

METHODS OF TESTING REFRACTORY MATERIALS

Part 1A. Sampling and physical tests

DECLARED
OBSOLESCE
JUL 1987

B.S. 1902 : Part 1A : 1966.

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THIS BRITISH STANDARD, having been approved by the Refractory Products Industry Standards Committee and endorsed by the Chairman of the Engineering Divisional Council, was published under the authority of the General Council on 6th April, 1966.

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The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

In order to keep abreast of progress in the industries concerned, British Standards are subject to periodical review. Suggestions for improvements will be recorded and in due course brought to the notice of the committees charged with the revision of the standards to which they refer.

A complete list of British Standards, numbering over 4000, fully indexed and with a note of the contents of each, will be found in the British Standards Yearbook. The B.S. Yearbook may be consulted in many public libraries and similar institutions.

This standard makes reference to the following British Standards:

- B.S. 410. Test sieves.
- B.S. 458. Xylenes.
- B.S. 600. Application of statistical methods to industrial standardization and quality control.
- B.S. 733. Density bottles.
- B.S. 1610. Methods for the load verification of testing machines.
- B.S. 2701. Rees-Hugill powder density flask.
- B.S. 3130. Glossary of packaging terms.

British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. It is important that users of British Standards should ascertain that they are in possession of the latest amendments or editions.

The following B.S.I. references relate to the work on this standard:
Committee reference RPE/1
Draft for comment D63/10770, D64/6458, D65/3597

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CO-OPERATING ORGANIZATIONS

The Refractory Products Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government department and scientific and industrial organizations:

- *British Ceramic Research Association
- British Chemical Plant Manufacturers' Association
- *British Iron and Steel Federation
- Chemical Industries Association
- Coke Oven Managers' Association
- Combustion Engineering Association
- *Council of British Manufacturers of Petroleum Equipment
- Engineering Equipment Users' Association
- *Gas Council
- Glass Manufacturers' Federation
- Institution of Gas Engineers
- *Ministry of Defence, Navy Department
- *National Federation of Clay Industries
- Society of British Gas Industries
- Society of Furnace Builders
- *Society of Glass Technology
- *Water-tube Boilermakers' Association

The Government department and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

British Cast Iron Research Association
British Investment Casters' Technical Association
Electricity Council, the Generating Board and the Area Boards
in England and Wales
Institute of Petroleum

BRITISH STANDARD

METHODS OF TESTING REFRACTORY MATERIALS

Part 1A. Sampling and physical tests

FOREWORD

The issue of B.S. 1902 published in 1952 was largely based on a series of tentative test methods drawn up by the Testing Committee of the British Ceramic Research Association. During the intervening period the Testing Committee, its Analytical Sub-Committee and their Working Groups have carried out co-operative investigations of many of the test methods and it has been considered desirable to revise these in the light of their findings and recommendations. To facilitate this revision and to allow appropriate groups of experts to prepare the various sections, the standard has been divided into two parts, Part 1 dealing with sampling and physical tests, Part 2 dealing with chemical analysis.

The original (1952) standard was concerned only with the chemical analysis of high silica and aluminosilicate materials and with methods of physical testing of general applicability to all classes of refractory materials; the new standard, however, will lay down analytical procedures and test methods for each of the main classes of refractory material. Part 1 and Part 2 of the new standard will therefore be subdivided and the subdivisions, each applicable to certain classes of material, will be published separately as they are completed.

Part 1A, on sampling and testing of refractories, deals with methods of general application (see list of contents, page 3). Appendix A describes the determination of volume with a mercury balance. Appendix B gives a method for determining resistance to thermal shock and Appendix C a method for determining resistance to abrasion, neither of which can yet be accepted as a standard method.

Work is now in hand on the physical testing of basic refractories and of mouldable and castable refractories, as well as on the chemical analysis of basic refractories. Subdivisions dealing with these fields will be published in due course.

The subject of classification is being studied separately, in view of the work now in progress in ISO/TC 33, 'Refractories', on that subject, as it may prove desirable for a detailed presentation of the U.K. views on this matter to be submitted in the form of a new British Standard. Similarly, several of the

chemical and physical methods of test in the revised B.S. 1902 will provide a basis for international discussion and their adoption will facilitate the work of the U.K. representatives in ISO/TC 33.

The testing of refractory materials presents many difficulties and the work should be carried out only by those having the necessary experience in the use and maintenance of the apparatus employed.

