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भारतीय मानक

चीनी मिट्टी और कांच उद्योग के लिए पेरिस प्लास्टर — विशिष्टि

(*तीसरा पुनरीक्षण*)

Indian Standard

PLASTER OF PARIS FOR CERAMIC AND GLASS INDUSTRY — SPECIFICATION

(*Third Revision*)

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(ICS 81.060.10)

Ceramicware Sectional Committee, CHD 09	Last date of comments: 19 January 2024

FOREWORD

(*Formal clauses to be added later*)

This standard was first published in 1963 and was covering requirements of plaster of paris for general purposes including that required for building, ceramic industry, surgical use and intra-oral impressions. However, with the publication of three separate standards, namely IS 2547: 1976 ‘Specification for gypsum building plaster’, IS 6555: 1972 ‘Specification for dental laboratory plaster’, IS 6556: 1972 ‘Specification for dental impression plaster’ and deletion of the requirement of surgical plaster from the standard, this standard was revised in 1981 to cover the demands of only ceramic industry in a comprehensive manner.

Second revision of this standard was brought in 1992, a new type of material, suitable for automatic machine jiggering and for roller head had been added. A new requirement for water absorption had also been incorporated and dry compressive strength had been modified for type 3. The procedures for test for normal consistency, modulus of rupture and dry crushing strength, had been modified. Compound water, for all types and expansion after setting for types 1 and 2 had been stipulated.

In this revision including glass in the title of the standard as type 3 is suitable for mounting optical glass items and the standard has been updated based on the experience of last three decades and on the currently available data.

In the formulation of this standard due weightage has been given to the standards and practices prevailing in different countries.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2: 2022 'Rules for rounding off numerical values (second revision)'. The number of significant places retained in the rounded off value should be the same as that of these specified value in this standard.

1 SCOPE

This standard peccaries requirements and method of sampling and test for plaster of Paris for use in ceramic, optics industries, jiggering and Toiler head of, automatic machines.

2 REFERENCE

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

IS No.	*Title*
196 : 1966	Atmospheric condition for testing (*first revision*)
460 (Part 1) : 2020	Test Sieves — Specification Part 1 Wire Cloth Test Sieves (*fourth revision*)
1070 : 2023	Reagent Grade Water — Specification (*fourth revision*)
2480 (Part 1) : 1983	Specification for general purpose glass thermometers Part 1 Solid-Stem thermometers (*second revision*)

3 TYPES

3.1 There shall be four types of the material as follows:

a) Type 1 — Suitable for moulds for slip casting;
b) Type 2 — Suitable for moulds for jiggering. Case and block making;
c) Type 3 — Suitable for mounting optical glass items; and
d) Type 4 — Suitable for automatic machine jiggering and for roller head.

4 REQUIREMENT

4.1 Description

The material shall be in the form of a fine white Pewter or of smooth texture, free from foreign matter and lumps. It shall be calcined gypsum and shall correspond essentially to the formula $CaSO_4 . \frac{1}{2} H_2O$.

4.2 Physical Requirements

The material shall comply with the physical requirements given in Table 1 when tested in accordance with the methods prescribed in **Annex A**. Reference to relevant clauses of **Annex A** is given in col **7** of Table 1

4.3 Chemical Requirements

The material shall also comply with the chemical requirements given in Table 2, when tested in accordance with the methods prescribed in **Annex B**. Reference to the relevant clauses of **Annex B** is given in co1 **7** of Table 2.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in clean moisture proof containers as agreed to between the manufacturer and the purchaser.

5.2 Marking

Each container shall be legibly and indelibly marked with the following:

a) Name of the material:
b) Type;

c) Net mass;
d) Indication on of source of manufacture:
e) Batch number and date of manufacture; and
f) Instructions for the use of the material.

5.2.1 *BIS Certification Marking*

The containers may also be marked with the BIS Certification Mark.

5.2.1.1 The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

6 SAMPLING

6.1 The method of drawing representative samples of the material and the criteria for its conformity to the requirements of this standard shall be as prescribed in **Annex C.**

Table 1 Physical Requirements for Plaster of Paris for Ceramic Industry

(*Clause* 4.2)

Sl No.	Characteristic	Requirement				Method of Test (Ref to Cl. No. in Annex A)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Fineness :					
	a) Material retained on 150 micron IS Sieve, percent by mass, Max	Nil	Nil	Nil	Nil	A-2
	b) Material retained on 75 micron IS Sieve, percent by mass, Max	7	Nil	Nil	Nil	A-2
ii)	Normal consistency	60 to 80	15 to 60	55 to 65	10 to 55	A-3
iii)	Setting Time, min					A-4
	a) Initial	8 to 15	8 to 15	8 to 15	8 to 15	
	b) Final	15 to 30	10 to 15	10 to 15	10 to 15	
iv)	Temperature rise during setting, °C, Max	12	12	12	12	A-4
v)	Expansion after setting, percent	0.2 to 0.4	0.2 to 0.4	—	—	A-5
vi)	Modulus of rupture, MPa, Min	4.0	5.0	5.0	7.0	A-6
vii)	Dry compressive strength, MPa, Min	9	15	17	20	A-6
viii)	Water absorption, percent	30 to 38	20 to 25	20 to 25	12 to 18	A-7

by mass

NOTE - Values given in this table are for normal consistency. For getting a higher water absorption: Water to plaster ratio should be higher

Table 2 Chemical Requirements for Plaster of Paris for Ceramic Industry
(*Clause* 4.3)

Sl. No.	Characteristic	Requirement				Method of Test (Ref to CL No. in Annex B)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
I)	Free moisture, percent by mass, *Max*	2.0	2.0	0.5	0.5	B-2
ii)	Carbonates (as $CaCO_3$ percent by mass, *Max*	3.0	3.0	1.0	1.0	B-3
iii)	Matter insoluble in hydrochloric acid, percent by mass, *Max*	7.0	7.0	2.0	2.0	B-4
iv)	Calcium sulphate (as $CaSO_4$), percent by mass, *Min*	85.0	85.0	90.0	90.0	B-5
v)	Compound water, percent	5.8 to 6.4	5.8 to 6.4	5.8 to 6.4	5.8 to 6.4	B-6

ANNEX A

(*Clause* 4.2)

METHODS OF PHYSICAL TESTS FOR PLASTER OF PARIS FOR CERAMIC INDUSTRY

A-1 TEST CONDITIONS

A-1.1 The ambient temperature and relative humidity in the room where tests are to be carried out shall be maintained (27 ± 2) °C with a relative humidity of (65 ± 5) percent (*see* IS 196). Unless specified otherwise distilled or demineralized water (*see* IS 1070) shall be used in the tests.

