भारतीय मानक

जलीय सीमेंट के भौतिक परीक्षणों की पद्धतियाँ

भाग 2 ब्लेन वायुगम्यता पद्धति द्वारा मलीनता ज्ञात करना

(दूसरा पुनरीक्षण)

Indian Standard

METHODS OF PHYSICAL TESTS FOR HYDRAULIC CEMENT

PART 2 DETERMINATION OF FINENESS BY BLAINE AIR PERMEABILITY METHOD

(Second Revision)

ICS 91.100.10

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

January 1999 **Price Group 4**

Cement and Concrete Sectional Committee, CED 2

FOREWORD

This Indian Standard (Part 2) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

Standard methods of testing cement are essential adjunct to the cement specifications. This standard in different parts lays down the procedure for the tests to evaluate the physical properties of different types of hydraulic cement. The procedure for conducting chemical test is covered in IS 4032 : 1985 'Methods of chemical analysis of hydraulic cement (*first revision*)'. Originally all the tests to evaluate the physical properties of hydraulic cement were covered in one standard but for facilitating the use of the standard and future revisions, the revised standard was brought out into different parts, each part covering different tests. This part covers determination of fineness by specific surface by Blaine air permeability apparatus.

The second revision of this standard has been prepared with a view to aligning this test method with European Standard EN 196 (Part 6) 'Method of testing cement: Determination of fineness'.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF PHYSICAL TESTS FOR HYDRAULIC CEMENT

PART 2 DETERMINATION OF FINENESS BY BLAINE AIR PERMEABILITY METHOD

(Second Revision)

1 SCOPE

This standard (Part 2) covers the procedure for determining by Blaine air permeability apparatus, the fineness of cement as represented by specific surface expressed as total surface area in cm^2/g.

NOTE — This method is also being used for determination of fineness of various other materials. However, it should be understood that the values obtained are relative rather than absolute.

2 REFERENCES

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

IS No.	*Title*
3535 : 1980	Methods of sampling hydraulic cement (*first revision*)
5516 : 1996	Specification for variable flow type air permeability apparatus (Blaine type) (*first revision*)

3 SAMPLING AND SELECTION OF TEST SPECIMEN

The samples of the cement shall be taken in accordance with the requirements of IS 3535 and the relevant standard specification for the type of cement being tested. The representative sample of the cement selected as above shall be thoroughly mixed before testing.

4 APPARATUS AND MATERIALS

4.1 Variable Flow Type Air Permeability Apparatus (Blaine Type)

Variable flow type air permeability apparatus (Blaine Type) and the accessories conforming to IS 5516 shall be used.

4.2 Timer

The timer shall have a positive starting and stopping mechanism and shall be capable of being read to the nearest 0.2 s or better. The timer shall be accurate to 1 percent or better over time intervals up to 300 s.

4.3 Balances

Balances capable of weighing about 3 g to the nearest 1 mg for the cement and about 50 g to 110 g to the nearest 10 mg for the mercury.

4.4 Standard Weights

4.5 Pyknometer

Pyknometer or other convenient means of determining the density of cement.

4.6 Manometer Liquid

The manometer shall be filled to the level of the lowest etched line with a non-volatile, non-hygroscopic liquid of low viscosity and density, such as dibutyl phthalate or light mineral oil.

4.7 Mercury

Mercury of reagent grade or better.

4.8 Reference cement of known specific surface.

4.9 Light oil, to prevent formation of mercury amalgam on the inner surface of the cell.

4.10 Circular discs of filter paper, having a smooth circumference adapted to the dimensions of the cell. The filter paper is of medium porosity (mean pore diameter 7 µm).

4.11 Light grease, for ensuring an airtight joint between cell and manometer, and in the stopcock.

5 TEST PROCEDURE

5.1 Test Conditions

The laboratory in which the air permeability test is carried out shall be maintained at a temperature of

27 ± 2°C and a relative humidity not exceeding 65 percent. All materials for test and calibration shall be at the laboratory temperature when used and shall be protected from absorption of atmospheric moisture during storage. A laboratory temperature of 20 ± 2°C may be maintained, if desired by the purchaser.

5.2 Compacted Cement Bed

5.2.1 *Basis*

The compacted cement bed comprises a reproducible arrangement of cement particles with a specified volume of air included between the particles. This air volume is defined as a fraction of the total volume of the bed and is termed the porosity, *e*.

