DRAFT TANZANIA STANDARD

Insulating Bricks- Specification

TANZANIA BUREAU OF STANDARDS

0 National Foreword

The Tanzania Bureau of standards is the statutory national standards body for Tanzania, established under the act.No.3 of 1975, amended by act.No.2 of 2009.

This draft Tanzania Standard is being prepared by BCDC 2 Masonry Technical Committee under the supervision of the Building and Construction Divisional Standards committee (BCDC).

In the preparation of this draft Tanzania Standard, reference was made to ***IS 2042:2006 Insulating bricks-specification, published by Bureau of Indian Standard.***

1 Scope

This draft Tanzania Standard covers requirements and test methods for insulating bricks.

2 Grades

The bricks shall be of three grades, namely:

- Grade A – Suitable for temperature up to 1500 ^{o}C
- Grade B – Suitable for temperature up to 1250 ^{o}C
- Grade C – Suitable for temperature up to 850 ^{o}C

3 General requirements.

3.1 The bricks shall be compact, of homogeneous texture and free from cracks, voids, pink or black cores and other flaws. They shall have sufficient mechanical strength and no soft comers.

4 Tolerance on size

Variations from specified dimensions, covering both warpage and shrinkage, shall be allowed to the extent of + 2 percent or + 1mm, whichever is greater.

5 Chemical composition

If required by the purchaser, when the bricks are to be used in direct contact with the heating elements or for a specific atmosphere in the furnace, the iron oxide content, shall be not more than 1 percent when determined in accordance with ANNEX G.

6 Physical requirements

6.1 The bricks shall conform to the requirements given in Table 1.

6.2 If required, the bricks shall be tested for refractoriness under load except that a load of 0.05N/mm^2 shall be applied instead of 0.20N/mm^2. In that case the test requirements shall be as agreed to between the purchaser and the manufacturer.

7 Sampling

Representative samples shall be drawn according to the scheme of sampling given in 1S 1528 (Part 1).

8 Marking

9.1 The bricks and/or their wrappers shall be suitably marked with the manufacturer's name or trade-mark, and type.

9.2 The refractory bricks may also be marked with the Standard Mark.

Table-1 Physical requirements For Insulating Bricks.

SI No.	Characteriscs	Requirement			Test methods
		Grade A	Grade B	Grade C	
i)	Pyrometric cone equivalent, Min	1717 ^{o}C (see Note)	1640 ^{o}C	_	ANNEX A
ii)	Bulk density, g/cm^3, Max	1.00	0.90	0.75	ANNEX B
iii)	Apparent porosity, percent (%), Min	60	60	65	ANNEX C
iv)	Cold crushing strength, N/mm^2, Min	1.96	1.47	0.69	ANNEX D
v)	Permanent change after reheating for 12 h, Max at 1500 ^{o}C	-1.5 percent at 1250 ^{o}C	-1.5 percent at 850 ^{o}C	-2.0 percent	ANNEX E
vi)	Thermal conductivity at 600 ^{o}C w/mk at hot face, Max	0.52	0.35	0.28	ANNEX F
NOTE — In the case of silica base Type I bricks (Refractory bricks suitable for general applications), the pyrometric cone equivalent shall be Standard Pyrometric Cone (ASTM) No. 28, Min.					

Annex A

(Informative)

DETERMINATION OF PYROMETRIC CONE EQUIVALENT (PCE) OR SOFTENING POINT

A.1 Object of Test

The object of this test is to determine the softening point of refractory materials by comparing the test cones prepared from the refractory material under test with standard pyrometric cones.

A.2 Heating Furnace

A furnace of the type in which a neutral or oxidizing atmosphere may be maintained shall be preferred.

A.3 Preparation of Samples

A.3.1 Raw Materials

Test pieces prepared from raw materials that are subject to considerable modification during reheating shall then be stabilized by heating before their refractoriness is determined. In particular, clays should be calcined at approximately 1000ºC; after calcination, the test pieces shall comply with the requirements of A.4.2.

A.3.2 Fired Shapes

Take 1 kg of the material. In the case of bricks or shapes, obtain a composite sample from bricks or portions of a number of test pieces taken for other tests, and reduce these fragments in size by means of rolls or jaw crusher adjusted to pass lumps no greater than 5 mm in diameter; take precautions to prevent contamination of the sample with steel particles during crushing or grinding. Reduce the quantity through quartering by different stages of grinding to about 50 g as test sample. Grind the full quantity of the material to pass through 212 microns IS Sieve by suitable grinder or agate mortar. Magnet should be used to separate the iron particles introduced during grinding and crushing operations except in the case of materials, which are themselves magnetic. In order to avoid excessive reduction of the fines, remove them frequently during the process of reduction by throwing the sample on the sieve and continue grinding of coarser particles until all the sample passes through the sieve. Subsequently make specimen as specified in A.4.1.

A.3.3 For Dry Monolithic Materials

Samples of unshaped materials, such as, plastic refractories, ramming materials and refractory cement shall be shaped and fired in a manner appropriate to the material and its condition of use; the firing temperature shall be stated in the test report.

A.3.4 For Mortar make specimen as per dry monolithic materials and add the liquid component, if any, in appropriate proportion. Subsequently make specimen as specified in A.4.1.

NOTE — At all states, crushing and grinding should be carried out so as to avoid the introduction of extraneous material. At all stages, mixing should be carefully carried out so that the contents of the test pieces are truly representative of the samples.

A.4 Preparation of Test Cone

A.4.1 Moulding

Mix thoroughly the sample prepared under A.3.2 and after adding sufficient alkali free dextrin or glue and water, form into test cones in a metal mould, preferably of brass, in the shape of tetrahedron measuring 8 mm on the sides of the base, and 25 mm high (*see* Figure 1).

A.4.2 Sintering

When dry, subject the test cones, if necessary, to a preliminary burn at a temperature not exceeding 1000°C for the purpose of sintering them into a firm condition to permit handling.

A.5 Procedure.

A.5.1 Mount the test cones and the standard pyrometric cones, the feature of typical standard cones used are reported in Table 1, on a plaque with the help of bonding material. Both the plaque and the bonding material should be of such composition, which will not affect the fusibility of the cones.