NOTE - The testing equipment and water should be kept at the above temperature at least for one day just before use.

A-1.2 The vessels used for mixing plaster of Paris shall be thoroughly cleaned after each use, as traces of hardened plaster of Paris accelerate the setting of fresh paste.

A-2 TEST FOR FINENESS

A-2.1 Apparatus

A-2.1.1 *Test Sieves* — one each of 150 micron and 75 micron apertures [*see* IS 460 (Part 1)].

A-2.1.2 *Vessel* — 3 to 5 cm larger in diameter than the test sieves and containing isopropyl alcohol/kerosene oil.

A-2.1.3 *Balance* **—** accurate to 0.1 g.

A-2.1.4 Brush of Camel Hair

A-2.2 Procedure

Weigh accurately about 50 g of the material and place it on 75-micron IS Sieve. Gently lower the sieve into the vessel containing isopropyl alcohol/kerosene oil to a depth not less than 50 mm. Lift the sieve out of the alcohol/oil, giving a swirling roil); l and permitting alcohol/oil to drain thoroughly the sieve back into the vessel. Repeat the process at least eight times until alcohol/oil passes freely through the sieve and the residue is essentially free from fines. Wash the residue with 100 ml of clean isopropyl Johal/kerosene oil, blot the bottom of the sieve with a soft, dry, lump free cloth and dry at (45 ± 1) °C. Shake the sieve for two minutes. Retrieve the residue, if any, with a camel hair brush on to a tared glazed sheet of paper and weigh immediately. Report the material retained on the sieve as percent by mass of the material taken for the test.

A-2.2.1 Carry out the test on sieves of 150 micron apertures also as in **A-2.2.**

A-2.2.2 Repeat the test on 150 micron and 75 micron sieves and report the mean of the results of two test. If the values of the two tests differ by more than 10 percent of the smaller value, a third sieving shall be carried out.

A-3 TEST FOR NORMAL CONSISTENCY (WATER-PLASTER RATIO)

A.3.1 Apparatus

A-3.1.1 *Glass Beaker*

Having internal height 66 mm, diameter 66 mm and height marks at 16 mm and 32 mm above the inside bottom surface.

A-3.1.2 *Porcelain Basin*

Having internal height 100 mm, diameter 120 mm.

A-3.1.3 *Stirrer*

Capable of stirring the plaster paste at a speed of 1 200 rpm.

A-3.1.4 *Hard Rubber Ring or a Suitable Metallic Ring*

Diameter 65 to 75 mm, height 40 mm and thickness 7 mm (*see* Fig. 1).

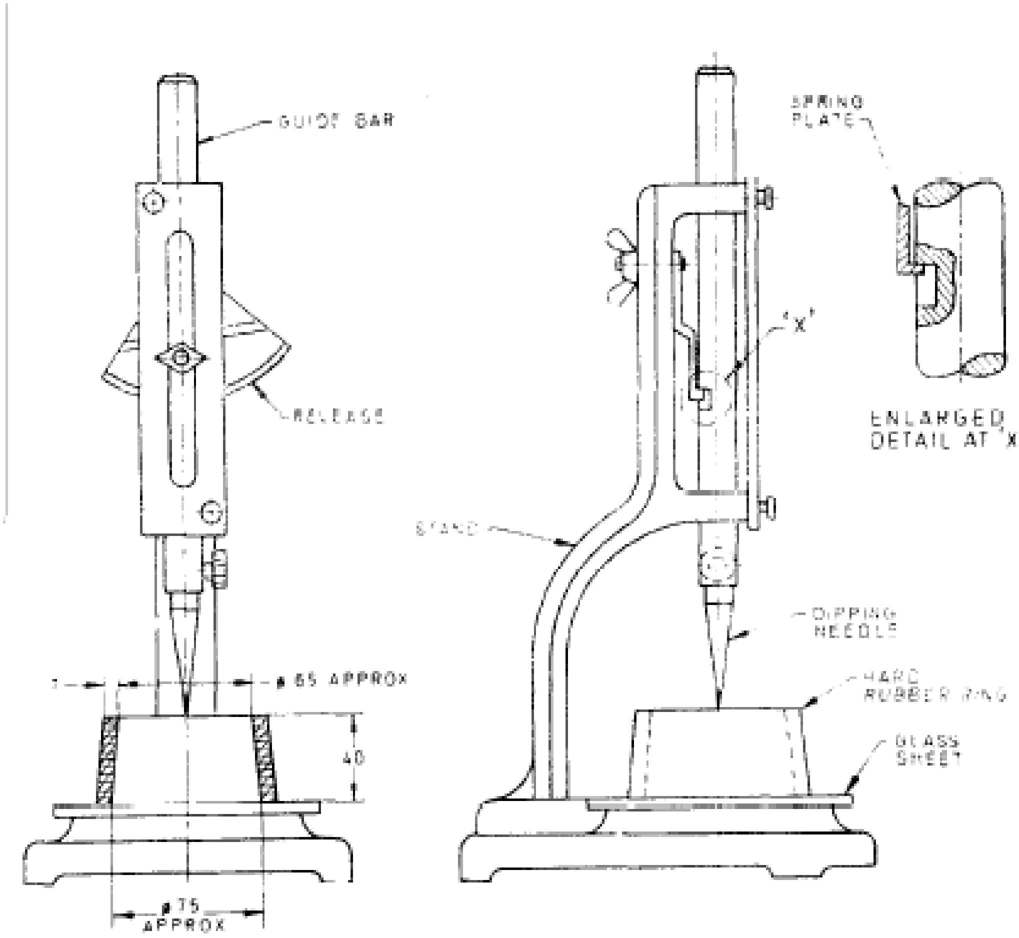
A-3.1.5 *Flat Glass Sheet*

250 mm × 250 mm with graph paper underneath.