NOTE. Where British units are stated, these are to be considered as the standard figure; the metric conversions are approximate. More accurate conversions can be made by reference to B.S. 350, 'Conversion factors and tables'.

SAMPLING FOR INDIVIDUAL TESTS

5. *a. Porosity.* Because porosity results may be required for various purposes, it has been found advantageous to divide the sampling problem into three parts.

(i) *Sample is a single brick.* At least three test pieces shall be taken. The positions from which they are to be cut will depend on the information required.

(ii) *Determination of mean of batch.* At least three test pieces shall be taken, one from each of three bricks. Two pieces shall be from outside positions and one from the interior. Where possible a larger sample consisting of test pieces from each of six bricks should be taken, four of the pieces being from the outside of the bricks and two from the interior. The porosity of the batch shall be reported as the average of the three or six determinations.

(iii) *Compliance with specified extreme limit.* Six pieces shall be taken as described in Subclause 5a(ii) and shall be considered as the first sample of six in the scheme for compliance with the limit *L*. If necessary, a second sample of six test pieces to complete the double sampling test shall be taken in the same way, using another set of six bricks, and the batch shall be judged on the combined sample results as described in Subclause 4d.

NOTE. The selection of test pieces advised here means that the sampling of positions within a brick is systematic rather than strictly random. However, since the variation in porosity between bricks is likely to be markedly greater than that within bricks, the use of the formula applicable to random variation is not likely to lead to serious errors.

b. Permanent dimensional change on reheating. If the results for a particular brick are in question, the values for the test pieces cut as described in Section Eight should be recorded, together with their average.

If acceptance of a batch of bricks depends on the average result for the three directions of cutting, the batch being accepted if the average does not exceed a specified value, it is inadvisable to rely on test pieces from only one brick. Unless the sample size is fixed by the user, it is recommended that four bricks should be taken and three test pieces cut from each as described in Section Eight. The results for the separate test pieces should be recorded together with their average. If the average does not exceed the specified value, accept the batch; if it does, take four more bricks and cut three test pieces from each as before. The results for the separate test pieces should be recorded, together with the average for the combined samples. If this average does not exceed the specified value, accept the batch; otherwise reject it.

c. Cold crushing strength. It is specified in Section Nine that at least six test pieces shall be taken and that, where possible, each test piece shall be cut from a different brick.

Where the user requires that the sample average shall not fall below a specified value, the following procedure should be adopted. Measure the strengths of pieces from six bricks; if the average strength does not fall below the specified value, pass the batch. If not, take test pieces from another six bricks for test and determine the average of the twelve results. If it does not fall below the specified value, pass the batch. Otherwise reject it.

Where compliance with a specified extreme limit is in question, a suitable procedure is the double sampling scheme described in Subclause 4d; each sample is of six test pieces, i.e. $n = 6$, and the specified limit L is here a lower limit.

SECTION FIVE: DETERMINATION OF PERMEABILITY TO AIR

(Unsuitable for materials of permeability greater than 1.0*)

DEFINITION

12. The permeability of a material to a gas is defined as the rate of flow of gas in millilitres per second through 1 square centimetre of the material under a pressure gradient of 1 centimetre head of water, per centimetre thickness. The method described measures the permeability to air at pressures near to atmospheric pressure. It should be noted that permeability and porosity are not directly related; permeability, unlike porosity, is a directional property.

METHOD OF TEST

13. *a. Apparatus.* The main item of apparatus is shown in Fig. 1. Water from a constant head supply flows into a metal cylinder and displaces air which passes through the test piece at a pressure indicated by a water manometer. The water flows first into an internal perforated metal tube, which eliminates ripples on the water surface when the flow is dropwise. The volume of air displaced in a measured time is indicated on a water-level tube at the side of the cylinder.

