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Indian Standard

**METHODS OF TEST FOR
POZZOLANIC MATERIALS**

(First Revision)

(Incorporating Amendment No. 1)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Group 10

Indian Standard

METHODS OF TEST FOR POZZOLANIC MATERIALS

(*First Revision*)

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(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 24 July 1967, after the draft finalized by the Pozzolanas Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 This standard was originally published in 1960. The various provisions of the standard have been under the review of the Pozzolanas Sectional Committee, for quite some time. The greater utilization of pozzolanic materials in the country, the experience with the use of this standard and also the latest developments in the field of pozzolanic materials has necessitated a change in the approach to the testing of pozzolanas. In the light of these, the Pozzolanas Sectional Committee has felt the need to issue this revision, to incorporate the modification necessary to bring the standard in line with the present thinking on the subject.

0.2.1 The revision incorporates a number of changes, the most important of them being — the method of test for specific surface of pozzolana by Blaine air permeability apparatus, the use of revised grading of standard sand for various tests, introduction of ‘absolute volume’ concept for the test mixtures for various pozzolanic activity tests, modified method of compaction of test specimens and a test for specific gravity of pozzolanas.

0.2.2 The design of mix for various pozzolanic activity tests on absolute volume basis was considered preferable as this would keep the binder plus pozzolana volume same as that of binder paste volume in control mix. This will control the workability, voids and yield of mortar which are affected by a change in the specific gravity of pozzolanas. If the proportions are by weight alone, any change in the specific gravity of pozzolana will alter the water requirements for the mix and this will have a significant effect on the strength with the result that a true assessment of the quality of pozzolana would not be possible. Proportioning by weight will also not give true comparison between the reactivity of two different pozzolanas because even if they had equal efficiency in respect of their chemical reactivity, an artificial difference in the strength will be observed due to difference in their specific gravities in case of proportioning by weight; on the other hand, if the absolute volumes occupied by both pozzolanas were kept constant (by batching by absolute volume) such an anomaly would not occur.

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0.2.3 Instead of equating workability on the basis of water requirements in a cement pozzolana paste, a suitable method to judge the performance of qualitative workability basis relative to the control mix has been introduced both for lime reactivity test as well as compressive strength test of cement pozzolana mortars. The Sectional Committee was of the view that the determination of water requirements merely on cement paste as specified earlier in the standard may not have sufficient bearing in reflecting the true workability of mortar because the workability and water requirements in cement mortar may be effected by the finer fractions of pozzolana reducing the particle friction in various sizes of sand grains.

0.3 There are different kinds of pozzolanas used for different purposes in various construction works. In general, pozzolanas improve certain properties of concrete, such as workability, cohesiveness, impermeability, resistance to aggressive waters and also assist in the reduction of heat of hydration and alkali-aggregate reactivity in hardened concrete. The relative importance of each of these properties will naturally depend upon the type and nature of construction. In mass concrete construction, for example, improvement in workability, reduction of segregation and low heat of hydration are relatively more important. In hydraulic structures imperviousness is of very great importance. Resistance to attack by acids and sulphate waters is necessary in marine construction in sewage disposal works and in laying roads over sulphate bearing soils.

0.4 While the only pozzolana which has been extensively used for centuries in this country is *SURKHI* or burnt clay pozzolana; the rapid industrialization of the country is yielding large quantities of industrial wastes like fly ash, coal ash, etc, which form additional sources for pozzolanas. The use of fly ash as a pozzolana is well established in a number of countries abroad, but it has come in vogue in India only recently. Besides these, there are a large number of pozzolanic clay deposits of stained and impure kaolins, ferruginous or ochreous earths, altered laterites and bauxites, certain shales, etc, available in different parts of the country, which yield highly reactive pozzolanic materials. The Central Road Research Institute, New Delhi have conducted an all India survey of pozzolanic clay deposits through the length and breadth of the country and prepared detailed report of such deposits in each State with suggestions for the mode of industrial utilization of each of them. The Sectional Committee has already published IS : 1344-1959*, IS : 3812 (Part I)-1966†, IS : 3812 (Part II)-1966‡ and IS : 3812 (Part III)-1966§. This standard lays down the methods of test for pozzolanic materials with a view to providing a uniform basis for the assessment of the properties and potentialities of the different pozzolanic materials.

*Specification for *SURKHI* for use in mortar and concrete. (Since revised).

†Specification for fly ash: Part I For use as pozzolana.

‡Specification for fly ash: Part II For use as admixture for concrete.

§Specification for fly ash: Part III For use as fine aggregate for mortar and concrete.

0.5 The tests to be performed on any particular pozzolanic material would depend on the purpose for which it is to be used and it is not necessary that all the tests should be performed on every pozzolana.

0.5.1 Though considered desirable, the test for mortar bar expansion to indicate reduction of alkali-aggregate reactivity has been omitted from this standard for two reasons; namely, there are very few localities in India where alkali-reactive aggregates would be encountered and the problem is not so serious as it is in some other countries, and the mortar bar test requires strictly controlled conditions which are available only in a few laboratories, and any lack of control might easily give misleading results.

0.6 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country.

0.7 This standard requires reference to the following Indian Standards:

- | | |
|-----------------|--|
| IS : 196-1966 | Specification for atmospheric conditions for testing
(<i>revised</i>) |
| *IS : 269-1958 | Specification for ordinary, rapid-hardening and
low heat Portland cement (<i>revised</i>) |
| IS : 460-1962 | Specification for test sieves (<i>revised</i>) |
| IS : 516-1959 | Methods of test for strength of concrete |
| IS : 650-1966 | Specification for standard sand for testing of
cement (<i>first revision</i>) |
| *IS : 712-1964 | Specification for building limes (<i>revised</i>) |
| IS : 1199-1959 | Methods of sampling and analysis of concrete |
| IS : 1607- 1960 | Methods for dry sieving |

0.8 This edition 2.1 incorporates Amendment No. 1 (October 1991). Side bar indicates modification of the text as the result of incorporation of the amendment.

0.9 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 : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard covers the following tests for pozzolanic materials:

- a) Chemical analysis,
- b) Fineness,
- c) Soundness,

*Since revised.

†Rules for rounding off numerical values (*revised*).

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- d) Setting time,
- e) Lime reactivity,
- f) Compressive strength,
- g) Transverse strength,
- h) Drying shrinkage,
- j) Permeability,
- k) Reduction in alkalinity and silica release, and
- m) Specific gravity.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definition of terms given in IS : 4305-1967* shall apply.

3. TEST MATERIALS

3.1 Hydrated Lime — Standard hydrated lime supplied by National Council for Cement and Building Materials, New Delhi shall be used in the tests. The standard hydrated lime shall have the following composition:

CaO	— 96 ± 0.5 percent
MgO	— 1 ± 0.5 percent
SiO ₂ , <i>Max</i>	— 1.0 percent
Insoluble residue, <i>Max</i>	— 1.0 percent

3.2 Cement — Cement for use in the tests shall be 33 grade ordinary Portland cement conforming to IS 269 : 1989†.

3.3 Standard Sand — Standard sand for use in the tests shall conform to IS : 650-1966‡.

4. TEST CONDITIONS

4.1 Temperature and Relative Humidity — Unless otherwise specified the temperature range within which physical tests may be carried out should be, as far as possible, 27 ± 2°C and the relative humidity 65 ± 5 percent (*see* IS : 196-1966§).

5. CHEMICAL ANALYSIS

5.1 Reagents

5.1.1 Quality of Reagents — Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977 ||) shall be used in the test.

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

*Glossary of terms relating to pozzolanas (Since revised).

†Specification for 33 grade ordinary portland cement (*fourth revision*).

‡Specification for standard sand for testing of cement (*first revision*).

§Specification for atmospheric conditions for testing (*revised*).

|| Specification for water for general laboratory use (*second revision*).

5.1.2 The following reagents and special solutions are required:a) *Reagents*

Hydrochloric acid	— sp gr 1.16
Nitric acid	— sp gr 1.42
Sulphuric acid	— sp gr 1.84
Phosphoric acid	— sp gr 1.7
Hydrofluoric acid	— 40 percent
Ammonium hydroxide	— sp gr 0.90

NOTE — The reagents shall be of the analytical reagent quality.

b) *Dilute solutions of reagents* — Prepare the following solutions by diluting the reagent with distilled water:

Hydrochloric acid — 50 percent, 25 percent and one percent (*v/v*)

Nitric acid — 3 percent (*v/v*)

Ammonium hydroxide — 50 percent (*v/v*)

c) *Stannous chloride solution* — Dissolve 50 g of stannous chloride in 100 ml of hydrochloric acid and dilute to one litre. Keep a few pieces of pure metallic tin in contact with the solution.d) *Mercuric chloride solution* — Prepare a saturated solution by dissolving 5.6 g in 100 ml of water at room temperature.e) *Manganese sulphate solution* — Dissolve 35 g of crystalline manganese sulphate in 250 ml of distilled water. Add 70 ml of phosphoric acid (sp gr 1.7) and 65 ml of sulphuric acid (sp gr 1.84). Dilute to 500 ml.f) *Standard potassium permanganate solution* — Approximately 0.05 N. Dissolve about 1.5 g of potassium permanganate in one litre of distilled water. Allow to stand for a week; filter through asbestos mat and keep in a dark place after standardizing it with pure sodium oxalate.g) *Ammonium nitrate solution* — Dissolve 2 g of ammonium nitrate in 100 ml of water.h) *Ammonium oxalate solution* — Dissolve 50 g of ammonium oxide in one litre of water. Also prepare a dilute solution containing one gram of salt per litre.j) *Ammonium hydrogen phosphate solution* — Dissolve 250 g of ammonium hydrogen phosphate in one litre of water.k) *Barium chloride solution* — Dissolve 100 g of barium chloride in one litre of water.**5.2 Preparation of Sample for Analysis** — About 100 g of the representative sample shall be air-dried and ground to pass 150-micron IS Sieve. The material shall be stored in an air-tight bottle.

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5.3 Loss on Ignition

5.3.1 Ignite 1.00 g of the air-dried, finely ground sample in a platinum crucible of 20 to 25 ml capacity by placing it in a muffle furnace at $1\,000 \pm 25^{\circ}\text{C}$ to constant weight (for about 20 to 30 minutes). Air should have free access to the sample to maintain oxidizing conditions. Cool and weigh the crucible to 0.1 mg and check the loss in weight by a second heating for 5 minutes and reweighing after cooling.

5.3.2 The percentage loss on ignition nearest to 0.1 shall be calculated as follows:

$$\text{Loss on ignition, percent} = \frac{A}{B} \times 100$$

where

A = loss in weight, and

B = weight of moisture free sample used.

NOTE — The significance of the determination of loss on ignition will vary according to the nature of the sample. The figure obtained may include loss due to the oxidation of any carbonaceous matter and gain due to the oxidation of any ferrous iron present.

5.4 Silica

5.4.1 Fuse one gram of the air-dried, finely ground, sample with about 7 g of anhydrous sodium carbonate in a covered platinum crucible. Raise the temperature slowly until frothing ceases, then complete the fusion at $1\,000^{\circ}\text{C}$ for 30 minutes, occasionally swirling the melt to ensure thorough mixing. Quench the melt by immersing the bottom half of the hot crucible in cold water, then place the crucible and lid in about 100 ml of hot water contained in a platinum or porcelain evaporating basin. Cover the basin with a clock glass and add a few drops of absolute alcohol. Then gradually add 30 ml of hydrochloric acid (sp gr 1.16). Warm until the melt is completely disintegrated and remove the crucible and lid, washing them thoroughly and scrubbing them with a rubber-tipped rod. Crush any lumps remaining in the solution.

5.4.2 Evaporate the solution, obtained from the fusion, to dryness breaking up from time to time the crust that forms and hinder evaporation. When the residue is completely dry, cover the basin with a clock glass and drench the residue with about 20 ml of hydrochloric acid (sp gr 1.16). Allow to stand for a few minutes, then add about 75 ml of hot water to dissolve the salts. Digest on a steam-bath for 5 minutes, then filter through an ashless filter paper (No. 40 Whatman paper or equivalent). Transfer the silica to the filter with a jet of hot water; it is necessary to scrub the basin. Wash the residue five times with hot dilute hydrochloric acid (one percent) followed by hot water until it is free from chlorides. Reserve the residue and paper for the subsequent ignition, and transfer the filtrate and washings back to the evaporating basin.

5.4.3 Again evaporate completely to dryness, cover the basin with a clock glass and bake in an air-oven for one hour at 110°C. Allow to cool, then drench the residue with about 20 ml of hydrochloric acid (sp gr 1.16). Allow to stand for a few minutes, add about 75 ml of hot water and digest for 5 minutes on a steam-bath. Filter through another ashless filter paper (No. 40 Whatman paper or equivalent) transferring the residue to the filter with a jet of hot water and scrubbing the basin with a rubber-tipped rod. Wash five times with hot dilute hydrochloric acid (one percent), followed by hot water until the residue is free from chlorides. Reserve the filtrate and washings for the determination of ferric oxide and alumina.

