduce 25 mL of the extract solution into a clean one-mark volumetric flask of 50 mL capacity.

Add 4 mL of the 0.25 M solution of ferric ammonium sulphate to the flask then add 4 mL of the saturated solution of mercuric thiocyanate. Dilute to the graduation mark with de-ionized water and allow to stand for 15 ± 1 min.

Measure the absorption at peak absorption wavelength against a freshly prepared blank sample.

Read off the chloride concentration from the calibration curve.

Calculate the percentage chloride (Cl') content based on the mass of the original sample of insulating material.

80.3.5 *Method of calculation.* If

- x is the mass of original sample of insulating material (in g);
- y is the mass of chloride as determined from the calibration curve (in mg);
- F is the dilution factor (10 as above);

then:

$$\text{content of chloride } (Cl') = \frac{100 yF}{1000 x} = \frac{y}{x} \%$$

⁷⁾ See BS 1752.

⁸⁾ Cellulose thimbles contain measureable amounts of chloride and thus in addition necessitate a blank determination.

81 Method 2

81.1 Apparatus for extraction

81.1.1 *Wide-necked round bottomed flask*, of 1 000 mL capacity.

81.1.2 *A Dimroth type condenser*, jacket length 250 mm.

81.1.3 *An electric heating mantle*, of 1 000 mL capacity.

81.2 Extraction procedure

Ensure that all apparatus is free from chloride and provide a supply of de-ionized water.

Accurately weigh 20 ± 1 g of sample into the 1 000 mL flask. Add approximately 350 mL of de-ionized water, connect the Dimroth condenser and boil under reflux conditions for 30 min. Allow to cool to ambient temperature and transfer the extract to a 500 mL glass beaker.

Acidify the extract by adding 1 mL of 1 : 1 nitric acid, stir and filter through an inert membrane filter (pore size 0.45 μm). If interfering substances are present (see **81.4**) add 0.5 g activated carbon (analytical grade) to the acidified extract, stir vigorously and filter off over an inert membrane filter (pore size 0.45 μm).

Transfer the extract to a 500 mL one-mark volumetric flask and make up to the mark with de-ionized water.

81.3 Determination of chloride ions

81.3.1 *General*. The chloride ions are determined by polarographic titration using the alternating current voltage method with amperometric indication. A weak a.c. voltage of constant amplitude is applied to two amalgamated silver electrodes immersed in the titration solution containing chloride ions producing a specific state of polarization at the electrode surface. The polarization resistance at the electrodes is dependent on the concentration of the chloride ions which react on titration with silver nitrate solution. As the titration nears its end point, the polarization resistance increases considerably, reaching a maximum at the end point.

If a constant a.c. voltage is maintained at the electrodes, any change in the polarization resistance is indicated by a corresponding current variation.

81.3.2 Apparatus

NOTE Other forms of polarographic equipment may be suitable.

81.3.2.1 *Schmidt Eltimeter* with control and measuring devices.

81.3.2.2 *Titration vessel* (glass).

81.3.2.3 *Micro burette*, of 5 mL or 10 mL capacity.

81.3.2.4 *Magnetic stirrer*

81.3.2.5 *Silver double electrode* (e.g. Schott 9004-21).

81.3.3 Reagents (analytical grades)

81.3.3.1 *Silver nitrate solution*, 0.1N.

81.3.3.2 *Silver nitrate solution*, 0.01N.

81.3.3.3 *Sodium chloride solution*, 0.1N.

81.3.3.4 *Sodium chloride solution*, 0.01N.

81.3.3.5 *Nitric acid*, 2N.

81.3.3.6 *Nitric acid*, 1 : 1 (v/v).

81.3.3.7 *Mercury (II) nitrate* [$\text{Hg}(\text{NO}_3)_2$].

81.3.3.8 *Etching solution*, 1.7 g [$\text{Hg}(\text{NO}_3)_2$] dissolved in 50 mL 2N nitric acid.

81.3.3.9 *Mercury* (polarographic quality).

81.3.4 Pre-treatment of the electrodes

81.3.4.1 *Amalgamation of the silver double electrode*. Immerse both electrode rods in the etching solution for 15 s. Wash with de-ionized water, and dry carefully with filter paper. Immediately dip the electrode rods in pure mercury for approximately 30 s. Remove any excess mercury by light tapping. The electrode rods should have a bright metallic appearance. The layer of amalgam is sufficient for 15 to 20 titrations with 0.01N AgNO_3 .

If there is doubt as to the condition of the electrodes a test titration should be undertaken with a solution of 5 mL 0.01N NaCl in 100 mL de-ionized water with 0.01N AgNO_3 . If the titration is slow and the end point not clearly defined then the electrode needs to be reamalgamated.

When not in use the electrode should be stored in de-ionized water.

81.3.4.2 *Activation of the amalgamated silver electrode*. Activation should be undertaken before the first chloride determination, after each new amalgamation or if the electrode has not been used for several hours.