A-3.2 Procedure

Pour 100 ml of water into the beaker without wetting the upper wall surface. Weigh about 200 g of plaster of Paris sample. Sprinkle portions of the sample with the fingers uniformly so that it reaches the first height mark (16 mm) After half a minute, the second height mark (32 mm) after one minute and stands about 2 mm below the water level after one and a half minute. In the next half a minute sprinkle just sufficient plaster of Paris so that the entire water surface up to the wall of the beaker is covered. If any small islands of plaster are formed, they should be fully moistened within 3 to 5 seconds; the entire sprinkling time being controlled within 2 minutes ± 5 seconds. Weigh the remaining quantity of the material and calculate the quantity of material sprinkled.

NOTE - In case of slowly sinking plaster, the height marks may not be reached in the prescribed periods of time. In that case sprinkling may be done so slowly that the plaster falls only on free water areas and not on plaster, the sprinkling time being indicated.



All dimensions in millimeter.

FIG. 1 VICAT APPARATUS WITH DIPPING NEDDLE AND RELEASE

A-3.2.1 Calculate the average quantity of material sprinkled from three individual values which shall not differ from each other by more than 5 g. Also measure the volume of the paste formed.

A-3.2.2 From the readings obtained in **A-3.2.1** calculate the volume of water and the mass of plaster of Paris which would give about 300 ml of paste. Next take the required volume of water, in the porcelain basin or in a 500 ml beaker and using a spoon, sprinkle within one and a half minutes the required quantity of plaster of Paris in such a way that the plaster is distributed uniformly over the water surface and does not form lumps which sink. Allow to soak for one and a half minutes and then stir the mass with the stirrer for one minute, taking care that no air is entrapped in the paste.

A-3.2.2.1 In the meantime place the rubber or metallic ring with its larger diameter down on the dry glass plate resting in a horizontal plane. Pour the paste prepared as in (**A-3.2.2)** into the ring so that it is flush with the edge of the ring and scrap off any excess paste without disturbing the paste or the glass plate, with a clean knife, completing the entire operation in one minute. After the completion of the period of 5 minutes from the start of sprinkling of plaster, Raise the rubber ring quickly by hand vertically upward and then move immediately sideways so that drops of paste do not fall on the plaster mass. Immediately read the diameter of the spread plaster mass from the graph paper along two mutually perpendicular directions correct to 1 mm.

A-3.2.3 If the spread diameter is within the value (210 ± 10) mm. calculate from the mass (or equivalent volume) of water required for 100 g of plaster of Paris.

A-3.2.3.1 If the two spread diameter measured perpendicular to each other differ by more than 10 mm, the test shall be repeated.

A-4 TEST FOR SETTING TIME AND TEMPERATURE RISE DURING SETTING

A-4.1 Apparatus

A-4.1.1 *porcelain Dish* — of capacity sufficient to hold paste for filling four rubber or metallic rings.

A-4.1.2 *Stirrer* **—** same as in (**A-3.1.3)**.

A-4.1.3 *Stop watch*

A-4.1.4 *Rubber or Metallic Rings* — 4 in number and of dimensions as given in (**A-3.1.4).**

A-4.1.5 *Flat Glass Sheet* — 4 in number and of dimensions as given in (**A-3.1.5).**

A-4.1.6 *Vicar Apparatus* — with dipping needle and release device as shown in Fig. 1.

A-4.1.7 *Thermometer* — having a range of 0 to 60°C [see Schedule Mark 4 of IS 2480 (Part 1):].

A.4.2 Procedure

A-4.2.1 *Preparation of the Test Blocks*

Pour 500 ml of water in the porcelain dish. Calculate the mass of plaster of Paris required for making paste of the right consistency from the result of (**A-3.2.3)** and weigh the quantity of plaster separately. Start the stop-watch and simultaneously start sprinkling plaster of Paris and prepare; past following the steps and time schedule as laid down in (**A-3.2.2)** and (**A-3.2.2.1)**, Place the four rubber: ring with their larger diameter on the fiat glass sheets resting in horizontal plant. Pour the paste into the four rings with light tamping. Scrap flush with the tip of the rings any excess paste using a straight edge knife and taking care that 11r pressure is exerted on the plaster mass.

A-4.2.2 *Time of Setting*

Place one of the glass sheets with the test block in the rubber ring on the base of the stand of the Vicar apparatus. Bring the tip of the needle Fitted to the guide bar of the Vicar apparatus, Carefully into contact with the surface of the Test block and release the guide bar to slide down Under its own weight. After it has come to rest/. Note the position of the tip of the needle above the glass sheet. Raise the guide bar, clean the needle and. repeat the penetration as above, Maintaining rhea distance between the positions of needle and from each other and from the edge of the rubber ring as at least 12 mm. After ascertaining approximate time of setting with these trial penetration determine *actual tint* start of setting on the second and completion of setting on the third test block contained in rubber rings, cleaning the dipping needle after each penetration. When the tip of the needle comes ‘o rest at a height of (18 ± 2) mm above surface of the glass sheet it indicates the eta of setting.