5.4.4 Place the two residues and papers, without drying, in a weighed platinum crucible and heat cautiously to dry the residue and char the papers. Then burn off the carbon and finally ignite at 1 100 to 1 200°C for 30 minutes and then to constant weight. Cool and weigh to obtain the weight of impure silica.

5.4.5 Moisten the weighed residue with a few drops of dilute sulphuric acid (50 percent) and add about 10 ml of hydrofluoric acid. Evaporate slowly to dryness on a sand-bath (or under a suitable radiant heater) in a fume-hood. Ignite the dry residue at 1 050 to 1 100°C for five minutes, allow the crucible to cool and weigh. Subtract the weight of this residue from the weight of impure silica to obtain the amount of silica in the sample taken. If the residue weighs more than 5 mg, repeat the treatment with hydrofluoric and sulphuric acids to ensure that all the silica is removed.

NOTE — For routine analysis, treatment with hydrofluoric acid may be omitted.

5.4.5.1 To this amount of silica, add the amount of silica recovered from the residue derived from the combined precipitates of alumina and ferric oxide as indicated in **5.5.4** and **5.6.1**.

5.4.6 Add 0.5 g of sodium or potassium persulphate to the crucible and treat below red heat until the small residue or impurities are dissolved in the melt. Cool, dissolve the fused mass in water, add it to the filtrate and washings reserved for the determination of the combined alumina and ferric oxide.

5.5 Combined Ferric Oxide and Alumina

5.5.1 To the solution obtained in **5.4.6** (approximately 300 ml) add 2 to 3 g of solid ammonium chloride, warm the solution to about 80°C and add diluted ammonia solution (50 percent) with stirring until precipitation appears to be complete. Render the solution just alkaline to methyl red. Boil the alkaline solution for two minutes, allow to stand for five minutes for the precipitate to settle and filter through an ashless filter paper (No. 41 Whatman paper or equivalent). Transfer the precipitate to the filter and wash five times with hot faintly

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ammoniacal ammonium nitrate solution (one percent). Reserve the filtrate and washings and transfer the precipitate back to the precipitation beaker. Moisten the paper with hot dilute hydrochloric acid (50 percent) and wash thoroughly with water adding these washings to the precipitation beaker. Reserve the paper for ignition. Dissolve the residue in a slight excess of hydrochloric acid, add one gram solid ammonium chloride and dilute the solution to about 300 ml with water. Repeat the precipitation as before and when the precipitation is complete add some macerated filter-paper pulp or filter accelerator and stir vigorously. Filter through another ashless filter paper (No. 41 Whatman paper or equivalent) and wash the precipitate free from chlorides with hot faintly ammoniacal ammonium nitrate solution and reserve the precipitate and paper for ignition.

5.5.2 Acidify the combined reserved filtrate with hydrochloric acid and evaporate to about 150 ml. Make it just alkaline to methyl red with dilute ammonia solution (50 percent). Any precipitate should be filtered off, reprecipitated, washed as before and reserved for ignition. Acidify the solution or the filtrate and washings with hydrochloric acid and reserve for the determination of lime and magnesia.

5.5.3 Place the precipitates and papers reserved for ignition in a weighed platinum crucible and heat, slowly at first, to dry the precipitates and char the papers. Burn off the carbon and finally ignite the residue at 1 050 to 1 100°C to constant weight taking care to prevent reduction. The weight of combined ferric oxide, alumina and titanium oxide (together represented as R_2O_3) is thus obtained.

5.5.4 If silica is suspected to be carried into the filtrate used for this estimation, proceed as follows:

Treat the residue in the crucible with a drop of water, about 5 ml of hydrofluoric acid and a drop of sulphuric acid and evaporate cautiously to dryness. Finally heat the crucible to 1 050 to 1 100°C for one or two minutes, cool and weigh. The difference between this weight and the weight previously obtained represents the amount of residue silica. Subtract this amount from the weight of combined ferric oxide and alumina found in **5.5.3**. Add the same amount to the silica already determined in **5.4.5**.

5.6 Ferric Oxide

5.6.1 A separate determination of the ferric oxide is often not required but where called for shall be done as follows:

Heat the precipitate (*see* Note below) obtained in **5.5.3** with fused potassium or sodium pyrosulphate until solution is complete. Dissolve the fused materials in 50 ml of sulphuric acid (10 percent) and evaporate to fumes. Cool, dilute with water, and filter off the silica washing with hot water. Reserve the filtrate for the determination of ferric and titanium oxides. Ignite the silica in a

platinum crucible and weigh. Treat to precipitate with 5 ml of hydrofluoric acid and 2 to 3 drops of sulphuric acid. Evaporate to dryness, ignite and weigh. The loss in weight represents extra silica which should be added to that determined previously in 5.4.5 and also deducted from the weight of the combined oxides, R_2O_3 , obtained in 5.5.3.

NOTE — Instead of fusing directly in the platinum crucible in which the R_2O_3 was ignited, the precipitate may be brushed into a porcelain crucible and then fused with potassium or sodium pyrosulphate. This avoids loss of platinum by the action of the pyrosulphate, and no platinum is present in the filtrate to interfere with the iron determination.

5.6.2 Evaporate the filtrate obtained in 5.6.1 to about 75 ml. Cool and dilute to 100 ml in a volumetric flask. Add 25 ml of dilute hydrochloric acid (50 percent) and heat to boiling. Reduce the iron by adding stannous chloride solution drop by drop from a pipette with constant swirling of the beaker until the solution is colourless. Then add one drop in excess. Cool quickly in running water, then add at one stroke 15 ml of saturated mercuric chloride solution. Allow to stand for 3 minutes and then transfer with the washing to a 1 000-ml beaker containing 300 ml of cold distilled water and 25 ml of manganese sulphate solution. Titrate with standard 0.04 N potassium permanganate solution, added very slowly while stirring constantly, until a permanent pink end point is obtained, and calculate the amount of ferric oxide present.

5.7 Alumina — Subtract the weight of ferric oxide calculated as in 5.6.2 and the small amount of silica found in 5.6.1 from the total weight of oxides found under 5.5.3. The remainder is the weight of alumina and of small amounts of other oxides which may also be reported as alumina for purpose of this analysis.

5.8 Calcium Oxide — To the filtrate from 5.5.1 reserved after determination of the combined ferric oxide and alumina, add one gram of ammonium oxalate. Boil the solution, then add, with stirring, dilute ammonia solution (50 percent) until the solution is alkaline, followed by an excess of 10 ml of dilute ammonia solution (50 percent). Cover the beaker with a clock glass and digest on a water-bath for two hours. Allow to cool and stand, preferably overnight, then filter through a filter paper (No. 42 Whatman paper or equivalent) and wash the precipitate four times with cold ammonium oxalate solution (one percent). Reserve the filtrate and washings for the determination of magnesia. Wash the precipitate back into the precipitation beaker. Moisten the filter paper with hot dilute nitric acid (50 percent) and dilute the solution to about 80 ml. Add about 0.2 g of solid ammonium oxalate, boil the solution and precipitate the calcium oxalate as before. Allow to cool and stand for at least three hours. Filter through a filter paper (No. 42 Whatman paper or equivalent) and wash the precipitate thoroughly with cold ammonium oxalate solution (one percent).

Combine the filtrate and washing with the solution reserved for the magnesia determination and acidify with hydrochloric acid.

5.8.1 Carefully ignite the precipitate and papers in a weighed platinum crucible slowly at first, to burn off the carbon and finally at 1 100°C for 15 minutes. Allow to cool over a good desiccant as the ignited precipitate is very hygroscopic. Weigh rapidly to obtain the weight of calcium oxide.

5.9 Magnesia — Acidify the filtrate set aside in 5.8 with hydrochloric acid and concentrate to about 150 ml. Add to this solution about 10 ml of ammonium hydrogen phosphate (150 g/l) and cool the solution by placing in a beaker of ice water. After cooling, add ammonium hydroxide drop by drop while stirring constantly until the crystalline magnesium ammonium phosphate begins to form, and then add the reagent in moderate excess (5 to 10 percent of the volume of the solution), the stirring being continued for several minutes. Set the solution aside for at least 16 hours in a cool atmosphere and then filter using filter paper (No. 42 Whatman paper or equivalent). Wash with ammonium nitrate wash solution. Place in a weighed platinum crucible, slowly char the paper and carefully burn off the resulting carbon. Ignite the precipitate at 1 100°C, to constant weight, taking care to avoid bringing the pyrophosphate to melting. From the weight of the magnesium pyrophosphate obtained, calculate the magnesia content of the material taken for test.

5.10 Sulphuric Anhydride — To one gram of the sample add 25 ml of cold water and, while the mixture is being stirred vigorously, add 5 ml of hydrochloric acid. If necessary, beat the solution and grind the material with the flattened end of a glass rod until it is evident that the decomposition of the pozzolana is complete. Dilute the solution to 50 ml and digest for 15 minutes. Filter and wash the residue thoroughly with hot water. Set aside the filter paper with the residue. Dilute the filtrate to 250 ml and heat to boiling. Add slowly, drop by drop, 10 ml of hot barium chloride solution and continue the boiling until the precipitate is well formed. Digest the solution on a steambath for four hours or preferably overnight. Filter the precipitate with filter paper (No. 42 Whatman paper or equivalent), wash, place the paper and contents in a weighed platinum or porcelain crucible, and slowly incinerate the paper without inflaming. Then ignite at 800 to 900°C, cool in a desiccator and weigh the barium sulphate. From the weight of the barium sulphate obtained, calculate the sulphuric anhydride content of the material taken for test.

5.11 Determination of Soluble Salts — Weigh 25 g of the sample and transfer to a 500-ml beaker. Add 100 ml of cold distilled water and stir the contents frequently for three hours. Decant through a filter paper (No. 42 Whatman paper or equivalent) into a 500-ml graduated

flask. Refilter the filtrate, if necessary. Return the filter paper to the beaker. Add 250 ml of distilled water and continue the extraction for another 90 minutes. Filter and wash the material on the filter paper three times with cold distilled water. Add the filtrate and washings to the first filtrate in the 500-ml flask and make up to the mark using distilled water. Shake the flask well and pipette out 200 ml to a weighed platinum dish (or porcelain dish, glazed inside and outside). Evaporate to dryness, and heat to constant weight at 105 to 110°C.

Calculate the percentage weight of the soluble salts as below:

$$\text{Soluble salts, percent} = 10 w$$

where

w = weight in grams of the dried material.

5.11.1 If the soluble salt content is more than 0.2 percent, analyse it further for sulphates, chlorides and nitrates in accordance with the relevant standard methods of chemical analysis.

5.12 Reproducibility of Results — Make blank determinations on the reagents for each constituent of the pozzolana and apply corrections, where necessary. In all cases, make check determinations and repeat if satisfactory checks are not obtained. The difference between check determinations shall not exceed 0.5 percent for silica and alumina and 0.2 percent for the other constituents.

6. DETERMINATION OF FINENESS

6.0 The fineness of pozzolana shall be determined by the procedure given in **6.1** or **6.2** or by both depending upon the requirements of the specification.

6.1 Determination of Specific Surface of Pozzolana

6.1.1 Scope — This method of test covers the procedure for determining by Blaine air permeability apparatus, the fineness of pozzolana as represented by the specific surface expressed as total surface area in square centimetres per gram of material.

6.1.2 Apparatus

6.1.2.1 Nature of apparatus — The Blaine air permeability apparatus consists essentially of a means of drawing a definite quantity of air through a prepared bed of definite porosity. The number and size of the pores in a prepared bed of cement of definite porosity is a function of the size of the particles and determines the rate of air flow through the bed. The apparatus, illustrated in Fig. 1, shall consist specifically of the parts described in **6.1.2.2** to **6.1.2.8**.