It follows that the volume fraction occupied by the cement particles is $(1-e)$. If V is the total volume of the bed, the absolute volume of cement is $V(1-e)$ (cm^3), and the mass of cement m is $\rho V(1-e)$ (g) where ρ is the solid density of the cement particles ρ (g/cm^3).

Thus, knowing ρ, a mass of cement can be weighed to produce a desired prosity, *e*, in the compacted bed of total volume V. The determination of ρ is described in **5.2.3** and that of V in **5.4.1**.

5.2.2 *Preparation of the Sample*

Agitate the sample of cement to be tested by shaking for 2 min in a stoppered jar to disperse agglomerates. Wait for 2 min. Stir the resulting powder gently using a clean dry rod in order to distribute the fines throughout the cement.

5.2.3 *Determination of Density*

Determine the density of the cement using a device such as a pyknometer or Le-chatelier flask. Use a non-reactive liquid in the determination. The quantity of cement used will depend on the nature of the apparatus but shall be such that the value of ρ determined is accurate to 0.01 g/cm^3. Verify this accuracy by a repeat determination and record the mean of the two determinations to the nearest 0.01 g/cm^3 as the density.

5.2.4 *Formation of the Bed*

To give a cement bed of porosity $e = 0.500$ weigh a quantity of cement, m_1, calculated from:

$$m_1 = 0.500\,\rho V \text{ (g)} \qquad \ldots(1)$$

where

ρ is the density of the cement (g/cm^3), and
V is the volume of the cement bed (cm^3).

This mass, correctly compacted, will produce a bed of porosity 0.500. Place the perforated disc on the ledge at the bottom of the cell and place on it a new filter paper disc. Ensure that the filter paper disc fully covers the perforated disc and is flat by pressing with a clean dry rod. Place the weighed quantity of cement, m_1, in the cell taking care to avoid loss.

Tap the cell to level the cement. Place a second new filter paper disc on the levelled cement. Insert the plunger to make contact with the filter paper disc. Press the plunger gently but firmly until the lower face of the cap is in contact with the cell. Slowly withdraw the plunger about 5 mm, rotate it through 90° and gently but firmly press the bed once again until the plunger cap is in contact with the cell. The bed is now compacted and ready for the permeability test. Slowly withdraw the plunger.

NOTE — Too rapid and vigorous pressing may change the particle size distribution and therefore change the specific surface of the bed. The maximum pressure should be that comfortably exerted by a thumb on the plunger.

5.3 Air Permeability Test

5.3.1 *Basis*

The specific surface, S, is given in **5.6.1** but is conveniently expressed as:

$$S = \frac{K}{\rho} \times \frac{\sqrt{e^3}}{(1-e)} \times \frac{\sqrt{t}}{\sqrt{0.1\eta}} \ (cm^2/g) \qquad \ldots(2)$$

where

K is the apparatus constant,
e is the porosity of the bed,
t is the measured time(s),
ρ is the density of cement (g/cm^3), and
η is the viscosity of air at the test temperature taken from Table 1 (P.s).

With the specified porosity of $e = 0.500$ and temperature:

a) at 27 ± 2°C

$$S = \frac{521.08\,K\sqrt{t}}{\rho} \ (cm^2/g)$$

b) at 20 ± 2°C

$$S = \frac{524.2\,K\sqrt{t}}{\rho} \ (cm^2/g)$$

Table 1 Density of Mercury *D*, Viscosity of Air (*n*) and $\sqrt{0.1\,v}$ as Function of Temperature

(*Clauses* 5.3.1, 5.4.1, 5.4.2 *and* 5.6.1)

Temperature °C	Mass Density of Mercury g/cm^3	Viscosity of Air pascal second Pa.s	$\sqrt{0.1\eta}$
(1)	(2)	(3)	(4)
16	13.56	0.000 017 88	0.001 337
18	13.55	0.000 017 98	0.001 341
20	13.55	0.000 018 08	0.001 345
22	13.54	0.000 018 18	0.001 348
24	13.54	0.000 018 28	0.001 352
26	13.53	0.000 018 37	0.001 355
28	13.53	0.000 018 47	0.001 359
30	13.52	0.000 018 57	0.001 363
32	13.52	0.000 018 67	0.001 366
34	13.51	0.000 018 76	0.001 370

NOTE — Intermediate value shall be obtained by linear interpolation.