A.5.2 Mount the cones with the base embedded approximately 3 mm deep in the plaque, and one of the faces inclined towards the centre of plaque and at an angle of 82° with the horizontal. Arrange the test cones around the outer edge of the plaque with standard cones in between them in the anticipated range, as far as practicable (*see* Figure. 2).

A.5.3 Place the test plaque with the test pieces and the pyrometric reference cones attached to it in the uniform temperature zone of the furnace. Avoid reducing conditions in the furnace during heating. Take care that the flame does not strike directly against the cones or the cone plaque. Check the furnace at intervals for uniformity of distribution of heat.

A.5.4 Over a period of 1.5 to 2 h, raise the temperature of the furnace to 200°C below the estimated refractoriness temperature of the test material.

A.5.5 Raise the temperature at the rate of 2.5°C/min or at the rate specified by the manufacturer of the cones. Maintain the heating so that at any moment the deviation from the specified temperature rise curve is less than 10°C.

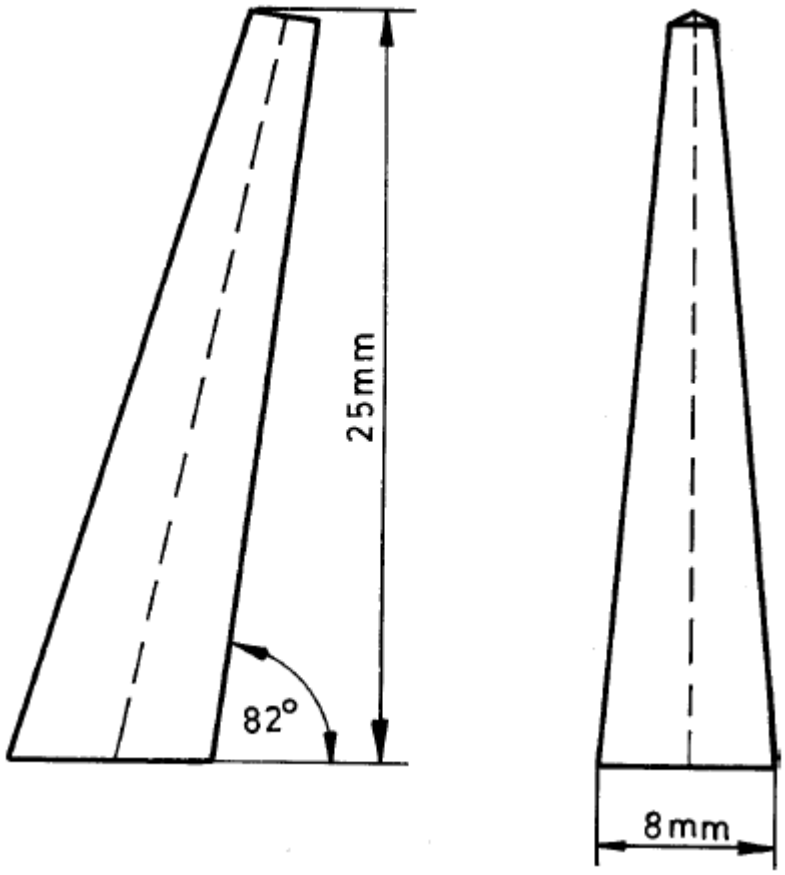


Figure 1-Standard Pyrometric Test Cone

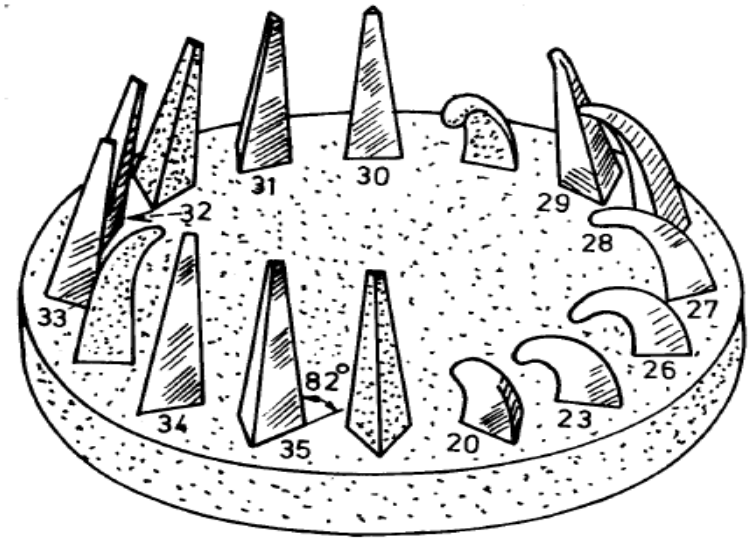


Figure 2-Method of Mounting Test Cone and Appearance After Testing.

A.6 Softening Point

Softening of the cone will be indicated by the top bending over and the tip of the cone touching the plaque surface.

A.7 Standard Cone Data

The standard pyrometric cone equivalents of standard cones are given in Table 1.

A.8 Report of Results

A.8.1 Report the softening point in terms of the standard pyrometric cone, it being that cone which most nearly corresponds in time of softening with the test cone.

A.8.2 If the test cone softens later than one cone but earlier than the next cone and approximately midway between, report the softening point, for example, Cone Number 31-32.

A.8.3 If the test cone starts bending at an early cone but it is not down until a later cone, report the fact.

Table 1 Reference Temperature and Cone Designations

Sl No.	Cone Designation	Temperature ^{O}C
i).	ISO 150	1 500
ii).	ISO 152	1 520
iii).	ISO154	1 540
iv).	ISO 156	1 560
v).	ISO 158	1 580
vi).	ISO 160	1 600
vii).	ISO 162	1 620
viii).	ISO 164	1 640
ix).	ISO 166	1 660
x).	ISO 168	1 680
xi).	ISO 170	1 700
xii).	ISO 172	1 720
xiii).	ISO 174	1 740
xiv).	ISO 176	1 760
xv).	ISO 178	1 780
xvi).	ISO 180	1 800

NOTES
1 The end point temperatures reported in the table have been obtained from the respective manufacturers' catalogue.
2 Any standard cone is acceptable.