6.1.2.2 Permeability cell — The permeability cell shall consist of a rigid cylinder 12.7 ± 1 mm in inside diameter, constructed of glass or

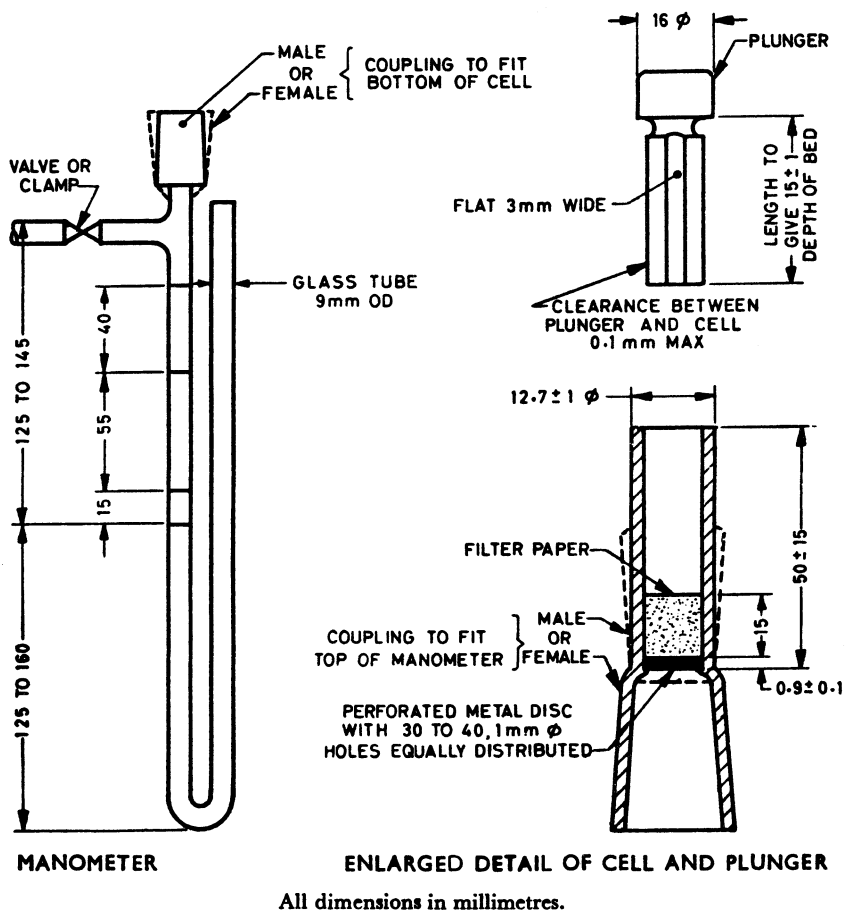


FIG. 1 BLAINE AIR PERMEABILITY APPARATUS

non-corroding metal. The top of the cell shall be at right angles to the principal axis of the cell. The bottom of the cell shall form an airtight connection with the top of the manometer. A ledge 0.5 to 1 mm in width shall be an integral part of the cell or firmly fixed in the cell 50 ± 15 mm from the top of the cell for support of the perforated metal disk.

6.1.2.3 Disk — The disk shall be constructed of non-corroding metal and shall be 0.9 ± 0.1 mm in thickness, perforated with 30 to 40 holes 1 mm in diameter equally distributed over its area. The disk shall fit the inside of the cell snugly.

6.1.2.4 Plunger — The plunger shall fit into the cell with a clearance of not more than 0.1 mm. The bottom of the plunger shall have sharp square edges and shall be at right angles to the principal axis. An air vent shall be provided either in the centre or on one side of the plunger. The top of the plunger shall be provided with a collar such that when the plunger is placed in the cell and the collar brought in contact with the top of the cell, the distance between the bottom of the plunger and the top of the perforated disk shall be 15.0 ± 1.0 mm.

6.1.2.5 Filter paper — The filter paper shall be medium retentive (corresponding to No. 40 Whatman). The filter paper disks shall be circular, with smooth edges, and shall have the same diameter as the inside of the wall. (Filter paper disks that are too small may leave part of the sample adhering to the inner wall of the cell above the top disk. When too large in diameter, the disks have tendency to buckle and cause erratic results.)

6.1.2.6 Manometer — The U-tube manometer shall be constructed according to the design indicated in Fig. 1, using nominal 9 mm outside diameter, standard-wall glass tubing. The top of one arm of the manometer shall form an airtight connection with the permeability cell. The manometer arm connected to the permeability cell shall have a line etched around the tube at 125 to 145 mm below the top of the side outlet and also others at distances of 15, 70 and 110 mm above that line. A side outlet shall be provided at 250 to 305 mm above the bottom of the manometer for use in the evacuation of the manometer arm connected to the permeability cell. A positive airtight valve or clamp shall be provided on the side outlet not more than 50 mm from the manometer arm. The manometer shall be mounted firmly and in such a manner that the arms are vertical.

6.1.2.7 Manometer liquid — The manometer shall be filled to the mid-point with a nonvolatile, nonhygroscopic liquid of low viscosity and density, such as dibutylphthalate (dibutyl 1, 2-benzenedicarboxylate) or a light grade of mineral oil.

6.1.2.8 Timer — The timer shall have a positive starting and stopping mechanism and shall be capable of being read to the nearest 0.5 second or less. The timer shall be accurate to 0.5 second or less for time intervals up to 60 seconds and to one percent or less for time intervals of 60 to 300 seconds.

6.1.3 Calibration of Apparatus

6.1.3.1 Sample — The calibration of the air permeability apparatus shall be made using National Bureau of Standards Standard Sample No. 114 or any other cement sample having specific surface equal to the appropriate certificate value of NBS Standard Sample No. 114. The sample shall be at room temperature when tested.

NOTE — In the absence of NBS Standard Sample No. 114, samples of Indian cements having specific surface equivalent to NBS Standard Sample No. 114 may be used; such samples may be obtained from the ACC Central Research Station, Bombay-Agra Road, P.O. Wagle Industrial Estate, (Thana).

6.1.3.2 Bulk volume of compacted bed of powder — The bulk volume of the compacted bed of powder shall be determined by the mercury displacement method as follows:

Place two filter paper disks in the permeability cell, pressing down the edges with a rod slightly smaller than the cell diameter until the filter disks are flat on the perforated metal disk; then fill the cell with mercury, removing any air bubbles adhering to the wall of the cell. Use tongs when handling the cell. If the cell is made of material that will amalgamate with mercury, the interior of the cell shall be protected by a very thin film of oil just prior to adding the mercury. Level the mercury with the top of the cell by pressing a small glass plate against the mercury surface until the glass is flush with the surface of the mercury. Check that no void or bubble exists between the mercury surface and the glass plate.

Remove the mercury from the cell, weigh and record the weight of mercury. Remove one of the filter disks from the cell. Using a trial quantity of 2.80 g of cement (*see* Note 1) compress the cement (*see* Note 2) in accordance with **6.1.3.5**, with one filter disk above and one below the sample. Fill the space remaining in the top of the cell with mercury, remove entrapped air, and level off the top as before. Remove the mercury from the cell, weigh and record the weight of mercury.

The bulk volume occupied by the cement shall be calculated to the nearest 0.005 cm^3 as follows:

$$V = \frac{W_a - W_b}{D} \dots\dots\dots(1)$$

where

V = bulk volume of cement in cm^3 ;

W_a = grams of mercury required to fill cell, no cement being in cell;

W_b = grams of mercury required to fill the portion of the cell not occupied by the prepared bed of cement in the cell; and

D = density of mercury at temperature of test in g/cm^3 (*see* Table 1).

At least two determinations of bulk volume of cement shall be made, using separate compactions for each determination. The bulk volume value used for subsequent calculations shall be the average of two values agreeing within plus or minus 0.005 cm^3 . The temperature in the vicinity of the cell shall be noted and recorded at the beginning and end of determination.

NOTE 1 — It is not necessary to use the standard sample for the bulk volume determination.

NOTE 2 — The prepared bed of cement shall be firm. If too loose or if the cement cannot be compressed to the desired volume, adjust the trial quantity of cement used.

TABLE 1 DENSITY OF MERCURY, VISCOSITY OF AIR (n) AND \sqrt{n} AT GIVEN TEMPERATURE

(Clause 6.1.3.2)

ROOM TEMPERATURE °C	DENSITY OF *MERCURY g/cm ³	VISCOSITY OF AIR, n , POISES	\sqrt{n}
16	13.56	0.000 178 8	0.013 37
18	13.55	0.000 179 8	0.013 41
20	13.55	0.000 180 8	0.013 44
22	13.54	0.000 181 8	0.013 48
24	13.54	0.000 182 8	0.013 52
26	13.53	0.000 183 7	0.013 55
28	13.53	0.000 184 7	0.013 59
30	13.52	0.000 185 7	0.013 62
32	13.52	0.000 186 7	0.013 66
34	13.51	0.000 187 6	0.013 69

*The density of the mercury shall be determined separately.

6.1.3.3 Preparation of sample — The contents of a vial of the standard cement sample shall be enclosed in a 125 gram jar and shaken vigorously for 2 minutes to fluff the cement and break up lumps or agglomerates.

6.1.3.4 Weight of sample — The weight of the standard sample used for the calibration test shall be that required to produce a bed of cement having a porosity of 0.500 ± 0.005 and shall be calculated as follows:

$$W = \rho V (1 - e) \dots\dots\dots (2)$$

where

W = sample required in grams;

ρ = specific gravity of test sample (for Portland cement, a value of 3.15 shall be used);

V = bulk volume of bed of cement in cm³, as determined in accordance with **6.1.3.2**; and

e = desired porosity of bed of cement (0.500 ± 0.005).

6.1.3.5 Preparation of bed of cement — The perforated disk shall be seated on the ledge in the permeability cell. A filter paper disk shall be placed on the metal disk and the edges pressed down with a rod slightly smaller than the cell diameter. A quantity of cement

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determined in accordance with **6.1.3.4** and weighed to the nearest 0.001 g shall be placed in the cell. The side of the cell shall be tapped lightly in order to level the bed of cement. A filter paper disk shall be placed on top of the cement and the cement compressed with the plunger until the plunger collar is in contact with the top of the cell. The plunger shall then be removed slowly. Use of fresh disks is required for each determination.

6.1.3.6 Permeability test — The permeability cell shall be attached to the manometer tube, making certain that an airtight connection is obtained (*see* Note 1) and taking care not to jar or disturb the prepared bed of cement.

The air in the one arm of the manometer U-tube shall be slowly evacuated until the liquid reaches the top mark, and the valve shall then be closed tightly. The timer shall be started as the bottom of the meniscus of the manometer liquid reaches the second (next to the top) mark and shall be stopped as the bottom of meniscus of liquid reaches the third (next to the bottom) mark. The time interval measured shall be noted and recorded in seconds. The temperature of test shall be noted and recorded in degrees centigrade.

In the calibration of the instrument, at least three determinations of the time of flow shall be made on each of three separately prepared beds of the standard sample (*see* Note 2). The calibration shall be made by the same operator who makes the fineness determination.

NOTE 1 — If a rubber stopper is used for the connection, it should be moistened with water; if a standard-taper joint is used, a little stopcock grease should be applied. The efficiency of the connection may be determined by inserting stopper at the top of the cell attached to the manometer and then partially evacuating the one arm of the manometer; then closing the valve. Any continuous drop in pressure indicates a leak in the system.

NOTE 2 — The sample may be re-fluffed and reused for preparation of the test bed, provided that it is kept dry and all tests are made within 4 hours of the opening of the sample.

6.1.3.7 Re-calibration — The apparatus shall be re-calibrated (*see* Note) at periodic intervals to check:

- a) for possible wear on plunger or permeability cell,
- b) if any loss in manometer fluid occurs, and
- c) if a change is made in the type or quality of the filter paper used for the test.

NOTE — It is suggested that a secondary sample be prepared and used as a fineness standard for the check determinations of the instrument between regular calibrations with the standard cement sample.

6.1.4 Procedure

6.1.4.1 Temperature of the test sample — The pozzolana sample shall be at room temperature when tested.

6.1.4.2 Size of test sample — The weight of the pozzolana sample used for the test shall be adjusted so that a firm, hard bed is produced in the cell by compaction process.

6.1.4.3 Preparation of the bed of test sample — The test bed of pozzolana sample shall be prepared in accordance with the method described in **6.1.3.5**.

6.1.4.4 Permeability test — The permeability test shall be made in accordance with the method described in **6.1.3.6** except that only one time of flow determination need be made on one prepared bed of pozzolana.

6.1.5 Calculations — Calculation of specific surface values shall be made according to the following formulae:

$$S = \frac{S_s \rho_s (1 - e_s) \sqrt{e^3} \sqrt{T}}{\rho (1 - e) \sqrt{e_s^3} \sqrt{T_s}} \dots\dots\dots (3)$$

$$S = \frac{S_s \rho_s (1 - e_s) \sqrt{n_s} \sqrt{e^3} \sqrt{T}}{\rho (1 - e) \sqrt{e_s^3} \sqrt{T_s} \sqrt{n}} \dots\dots\dots (4)$$

where

S = specific surface in cm^2/g of the test sample;

S_s = specific surface in cm^2/g of the standard sample used in calibration of the apparatus;

ρ_s = specific gravity of standard sample used in calibration of apparatus (assumed to be **3.15**);

e_s = porosity of prepared bed of standard sample used in calibration of apparatus;

e = porosity of prepared bed of test sample;

T = measured time interval, in seconds, of manometer drop for test sample;

ρ = specific gravity of test sample (for Portland cement value of **3.15** shall be used);

T_s = measured time interval in seconds of manometer drop for standard sample used in calibration of the apparatus;

n_s = viscosity of air in poises at the temperature of test of the standard sample used in calibration of the apparatus; and

n = viscosity of air in poises at the temperature of test of the test sample.

NOTE 1 — Values for \sqrt{n} and $\sqrt{e^3}$ may be taken from Tables 1 and 2 respectively.

NOTE 2 — Equation 3 shall be used when the temperature of test and of the test sample is within $\pm 3^\circ\text{C}$ of the temperature of calibration test of the standard fineness sample, and equation 4 shall be used if the temperature of tests is outside this range.