5.3.2 *Procedure*

Insert the conical surface of the cell into the socket at the top of the manometer, using if necessary a little light grease to ensure an airtight joint. Take care not to disturb the cement bed.

Close the top of the cylinder with a suitable plug. Open the stopcock and with gentle aspiration raise the level of the manometer liquid to that of the highest etched line, close the stopcock and observe that the level of the manometer liquid remains constant. If it falls, remake the cell manometer joint and check the stopcock, repeat the leakage test until the improved sealing produces a steady level of the liquid. Open the stopcock and by gentle aspiration adjust the level of the liquid, to that of the highest etched line. Close the stopcock. Remove the plug from the top of the cylinder. The manometer liquid will begin to flow. Start the timer as the liquid reaches the second etched line and stop it when the liquid reaches the third etched line. Record the time t, to the nearest 0.2 s and the temperature to the nearest 1°C.

Repeat the procedure on the same bed and record the additional values of time and temperature. Prepare a fresh bed of the same cement with a second sample following the procedure of **5.2.4** or, where there is little cement available, by breaking up the first bed and reforming it as in **5.2.4**. Carry out the permeability test twice on the second bed, recording the values of time and temperature as before.

5.4 Calibration of Apparatus

5.4.1 *Determination of the Bed Volume*

Owing to the need for clearance between the cell and the plunger, the volume of the compacted cement bed varies for each cell-plunger combination. The volume of the compacted cement bed shall be established for a given cell-plunger clearance, this volume is to be determined as follows.

Apply a very thin film of light mineral oil to the cell interior. Place the perforated disc on the ledge in the cell. Place two new filter paper discs on the perforated disc and ensure that each covered the base of the cell whilst lying flat by pressing with a rod.

Fill the cell with mercury. Remove any air bubbles with a clear dry rod. Ensure that the cell is full by pressing a glass plate on the mercury surface until it is flush with the cell top. Empty the cell, weigh the mercury to the nearest 0.01 g, m_2, and record the temperature. Remove one filter paper disc. Form a compacted cement bed by the method described in and place on it a new filter paper disc. Refill the cell with mercury, removing air bubbles and levelling the top as before. Remove the mercury, weigh it to the nearest 0.01 g, m_3, and check the temperature.

The bed volume V is given by:

$$V = \frac{m_2 - m_3}{D} \ (\text{cm}^3)$$

where D is the density of mercury at the test temperature taken from Table 1.

Repeat the procedure with fresh cement beds until two values of V are obtained differing by less than 0.005 cm^3. Record the mean of these two values as V.

NOTE — Care should be taken to avoid spilling or splashing the mercury and any contact between it and the operator's skin and eyes.

5.4.2 *Determination of the Apparatus Constant*

From a supply of reference cement of known specific surface prepare a compacted cement bed and measure its permeability by the procedures given in **5.2.2** to **5.2.4** and **5.3.2**. Record the time t, and the temperature of test using the same bed 1; repeat twice the procedure of **5.3.2** and record the two further values of time and of temperature. Repeat the whole procedure on two further samples of the same reference cement. For each of the three samples calculated the means of the three times and temperatures. For each sample calculate:

$$K = \frac{S_0 \rho_0 (1 - e) \sqrt{0.1 \eta_0}}{\sqrt{e^3} \sqrt{t_0}} \qquad \ldots(3)$$

where

S_0 is the specific surface of the reference cement (cm^2/g),

ρ_0 is the density of the reference cement (g/cm^3),

t_0 is the mean of the three measured times (s), and

η_0 is the air viscosity at the mean of the three temperatures (Pa.s) (Table 1),

with the specified porosity of $e = 0.500$

$$K = 1.414 \ S_0 \rho_0 \frac{\sqrt{0.1\eta_0}}{\sqrt{t_0}}$$

Take the mean of the three values of K as the constant K for the apparatus.

5.4.3 *Recalibration*

Repeated use of apparatus may cause changes in the cement bed volume and in the apparatus constant (because of the wear of cell, plunger and perforated disc). These changes can be determined with the help of a so-called secondary reference cement whose specific surface has been measured.

The cement bed volume and the apparatus constant shall be recalibrated with the reference cement:

a) after 1 000 tests;
b) in the case of using:
   —another type of manometer fluid,

—another type of filter paper, and
—a new manometer tube; and

c) at systematic deviations of the secondary reference cement.