ANNEX B
(Informative)

Method for Determination of Bulk Density

B.1 Principle
The mass of a dry test piece of a specified geometrical form is determined by weighing and the dimensions are measured. From these values and from the true density of the material, (determined by the method specified in ISO 5018, the volume, bulk density is determined by calculation.

NOTE — The method of immersion in a liquid and determination of the mass of the test piece when immersed and when soaked is not suitable for insulating refractory products because of their very open texture, which can lead to serious errors in the determination of the mass of the test piece when soaked. The precision of the results does not require any correction to be made for the fact that weighing is carried out in air, not in a vacuum.

B.2 Apparatus

B.2.1 Callipers, graduated in 0.5 mm, or flat metal rule, graduated in 0,5 mm and having a square at one end which can be fitted to the edge of the test piece.

B.2.2 Drying oven, capable of being controlled at (110 ± 5) ^{O}C.

B.2.3 Balance, with an accuracy of ± 0.1 g.

B.2.4 Desiccator.

B.3 Test pieces

B.3.1 The number of items (for example, bricks or blocks) to be tested shall be determined by agreement between the interested parties.

B.3.2 If several items are tested, the same number of test pieces shall be taken from each item, so as to facilitate statistical calculation.

NOTE — The number of test pieces per item should be the subject of agreement between the interested parties.

B.3.3 The test pieces shall be rectangular with plane and parallel surfaces. The volume of each test piece shall be not less than 500 cm^3 and no dimension of a test piece shall be less than 50 mm. The faces of each test piece shall be precisely formed to obtain a parallelepiped. For the purposes of this test, the test piece shall be considered to be a paralleled piped if, for each pair of opposite faces, the four measurements made along the centre lines of the faces that separate them do not differ by more than 1.0 mm.

B.3.4 In the case of insulating bricks that have been finished by sawing, the whole brick maybe used as a test piece, provided that the faces are plane and parallel, the parallelism tolerance being as specified in B.3.3.

B.4 Procedure
B.4.1 Using the callipers or the flat metal rule (B.2.1), measure the three principal dimensions (length 1, breadth *b,* thickness d) of each test piece to within 0.5 mm, the measurements shall be made at the centre line of each face (i.e., four times for each dimension) and the mean of the four measurements shall be noted for each of the three dimensions.

B.4.2 Dry the test pieces carefully in the drying oven (B.2.2), controlled at (110 + 5) ^{O}C, allow to cool to ambient temperature in the desiccator (B.2.4), and weigh each test piece to the nearest 0.1 g.

B.4.3 Repeat the drying, cooling and weighing operations until constant mass is reached, i.e., until two successive weighing made before and after at least 2 h in the drying oven do not differ by more than 0.1%.

B.4.4 Determine the true density in accordance with IS0 5018.

B.5 Expression of results

B.5.1 The bulk volume V_b of the test piece is given, in cubic centimetres, by the equation

$V_b = lbd$

Where l, *b* and *d* are the length, breadth and thickness, respectively, in centimetres, of the test piece.

B.5.2 The bulk density ρ_b of the test piece is given, in grams per cubic centimetre, by the equation

$$\rho b = \frac{\mathrm{M}}{\mathrm{Vb}}$$

where

m is the dry mass, in grams;
Vb is the bulk volume, in cubic centimetres.

B.5.3 The bulk density shall express in gram per cubic centimetre or in kilograms per cubic metre (by multiplying the result in B.5.2 by10^3). The calculation shall be made to three significant digits.

B.6 Test report
The test report shall include the following information"

a) the name of the testing establishment;
b) the date of the test;
c) reference to this Tanzania Standard;
d) the designation of the material tested (manufacturer, type, batch number);
e) the number of test pieces per item; alternatively, a statement that a whole brick was used (see B.3.*2* and B.3.4);
f) the individual values and the mean value of the bulk density.

ANNEX C

(Informative)

METHOD FOR DETERMINATION OF APPARENT POROSITY

C.1 Principle

C.1.1 The following are determined by weighing:

- The mass of a dry test piece;
- Its apparent mass when immersed in a liquid with which it has been impregnated under vacuum
- Its mass in air while still soaked with the liquid.

C.1.2 the precision of the results does not require any correction to be made for the fact weighing are carried out in air, not in a vacuum.

C.2 Apparatus and materials

C.2.1 Drying oven, capable of being controlled at 110 ^{O}C ± 5 ^{O}C.

NOTE—A fan-assisted oven with ventilation would assist in attaining an even temperature distribution and efficient drying of the test pieces.

C.2.2 Balance, with an accuracy of ±0.01g that can be arranged so that test pieces can be suspended in the immersion liquid (see Figure 1)

C.2.3 Beakers, of a suitable size for containing the samples during soaking (see C.3.2) and when determining the apparent immersed mass (see C.3.3).

C.2.4 Evacuating equipment, capable of reducing the absolute pressure to a value not greater than 2500Pa (0.025 bar) and a means of measuring the pressure used (see figure 1).

C.2.5 Thermometer, accurate to ±1 ^{O}C.

C.2.6 Immersion liquid: For materials that do not react with water, the immersion liquid may be cold distilled water. For materials that are sensitive to contact with water, a suitable organic liquid shall be used. The immersion liquid shall not fractionate at a pressure above the absolute pressure attained in the test.

NOTE-Distilled may be used for hydratable materials.

C.2.7 Desiccator

C.2.8 Number and shape of test pieces.

C.2.9 The number of items (for example, bricks, shapes, nozzles) to be tested shall be determined by agreement between the interested parties.

C.2.10 The number of test pieces to be tested per item shall be agreed between the parties; it shall be stated in the test report. If the test pieces are cut out of bricks or blocks, the same number shall be cut from each one, in order to facilitate statistical analysis.

C.2.11 Test pieces shall be cutting the form of prisms or cylinders, the bulk volume of a test piece shall be not less than 50cm^3, and shall be not more than 200cm^3. The ratio of the longest to the shortest dimension of a test piece shall not exceed 2:1.

NOTES

1. Where it is not possible to obtain the given size and volume from the item, test piece so for the other dimensions and volume may be used by agreement between parties, and are to be reported.

2. If test pieces are to be cut from an item in which variations in density could occur, the position of the test pieces should be agreed between parties and stated in the report.