NOTES

1 The evaluation is made easy by plotting a graph showing distance of the tip of the needle from the surface of glass sheet against time.

2 Completion of rye process of setting of plaster is indicated when the tip of the needle does not leave any visible impression on the surface of the test block.

A-4.2.2.1 Note the time consumed between the start of sprinkling of plaster of Paris (**A-4.2.1**), Report the result as the mean of two value rounding off the time nearest to the minute.

A-4.2.3 Temperature Rise during Setting

Simultaneously with starting test for time of setting (**A-4.2.2**), insert the thermometer into the fourth test block reserved for this test and note the reading of the thermometer observing the gradual rise of temperature and finally note the reading of the thermometer when the mercury column in the thermometer does not move up further.

A-4.2.3.1 Report the result as the difference between the initial and final readings.

A-5 DETERMINATION OF LINEAR EXPANSION ON SETTING

A-5.1 Apparatus

A-5.1.1 *Extensometer*

It shall be as illustrated in Fig. 2, the dial gauge of which is essentially free-moving with no internal mechanism or springs which could effectively, influence the expansion of plaster in the cradle. To prevent plaster sticking to the sides of the cradle, grease the interior surface before use and line with thin non-absorbent paper which has a glazed surface. Renew the paper lining for each test.

A-5.1.2 *Contriver*

The container into which the extensometer may be placed and stored in an atmosphere of high relative humidity. It is suggested that a plastic box with an air-tight lid, containing water to a depth of approximately 3 mm, be used, but any enclosed space which will effectively prevent dehydration of the test specimen during the test procedure may be used.

A-5.2 Procedure

Carry out the test in triplicate. Fill the cradle of the extensometer with plaster of standard consistency and strike of level. Ensure that the movable end plate is slightly clear of the cradle and the plaster is in close contact with this plate. Zero the dial gauge, place the extensometer in the container (see **A-5.1.2**), add the water and close the lid. Leave undisturbed at a temperature of (27 ± 2) °C for a period of two hours, measured from the first contact of plaster and water, and then take a dial reading.

A-5.2.1 *Calculate the test result as follows:*

$$\text{Setting expansion, percent} = \frac{\text{Dial reading in 0.01 mm}}{100}$$

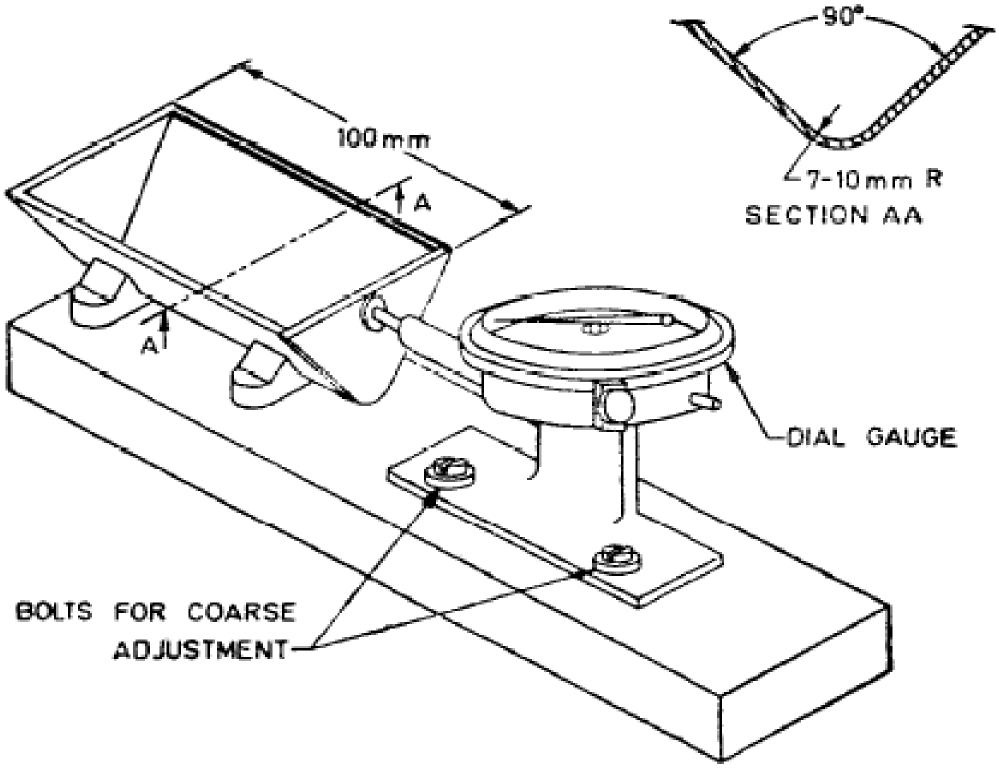


FIG. 2 APPARATUS FOR DETERMINATION LINER EXPANSION ON SETTING

A-5.2.2 Report the setting expansion as the mean of three determinations. If any result diverges by more than 20 percent from the mean, repeat the whole test.

A-6 DETERMINATION OF MODULUS OF RUPTURE AND DRY CRUSHING STRENGTH

A-6.1 Apparatus

A-6.1.1 *Disk* **—** of capacity about 1.5 liters and made of either porcelain or vitreous enamel.

A-4-6.1.2 *Spoon* — for mixing plaster of Paris for making paste.

A-6.1.3 *Knife* — having straight edge.

A-6.1.4 *Molds* — made of brass; three in Number; for making bars of dimensions (200 ± 0.8) mm × (40 ± 0.2) mm × (40 ± 0.2) mm.

A-6.1.5 *Air-Oven* — for drying test at (45± 2) °C.