TABLE 2 VALUES FOR POROSITY OF CEMENT BED

(Clause 6.1.5)

POROSITY OF BED, e	$\sqrt{e^3}$
0.495	0.348
0.496	0.349
0.497	0.350
0.498	0.352
0.499	0.353
0.500	0.354
0.501	0.355
0.502	0.356
0.503	0.357
0.504	0.358
0.505	0.359
0.506	0.360
0.507	0.361
0.508	0.362
0.509	0.363
0.510	0.364
0.525	0.380
0.526	0.381
0.527	0.383
0.528	0.384
0.529	0.385
0.530	0.386
0.531	0.387
0.532	0.388
0.533	0.389
0.534	0.390
0.535	0.391

6.1.6 Retests — A retest shall consist of two determinations made as prescribed in 6.1.4 and 6.1.5, using a freshly prepared test bed of the sample for each determination. Care shall be exercised in the preparation of the test beds, and precautions shall be taken to ensure an airtight connection between the permeability cell and the manometer arm. The fineness value reported shall be the average of the values calculated from retest determinations agreeing within 2 percent of each other.

NOTE — If tests are done at e other than 0.5, the actual porosity value shall be reported along with fineness value, since it is known that the indicated value for fineness changes with the decrease or increase of the value of e .

6.2 Determination of Fineness by Sieving

6.2.1 Scope — This method of test covers the procedure for determining the fineness of particles by a sieve analysis.

6.2.2 Apparatus — Following sieves conforming to IS : 460-1962* shall be used:

300-micron IS Sieve	For dry sieving
150-micron IS Sieve	For dry sieving
75-micron IS Sieve	For wet sieving
45-micron IS Sieve	For wet sieving

NOTE — Where the requirements in the individual specification for a pozzolanic material differ from the provisions of this clause, the former shall prevail.

6.2.3 Preparation of Samples — A representative sample shall be taken in accordance with the sampling procedure given in the respective specification for the material, and shall be thoroughly mixed. The weight of the representative sample shall not be less than 200 g. The sample for sieving shall be prepared from the laboratory samples as described in IS : 1607-1960†.

6.2.4 Drying Sieving — The sample shall be passed successively through 300-micron IS Sieve and 150-micron IS Sieve, starting with the largest. Sieving shall be done and the results reported in accordance with IS : 1607-1960†.

6.2.5 Wet Sieving — Separate determinations shall be made for the percentage of material passing 75-micron IS Sieve and 45-micron IS Sieve by wet sieving.

6.2.5.1 Place 100 g of the material over each of the two sieves. Wash the material with a jet of water and keep it well agitated. The washing shall continue till the washings appear no more turbid. Dry the sieve and the residue in an oven. Brush the residue from the sieve after drying, and weigh on a balance sensitive to 0.1 percent of the weight of the test sample. The percentage of material passing each sieve on wet

*Specification for test sieves (revised).

†Methods for dry sieving.

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sieving shall be reported to the nearest 0.1 percent by weight of the test sample.

7. DETERMINATION OF SOUNDNESS

7.0 Either of the methods given in **7.1** and **7.2** may be used for determining soundness of pozzolanic test mixtures.

7.1 ‘Le Chatelier’ Method — The test shall be done as specified in IS : 4031-1968* Methods of Physical Tests for ‘Hydraulic Cement’ except that in place of cement, a mixture of pozzolana and cement in the proportion 0.2 *N* : 0.8 by weight, blended intimately shall be used:

where

$$N = \frac{\text{Specific gravity of pozzolana}}{\text{Specific gravity of cement}}$$

7.2 Autoclave Test — The test shall be done as specified in IS : 4031-1968* except that a mixture of pozzolana and cement in the proportion as indicated in **7.1** shall be used in place of cement.

7.2.1 When determining soundness by this method, not less than six specimens shall be tested at a time for arriving at an average result.

8. DETERMINATION OF INITIAL AND FINAL SETTING TIME

8.1 The test for initial and final setting time of pozzolana with lime and cement shall be done in accordance with the requirements of IS : 4031-1968* except that a mixture of pozzolana and cement in the proportion as indicated in **7.1** shall be used in place of cement for making the paste. The determination of standard consistency shall also be carried out in accordance with IS : 4031-1968* using the mixture of pozzolana and cements in the proportion as indicated in **7.1** instead of cement for making the paste.

9. DETERMINATION OF LIME REACTIVITY

9.1 Scope — This method of test covers the procedure for determining the reactivity of the pozzolanic material with hydrated lime, as represented by compressive strength of standard mortar test cubes prepared and tested under specific conditions.

9.2 Size and Number of Test Specimens

9.2.1 The tests specimen shall be 50-mm cubes.

*Methods of physical tests for hydraulic cement.

9.2.2 Unless otherwise specified in the relevant specification for the pozzolana, three or more specimens shall be made for each period of test specified.

9.3 Apparatus

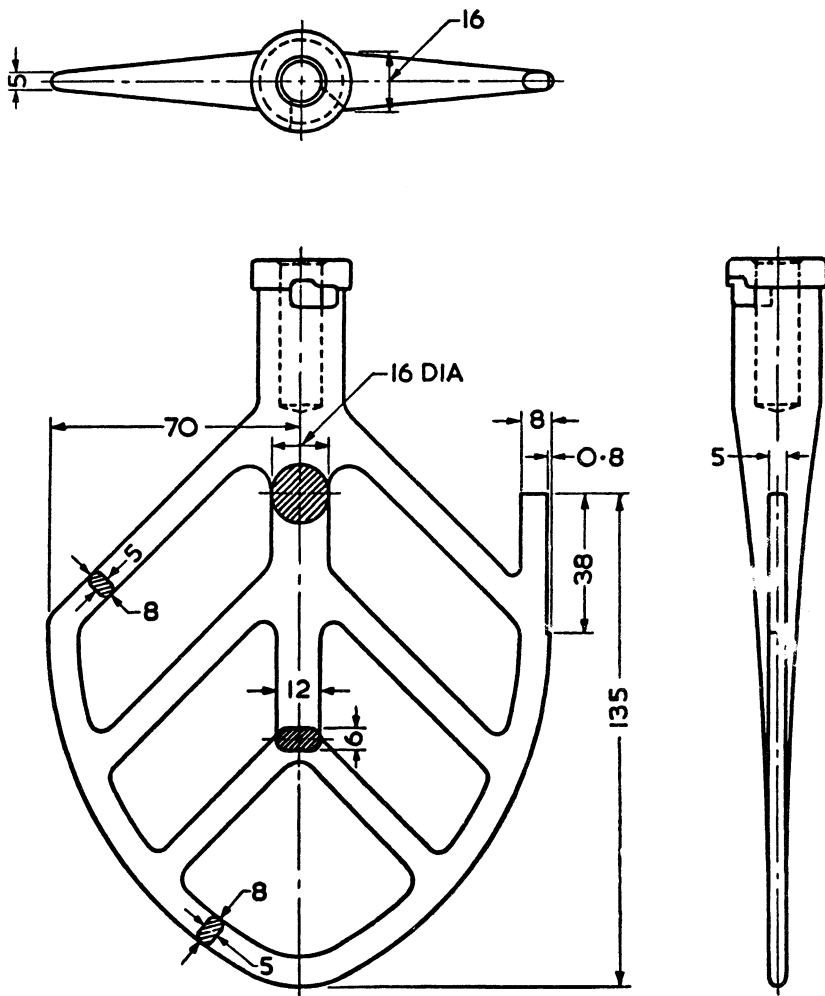
9.3.1 Cube Moulds — Moulds for the 50-mm cube specimens shall be of metal not attacked by cement-pozzolana or lime-pozzolana mortar and there shall be sufficient strength and stiffness to prevent spreading and wrapping. The moulds shall be rigidly constructed in such a manner as to facilitate the removal of the moulded specimen without damage. The moulds shall be machined so that when assembled ready for use the dimensions and internal faces shall be accurate to the following limits:

The height of the moulds and the distance between the opposite faces shall be 50 ± 0.1 mm for new moulds, and 50 ± 0.5 mm for moulds in use. The angle between adjacent interior faces and between interior faces and top and bottom planes of the mould shall be 90 ± 0.5 degrees. The interior faces of the moulds shall be plane surfaces with a permissible variation of 0.02 mm for new moulds and 0.05 mm for moulds in use. Each mould shall be provided with a base plate having a plane surface machined to a tolerance of 0.1 mm and made of non-absorbent and non-corrodable material. The base plate shall be of such dimensions as to support the mould during the filling without leakage.

9.3.2 Mixing Apparatus — This shall have components and accessories as described in **9.3.2.1** to **9.3.2.4**.

9.3.2.1 Mixer — The mixer shall be an electrically driven mechanical mixer of the epicyclic type, which imparts both a planetary and a revolving motion to the mixer paddle. The mixer shall have at least two speeds, controlled by definite mechanical means. (Rheostat adjustment of speed shall not be acceptable.) The first or slow speed shall revolve the paddle at a rate of 140 ± 5 rev/min, with a planetary motion of approximately 62 rev/min. The second speed shall revolve the paddle at a rate of 285 ± 10 rev/min, with a planetary motion of approximately 125 rev/min. The electric motor shall have a power of about 150 W. The mixer shall be capable of adjustment so that when the bowl is in the mixing position the clearance between the lower end of the paddle and the bottom of the bowl shall be approximately 2.5 mm but not less than the approximate diameter of a grain of the standard sand (see **3.3**).

9.3.2.2 Paddle — The paddle shall be readily removable, made of stainless steel, and shall conform to the basic design shown in Fig. 2. The dimensions of the paddle shall be such that when it is in the mixing position the paddle outline conforms to the contour of the bowl used with the mixer, and the clearance between corresponding points



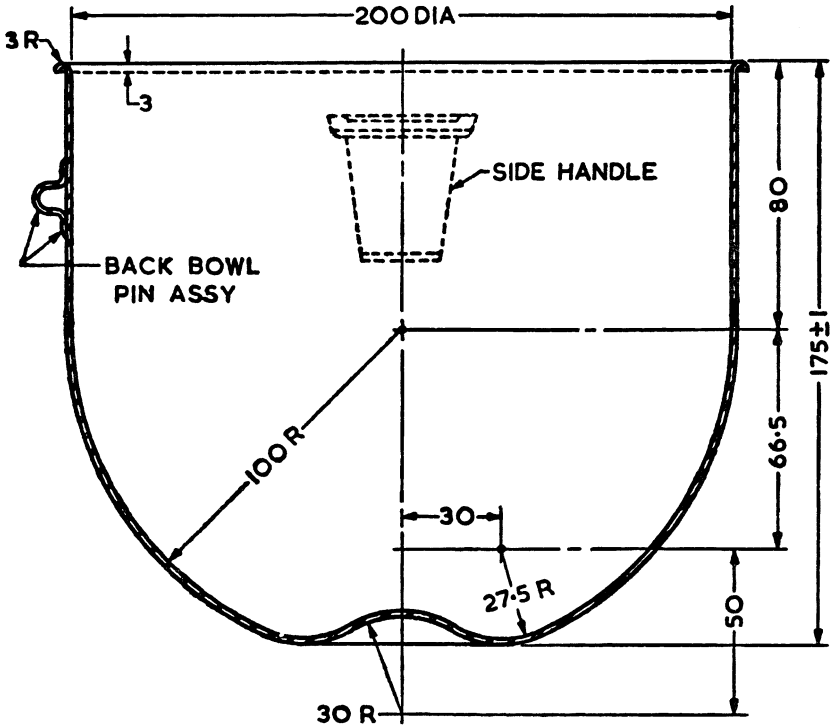
All dimensions in millimetres.

FIG. 2 PADDLE

on the edge of the paddle and the side of the bowl in the position of closest approach shall be approximately 4 mm but not less than 0.85 mm.

9.3.2.3 Mixing bowl — The mixing bowl shall be removable and shall have a nominal capacity of about six litres. It shall be of the general shape shown in Fig. 3. It shall comply with the limiting dimensions

shown in Fig. 2 and shall be made of stainless steel. The bowl shall be so equipped that it will be positively held in the mixing apparatus in a fixed position during the mixing procedure. It shall be provided with a lid made of non-absorbing material and not attacked by the pozzolanic mixture.



All dimensions in millimetres.

FIG. 3 MIXING BOWL

9.3.2.4 Scraper — The scraper shall consist of a semi-rigid rubber blade attached to a handle about 150 mm long. The blade shall be about 75 mm long, 50 mm wide, and tapered to a thin edge about 1.5 mm thick.

9.3.3 Flow Table and Accessories — The flow table and accessories shall conform to the requirements given in IS : 5512-1969*.

*Flow table for use in tests of hydraulic cement and pozzolanic materials.

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9.3.4 Tamping Rod — The tamping rod shall be made of a non-absorptive, non-abrasive, non-brittle material, such as a rubber compound having a Shore A durometer hardness of 80 ± 10 or seasoned teak wood rendered non-absorptive by immersion for 15 minutes in paraffin at approximately 200°C , and shall have a cross-section of 12×25 mm and a convenient length (125×150 mm). The tamping face shall be flat and at right angles to the length of the tamping rod.