C.2.12 Any test piece showing cracks shall be eliminated, since these might falsify the determination of the bulk volume.

C.3 Procedure

C.3.1 Determination of mass of dry test piece (m_1)

See Figure 3.

Dry the test piece at 11O°C ± 15 ^{O}C to constant mass, i.e., until two successive weighing made before and after at least 2h in the oven (5.1) do not differ by more than 0.1%. Before each weighing, place the test piece in a desiccator (**C.2.7**) until it has cooled to room temperature. Weigh each test piece to the nearest 0.01g. The mass determined is the mass of the dry test piece (m_1).

C.3.2 Soaking of test piece

Carry out a check test to ensure that the apparatus will hold a vacuum. Place the cooled and dried test piece in an air-tight vessel. After sealing the vessel, evacuate it until a pressure of not more than 2500Pa is attained; maintain this vacuum for at least 15min. In order to ensure that all the air has been removed from the open pores, isolate or disconnect the vessel from the vacuum pump and check that pressure does not rise through any de-gassing of the test piece. Re-connect the vessel to the vacuum pump and progressively introduce the immersion liquid (C.2.6) so that, after 3 min, the test piece is covered by about 20 mm of liquid. Maintain this reduced pressure for 30 min, then switch off the pump and open the vessel. Wait a further 30 min to ensure that the liquid penetrates into all the open pores. The test piece or test pieces shall remain covered by the immersion liquid throughout the impregnation and until removed for subsequent weighing (see C.3.3 and C.3.4).

NOTE – certain fine porosity materials such as refractories containing carbon and some clay products may require longer periods of evacuation and soaking. If a different soaking time is used this time should be stated in the report.

C.3.3 Determination of apparent mass of immersed test piece (m_2)

See Figure 4.

Suspend the test piece by a thin thread from the load-pan suspension point of a balance (C.2.2) and weight while completely immersed in a quantity of the immersion liquid, contained in a beaker (C.2.3) standing on the bridge, if used. In this way, the apparent mass of the immersed test piece is obtained (m_2). The weighing shall be made to the nearest 0.01g Determine the temperature of the immersion liquid to an accuracy of ±1 ^{O}C.

C.3.4 Determination of mass of soaked test piece (m_3)

Remove the test piece from the liquid and immediately sponge it quickly and carefully with a damp sponge or cloth to remove droplets and the surface film of liquid. Be sure not to draw liquid out of any of the pores.

NOTE—Consistent results have been obtained by keeping- for this purpose alone-linen cloth which, having been washed two or three times when new to remove the dressing, is immersed in the immersion liquid and lightly wrung out by hand before each use.

Immediately weigh the test piece in air to the nearest 0.01g. Take care to ensure that evaporation of the immersion liquid does not lead to any appreciable loss in mass during the weighing operation. In this way, the mass of the soaked test piece is obtained (m_3).

C.4 Expression of results.

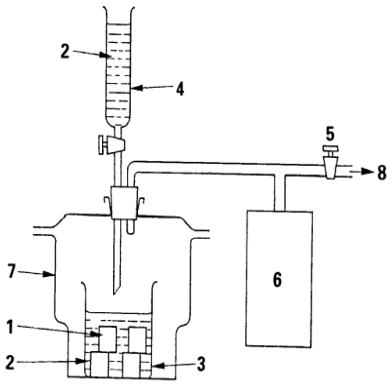
C.4.1 The apparent porosity π_a expressed as a percentage by volume, is given by equation.

$$\pi_a = \frac{M3-M1}{M3-M2} X\ 100$$

C.4.2 Test report

The test report shall include the following information:

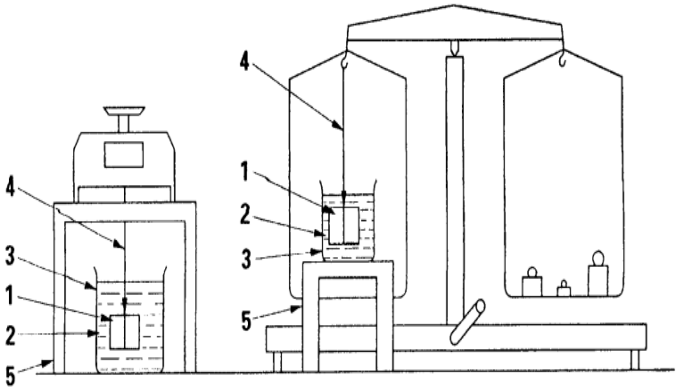
a) The name of testing establishment;
b) The date of the test;
c) The reference to this Tanzania Standard;
d) The description of the test material (manufacturer, type, batch number);
e) The number of items tested;
f) The number of test pieces per item and if relevant their position;
g) The pressure to which the vacuum chamber was reduced;
h) The immersion liquid used;
i) The individual value and the mean value of the apparent porosity for each item



Key

1 Test pieces
2 Immersion liquid
3 Beaker
4 Tap funnels
5 Pump isolation
6 Pump isolation valves
7 Pressure measuring device (e.g, manometer)
8 Desiccator Air outlet (to vacuum pump)

Figure 3 — Example of a vacuum system for soaking test pieces



Key
1 Test piece
2 Immersion liquid
3 Beaker
4 Suspension thread
5 Bridge

Figure 4 — Arrangement for the determination of apparent mass of immersed test piece using single- and double-pan balances

ANNEX D
(Informative)

DETERMINATION OF COLD CRUSHING STRENGTH

D.1 Principle

At ambient temperature, a test piece of specified dimensions is subjected, in a compression test machine, to an increasing load until either the test piece collapses or its height is reduced to 90% of its original value. During testing, the load is increased at a specified rate. The cold crushing strength is calculated from the maximum force recorded and the dimensions of the test piece.

D.2 Apparatus

D.2.1 Mechanical or hydraulic crushing strength machine, that will enable the load to be increased progressively and smoothly, and with a system of measurement that will enable the force exerted on the test piece to be known to within 2%. The range of the machine shall be such that the maximum force exerted in the test is greater than 10% of the maximum force of which the machine is capable. One of the platens of the machine shall be mounted on a spherical seating that will compensate for any small error of parallelism between the load-bearing faces of the test pieces. The platens of the machine shall be ground and the lower one shall be marked so as to facilitate placing the test piece at its centre.