The complete assembly is shown in Fig. 3.

b. Test material. The air-dried test piece shall be of square or cylindrical section, at least 5 cm high where the brick allows and at least 20 cm² in cross section. It shall be cut from the brick in such a manner that the height of the test piece is parallel to the direction in which the permeability is required to be known. It shall be stated in the report whether any 'skin' is included in the test piece and whether such a 'skin' is perpendicular to the direction of gas flow.

c. Procedure. The test piece shall be fixed in a holder, which shall be designed to ensure that the sides of the test piece are completely sealed and that the whole of the upper and lower surfaces are exposed for the free passage of air. A suitable holder is shown in Fig. 2. The metal cylinder shall then be emptied by means of the drainage tap and water shall be admitted from the constant-head supply. When the manometer reading has become steady, the rate of flow shall be measured by noting, to the nearest second, the time required for the water level in the side tube to rise from one chosen graduation to another. These graduations shall previously have been calibrated in terms of the volume of air displaced, by pouring water into the cylinder through the inlet tube from a 100 ml graduated flask and noting the levels corresponding to 100 ml incre-

* For permeabilities above 1.0 the method recommended by the American Foundrymen's Society (Foundry Sand Handbook, 1963) is suitable.

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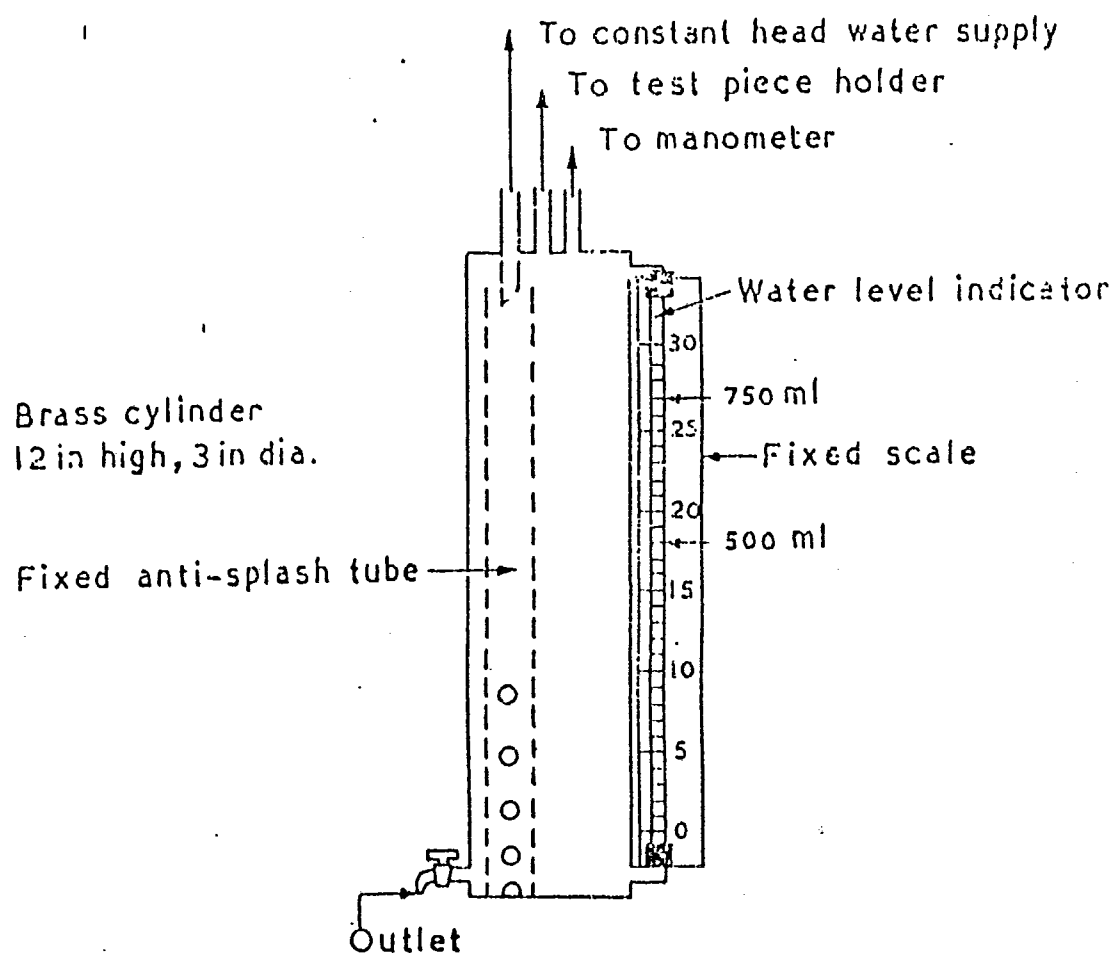