9.3.5 Trowel — This shall have a steel blade 100 to 150 mm in length with straight edge.

9.4 Preparation of Moulds — The interior faces of the specimen moulds shall be thinly covered with mineral oil or light cup grease. After assembling the moulds, excessive oil or grease shall be removed from the interior faces and the top and bottom surfaces of each mould. Moulds shall then be set on plane, non-absorbent base plates that have been thinly coated with mineral oil, or light cup grease.

9.5 Preparation of Mortar

9.5.1 Clean appliances shall be used for mixing. The temperature of the water and that of the test room at the time when the mixing operation is being performed shall be from $27 \pm 2^{\circ}\text{C}$ (*see* IS : 196-1966*).

9.5.2 The dry materials of the standard test mortar shall be lime: pozzolana: standard sand in proportion 1 : $2M$: 9 by weight blended intimately:

where

$$M = \frac{\text{Specific gravity of pozzolana}}{\text{Specific gravity of lime}}$$

The amount of water for gauging shall be equal to that required to give a flow of 70 ± 5 percent with 10 drops in 6 seconds as determined in **9.5.3**.

9.5.2.1 The following quantities of materials are suggested for preparation of mortar:

150 g	Hydrated lime
$300 \times M$ g	Pozzolana
1350 g	Standard sand

These quantities will suffice for preparing six test specimens according to **9.6**.

9.5.3 Determination of Flow

9.5.3.1 Trial mixing — With dry material as specified in **9.5.2** make trial mortars with different percentages of water until specified flow is

*Specification for atmospheric conditions for testing (*revised*).

obtained. Make each trial with fresh mortar. The mixing shall be done mechanically by means of mixing apparatus as described in 9.3.2. Place the dry paddle and the dry bowl in the mixing position in the mixer. Then introduce the materials for batch into the bowl and mix in the following manner:

- a) Place all the mixing water in the bowl.
- b) Add the pozzolanic mixture to the water, then start the mixer and mix at the slow speed (140 ± 5 rev/min) for 30 seconds.
- c) Add the entire quantity of sand slowly over a period of 30 seconds, while mixing at slow speed.
- d) Stop the mixer, change to medium speed (285 ± 10 rev/min), and mix for 30 seconds.
- e) Stop the mixer, and let the mortar stand for one and a half minutes. During the first 15 seconds of this interval, quickly scrape down into the batch any mortar that may have collected on the side of the bowl, then for the remainder of this interval, cover the bowl with the lid.
- f) Finish by mixing for one minute at medium speed (285 ± 10 rev/min).
- g) In any case requiring a remixing interval, any mortar adhering to the side of the bowl shall be quickly scraped down into a batch with the scraper prior to remixing.

Upon the completion of mixing the mixing paddle shall be shaken to remove excess mortar into the mixing bowl.

9.5.3.2 Carefully wipe the flow-table top clean and dry and place the mould at the centre. Place a layer of mortar mixed in accordance with **9.5.3.1** about 25 mm in thickness in the mould and tamp 20 times with the tamping rod. The tamping pressure shall be first sufficient to ensure uniform filling of the mould. Then fill the mould with mortar and tamp as specified for the first layer. Cut off the mortar to a plane surface flush with the top of the mould by drawing the straight edge of a trowel (held nearly perpendicular to the mould) with a sawing motion across the top of the mould. Wipe the table top clean and dry, particularly taking care to remove any water from around the edge of the flow mould. Lift the mould away from the mortar one minute after completing the mixing operation. Immediately drop the table through a height of 12.5 mm, ten times in six seconds.

The flow is the resulting increase in average base diameter of the mortar mass, measured on at least four diameters at approximately equispaced intervals expressed as a percentage of the original base diameter.

9.5.4 The materials for each batch of moulds shall be mixed separately using the quantities of dry materials, conforming to the proportions

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specified in **9.5.2** and the quantity of water as determined in **9.5.3**. Mixing of mortar shall be done mechanically as described in **9.5.3.1**.

9.6 Moulding Test Specimens — Immediately following the preparation of the mortar in accordance with **9.5.4** place the mortar in a 50-mm cube mould in a layer of about 25 mm thickness and tamp 25 times with the tamping rod. The tamping shall be just sufficient to ensure uniform filling of the mould. Then fill the mould to overflow and tamp as specified for the first layer. On the completion of the tamping, the tops of all cubes shall extend slightly above the tops of the moulds. Cut off the mortar to a plane surface flush with the top of the mould by drawing the straight edge of a trowel (held nearly perpendicular to the mould) with a sawing motion across the top of the mould.

9.7 Storage and Curing of Specimens — Cover the surface of the specimen in the mould with a smooth and greased glass plate. Keep the specimens with the moulds along with the cover plates under wet gunny bags for 48 hours. Then remove the specimens from the moulds and cure at 90 to 100 percent relative humidity at $50 \pm 2^\circ\text{C}$ for a period of eight days. Samples shall not be cured under water.

NOTE — For curing the specimen at the specified relative humidity and temperature as given above, it is recommended that the controlled humidity and temperature chamber developed by National Council for Cement and Building Materials may be used for proper performance.

9.8 Procedure of Test — Remove the specimens after curing for eight days in the incubator as described above and test for compressive strength after they reach the room temperature. Test not less than three cubes. The cubes shall be tested on their sides without any packing between the cube and steel platens on the testing machine. One of the platens shall be carried on a ball and shall be self-adjusting, and the load shall be steadily and uniformly applied, starting from zero and at a rate of $35 \text{ kg/cm}^2/\text{min}$.

9.9 Calculation — Calculate the compressive strength from the crushing load and the average area over which the load is applied. Express the result nearest to 1 kg/cm^2 .

9.10 Faulty Cubes and Retests — Cubes that are manifestly faulty or that give strengths differing by more than 15 percent from the average value of all test specimens, made from the same sample and tested at the same period, shall not be considered in determining the compressive strength. After discarding such cubes, if less than two strength test values are left for determining the compressive strength at any given period, a retest shall be made.

10. DETERMINATION OF COMPRESSIVE STRENGTH OF POZZOLANA-CEMENT MORTAR

10.1 Scope — This method of test covers the procedure for determining the compressive strength of pozzolana mortar as obtained

in standard mortar test cubes prepared and tested under specified conditions.

10.2 Size of specimens — The test specimens shall be 50 mm cubes.

10.3 Apparatus — The cube moulds, mixing apparatus, flow table, tamping rod and trowel shall be the same as specified in 9.3.

10.4 Preparation of Moulds — The moulds shall be prepared in the same manner as described in 9.4.

10.5 Preparation of Mortar

10.5.1 Clean appliances shall be used for mixing and the temperature of the water and that of the test room at the time when the mixing operation is being performed shall be $27 \pm 2^\circ\text{C}$ (see IS : 196-1966*).

10.5.2 The dry materials for the standard test mortar shall be pozzolana : cement : standard sand in proportion 0.2 *N* : 0.8 : 3 by weight, blended intimately,

where

$$N = \frac{\text{Specific gravity of pozzolana}}{\text{Specific gravity of cement}}$$

The amount of water for gauging shall be equal to that required to give a flow of 105 ± 5 percent with 25 drops in 15 seconds as determined in 9.5.3.

10.5.2.1 The following quantities of materials are suggested for preparation of the mortar:

100 × <i>N g</i>	Pozzolana
400 g	Cement
1500 g	Standard sand

These quantities will suffice for preparing six test specimens according to 10.6.

10.5.3 The materials for each batch of moulds shall be mixed separately using the quantities of dry materials, conforming to the proportions given in 10.5.2 and the quantity of water as determined in accordance with the procedure given in 9.5.3 to give a flow of 105 ± 5 percent with 25 drops in 15 seconds. Mixing of mortar shall be done mechanically as described in 9.5.3.1.

10.6 Moulding Test Specimens — Immediately after mixing the mortar in accordance with 9.5.3, place the mortar in 50-mm cube moulds and prepare test cubes in accordance with the method as given under 9.6.

10.7 Storage and Curing of Specimens — Keep the cubes at a temperature of $27 \pm 2^\circ\text{C}$ in an atmosphere of at least 90 percent

*Specification for atmospheric conditions for testing (revised).

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relative humidity for 24 hours after completion of compaction. At the end of that period, remove them from the moulds and immediately submerge in clean fresh water and keep there until taken out just prior to breaking. The water in which the cubes are submerged shall be renewed every seventh day and shall be maintained at a temperature of $27 \pm 2^{\circ}\text{C}$. After they have been taken out and until they are broken, the cubes shall not be allowed to become dry.

10.8 Procedure of Test

10.8.1 Test not less than three cubes for compressive strength for each of the periods of 7, 28 and 90 days, the periods being reckoned from the completion of compaction. The compressive strength shall be the average of the strengths of the three cubes for each period respectively.

10.8.2 The cubes shall be tested on their sides without any packing between the cube and the steel platens of the testing machine. One of the platens shall be carried on a base and shall be self-adjusting, and the load shall be steadily and uniformly applied, starting from zero at a rate of $140 \text{ kg/cm}^2/\text{min}$.

10.9 Calculation — Calculate the compressive strength from the crushing load and the average area over which the load is applied. Express the result nearest to 1 kg/cm^2 .

10.10 Faulty Cubes and Retests — Cubes that are manifestly faulty or that give strengths differing by more than 15 percent from the average value of all test specimens, made from the same sample and tested at the same period, shall not be considered in determining the compressive strength. After discarding such cubes, if less than two strength test values are left for determining the compressive strength at any given period, a retest shall be made.

10.11 Corresponding Test on Plain Cement-Sand Mortar Cubes — For the purpose of comparison, similar tests with cubes from mortar containing no pozzolana shall be carried out as follows:

Not less than three mortar cubes shall be prepared out of a mix with one part of ordinary Portland cement used in this test and three parts of standard sand, stored and tested in the same manner as described in **10.5** to **10.9** and their average strength calculated.

11. DETERMINATION OF TRANSVERSE STRENGTH OF THE POZZOLANA-CEMENT MORTAR

11.1 Scope — This method of test covers the procedure for determining the transverse strength of the pozzolana mortars as obtained on standard rectangular specimens prepared and tested under specified conditions.

11.2 Apparatus

11.2.1 *Mixing Apparatus* — The mixing apparatus shall be same as specified in 9.3.2.

11.2.2 *Jolting Apparatus* — A typical design of the jolting apparatus is shown in Fig. 4. The apparatus shall consist essentially of a rectangular table rigidly connected by two light arms to a spindle at a horizontal distance of 800 mm from the centre of the table. The table shall incorporate on its lower face a projecting lug with a plane face, beneath which is a small stop with a rounded upper surface. When the projecting lug rests on the stop, its plane face and that of the table shall be horizontal. There shall be provided a cam made of hard or case-hardened steel, by means of which the table can be raised and then allowed to fall freely from a height of 15 mm before the lug strikes the stop.

11.2.2.1 The cam shall be driven by means of an electric motor of about 250 W and a reduction gear at a speed of one revolution per second. It is recommended that the motor be provided with a device which automatically stops it after 60 jolts.

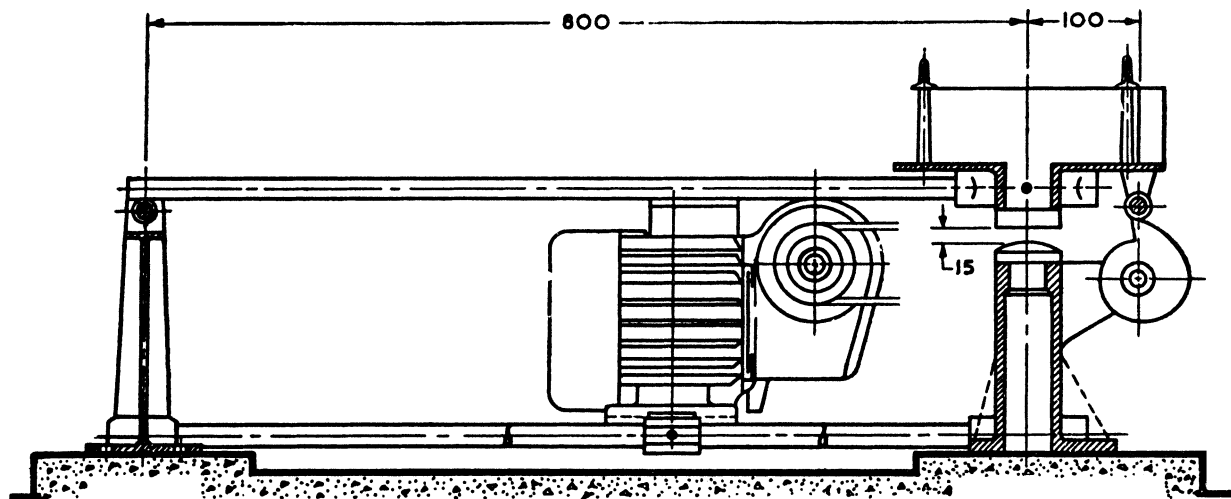
11.2.2.2 There shall be arrangements to place the mould shell on the table in such a way that the length of the three compartments is perpendicular to the axis of rotation of the cam. The mould shall be located on the table with the aid of suitable reference marks, so that the centre of the central compartment is directly above the point of percussion. The mould surmounted by its hopper shall be clamped rigidly to the table, for example, by means of wing nuts.