D.2.2 Micrometer, or other suitable instrument, to measure the deformation of the test piece during the test.
D.2.3 Measuring equipment, accurate to 0.1 mm, to measure the size of each test piece and to verify its geometrical form.

D.2.4 Drying oven, capable of being controlled at 110 ^{O}C ±5 ^{O}C.

D.2.5 Steel rule.

D.2.6 0.5mm feeler gauge.

D.3 Test pieces

D.3.1 The number of items (e.g., bricks or blocks) to be tested shall be determined in accordance with ISO 5022 or with an alternative sampling plan agreed between the parties concerned.

D.3.2 One test piece shall be taken from each brick of standard size.

NOTE. The number to be taken from larger items is a matter for agreement between the parties concerned. To facilitate further statistical evaluation, the same number of test pieces should be taken from each item.

D.3.3 Each test piece shall be nominally the size of half a standard brick, i.e.; 114mm x 114 mm x 76 mm or 114 mm x 114 mm x 64 mm.

D.3.4 In the case of special shapes, the test pieces shall be dry cut to one of the sizes specified in D.3.3.

NOTE. If possible, the test report should indicate the relationship of the direction of loading to the direction of pressing or extrusion during manufacture.

D.3.5 The load-bearing faces of each test piece shall be flat within a tolerance of 0.5 mm. This condition shall be checked across both diagonals of each load-bearing face with a steel rule (D.2.5) and a feeler gauge (D.2.6).

D.3.6 The load-bearing faces of each test piece shall be parallel within a tolerance of 1 mm. This condition shall be checked by making four measurements of the height of the test piece, one at the centre of each of its four sides; the measurements shall not differ among themselves by more than 1 mm.

D.3.7 The perpendicular of each of the four sides of the test piece, with respect to the base, shall be within a tolerance of 1 mm. This condition shall be checked by placing the test piece on a flat, smooth surface and presenting a set square to the centre of a horizontal edge of the side; any gap between the set square and the side of the test piece shall not exceed 1 mm.

D.4 Procedure

D.4.1 Measure the length and breadth of each load-bearing face of the test piece, and its height at the mid-point of each of its four sides, in each case to the nearest 0.5 mm.

D.4.2 Dry the test piece to constant mass in the drying oven (D.2.4), controlled at 110 ^{o}C ±5 ^{o}C, cooling it each time in a dry atmosphere.
D.4.3 Place the test piece on one of its larger faces (114 mm x 114mm) in the centre of the lower platen of the testing machine (D.1). No packing material shall be used between the test piece and the platens. Mount the measuring instrument (D.2) on the lower platen to measure the deformation occurring in the test piece.

D.4.4 Gradually and continuously increase the load at such a rate that

a) If the expected cold crushing strength is less than 10MPa, the rate of increase of stress in the test piece is 0.05MPa/s ± 0.005MPa/s or
b) If the expected cold crushing strength is equal to or greater than 10 MPa, the rate of increase of stress in the test piece is 0.2 MPa ± 0.002MPa.

D.4.5 Continue increasing the load at the rate given in D.4.4 until either the test piece collapses (fails to support the load) or its height is reduced to 90% ± 1% of its original height. Record the maximum load indicated during the test.

D.5 Expression of results

Calculate the cold crushing strength, S, in megapascals, using the equation:

$$S = \frac{\mathrm{Fmax}}{\mathrm{l\ x\ b}}$$

Where

F_{max} is the maximum load, in newtons, indicated during the test;
l is the mean of the four measurements of the length, in millimetres, of the test piece;
b is the mean of the four measurements of the breadth, in millimetres, of the test piece.

Report the cold crushing strength to the nearest.

D.6 Test report

The test report shall include the following information:

a) all information necessary for identification of the sample tested including the designation of the material tested (manufacturer, type, batch number);

b) reference to this Tanzania Standard;

c) the sampling procedure, including:

1) the number of items tested (see D.3.1);
2) the number of test pieces cut from each item, if more than one (see D.3.2);
3) the dimensions of the test pieces (see D.3.3); their positions in the brick (see D.3.2) and the presence and location of any fired surface;
4) where possible, the relationship between the direction of loading and the direction of pressing or extrusion during manufacture (see Note to 6.4);

d) the results of the test, including:

1) the rate of stress increase (see D.4.4);
2) whether the test was terminated by the collapse of the test piece or by the height being reduced to of the original (see D.4.5);
3) the individual values of the cold crushing strength for each test piece, calculated as specified in Clause D.5 and, if appropriate (see D.3.2), the mean value for each item tested;

e) The name of the testing establishment;
f) Any information which might help in the interpretation of the test;
g) Any deviations from the procedure specified.

ANNEX E
(Informative)

Determination of Permanent Linear Change After Reheating

E.1 PRINCIPLE

Test pieces prepared out of the bricks are dried. Initial dimension with respect to length in case of Insulating product and initial volume in case of dense shaped product are determined. The test pieces are heated in a furnace at a prescribed rate to a specified temperature, which is maintained for a specified time. After cooling to ambient temperature, test pieces are measured again for final length or final volume and permanent linear change is calculated.

E.2 APPARATUS

E.2.1 Furnace — Either electric or gas fired, capable of firing the test pieces at the specified rate and of maintaining the test temperature for the required time. The furnace should be equipped with temperature controlling system which shall be able to comply the heating schedule mentioned in E 8.

NOTE — Use of electric furnace is recommended.

E.2.2 Thermocouple — Thermocouple(s) to measure the temperature.

E.2.3 Length Measuring Device — Vernier callipers or a dial gauge comparator with an accuracy of 0.1mm.

E.2.4 Drying Oven — Oven shall be fan assisted and shall have openings, which permit efficient ventilation

E.2.5 Drying Oven — Oven shall be fan assisted and shall have openings, which permit efficient ventilation.

E.3 TEST PIECES

E.3.1 Size of Test Pieces

D.3.1 For Insulation Bricks 114 mm × 114 mm × 64 mm or 76 mm, that is, half of standard brick.