A-6.1.6 *Testing Machine for modulus of Rupture*

Any standard testing machine may be used. Its bearing edges shall be in the form of V-support s rounded to a 15.875 mm radius or cylindrical pieces 31.750 mm in diameter. That shall he straight and of a length at least equal to this width of the test specimen. The supporting members of the lower bearing edges shall be constructed so as to provide a means for the alignment of the bearing edges with the under surface of the test specimen. Suitable designs for modulus of rupture test assembly are illustrated in Fig.3.

A-6.1.7 *Testing Machine for crushing strength*

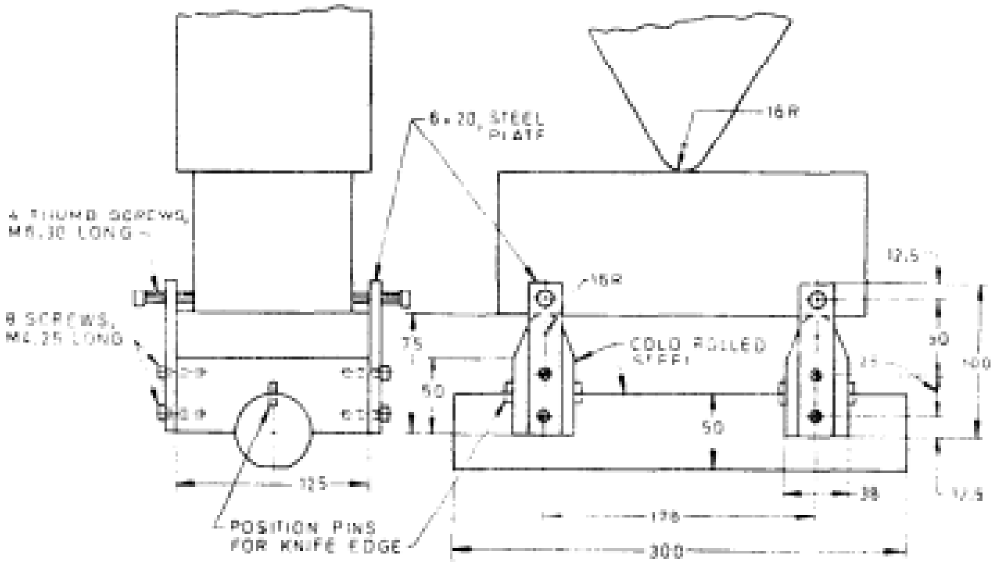
Any standard mechanical or hydraulic testing machine may be used. Its bearing blocks shall be spherical with plane bearing surface, the area which is equal 10 or greater than to the area of the surfaces of the specimen subjected to compression. A suitable design for crushing strength test assembly is illustrated in Fig. 4.

A-6.2 Procedure

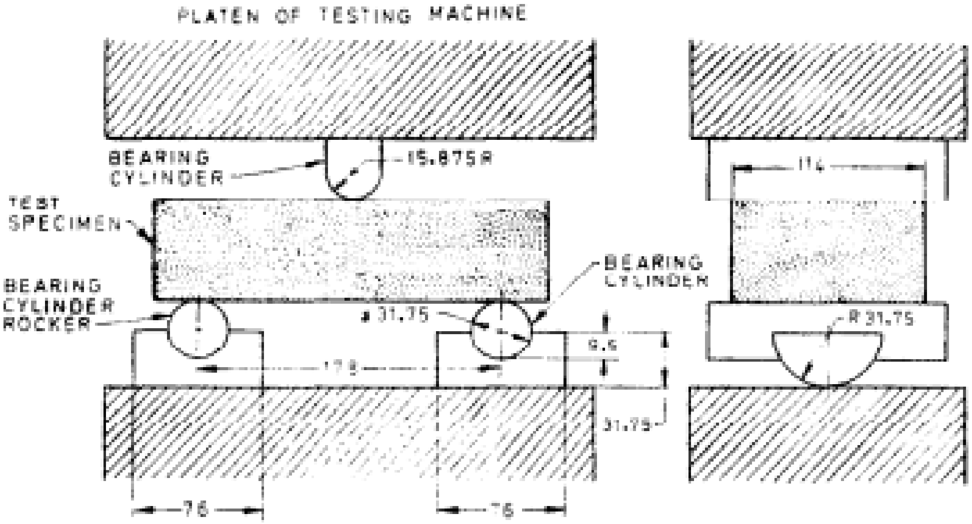
A-6.2.1 *Preparation of Test Specimen*

Weigh accurately 450 g of plaster of Paris per bar. Pour into the dish the required quality of water weighed out correctly to 0.5 percent or measured our volumetrically for preparing paste of right consistency on the basis of result obtained in (**A-3.2.3)** Sprinkle the weighed plaster of Paris with the spoon over a period of one minute. Allow to soak for half a minute followed by mechanical sting (**A-3.1.3**) for one minute so that a uniform past is made.

A-6.2.1.1 Apply a thin coaring of oil org case to the inter walls of the moulds, assembled in advance, and immediately pour the paste into the three moulds with light tamping and taking care that air is not entrapped in the test specimens. Fill the moulds within 5 minute after the start of sprinkling of plaster. That is. Before the setting of start of plaster of Paris. If any excess paste starts setting remove it by scrapping with Straight edge knife without applying any pressure on the plaster mass. After the specimen have become rigid. As checked by pressing with finger mark them on the top side and take them out of the mould.



3A Recommended Design of Bearing Cylinders for Modulus of Rupture Test



3B Alternative Design of Bearing Cyanides for Modulus of Rupture Test

All dimensions in millimeters.

FIG. 3 SUITABLE DESIGNS OF MODULUS OF RUPTURE TESTING ASSEMBLY

A-6.2.1.2 Store the test specimens in the ambient condition (**A-1.1**) for three days and dry them at (50 ± 2) °C in the air oven to constant mass. After drying, cool the specimens in a dessicator.