The electrodes should be activated in a titration with 0.01N AgNO_3 . Titrate a solution of 5 mL 0.1N NaCl and 100 mL de-ionized water with 0.1N AgNO_3 to the end point. Newly amalgamated electrodes should be left in the titrated solution for 30 min and then rinsed with de-ionized water before an actual titration.

81.3.5 Chloride titration procedure. Place 200 mL of the nitric acid extract together with a magnetic stirring rod into the titration vessel.

Add 2.0 mL 0.01N sodium chloride to aid detection of the end point. Immerse the double silver electrode as deeply as possible into the solution and away from the inlet of the titrant. Switch on the instrument and magnetic stirrer. Adjust the micro-ammeter needle to the centre of the scale and when the needle has steadied titrate the solution with 0.01N AgNO₃. The end point is reached when the needle moves clearly to the left.

Determine the blank value with 2.0 mL 0.1N NaCl solution:

$$\text{mL test solution} - \text{mL blank value} = x \text{ mL}$$

81.3.6 Expression of results. Calculate the chloride content from the following expression:

$$\frac{500 \times F}{200 E} = \text{mg Cl/kg}$$

where

E is the mass of insulating material (in g);

F is the stoichiometric factor for calculating chloride content (in mg/kg) = 355 when using 0.01N AgNO₃ solution.

NOTE Outlying results (statistical outliers) should be determined using Dixon's test as described in clause 13 of BS 5497-1:1987. If more than one outlier is found, the chloride determination has to be repeated.

81.4 Impurities and interferences

As a check on the titration result add a known volume of 0.01N NaCl solution to the completely titrated test solution. Titrate with 0.01N AgNO₃. This titration has to give an identical volume of AgNO₃ to the NaCl volume that was added. If the volume of AgNO₃ is less than the volume of NaCl added then the titration is incorrect because of the presence of impurities. In this case it is essential that pretreatment as in 81.2 is carried out.

Section 22. pH value of water extract

82 Preparation of sample

From the bulk sample, taken in accordance with clause 3, cut five pieces, each of approximate mass 5 g, from separate units where possible. Disintegrate these pieces and mix thoroughly.

83 Determination of pH value of water extract

Weigh 2 g of the prepared sample and shake well for 10 min with 100 mL of distilled or de-ionized water (pH 6.5 to 7.5) at room temperature. Leave to settle for 5 min and decant the solution if necessary. Measure the pH of the mixture using a standard pH meter as specified in BS 3145. Repeat the test on a further 2 g of the sample and report both values.

Publications referred to

- BS 410, *Specification for test sieves.*
- BS 476, *Fire tests on building materials and structures.*
- BS 572, *Specification for interchangeable conical ground glass joints.*
- BS 874, *Methods of determining thermal insulating properties.*
- BS 903, *Methods of testing vulcanized rubber.*
- BS 903-A19, *Accelerated ageing tests.*
- BS 1752, *Specification for laboratory sintered or fritted filters including porosity grading.*
- BS 1792, *Specification for one-mark volumetric flasks.*
- BS 1902, *Methods of testing refractory materials.*
- BS 1902-6, *Ceramic fibre products.*
- BS 2071, *Specification for Soxhlet extractors.*
- BS 2690, *Methods of testing water used in industry.*
- BS 2690-6, *Chloride and sulphate.*
- BS 3145, *Specification for laboratory pH meters.*
- BS 3177, *Method for determining the permeability to water vapour of flexible sheet materials used for packaging.*
- BS 3533, *Glossary of thermal insulation terms.*
- BS 3958, *Thermal insulating materials.*
- BS 3958-1, *Magnesia preformed insulation.*
- BS 3958-2, *Calcium silicate preformed insulation.*
- BS 3958-3, *Metal mesh faced man-made mineral fibre mattresses.*
- BS 3958-4, *Bonded preformed man-made mineral fibre pipe sections.*
- BS 3958-5, *Specification for bonded man-made mineral fibre slabs.*
- BS 4364, *Specification for industrial oxygene.*
- BS 4370, *Methods of test for rigid cellular materials.*
- BS 4370-2, *Methods 6–10.*
- BS 5497, *Precision of test methods.*
- BS 5497-1, *Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*
- BS 5803, *Thermal insulation for use in pitched roof spaces in dwellings.*
- BS 5803-1, *Specification for man-made mineral fibre thermal insulation mats.*
- BS 5803-2, *Specification for man-made mineral fibre thermal insulation in pelleted or granular form for application by blowing.*
- BS 5922, *Specification for glass condensers for laboratory use.*
- PD 6467, *Arrangement of vibration tester.*
- ASTM-C351-82, *A test method for the specific heat of thermal insulation.*
- National Physical Laboratory, Notes on Applied Science, No. 4, Measurement of humidity.

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