11.2.2.3 The combined weight of the table, mould, hopper and clamps shall be 20 ± 1 kg.

11.2.2.4 The apparatus shall be fixed on a concrete base $1\ 000 \times 300$ mm and 800 mm high. The base plates of the two frames carrying the cam and the spindle about which the table rotates shall each be fixed to the concrete base by means of four anchor bolts, and when fixing them, a thin layer of rich mortar shall be placed between the base plates and the concrete base in order to ensure perfect contact.

11.2.2.5 To reduce noise, the concrete base shall be placed on four rubber pads of suitable size. The table shall be horizontal when at rest, and the common normal through the point of contact of the lug and stop shall be vertical. The lug striking face and the stop shall be replaced as soon as the above condition is no longer met. Ball bearings shall be employed for the spindles about which the table and the cam rotate. If plain bearings are used, the play of the spindles in them shall not exceed 0.1 mm.

11.2.3 *Moulds* — The moulds shall be made of hard steel not readily attacked by the pozzolana mortar and shall embody three



All dimensions in millimetres.

FIG. 4 JOLTING APPARATUS

compartments permitting the preparation of three specimens at a time. The internal dimension of each compartment, when assembled ready for use, shall be:

Length	160 ± 0.4 mm
Width	40 ± 0.1 mm
Height	40 ± 0.1 mm

11.2.3.1 The mould walls shall be at least 10 mm thick, the internal opposite faces of 40 × 160 mm shall be plane to within 0.02 mm and the angle between the base and the mould shall be 90 ± 0.5°.

11.2.3.2 The mould shall rest on a machined steel base plate to which it shall be securely clamped. The mould shall be rigidly constructed but in such a manner as to facilitate the removal of the moulded specimen without damage. The base plate shall accurately fit the mould to prevent any leakage during filling and the parts of the mould when assembled shall be ensured by suitable methods to be positively held together both during the filling and subsequent removal of the filled mould from the jolting apparatus, in order to prevent any damage to the moulded specimen.

11.2.3.3 Each compartment shall, in turn, be surmounted by a hopper made of steel or non-ferrous metal, with vertical walls 20 to 40 mm in height. During filling, the interior vertical surfaces of the hopper shall be within those of the compartment by a distance not exceeding 0.4 mm.

11.2.4 Apparatus of Transverse Test — The apparatus for testing transverse strength shall consist of two supports in the form of rollers 10 mm in diameter and spaced 100 mm apart, where the test prism is placed centrally with one of its faces resting on them, and of a third roller of the same diameter equidistant from the first two and transmitting the applied load to the opposite face of the prism. One of the supporting rollers shall be self-aligning. The apparatus shall be fixed on to a testing machine which is capable of applying and measuring small loads (less than 1.00 kg) with an accuracy of one percent in the upper four-fifths of its range.

11.3 Size and Number of Test Specimens

11.3.1 The specimens shall be in the form of a rectangular bar of dimensions 160 × 40 × 40 mm.

11.3.2 Unless otherwise specified in the relevant specification for the pozzolana, three or more specimens shall be made for each period of test specified.

11.4 Preparation of Moulds — In assembling the moulds ready for use, cover the joints between the parts of the mould with a thin film of petroleum jelly and apply a similar coating of petroleum jelly between the contact surfaces of the bottom of the mould and its base plate in order to ensure that no water escapes during vibration. Treat the interior faces of the mould with a thin coating of mould oil.

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11.4.1 The mould and its hopper shall be fixed securely on the table of the jolting apparatus by means of the clamp.

11.5 Preparation of Mortar

11.5.1 Clean appliances shall be used for mixing and the temperature of the water and that of the test room at the time when the mixing operation is being performed shall be $27 \pm 2^\circ\text{C}$.

11.5.2 The dry materials of the standard test mortar shall be pozzolana : cement : standard sand in proportion 0.2 N : 0.8 : 3 by weight, blended intimately:

where

$$N = \frac{\text{Specific gravity of pozzolana}}{\text{Specific gravity of cement}}$$

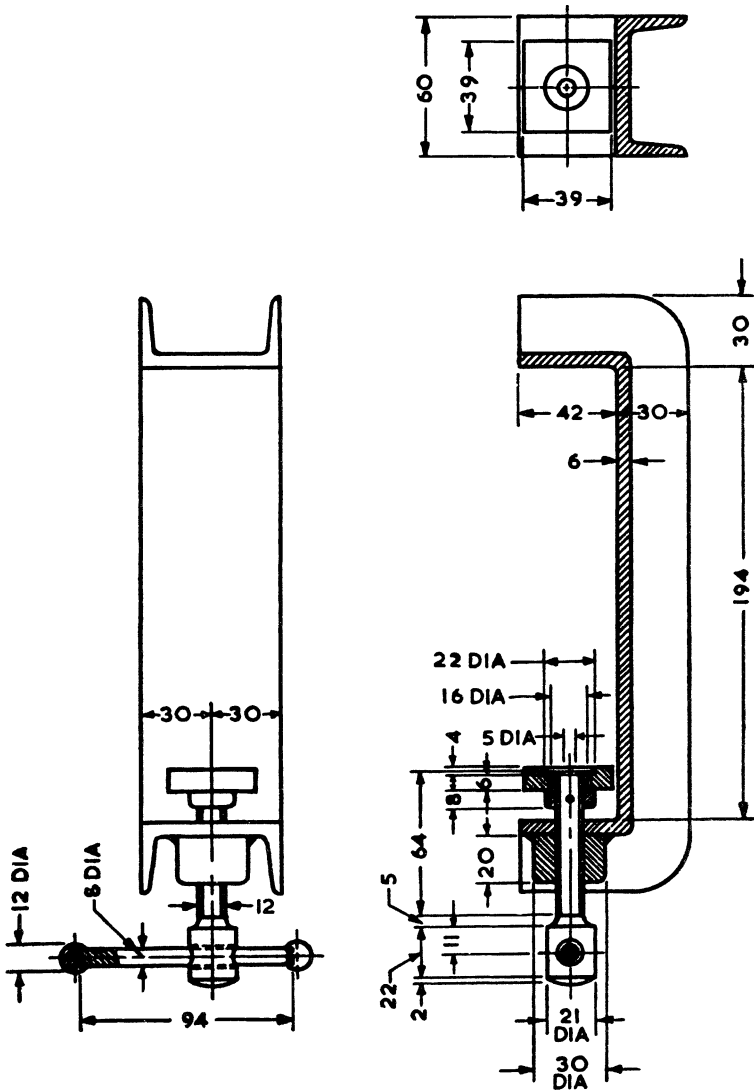
The weight of water shall be equal to half the weight of pozzolana plus cement in the above mixture.

11.5.3 The materials for each batch of moulds shall be mixed separately using the quantities of materials, conforming to the proportions given in **11.5.2**. Mixing of mortar shall be done mechanically as described in **9.5.3.1**.

11.6 Moulding Test Specimens — After fixing the mould and its hopper on the jolting table, introduce directly from the mixer the first layer of mortar of about 320 g into each of the moulding compartment (for example, by using a spoon of known capacity). Spread this layer by means of a steel plate levelling tool which is to be drawn twice forward and backward along the mould while pressing its flanges against the top of the hopper. Give 60 jolts to the first mortar layer in 60 seconds. Then introduce a second identical layer of mortar, level and compact as before. Lift the mould from the jolting table and remove its hopper. Strike off the excessive mortar with a metal straight edge held nearly vertical and moved slowly along the length of the mould with a transverse sawing motion. Then smooth lightly the surface, using the straight edge held almost flat.

11.7 Storage and Curing of Specimens — After completion of the jolting, cover the specimens in the mould by steel or rubber sheet and keep them at a temperature of $27 \pm 2^\circ\text{C}$ in an atmosphere of at least 90 percent relative humidity for 24 hours. At the end of that period, remove them from the mould with due precautions, preferably with the apparatus shown in Fig. 5. Immediately keep them submerged, preferably standing on end, in saturated still water until the time for testing. The specimens shall be kept apart from each other allowing free access of water to all their faces.

11.7.1 The volume of curing water shall be at least four times that of the test specimens. Half the curing water shall be replaced every fifteenth



All dimensions in millimetres.

FIG. 5 APPARATUS FOR DEMOULDING SPECIMENS

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day. The temperature of curing water shall be maintained at $27 \pm 2^\circ\text{C}$. The specimens shall be taken from the water only less than 15 minutes before test. In order to satisfy this condition, they shall be transported to the test machine in a container full of water. They shall then be wiped with a clean cloth so that any deposit that might have accumulated on them is removed. The specimens shall not be allowed to become dry until they are tested.

11.8 Procedure of Test — Test not less than three specimens for transverse strength in the apparatus described in 11.2.4 for each of the periods of 7,28 and 90 days, the period being reckoned from the completion of jolting. The load shall be progressively increased during application at the rate of 5 ± 1 kg/sec. The transverse strength shall be the average of the strengths of the three specimens for each period respectively.

11.9 Calculation — The transverse strength of the specimen T , shall be calculated by the formula:

$$T = 0.234 P$$

where

P = applied load in kg at the time of breaking.

The value of T shall be expressed nearest to 1 kg/cm^2 .

11.10 Faulty Specimens and Retests — Specimens that are manifestly faulty or that give strengths differing by more than 15 percent from the average value of all test specimens, made from the same sample and tested at the same period shall not be considered in determining the transverse strength. After so discarding, if less than three values are left for determining the transverse strength at any given period, a retest shall be made.

12. DETERMINATION OF DRYING SHRINKAGE IN POZZOLANA-CEMENT MORTAR

12.1 Scope — This method of test covers the procedure for determining the drying shrinkage of pozzolana-cement mortar as obtained on rectangular specimens prepared and tested under specified conditions.

12.2 Apparatus

12.2.1 Scale — The scales shall conform to the following requirements:

On scales in use the permissible variation at a load of 1 000 g shall be ± 1.0 g. The permissible variation on new scales shall be one-half of this value. The sensibility reciprocal shall be not greater than twice the permissible variation.

12.2.2 Weights — The permissible variations on weights in use in weighing the cement shall be as prescribed in Table 3.

TABLE 3 PERMISSIBLE VARIATIONS ON WEIGHTS
(Clause 12.2.2)

WEIGHT	PERMISSIBLE VARIATION ON WEIGHTS IN USE, PLUS OR MINUS	WEIGHT	PERMISSIBLE VARIATION ON WEIGHTS IN USE, PLUS OR MINUS
g	g	g	g
500	0.35	20	0.05
300	0.30	10	0.04
250	0.25	5	0.03
200	0.20	2	0.02
100	0.15	1	0.01
50	0.10		

12.2.3 Trowel — This shall have a steel blade 100 to 150 mm in length with straight edges.

12.2.4 Length Comparator — Changes a length of the test specimen shall be measured by a dial gauge or micrometer comparator having a range of at least 7.6 mm. The instrument shall be graduated to at least 0.025 mm and, when tested at any point throughout its range, the error shall be not greater than ± 0.050 mm. The difference between repeated measurements shall be not greater than 0.025 mm. The comparator shall be equipped with a steel reference bar having an insulating grip and shall be frequently checked with this reference bar.

12.2.5 Flow Table — Components, accessories (including mould), mounting and lubrication of flow table shall conform to IS : 5512-1969*.

12.3 Size and Number of Test Specimens

12.3.1 The test specimens shall be in the form of rectangular bars 25×25 mm in section and 250 mm long.

12.3.2 Unless otherwise specified in the relevant specification for the pozzolana, three or more specimens shall be made for each period of test specified.

12.4 Preparation of Moulds — The moulds shall be thinly covered with mineral oil; after this operation the stainless steel or non-corroding metal reference points shall be set, care being taken to keep them clean and free of oil.

12.5 Preparation of Mortar

12.5.1 Clean appliances shall be used for mixing, and the temperature of the water and that of the test room at the time when the mixing operations is being performed shall be $27 \pm 2^\circ\text{C}$.

*Flow table for use in tests of hydraulic cement and pozzolanic materials.

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12.5.2 The dry materials for the standard test mortar shall be pozzolana : cement : standard sand in proportion $0.2N : 0.8 : 3$ by weight, blended intimately:

where

$$N = \frac{\text{Specific gravity of pozzolana}}{\text{Specific gravity of cement}}$$

The amount of water for gauging shall be equal to that required to give a flow between 100 and 115 percent with 25 drops in 15 seconds, as determined in **9.5.3**.