A-6.2.2 .*Modulus of Rupture*

Place the dried and cooled specimen in such a way that one of the surfaces which was in contact that the sides of the mould rests centrally on the V-supports of the testing machine. Then apply load on the test specimen at a uniform rate of 1 kN/min till it breaks.

Note the breaking loud and calculate modulus of rupture from the following formula:

$$R= 3\,W/2\,h.d^2$$

where

R = modulus of rupture in mega pascal (MPa).
W = total load in newton (N) at which specimen failed.
I = distance in millimeters between the v-edge supports.
B = width of the test specimen in mm and
D = thickness of the test specimen in mm.

and report mean of the three test result rounded off to one decimal place

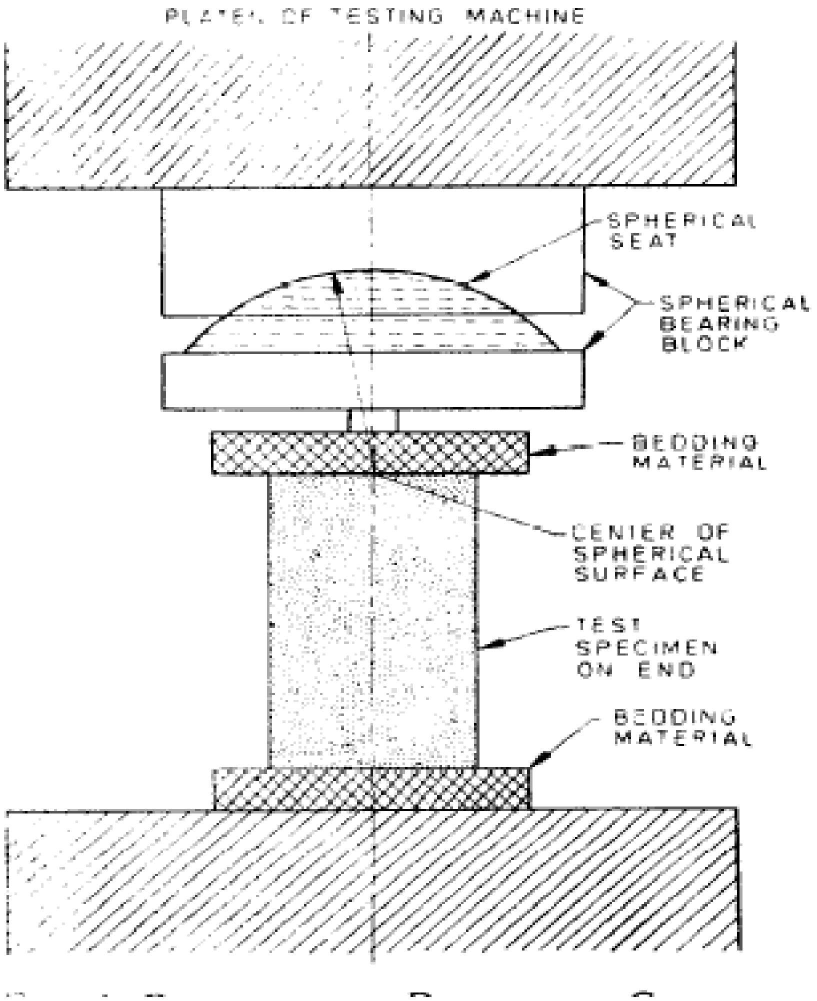


FIG. 4 RECOMMENDED DESIGN FOR CRUSHING TEST ASSEMBLY, INCLUDING BEARING BLOCK

A-6.2.3 *Crushing Strength*

Collaret the broken halves from the test for modulus of rupture and cut them into lengths of 62.5 mm. Price them, one at a time, with the side surface resting on the bearing block in the compressive strength testing machine in such a way that compression is applied to the surface measuring 40 mm × 62.5 mm. Sandwich a cellulose fiber wall board 5 mm in thickness and extending 50 mm or mare over the edges between the specimen and bearing blocks and applying the load at a uniform rate of 1 kN/min till the test piece breaks. Note the breaking load and calculate the crushing strength from the following formula:

$$S = W/A$$

S = crushing strength in MPa.

W = total load in Newton (N) at which the specimen failed, and

A = average of the gross areas in mm^2 of the top and bottom side surface of the specimen subjects to load.

And report mean of the six test results rounded off to one decimal place.

A-7 DETERMINATION OF WATER ABSORPTION

A-7.1 Apparatus

A-7.1.1 *Vacuum Desiccator/Vacuum Oven* — fitted with vacuum pump and gauge, etc.

A-7.1.2 *Blower* — capable to blow air at constant pressure.

A-7.1.3 *Balance* — accurate to 0.01 g.

A-7.2 Procedure

A-7.2.1 Preparation of Test Specimen

Cut a 15 mm length from the rectangular bar prepared by the method in (**A-6.2.1**) Shape it to a regular rectangular form with sand paper. Blow out the dust from the surface of the specimen.

A-7.2.2 Weigh accurately three individual test pieces nearest to 0.01 g. Let this be dry mass M, Place the test bar in a beaker inside a vacuum desiccator/vacuum oven and set the pump for obtaining vacuum to nearly 30 cm of mercury. Maintain the vacuum for 1 hour to completely evacuate the air, contained in the pores of the specimen. Stop the pump and flow kerosene into the beaker through the thistle funnel drop by drop, until the specimen becomes completely immersed under the level of kerosene. Run the pump for further 30 minutes and then stop. Leave the specimen to remain under. Vacuum condition for 24 hours to allow complete soaking of kerosene into the pores of the specimen. After this the vacuum is released and the sample is taken out.

A-7.2.3 Take out the piece and wipe out all the adhering film of kerosene with a soaked blotting paper/cloth-pad so that only the outer film of kerosene on the specimen is blotted out without soaking the same from within. Let the soaked mass be denoted as M_2. Determines the density of kerosene and water used at the temperature of experiment and denoted as d_1 and d_2, respectively.