E.4 PROCEDURE

E.4.1 Dry the test specimen in a drying oven at 110 ± 5°C to constant mass.

E.4.2 Measurement of Test Pieces

E.4.3 For Insulating Refractory Shaped Product

Calibrated length measuring device shall be used for measurement of dimensions. Make measurements on each test piece to the nearest 0.2 mm of the distance *L*o between the opposite faces nominally 114 mm apart. Make two of these measurements parallel to the centrelines (EF and *GH* in Figure 5) of the top and bottom faces of the test pieces, 15 mm from the edges of those faces, and two parallel to the centrelines (*AB* and *CD*) of the front and rear faces of the test pieces 15 mm from the edges of those faces. Mark the position of the measurements with refractory paint.

E.5 Mounting of Test Pieces in The Furnace

E.5.1 Furnace

Place the test pieces in the furnace as given as follows:

For Insulation Bricks — Place each sample on a 114 mm × 76 mm or 114 mm × 64 mm face. The samples shall be protected from direct radiation in an electrically heated furnace or from the flame of the gas burner in a gas fired furnace. Do not superimpose test pieces one on another. To allow free circulation of the hot gases, the test pieces shall be separated from each other by a distance of not less than 20 mm and shall be not nearer than 50 mm to the wall of the furnace to avoid direct heat impingement. The test pieces shall be placed in the furnace on bricks of 30 to 65 mm thick preferably of same quality.

E.6 Test Temperature

Unless otherwise agreed, the test temperature shall be 800°C or a higher temperature in multiples of 50°C.

E.7 Temperature Measurement

Thermocouple shall be placed away from the walls of the furnace, away from the heaters so as not to be in contact with any flames or direct heating from elements.

E.8 Heating

Raise the temperature in the furnace at one of the following rates:

a) For test temperatures up to 1250°C:
   - From ambient temperature up to 50°C below the test temperature: between 5 to 10°C/min
   - For the last 50°C: between 1 to 5°C/min.

b) For test temperatures above 1250°C:
   - From ambient temperature up to 1200°C: between 5 to 10°C/min.
   - From 1200°C up to 50°C below the test temperature: between 2 to 5°C/min.
   - For the last 50°C: between 1 to 5°C /min.

E.9 Cooling

Switch off the furnace after reaching and holding at the test temperature. Allow it to cool at its natural rate, the test pieces being allowed to cool in the furnace.

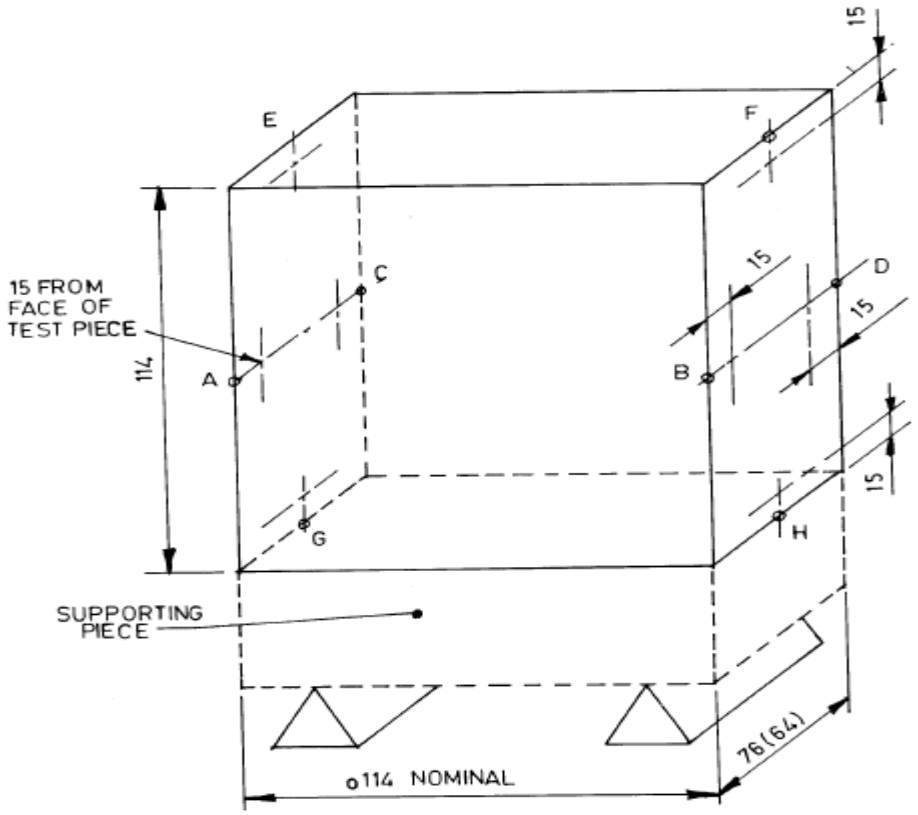


FIGURE 5- Position of Measurements and Mounting of the Test Pieces in The Furnace

E.10 Measurement of Test Pieces After Firing

First examine the test pieces, noting particularly any blisters or accretions produced during firing.

E.10.1 Linear Measurement for Insulating Brick Samples

Measure the distance between the two opposite faces of each test piece as described in E.4.2. If any of the measuring points might be affected by such a defect, measure at the nearest point unaffected. If necessary, rotate the test piece to avoid contact between defects.

E.10.2 Volume Measurement

The bulk volume V_b of the test piece is given, in cubic centimetres, by the equation

$V_b = lbd$

Where l, *b* and *d* are the length, breadth and thickness, respectively, in centimetres, of the test piece.

E.11 Expression of Results

For Insulation refractory quality, calculate the change in the relevant length, ΔL as a percentage of original value L_o, that is, $100 \times L / L_o$. Calculate the change in length for each measuring point.

E.12 Test Report

The test report shall include the following:

a). Description of the material tested.

b). Number of items tested.
c). Number of test pieces per item or brick,
d). Type of length measuring device used,
e). Type of furnace used,
f). The heating schedule used,
g). Test temperature,
h). Period of the actual holding time at test temperature, and
i). Appearance of test pieces after firing.