12.5.2.1 The following quantities of materials are suggested for preparation of the mortar:

$60 \times N$ g	Pozzolana
240 g	Cement
900 g	Standard sand

12.5.3 The materials for each batch of moulds shall be mixed separately using the quantities of dry materials, conforming to the proportions given in **12.5.2** and the quantity of water as determined in accordance with the procedure given in **9.5.3** to give a flow of 100 to 115 percent with 25 drops in 15 seconds. Mixing shall be done mechanically as described in **9.5.3.1**.

12.6 Moulding Specimens — Immediately following the completion of mixing, the test specimen shall be moulded in two layers, each layer being compacted with the thumbs and forefingers by pressing the mortar into the corners, around the reference inserts and along the surfaces of the moulds until a homogeneous specimen is obtained. After the top layer has been compacted, the mortar shall be levelled off flush with the top of the mould and the surface smoothed with a few strokes of the trowel. During the operations of mixing and moulding, the hands shall be protected by rubber gloves.

12.7 Procedure of Test

12.7.1 After filling the moulds, place them immediately in a moist room or moist closet for 24 ± 2 hours. Then remove the specimens from the moulds and immediately immerse in water at $27 \pm 2^\circ\text{C}$ and allow them to remain there for six days.

12.7.2 Remove the specimens from the water and measure for length using a length comparator. Protect specimens against loss of moisture prior to reading for initial length. The temperature of the test specimens at the time of initial measurement shall be $27 \pm 2^\circ\text{C}$. Store the specimens in a closet or room maintained at $27 \pm 2^\circ\text{C}$ and 50 ± 5 percent relative humidity. Measure the length of the specimens again

28 days after the initial measurement. Place the specimens in the comparator with the same end uppermost with respect to the position of the specimens as when the initial measurement was made. When making the measurements, the specimens, comparator, and the reference bar shall be at a temperature of $27 \pm 2^\circ\text{C}$.

12.8 Calculation — After the specimens are measured as in **12.7.2** at the age of 7 and 35 days, calculate the average difference in length of the three specimens to the nearest 0.01 percent of the effective gauge length, and report this difference as the drying shrinkage.

13. DETERMINATION OF THE PERMEABILITY OF THE POZZOLANA-CEMENT MORTAR

13.1 Scope — This method of test covers the procedure for determining the permeability to water of the pozzolana mortar prepared and cured under specified conditions, by measuring the percolation of water through standard cylindrical specimens.

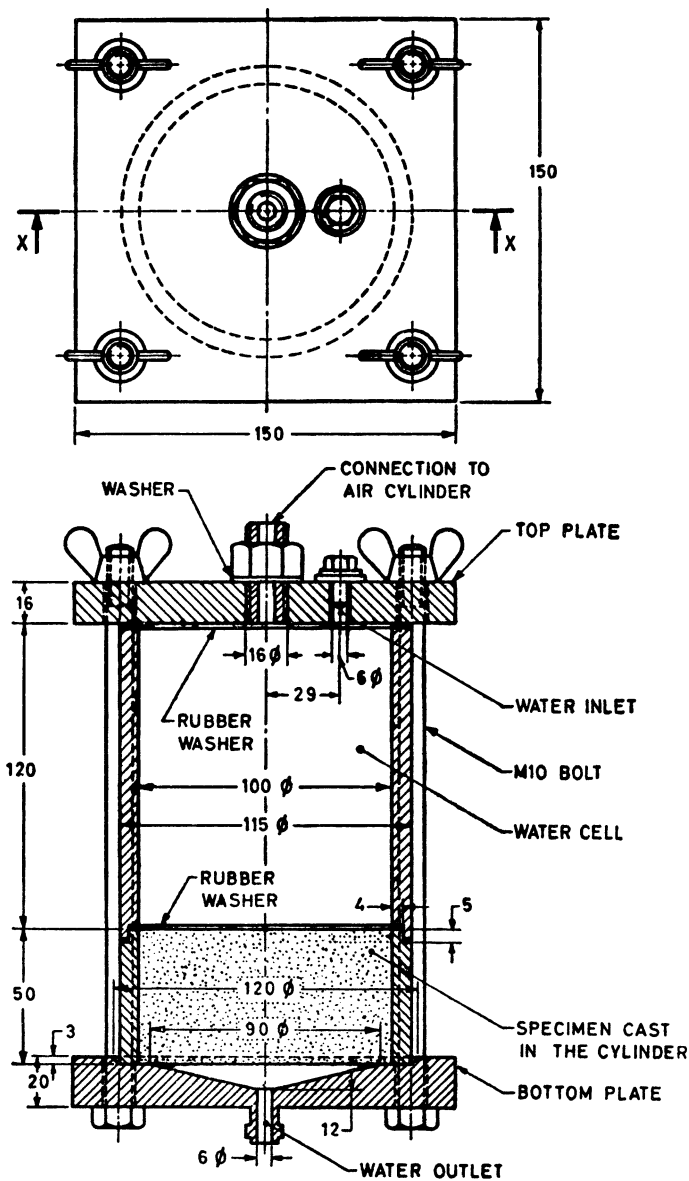
13.2 Apparatus

13.2.1 Permeability Unit — The permeability unit shall consist of a specimen container ring of 100 mm diameter and 50 mm high, held between a bottom plate and a water cell. The hydraulic head for testing shall be obtained by connecting the unit to a compressor through a water pressure vessel. A pressure regulator and pressure gauge shall be included between the compressor and the water pressure vessel to indicate the test pressure. Water percolating through the specimen shall be collected in a container. Typical details of an individual unit are given in Fig. 6 and 7. The water cell shall be 100 mm diameter brass cylinder and the top and bottom plates shall be either of brass or any other non-corroding metal. The connecting pipeline from the water cell to the water pressure vessel shall also be of non-corroding metal or of hard board polythene. This requirement is necessary since the tests last over a number of days in which mild steel or similar materials will get corroded and the rust formed will coat the top of the specimens and affect the permeability. The connection of units to the air cylinder shall be done with armoured heavy-duty rubber hose.

13.2.2 Rammer — A rammer of the type shown in Fig. 8 shall be used to compact the mortar. The rammer shall consist of a plunger weighing 500 ± 5 g which shall fall freely through a height of 150 ± 1 mm in a tubular guide. The base of the plunger shall have a diameter of 50 ± 1 mm.

13.3 Materials

13.3.1 The dry materials for the standard test mortar shall be pozzolona:



SECTION XX

All dimensions in millimetres.

FIG. 6 DETAILS OF PERMEABILITY UNIT

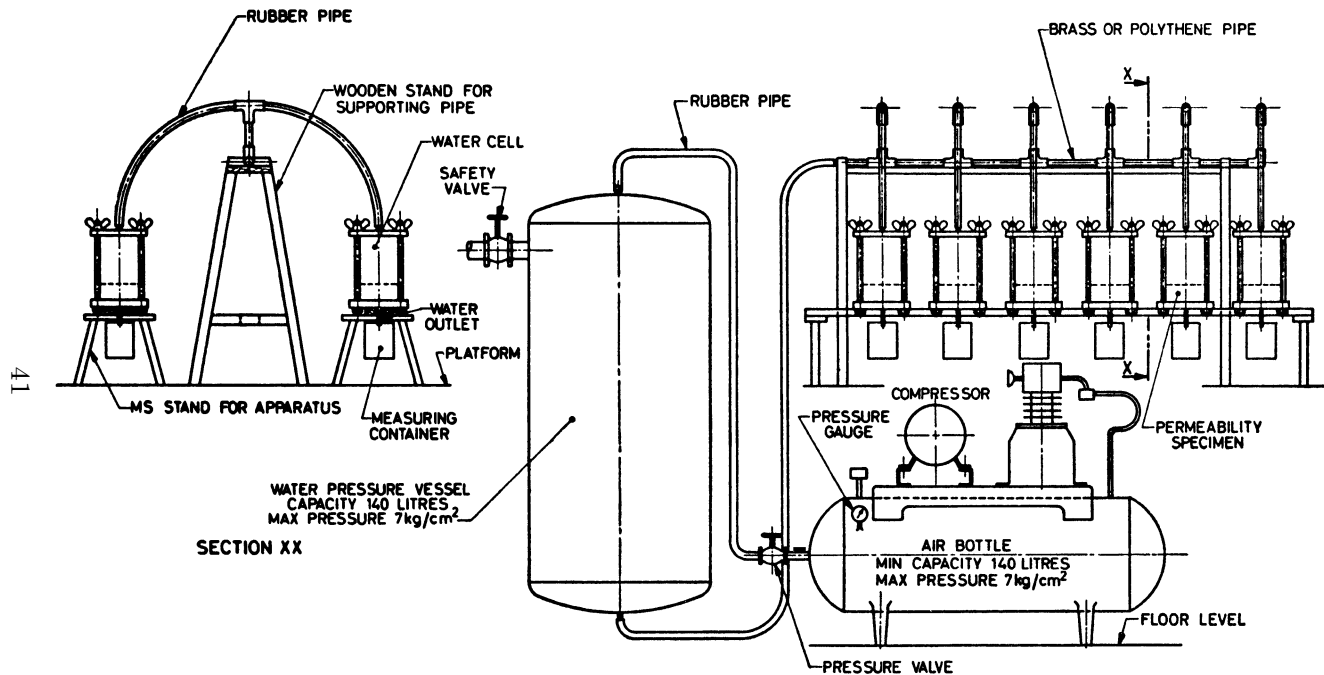
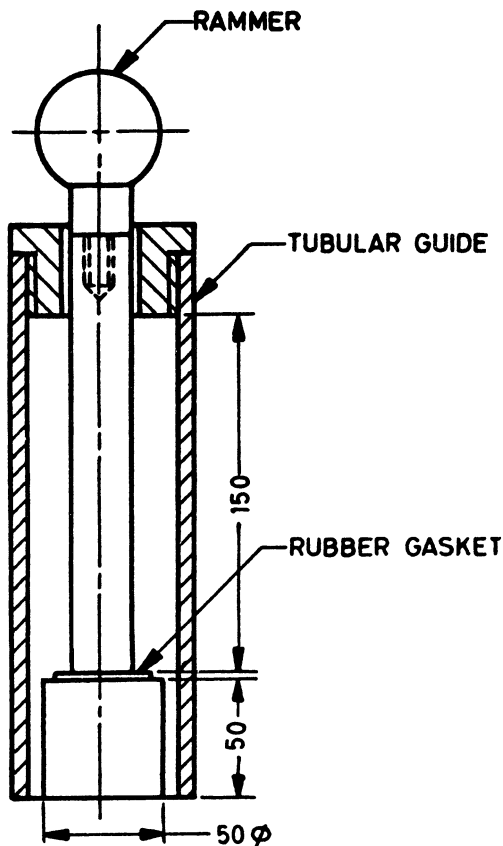


FIG. 7 GENERAL ARRANGEMENT OF PERMEABILITY TEST UNIT



All dimensions in millimetres.

FIG. 8 SPECIMEN COMPACTING APPARATUS

cement : sand in proportion $0.2N : 0.8 : 5$ by weight, blended intimately,
where

$$N = \frac{\text{Specific gravity of pozzolana}}{\text{Specific gravity of cement}}$$

13.3.2 The amount of water for gauging shall be equal to that required to give a flow of 105 ± 5 percent with 25 drops in 15 seconds as determined in 9.5.3.

13.3.3 Sand — The sand to be used in the preparation of mortar for this test shall be natural, rounded siliceous sand with a maximum possible amount of quartz. The sand shall preferably be obtained from

Ennore. The particle size distribution, determined by sieving, shall be between the limits given below:

<i>Sieve</i>	<i>Cumulative Retained (Percent)</i>
2.00-mm IS Sieve	0
1.70-mm IS Sieve	5 ± 5
1.00-mm IS Sieve	33 ± 5
500 micron IS Sieve	67 ± 5
150 micron IS Sieve	88 ± 5
75 micron IS Sieve	98 ± 2

13.4 Preparation of Mortar

13.4.1 Clean appliances shall be used for mixing, and the temperature of the water and that of the test room at the time when the mixing operation is being performed shall be $27 \pm 2^\circ\text{C}$.

13.4.2 The materials for each batch of moulds shall be mixed separately using the quantities of dry materials, conforming to the proportions given in **13.3.1** and the quantity of water as determined in accordance with the procedure given in **9.5.3** to give a flow of 105 ± 5 percent with 25 drops in 15 seconds. Mixing shall be done mechanically as described in **9.5.3.1**.

13.5 Preparation of Specimens — The container ring shall be given a thin coating of neat cement slurry and the mortar shall be placed in it in two layers. Each layer shall be compacted with 10 blows of the rammer described in **13.2.2**, falling freely through a height of 150 ± 1 mm in its tubular guide. During compaction the guide of the rammer shall remain in contact with the wall of the container ring. It shall be moved through a uniform distance after each blow so as to cover the entire periphery of the container ring after 10 blows. After compaction the top of the specimen shall be levelled off with a 12 mm diameter rod slowly and firmly moved over the surface. No trowelling or other type of surface finish shall be allowed.