Fig. 1. Displacement cylinder for permeability apparatus

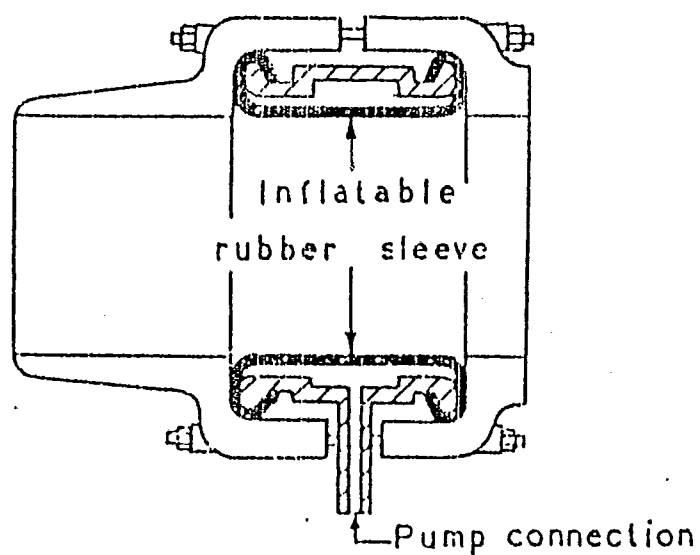


Fig. 2. Example of suitable test piece holder
for cylindrical specimens

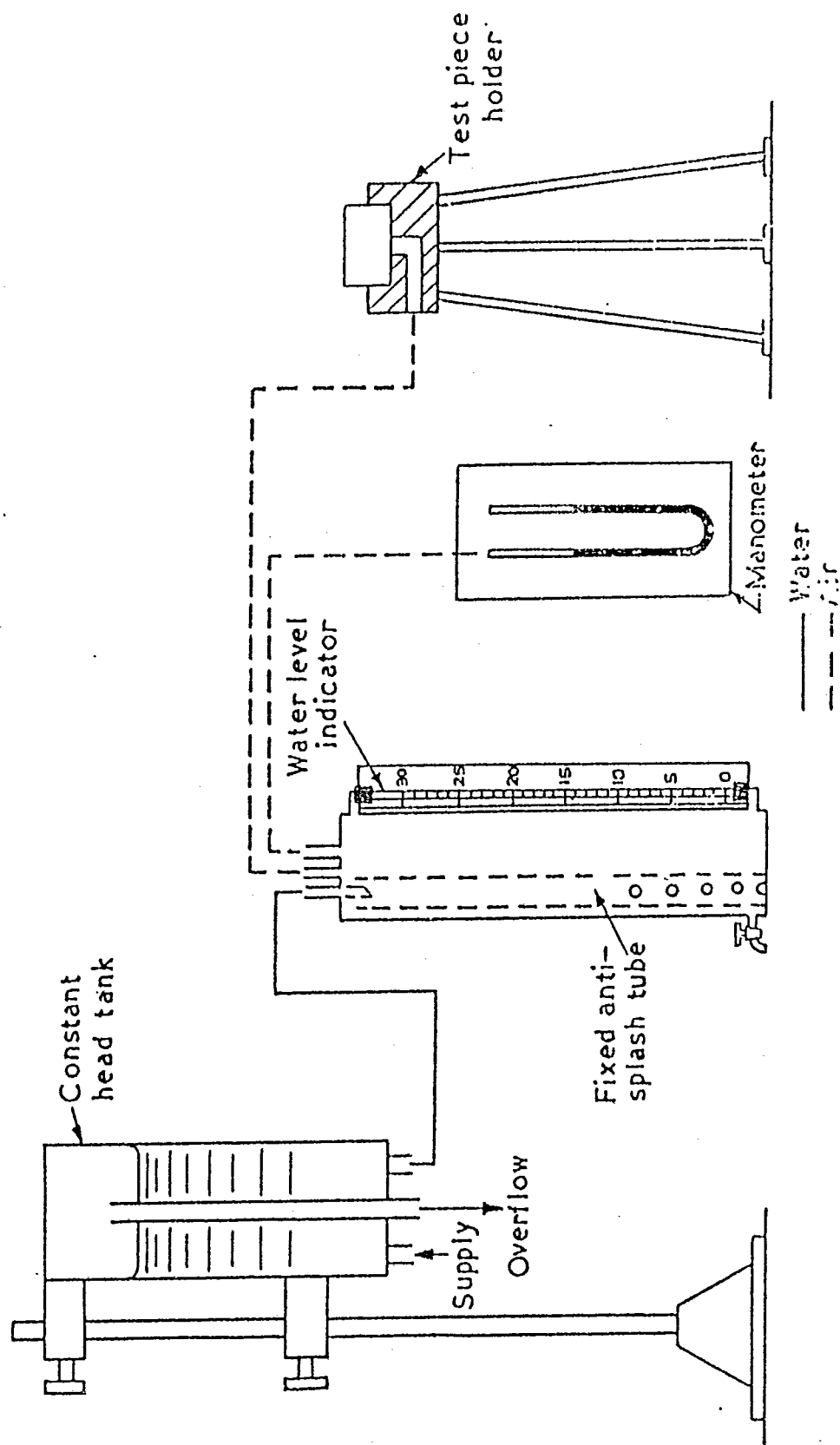


Fig. 3. Assembly of permeability apparatus

ments. With test pieces of medium or high permeability, the volume of water run into the cylinder should be about 500 ml or 600 ml, but with low-permeability pieces water shall be run in for at least six minutes or until at least 100 ml has entered the cylinder, whichever is the longer period. The rate of flow with a given test piece can be adjusted by raising or lowering the constant-head device, but the pressure registered on the manometer shall in no case exceed 20 cm.

During the timed period the pressure difference registered on the manometer shall be read every half minute (see Note); the average value of the pressure difference shall be calculated.

NOTE. The apparatus operates in principle at constant pressure, but a slight fall in pressure during the timed period will often be noticed. This is due to cooling of the cylinder by the entering water, which will usually be below room temperature. If a supply from an internal storage tank is available, this should be used in preference to the mains supply.

CALCULATION

14. If V = volume of air passed through the test piece (ml),
 H = height of test piece (cm),
 T = time of flow (seconds),
 A = area of cross section of test piece (cm²),
 P = pressure head under which the air has passed (cm of water),

$$\text{the permeability* of the sample} = \frac{V \times H}{T \times A \times P} \text{ units.}$$

* This value is in arbitrary units and may be converted to c.g.s. units by dividing by 981.

SECTION SEVEN: DETERMINATION OF REFRACTORINESS UNDER LOAD (R.U.L.)

GENERAL

17. This method of test describes the procedure for the determination of the high temperature deformation of refractory materials when subjected to a specified compressive load. Two forms of the test are described:

TEST A. *Rising temperature test.* This involves heating the loaded specimen at a prescribed rate until either collapse or a specified amount of deformation occurs.