ANNEX F
(Informative)

Method for Determination of Thermal Conductivity of Thermal Insulation Materials (Water Calorimeter Method)

F.1 Principle of measurement

F.1.1 A specimen of the material to be tested is placed on a horizontal heater assembly. On the specimen a water flow calorimeter is placed along with a guard ring. The assembly is suitably insulated to minimize heat losses. The amount of heat flowing through the specimen is deter-mined by the flow rate of water through the calorimeter and its temperature rise. Thermal conductivity is measured using temperature drop and heat flow through the specimen, and its area and thickness.

F.2 Equipment

F.2.1 Heater Assembly — The heater assembly shall consist of a heater resistance wire coil laid in a suitable refractory material with grooves, meant for the purpose, deep enough so that the coil does not touch the sample (or plate) placed on it, and produces uniform heating. The material shall be strong enough to withstand the load of specimen, calorimeter, etc., placed on it. Its dimensions shall be about 450 × 450 mm.

F.2.2 The heater shall be fed from stabilized lead acid batteries of sufficient ampere-hour capacity or through a stabilized and regulated power supply from the mains, so that its temperature does not fluctuate in one hour of a test period by more than 0.5 percent of the temperature difference between the hot and cold plates.

F.2.3 Calorimeter and Guard Ring — The water flow calorimeter plate shall be about 230 × 230 mm. The guard ring shall be co-planar, about 110 mm wide, fitted around the calorimeter plate, with a gap not more than 3 mm wide maintained between the guard plate and the calorimeter plate in order to minimize thermal contact between the two. In order to ensure that lower surfaces of the calorimeter and guard ring are co-planar and that the calorimeter remains centered in the guard ring, the two shall be placed on a levelling plate and the gap between them filled with strips of blotting paper or any other suitable material. The two shall then be linked suitably so as to lock them together. The calorimeter and the guard ring shall consist of a manifold of high conducting metallic tube of not less than 7 mm bore packed as tightly as possible and brazed or clamped continuously to a thick high conducting metallic plate on one side and a strong iron plate on the other. The high conducting metallic surface plate shall be flat to within 0.25 mm/m and shall be not less than 5 mm in thickness. A twin-tube manifold is preferable in so far as every portion of the plate will then have the same temperature. The calorimeter and guard ring may also be made of high conducting metallic plates, with suitably designed cut-in counter flow channels, which on assembly would form continuous tube space inside the plate assembly so that uniform temperature could be maintained in the entire plate surface by circulation of water.

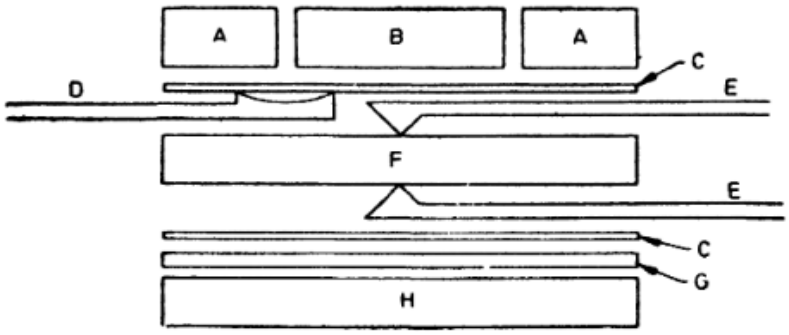
F.2.3 The calorimeter and the guard ring shall be supplied with water at a steady flow rate from a constant-head tank. The accuracy of measurement of flow rate of water shall be within ± 0.2 percent. The rate of flow shall not vary by more than 1 percent during the test period.

F.2.4 Thermocouples — Calibrated, similar thermocouples shall be used to measure temperatures to 0.02 K or better in conjunction with a calibrated potentiometer having an accuracy of ± 0.1 percent or better. Cold junctions of the thermocouples shall be maintained at 273 K, for example, in an ice cell containing mixture of ice and water. At least 4 thermocouples shall be placed on the heating side, and 2 under the calorimeter. Preferably, for precise detection of temperature imbalance between the guard surface and the calorimeter surface, additional thermo-couples may be fitted across the gap at each face along the periphery of the gap. These thermocouples across the gap may be fitted at a distance of about 10 mm from

the edge of the calorimeter and guard surface. Thermocouples shall be made of wire not larger than 0.46 mm in diameter (No. 26 SWG).

2

F.2.5 Assembly — General arrangement of the equipment is shown in Figure 6. A flat plate of a heat resistance alloy measuring 450 × 450 mm and about 3 mm thick and flat to within 0.25 mm/m shall be placed on the heater assembly. This plate shall have one cut, about 100 mm long, in each corner and edge to reduce warping. The sample shall be placed on this plate, sandwiched between two sheets of asbestos paper about 0.3 mm thick. The thermocouples shall be placed between the sample and the asbestos sheets. Calorimeter and guard ring shall be placed on the top asbestos sheet. The edges of the test specimen shall be covered with thermal insulating material at least 70 mm thick to minimize heat losses. The exposed surfaces of the calorimeter and guard ring shall also be covered with at least 70 mm of insulating material.



A — Guard ring
B — Water calorimeter
C — Asbestos paper
D — Thermocouples for detecting temperature imbalance
E — Thermocouples
F — Specimen
G — Plate
H — Heater assembly

Figure 6- Arrangement of Water Calorimeter Apparatus

F.3 Procedure for Thermal Conductivity Measurement

F.3.1 Samples — A test specimen, 450 × 450 mm and of uniform thickness 50 mm to 80 mm shall be used. The materials to be tested in the apparatus may be of various types and shall be suitably supported horizontally in the apparatus as given in F.2.1 to F.2.3.