A-7.2.4 Calculation

$$\text{Water absoption percent by mass}, W = \frac{(M_2 - M_1) \times d_2 \times 100}{M_1 \times d_3}$$

where

M_2 = mass in g of test piece soaked in kerosene,

M_1 = mass in g of dry test piece,

d_1 = the density of kerosene used at the temperature of experiment; and

d_1 = the density of water used at the temperature of experiment

ANNEX B

(*Clause* 4.3)

METHODS OF CHEMICAL TESTS FOR PLASTER OF PARIS FOR CERAMIC INDUSTRY

B-1 QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1670), shall be used in the tests.

Note – pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

B-2 DETERMINATION OF FREE MOISTURE

B-2.1 Procedure

Weigh accurately about 5 g of the material and spread it in a thin layer in a petri dish. Dry in an air oven maintained at (45 ± 1) ºC to constant mass, cool in a desiccator and weigh.

B-2.2 Calculation

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

where

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

M_1 = mass in g of the material after drying.

B-3 DETERMINATION OF CARBONATES

B-3.1 General

Two methods are prescribed for the determination of carbonate, namely, evolution method and volumetric method. Volumetric method is recommended for routine analysis. In case of dispute, evolution method shall be the referee method.

B-3.2 Evolution Method

B-3.2.1 *Apparatus*

The apparatus shown in Fig. 5 consists of the following.

B-3.2.1.1 *Round-bottom* — flask 100 ml capacity and provided with a two holed rubber stopper.

B-3.1.2 *Dropping funnel* — A of 50 ml capacity.

B-3.1.1.3 *Bubblers* — B, two, containing concentrated sulphuric acid.

B-3.1.1.4 *Hydrochloric acid absorption tube — C,* U-shaped tube containing anhydrous copper sulphate.

B-3.1.1.5 *Soda* — lime tubes - *E, F* and H, U shaped tubes containing soda-lime (or soda asbestos) and magnesium perchlorate. The tubes shall be filled as shown in Fig. 6 and shall be so used that the gases on entry pass through them.

B-3.1.1.6 *Water absorption tubes — D* and G. U- shaped tubes filled with magnesium Perchloric previously saturated with carbon dioxide.

B-3.1.2 *Reagent*

B-3.1.2.1 *Dilute hydrochloric acid* — 1 : 4 (*v/v*)

B-3.1.3 *Procedure*

Assemble the apparatus as shown in FIG. 5. Weigh accurately about 5 g the material into the round bottom flask. Remove the two soda-lime tubes *E* and *F,* wipe them with clean, dry cloth and leave them with the taps closed, in the balance cache or 45 minutes. Sweep the remaining apparatus with a current of air free from carbon dioxide till all the residual carbon dioxide driven out. Open the taps momentarily in the balance case, weigh them separately and replies them in the assembly. Pour about 50 ml of dilute hydrochloric acid in the dropping funnel A. Open the taps of the U-tube run in the acid to cover the material, close the tap of the lopping funnel and heat the flask carefully, so that not more than two bubbles gas pass through the bubbler *B* per second. After about 30 minutes boil the contents of the flask for 2 to 3 minutes. Attach a second bubbler (not shown In in Fig. 5) to tube G; remove the flame immediately apply suction for 20 minutes, opening the tap of the dropping funnel at the same time. Regulate the suction so that not than two bubbles are seen passing through ice secular bubbler per second. Remo the soda lime tubes *E* and *F. Close* the taps treat them as before and weigh.

B-3.1.4 *Calculation*

$$\text{Carbonate (as CaC}O_3\text{), percent by mass} = \frac{2272 \times M_1}{\text{M}}$$

where

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

M_1 = increase in mass in g of tubes E and F.

B-3.2 Volumetric Method

B-3.2.1 *Reagents*

B-3.2.1.1 *Standard hydrochloric acid* — approximately 0.2 N.