TEST B. *Maintained temperature test.* In this test, the loaded specimen is heated at a prescribed rate to a predetermined temperature which is maintained either for a specified time or until collapse (or a predetermined deformation) occurs.

Clause 18 describes the procedure for both forms of the test.

METHOD OF TEST

18. a. Apparatus.

(i) *General.* The apparatus shall comprise a suitable furnace and loading device. If the furnace is fired by oil or gas, the burners shall fire tangentially to the test piece. Means shall be adopted to prevent flame impingement on the test piece and thermocouples.

The furnace shall be capable of being heated at a controlled rate with an oxidizing atmosphere and the apparatus shall be fitted with a suitable device for applying a predetermined load to the test piece.

(ii) *Measurement of deformation of test piece.*

TEST A. *Rising temperature test.* The length changes to be reported in the rising temperature test are those of the test piece only, i.e. they shall not include the length changes of the loading and supporting columns.

either A recording or indicating system shall therefore be fitted which measures the length change of the test piece by the differential (dilatometer) method described in Clause 19;

or measures by means of a lever or other magnifying device the movement of the top of the loading column, in which case a correction shall be applied as described in Clause 20.

The recording or indicating system shall be capable of detecting a change in the length of the test piece or the height of the loading column of 0.001 in or less.

TEST B. *Maintained temperature test.* The length changes to be reported in the maintained temperature test are those measured at the top of the loading column by the indicating system of the apparatus.

A recording or indicating system shall therefore be fitted which records the movement of the top of the loading column by means of a lever or other magnifying device and is capable of detecting a movement of 0.001 in or less.

NOTE. It is recommended that the dilatometer described in Clause 5 should not be used in a maintained temperature test until further information is available.