F.3.1.1 Rigid Slabs *— Rigid slabs such as those of cellular concrete or calcium silicate insulation or slabs built up of bricks bonded with cement may be held in a frame of angle iron about 600 mm square, if necessary, which is fitted with screws which support the edges of the slab. Shallow grooves shall be made on the surface to place the thermocouples. Care shall be taken to ensure that the faces of the slab are flat to within 0.25 mm/m.*

F.3.1.2 Semi-rigid Slabs *— Semi-rigid slabs and similar materials such as fibrous mats or plastic compositions shall be placed in the apparatus on the flat plate. Where the compressibility of the material makes it necessary the thickness of the test specimen shall be maintained by supporting the calorimeter and guard ring on small distance pieces inserted in holes in the test specimen. A correction shall be applied for the heat flow through the distance pieces.*

F.3.1.3 Loose Fill Materials *— Loose fill materials, such as powders or granules, shall be packed as uniformly as practicable into a frame about 450 × 450 mm resting on the plate. Where necessary, the thickness of test specimen shall be maintained as in F.3.1.2 or by means of a grid of thin mica sheets. A correction shall be applied for the heat flow through the distance pieces.*

F.3.2 Observations — After assembling the apparatus, operate it, and when conditions are steady, make a series of observations of temperatures, the water-flow rate and water temperature rise through the calorimeter. The heat flow shall be calculated from these observations. For each hot face temperature condition, make observations using at least two different water flow rates. The thermal conductivity of the central section of the specimen shall be calculated from the heat flow through the central section, regarded as flowing through a square of area half way between that of the calorimeter and that of the central hole in the guard plate, together with the observed hot and cold face temperatures and thickness of the central section. The test observations shall be made at intervals of not less than 30 minutes until four successive sets of observations give thermal conductivity values differing by not more than 1 percent.
Thermal conductivities at high cold face temperatures shall be determined by measuring the temperature by means of thermocouples fixed parallel to the faces at known positions inside the test specimen.

F.4 Calculation

F.4.1 Thermal conductivity shall be calculated as follows:

$$\lambda = \frac{f\,\iota s(t_0 - t_i)}{A(t_h - t_c)}$$

λ = thermal conductivity of the material, W/mK;
f = flow rate of water, g/s;
ι = thickness of sample (or distance between hot face and cold face), m;
s = specific heat capacity of water in J/gK;
t_0 = outlet temperature of water, K;
t_i = inlet temperature of water, K;
A = Area of heat flow (central section), m^2;
t_h = hot face temperature, K; and
t_c = cold face temperature, K.

F.5 Report

F.5.1 The report of the results of each test shall include the following:

a) Name and other identifications of the materials;
b) Thickness of the specimen tested (or distance between hot face and cold face);
c) Mass of the specimen, after drying, used for the test;
d) Density before test just after placing the specimen in the apparatus of the specimen used for the test;
e) Moisture, as received, in the specimen used for the test;
f) Moisture regains, during test, of the specimen;
g) Hot face temperature;
h) Gold face temperature;
i) Mean temperature of the test;
j) Heat input per unit area;
k) Thermal conductivity;
l) Flow rate of water; and
m) Special remarks, if any. Here mention should also be made of the following:
    1) Whether some metallic portions, or coatings of insulation, were removed during test;
    2) Whether thermocouples were fixed inside the specimen for determining thermal conductivity at higher cold face temperature; and

3) Any other special point that may be relevant from the scientific or application point of view.

ANNEX G

(Informative)

DETERMINATION OF IRON BY THE ORTHO-PHENANTHROLINE (PHOTOMETRIC) METHOD.

G.1 Outline of the Method- The orange-red complex produced in acid solution of the sample (pH 4 to 6) by the addition of O-phenanthroline is determined photometrically at approximately 510 mm.

G.2 Reagents

G.2.1 Tartaric Acid Solution-10 percent (m/v).
G.2.2 Hydroxylamine Hydrochloride Solution (10 g/l) - Dissolve 1 g of the reagent in water. Transfer the solution to 100-ml volumetric flask, dilute to the mark and mix well.

G.2.3 Ortho-Phenanthroline Solution (One Percent) - Dissolve one gram of the 0rtho-phenanthroline monohydrate in 90 ml of water with gentle heating and constant stirring. Cool and dilute to 100 ml.

G.2.4 Acetate Buffer - Dissolve 21.5 g of sodium acetate (CH_3COONa, $3H_2O$) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 litre.

G.2.5 Standard Iron Solution (1 ml = 0.02 mg Fe) - Dissolve 0.2 g of pure iron in concentrated hydrochloric acid. Dilute the solution to 100 ml. Take the solution in a 1-litre volumetric flask, dilute to the mark and mix well. Transfer 100 ml of this solution to 1-litre measuring flask and dilute to the mark with water mix well.

G.3 Procedures

G.3.1 Solution of the Sample - Weigh accurately about 2 g of the finely ground test sample in a platinum dish. Moisten the sample with 5 ml of water. Add 3 ml of the dilute sulphuric acid and 20 ml of the hydrofluoric acid and evaporate slowly to dryness on the sand bath. or hot plate in a fume cupboard, taking care to avoid spurting. Repeat the process with 10 ml of hydrofluoric acid. Cool, add 2 ml of the dilute sulphuric acid and evaporate as before. Heat the dry residue cautiously until fumes of Sulphur trioxide cease to be evolved. Raise the temperature to 1000°C and ignite for five minutes. Cool and fuse the residue with about 3 g of sodium carbonate. Dissolve the residue in 30 to 40 ml dilute hydrochloric acid, cool and dilute to 250 ml in a calibrated flask, Reserve the solution for determination of iron.

G.3.2 Transfer a 5 ml aliquot of the solution to a 100-ml measuring flask. Add 2 ml of tartaric acid solution, and 2 ml of the hydroxylamine hydrochloride solution. Stir well. To this, add 5 ml of the ortho-phenanthroline solution and 10 ml of acetate buffer solution. Allow to stand for 15 minutes and dilute to 100 ml. Shake well.

G.3.3 Transfer a suitable portion of the solution to an absorption cell and take photometric reading using a light band centred approximately at 510 mm against a reference test blank solution.

G.3.4 Calibration Curve - Transfer 0, 1,2, 4, 8 and 10 ml of the standard iron solution in 100-ml volumetric flasks, using same quantities of reagents. Carry out the entire stages of procedures as described under **G.3.2** and **G.3.3** and record the photometric readings of all the standard solutions against the blank.

G.3.4 Calculation -Convert the photometric reading of the sample to milligrams of iron by means of a calibration curve and calculate the percentage of iron as follows:

$$Ferric\ oxide, percent = \frac{A}{B} \times 0.1429$$

Where;

A= mass in mg of iron found in aliquot, and

B= mass in g of the sample represented by the aliquot taken

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