13.6 Curing and Storage of Specimens — Keep the container ring with the specimens at a temperature of $27 \pm 2^\circ\text{C}$ in an atmosphere of at least 90 percent relative humidity for 24 hours. At the end of that period, submerge the specimens in clean fresh water and keep there for 27 days, and take it out just prior to testing. The water in which the specimens are submerged shall be renewed every seventh day and shall be maintained at a temperature of $27 \pm 2^\circ\text{C}$.

13.7 Procedure of Test — The specimens shall then be fitted into the testing apparatus as shown in Fig. 6. Water shall be poured into the water cell till it is nearly filled. The water inlet shall then be closed with the lid screwed tight.

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An initial pressure of about 0.5 kg/cm^2 shall be applied to the water and from time to time the collecting container shall be taken out and weighed to determine the rate of percolation. The rate of percolation will be comparatively high in the initial stages and will then become stabilised. At this stage the pressure shall be increased again by 0.5 kg/cm^2 and this procedure repeated until a final pressure of 2 kg/cm^2 is reached. When a stable flow has been reached at this pressure readings of the percolation shall be taken at fixed intervals of time for 8 hours.

The test shall be carried out at a temperature of $27 \pm 2^\circ\text{C}$. For each test three specimens shall be tested at a time.

13.8 Calculation — The coefficient of permeability shall be calculated from the formula:

$$K = \frac{Qs}{waTh}$$

where

K = coefficient of permeability in cm/sec/unit gradient,

Q = quantity of water collected in g,

s = thickness of the specimens in cm,

w = density of water in g/cm^3 normally equal to one,

a = area of cross-section of the specimen in cm^2 ,

T = time of collection in seconds, and

h = net pressure head on the specimen in cm of water.

The average for three specimens shall be reported as the permeability of the mortar.

13.9 Faulty Specimens and Retests — Specimens that are manifestly faulty or that give permeability values differing by more than 15 percent from the average shall not be considered. In such cases, a retest shall be made.

14. REDUCTION IN ALKALINITY AND SILICA RELEASE BY POZZOLANA

14.1 Scope — These tests will help to find out the effectiveness of a few pozzolanas in reducing the harmful effects of alkali-aggregate reaction in concrete. However, the tests are not applicable to all types of pozzolana.

14.2 Apparatus

14.2.1 Reaction Containers — These shall be of 50 to 75 ml capacity, made of stainless steel or other corrosion-resistant material and fitted with air-tight covers.

14.3 Reagents

- a) Unless otherwise indicated, all reagents used shall be analytical reagents. Water used shall be distilled water, unless otherwise stated.
- b) *Standard sodium hydroxide* — Prepare a 1.000 ± 0.010 N sodium hydroxide solution and standardize to ± 0.001 N. Use water that has been boiled until free of carbon dioxide for preparing the solution.
- c) *Hydrochloric acid* — sp gr 1.16.
- d) *Standard hydrochloric acid* — Prepare approximately 0.05 N hydrochloric acid and standardize to ± 0.0001 N. Standardize the hydrochloric acid, for each day's tests, against an approximately 0.05 N standardized sodium hydroxide solution.
- e) *Phenolphthalein indicatory solution* — Dissolve one gram of phenolphthalein in 100 ml of ethanol (1 : 1).

14.4 Initial Preparation

14.4.1 Weigh out three representative portions of the pozzolana, each of 12.50 ± 0.05 g. Place one portion in each of the three reaction containers and add 25 ml of the 0.001 N sodium hydroxide solution. To a fourth reaction container, add 25 ml of the standard sodium hydroxide solution to serve as a blank. Seal the four containers and gently swirl them to liberate trapped air.

14.4.2 Immediately after the containers have been sealed, place them in a liquid bath maintained at $80.0 \pm 1.0^\circ\text{C}$. After $24 \pm \frac{1}{4}$ h, remove the containers from the bath and cool them under running water for 15 ± 2 minutes to below 30°C .

14.4.3 Immediately after the containers have been cooled, open them and filter the solution from the total residue. Use a porcelain Gooch crucible fitted with a disk of rapid low-ash filter paper, setting the crucible in a rubber holder in a funnel. Place a dry test tube of 35 to 50 ml capacity in the filter flask to collect the filtrate and seal the funnel in the neck of the filter flask. With the aspirator in operation or the vacuum line open, decant a small quantity of the solution into the filter paper so that it will seat properly in the crucible. Without stirring the contents of the container, decant the remaining free liquid into the crucible. When the decantation of the liquid has been completed, discontinue the vacuum and transfer the solids remaining in the container to the crucible and pack in place with the aid of a stainless steel spatula. Then apply and adjust the vacuum to approximately 380 mm of mercury. Continue the filtration until further filtration yields filtrate at the approximate rate of one drop

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every 10 seconds. Reserve the filtrate for further tests. Record the total time for which the vacuum is applied as the filtration time. Every effort shall be made to achieve an equal filtration time for all samples in a set, by uniformity of procedure in the assembly of the filtration apparatus and the packing of the solids in the crucible.

14.4.4 Filter the blank according to the procedure described in **14.4.3**. Apply the vacuum for the same length of time as the average filtration time for the three specimens.

14.4.5 Immediately following the completion of filtration, stir the filtrate to assure homogeneity. Then take an aliquot of 10 ml of the filtrate and dilute with water to 200 ml in a volumetric flask. Reserve this diluted solution for the determination of dissolved silica and reduction in alkalinity.

NOTE — In some cases, it may become necessary to add more than 25 ml of sodium hydroxide solution either due to the solution becoming viscous or to the formation of a gel. In such cases, the quantity of sodium hydroxide may be increased.

14.5 Silica Release Test

14.5.1 Transfer 100 ml of the dilute solution to an evaporating dish, preferably of platinum. For the sake of celerity in evaporation, add 5 to 10 ml of hydrochloric acid and evaporate to dryness on a steam-bath. Without heating the residue any further, treat it with 5 to 10 ml of hydrochloric acid, and then an equal amount of water, or at once pour 10 to 20 ml of hydrochloric acid (50 percent) upon the residue. Cover the dish and digest for 10 minutes on the bath or a hot-plate. Dilute the solution with an equal volume of hot water (*see Note*) and reserve the residue.

NOTE — The washing of silica precipitates can be made more effectively by using hot hydrochloric acid (one percent) and then completing the washing with hot water.

14.5.2 Again evaporate the filtrate to dryness, baking the residue in an oven for one hour at 105°C to 110°C. Take up the residue with 10 to 15 ml of hydrochloric acid (50 percent) and heat on the bath or hot-plate.

14.5.3 Dilute the solution with an equal volume of hot water and obtain and wash the small amount of silica it contains on another filter paper.

14.5.4 Transfer the papers containing the residue on to a platinum crucible (*see Note*). Dry and ignite the papers, first at a low heat until the carbon of the filter paper is completely consumed without inflaming, and finally at 1 100°C to 1 200°C until a constant weight is reached.

NOTE — The empty crucible may be weighed if one wishes to know for his own information, the magnitude of impurities in the residue of silica.

14.5.5 Treat the silica thus obtained, which will contain small amounts of impurities, in the crucible with a few drops of water, about 10 ml of hydrofluoric acid, and one drop of sulphuric acid, and evaporate cautiously to dryness. Finally heat the residue at 1 050°C to 1 100°C for

1 to 2 minutes cool and weigh. The difference between this weight and the weight previously obtained represents the amount of silica.

14.5.6 Make a blank determination, following the same procedure as described in **14.5.1** to **14.5.4** using the same amount of dilute solution from the blank and the same amounts of reagents.

14.5.7 Calculate the silica concentration in the solution originally filtered (*see* **14.4.3**) as follows:

$$Sc = (W_1 - W_2) \times 3\,330 \text{ (see Note)}$$

where

Sc = concentration of silica in millimoles/litre in the original filtrate,

W_1 = grams of silica found in 100 ml of the dilute solution, and

W_2 = grams of silica found in the blank.

NOTE — The dilute solution analysed is equivalent to 5 ml of the original or $\frac{(W_1 - W_2) \times 1\,000}{60.06}$ millimoles of silica consequently one litre of the original filtrate

would contain $\frac{1\,000}{5} \times \frac{1\,000}{60.06} \times (W_1 - W_2)$ or $3\,330 (W_1 - W_2)$ millimoles of silica.

14.6 Reduction in Alkalinity

14.6.1 Transfer a 20 ml aliquot of the dilute solution obtained as in **14.4** to a 125 ml Erlenmeyer flask. Add 2 or 3 drops of phenolphthalein solution, and titrate with 0.05 N hydrochloric acid to the phenolphthalein end point.

14.6.2 Calculate the reduction in alkalinity as follows:

$$R_c = \frac{20\,N}{V_1} (V_3 - V_2) \times 1\,000$$

where

R_c = the reduction in alkalinity, millimoles/litre;

N = normality of the hydrochloric acid used for titration;

V_1 = volume of dilute solution used in ml;

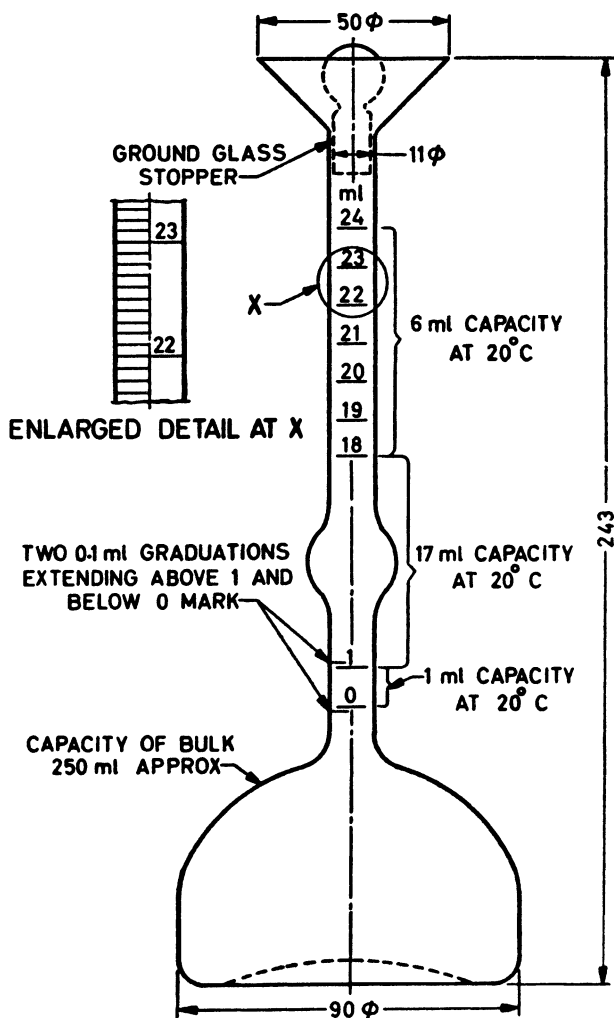
V_3 = volume of hydrochloric acid used in ml to attain the phenolphthalein end point in the blank; and

V_2 = volume of hydrochloric acid used in ml to attain the phenolphthalein end point in the test sample.

15. DETERMINATION OF SPECIFIC GRAVITY

15.1 Apparatus

15.1.1 The standard 'Le Chatelier' flask conforming to the dimensions shown in Fig. 9 shall be used.



NOTE — Variations of a few millimetres in such dimensions as total height of flask, diameter of base, etc, are to be expected and will not be considered sufficient cause for rejection.

All dimensions in millimetres.

FIG. 9 'LE CHATELIER' FLASK FOR SPECIFIC GRAVITY TEST

15.1.2 Water Bath — A suitable water bath to accommodate the ‘Le Chatelier’ flask shall be used.

15.2 Material — Kerosine free of water or naphtha having a specific gravity not less than 0.7313 shall be used.

15.3 Procedure

15.3.1 Specific gravity of pozzolana shall be determined on the material as received, unless otherwise specified.

15.3.2 Fill the flask with kerosine or naphtha to a point on the stem between the zero and the 1 ml mark and replace the stopper. Then immerse the flask in a constant temperature water bath, maintained at about room temperature, for sufficient interval to avoid variations greater than $\pm 0.2^{\circ}\text{C}$ in the temperature of the liquid in the flask. Take the reading of the liquid in the flask.

15.3.3 Introduce a weighed quantity of pozzolana into the flask, taking care that no portion of it adhere to the inside of the flask above the liquid, by slightly vibrating the flask. Replace the stopper and roll the flask in an inclined position to expel any air bubble in the pozzolana. The level of the liquid will be in its final position at some point of the upper series of graduations. The reading shall be taken after the flasks immersed in the water bath.

NOTE 1 — A rubber pad on the table may be used when filling or rolling the flask.

NOTE 2 — The flask may be held in a vertical position in the water bath by means of a burette clamp.

15.4 Calculation — The difference between the first and final readings represents the volume of liquid displaced by the weight of cement used in the test. Specific gravity shall be calculated as follows:

$$\text{Specific gravity} = \frac{\text{Weight of pozzolana in g}}{\text{Displaced volume in ml}}$$

15.5 Reproducibility — Duplicate determination of specific gravity by this method shall agree within 0.01.

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