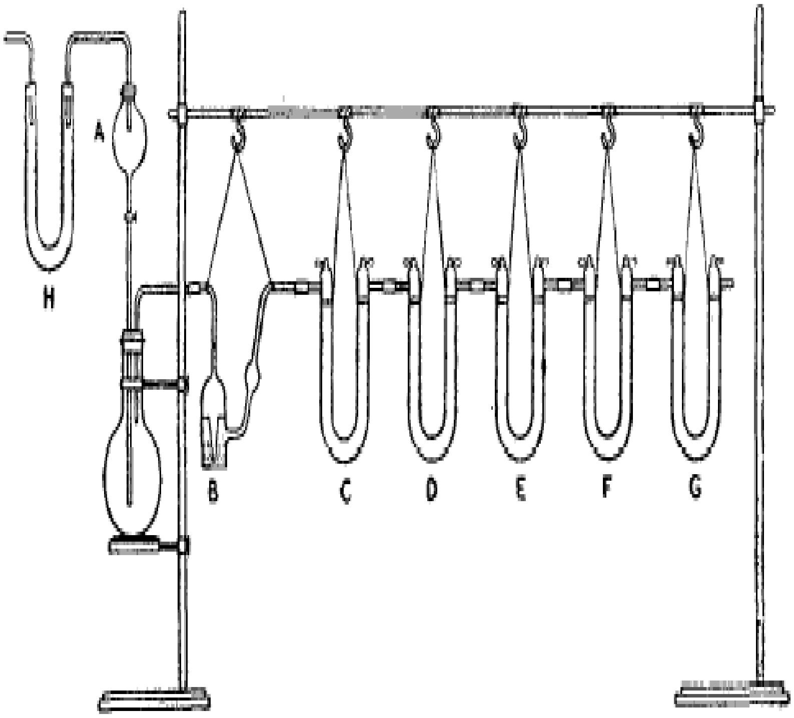


FIG. 5 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF CARBONATES

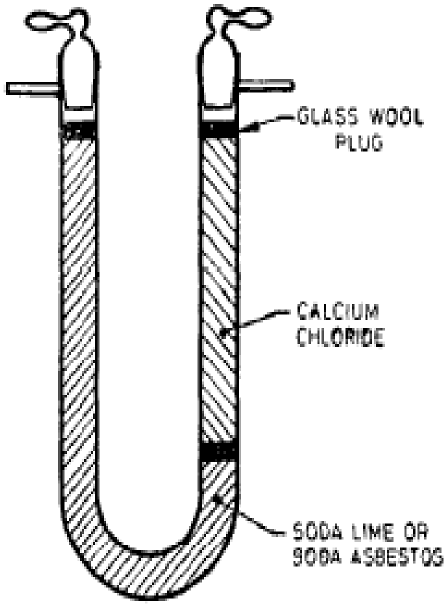


FIG 6. SODA-LIMETUBES FOR CARBON-DIOXIDE ABSORPTION

Approximately 0.1 N, nearly free from carbonate, made immediately before use, by appropriately diluting with boiled and cooled water, about 12 N solution of sodium hydroxide which have been filtered through glass wool.

B-3.2.1.3 *Bromophenol blue indicator solution* — 0.1 percent (*m/v*) in water.

B-3.2.2 *Procedure*

Weigh accurately about 5 g of the material, transfer to a 100 ml conical flask; add 25 ml of water and stir well. Add 25 ml of standard Hydrochloric acid and boil for 15 minutes to expel all carbon dioxide. Cool, wash down the sides of the flask, add a few drops of the side of the acid with standard sodium hydroxide solution till the end point is reached.

3.2.3 *Calculation*

$$\text{Carbonates (as Ca}CO_3\text{)percent by mass,} = \frac{5\ (25\ N_1\ - V\ N_2)}{\mathrm{M}}$$

where

N_1 = normality of standard hydrochloric acid,

N_2 = normality of standard sodium hydroxide solution,

V = volume in ml of standard sodium hydroxide solution used, and

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

B-4 DETERMINATION OF MATTER INSOLUBLE IN HYDROCHLORIC ACID

B-4.1 Reagents

B-4.1.1 *Dilute Hydrochloric Acid* — 1 : 2 (*v/v*)

B-4.2 Procedure

Weigh accurately about 0.5 g of the material and dissolve in 30 ml of dilute hydrochloric acid in a porcelain dish. Evaporate to dryness on a water-bath. Heat for two hour at 120 °C and again add 20 ml of hydrochloric acid; warm for a few minutes and filter. Wash the residue with warm water till free from chloride. Dry the residue, ignite and coal in a desiccator and weigh.

B-4.3 Calculation

Matter insoluble in hydrochloric acid, percent by mass= $\frac{M_1}{M} \times 100$

where

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

M_1 = mass in g of the ignited residue.

B-5 DETERMINATION OF SULPHATES

B-5.1 Reagents

B-5.1.1 *Dilute Hydrochloric Acid* — 1: 5 (*v/v*).

B-5.1.2 *Barium Chloride Solution*

Dissolve 10 g of barium chloride in 100 ml of water.

B-5.2 Procedure

Weigh accurately about 0.5 g of the material and dissolve in 50 ml of dilute hydrochloric acid. Boil the solution and add about 100 ml of water in a 500 ml beaker. Continue boiling for 5 minutes. Filter immediately and wash the filter paper thoroughly with hot water. Boil the filtrate and add 20 ml of hot barium chloride solution with stirring. Put the beaker on a hot plate and allow the precipitate to settle. Filter the precipitate (Whatman No. 42 or equivalent) and wash with hot water till free from chlorides. Dry the precipitate carefully. Ignite over a Bunsen burner at the lowest heat, or in a furnace at 300 ºC to 400 ºC, until the filter paper is carbonized, without catching fire. Then ignite for about 5-10 minutes until most of the carbon is burnt off. Add a drop or two of concentrated sulphuric acid to wet the entire residue, heat gently until fumes of sulphuric acid cease and then ignite for 15 minutes. Cool in a desiccator and weigh to a constant mass.

B-5.3 Calculation

$$x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}$$

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

M_1 = mass in g of the precipitate.

B-6 DETERMINATION OF COMPOUND WATER

B-6.1 Procedure

Take 5 g of the sample, spread it out in a thin layer in a suitable vessel and put the vessel in an oven, dry it at 45°C. Then cool it in the dessicator over fused calcium chloride and weigh till constant weight. Take the sample again in the oven and dry it to constant weight at 215°C to 230°C and weigh.

B-6.2 Calculation

$$\text{Percent compound water} = \frac{\mathrm{M} - M_1}{\mathrm{M}} \times 100$$

where

M = mass of the sample after drying at 45°C and

M_1= mass of the sample after drying at 215°C to 230°C.

ANNEX C

(*Clause* 6.1)

SAMPLING OF PLASTER OF PARIS

C-1 GENERAL

C-1.1 In drawing, preparing and handling test samples the following precautions and directions shall be observed.

C-1.2 Samples shall be taken in a protected place.

C-1.3 The sampling instrument shall be clean and dry.

C-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from moisture and other adventitious contamination.

C-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

C-1.6 The samples shall be placed in clean, dry, air-tight glass or other suitable containers on which the material has no action.

C-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.8 Each sample container, after filling, shall be sealed air-tight with a stopper and marked with full details of the material and the date of sampling.

C-2 SCALE OF SAMPLING

C-2.1 Lot

All the containers of the same type in a single consignment of the material draw from a Single batch of manufacture shall constitute a lot. If a consignment is declared or known to consort of different batches of manufacture, the container belonging to the same batch shall be grouped together and each group shall constitute a separate lot.

C-2.1.1 Samples shall be tested from each Lot for ascertaining conformity of the material to the requirements of this specification.