(iii) *Conditions of temperature uniformity.* The uniformity of heating shall be such that the highest and lowest temperatures indicated by the four thermocouples attached to the sides of the test piece, as described in Subclause b(i), shall not differ by more than 25 degC at any time after the mean surface temperature has reached 1000°C.

The length of the hot zone shall be such that, in a rising temperature test, the temperatures at the top and the bottom of the test piece, measured as indicated in Subclause b(ii), shall comply with the conditions laid down in Table 1 for the heating schedule employed.

In a maintained temperature test the temperature at the top or bottom of the test piece shall not differ from the mean surface temperature by more than 25 degC at any time during the soaking period and the mean of the top and bottom temperatures shall not differ from the mean surface temperature by more than 20 degC during this period.

NOTE. If the above conditions of temperature uniformity cannot be met, a rotating or rotating and reciprocating tube should be fitted (see References 1 and 2 at the end of this Clause).

b. Temperature measurement.

(i) *Surface temperature.* The temperature to be used in controlling the test and in reporting the results shall be the mean surface temperature of the test piece. It shall be measured by means of four platinum v. platinum rhodium thermocouples, attached one on each $2\frac{1}{2}$ in by $1\frac{3}{4}$ in face of the test piece if this is a prism, or one at each end of two diameters mutually at right angles if the test piece is cylindrical (see Subclause 13d). The couples shall be parallel to the principal axis of the test piece and their junctions shall be at the mid-point of each face or at the mid-height of a cylindrical test piece. They shall be fitted with pure alumina insulators but not sheathed and shall be tied to the test piece with one or more strands of platinum/rhodium wire* of about 0.5 mm diameter. To ensure that the hot junction of each thermocouple is sensibly at the same temperature as the surface of the test piece, the junction should be tucked under

* Pure platinum wire is of insufficient tensile strength.

the end insulator before the couple is bound to the test piece. A very shallow recess (about equal in depth to the diameter of the couple junction) is made in the surface of the test piece to locate the junction.

To facilitate the insertion of the test piece into the furnace, the loading column can be placed in position on the test piece at this stage and the assembly bound securely with transparent adhesive tape to form a transportable unit. It is usually desirable to bind the thermocouples to the loading column with platinum/rhodium wire as well as with transparent tape.

Care shall be taken to ensure that none of the binding wires make contact with the couple wires at any point, e.g. at the junctions between insulators.

(ii) *Top and bottom temperatures.* On the occasions mentioned in Subclause a(iii), the temperature at the centres of the top and bottom faces of the test piece shall be measured by means of platinum v. platinum/rhodium thermocouples. A groove approximately 1 mm deep and 1 mm wide shall be cut across the centre line or diameter of each end face and a thermocouple having no insulation for about 1 in on each side of the junction shall be laid in each groove so that the wires emerge on opposite sides of the test piece and the junction coincides with the geometrical centre of the face. These thermocouples need not be cemented in the grooves. The emergent wires shall be fitted with pure alumina insulators, which should be bound to the test piece and loading column with the strands of wire holding the surface couples.

(iii) *Temperature recording and indicating.* The thermocouples are connected to a potentiometer or potentiometric recorder by means of compensating leads, which should be appropriate to the composition of the thermocouples*. Care shall be taken to ensure that the connections between the thermocouple wires and the compensating leads are not subjected to a temperature exceeding 80°C at any stage of the test.

The four surface thermocouples shall be connected in parallel to the indicating or recording instrument. Provided that the thermocouples are of approximately equal lengths, the readings so obtained will be very nearly equal to the arithmetic mean of the individual readings of the four thermocouples.

The top and bottom thermocouples shall be connected individually to the potentiometer by a suitable switching arrangement or, if a recorder is used to separate recorder points.

Provision shall be made by means of a suitable switching system to disconnect the surface thermocouples from the potentiometer or recorder and to connect each thermocouple individually and consecutively to the instrument. The individual temperatures so indicated, when referred by calculation to the same instant of time, shall comply with the requirement of Subclause a(iii). This

* Certain types of thermocouple, such as platinum/5 per cent rhodium v. platinum/20 per cent rhodium, generate a negligible e.m.f. at low temperatures and do not require compensating leads.

check on the uniformity of heating shall be made at intervals of 20 minutes after the mean surface temperature has reached 1000°C in the rising temperature test, and at least three times during a maintained temperature test, once immediately before the soaking temperature is reached and twice during the soaking period.

On each of these occasions readings shall also be taken on the top and bottom thermocouples; the temperatures so indicated, when referred to the same instant of time, shall comply with Subclause *a(iii)*.

If the temperatures in a referee test are measured on a potentiometric recorder, provision shall be made to disconnect the four parallel-connected couples from the recorder and to connect them collectively to a potentiometer. The temperatures indicated by the two instruments, when referred to the same instant of time, shall not differ by more than 5 degC. This check on the accuracy of the recorder shall be made during every referee test, at a temperature between 1500°C and 1600°C in a rising temperature test, and during the soaking period in a maintained temperature test.

(iv) *Life of thermocouples.* Thermocouples deteriorate more quickly at the temperatures encountered in these tests than they do at the lower temperatures for which they are normally used. The rate of deterioration of a thermocouple, and hence the number of tests for which it can be used before it requires to be re-calibrated, will depend on the temperature to which it has been exposed and on the duration of the exposure. The following estimates of the life of thermocouples are offered as a guide in deciding on the necessary frequency of re-calibration:

Platinum v. platinum/13 per cent rhodium:	5 rising temperature tests to temperatures not exceeding 1650°C, or 3 maintained temperature tests at temperatures not exceeding 1600°C, or equivalent combinations of these tests.
Platinum/5 per cent rhodium v. platinum/20 per cent rhodium and platinum/20 per cent rhodium v. platinum/40 per cent rhodium	5 rising temperature tests to temperatures not exceeding 1700°C, or 3 maintained temperature tests at temperatures not exceeding 1650°C, or equivalent combinations of these tests.

The above estimates refer to wire of about 0.5 mm diameter; a shorter life may be expected with thinner wire. Estimates of thermocouple life cannot be given for other compositions but if previous experience offers no guide the estimate for platinum v. platinum/13 per cent rhodium should be used.

Also, the above estimates refer to tests on clean materials, i.e. those that are not likely to contaminate the thermocouples. A shorter thermocouple life is to

be expected in tests on basic materials containing chromium and iron, and on iron-spotted fireclay materials, as well as in tests at temperatures in excess of those quoted above.

(v) *Calibration of thermocouples.* Thermocouples shall be calibrated by comparison with a standard, which shall be a new thermocouple of the same composition and of known accuracy. If it is thought desirable, the accuracy of this thermocouple may be checked, before it is used for the calibration, against the melting point of pure palladium, by the wire method (see Reference 3 at the end of this Clause).

The calibration shall be carried out by binding the new thermocouple to the used ones with platinum wire, the junctions being as close together as possible. Up to four thermocouples can be calibrated together. Care shall be taken that the binding wire does not make contact with the thermocouple wires at the junctions between insulators. The assembly is then placed in the furnace, with the junctions suspended in a small refractory crucible having a lid with a hole through which the thermocouples pass and which acts as a guide to keep the junctions clear of the walls. The crucible shall occupy the normal position of the test piece. The temperature shall be raised to 1550°C at approximately 10 degC/min and held constant at this temperature within ± 5 degC while the reading of each thermocouple on a potentiometer is compared with that of the standard thermocouple, readings being taken quickly in the sequence: standard, thermocouple 1, standard, thermocouple 2, standard, etc. The reading of each thermocouple shall be compared with the mean of the standard readings taken immediately before and after the reading concerned.

A thermocouple shall not be used again if it differs by more than 5 degC from the standard thermocouple*.

NOTE. Thermocouples which have been used for this test should not be calibrated under conditions of temperature gradient which differ from those of the test furnace.

c. *Atmosphere.* The furnace atmosphere shall contain free oxygen.

d. *Test piece.* The test piece shall be cut or ground to the shape of a prism $2\frac{1}{2}$ in high having a base $1\frac{3}{4}$ in square or, alternatively, bored or ground to the shape of a cylinder $2\frac{1}{2}$ in high and 2 inches in diameter. The position of the test piece in the original block and the direction in which pressure is applied shall be stated.

Care shall be taken to ensure that the square or circular faces of the test piece are plane and perpendicular to the major axis (height) of the test piece.

The height of the test piece shall be measured before the test along the two

* The life of thermocouples which have been used may sometimes be prolonged by cutting off the junction together with about 1 in of each limb, then reversing the thermocouples and welding a new junction at the former cold ends. Such thermocouples should be re-calibrated before use.