5.5 Special Cements

Certain cements having unusual particle size distributions and in particular, fine cements of higher strength grades may prove difficult to form into a compacted bed of porosity $e = 0.500$ by the method of **5.2.4**. Should thumb pressure on the plunger cap fail to bring it in contact with the top of the cell or if, after making contact and removing the pressure the plunger moves upwards, the porosity of $e = 0.500$ shall be considered unattainable.

For such cases the porosity required for a well-compacted bed shall be determined experimentally. The mass of cement, m_4 weighed to make the bed as in **5.2.4** then becomes

$$m_4 = (1 - e_1)\ \rho_1\ V\ (\text{g}) \qquad \ldots(4)$$

Where e_1 is the porosity determined experimentally.

5.6 Simplification of the Calculation

5.6.1 *Basic Formula*

The specific surface, S, of the cement under test is calculated from the formula:

$$S = \frac{\rho_0}{\rho} \times \frac{(1 - e_0)}{(1 - e)} \times \frac{\sqrt{e^3}}{\sqrt{e_0^3}} \times \frac{\sqrt{0.1\eta_0}}{\sqrt{0.1\eta_0}} \times \frac{\sqrt{t}}{\sqrt{t_0}}\, S_0 \quad (\text{cm}^2/\text{g}) \qquad \ldots(5)$$

where

S_0 is the specific surface of the reference cement (cm^2/g),

e is the porosity of the bed of cement under test,

e_0 is the porosity of the bed of reference cement,

t is the measured time for the cement under test(s),

t_0 is the mean of the three times measured on the reference cement(s),

ρ is the density of the cement under test (g/cm^3),

ρ_0 is the density of the reference cement (g/cm^3),

η is the air viscosity at the test temperature taken from Table 1 (Pa.s), and

η_0 is the air viscosity at the mean of the three temperatures (Table 1) for the reference cement (Pa.s).

5.6.2 *Effect of Specified Porosity*

Use of the specified porosity, $e = 0.500$ for both the reference and test cements simplifies formula, 5 to

$$S = \frac{\rho_0}{\rho}\ \frac{\sqrt{0.1\ \eta_0}}{0.1\ \eta} \times \frac{\sqrt{t}}{\sqrt{t_0}} \times S_0\ (\text{cm}^2/\text{g}) \qquad \ldots(6)$$

In the case of cements requiring a porosity other than $e = 0.500$ formula 6 cannot be used unless a reference cement has been tested at that porosity.

5.6.3 *Effect of Density of Cement*

The only remaining possibility of simplification is the elimination of the density (ρ) terms. This has previously been done where the only cements in question were pure portland cements for which a value of ρ of 3.15 was assumed to apply. That assumption is known to produce errors of up to 1 percent.

5.6.4 The formulae given in Table 2 may be used, calculate appropriate apparatus constant (K) as shown in col 3 of Table 2.

Table 2 Formulae for Apparatus Constant

Sl No.	Formula for Fineness S	Apparatus Constant K	Porosity: Calibrated Sample	Porosity: Test Sample	Temperatures Difference Between Time of Testing of Test Sample and Calibrated Sample (27°C)	Cement to be Tested
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	$K\sqrt{t}$	$\frac{S_0}{t_0}$	0.5	0.5	Within ±3°C	Ordinary Portland cement
ii)	$\frac{K\sqrt{t}}{\sqrt{0.1n}}$	$\frac{0.001\ 36 \times S_0}{t_0}$	0.5	0.5	Outside ±3°C	do
iii)	$\frac{K\sqrt{t}\ \sqrt{e^3}}{1-e}$	$\frac{1.414 \times S_0}{t_0}$	0.5	Other than 0.5	Within ±3°C	Cements other than OPC
iv)	$\frac{K\sqrt{t}\sqrt{e^3}}{\sqrt{0.1n}\ (1-e)}$	$\frac{0.001\ 92 \times S_0}{t_0}$	0.5	do	Outside ±3°C	do
v)	$\frac{K\sqrt{t}\sqrt{e^3}}{\rho\ (1-e)}$	$\frac{4.455 \times S_0}{t_0}$	0.5	do	Within ±3°C	do
vi)	$\frac{K\sqrt{T}\sqrt{e^3}}{\rho\ (1-e)\ \sqrt{0.1n}}$	$\frac{0.006\ 05 \times S_0}{t_0}$	0.5	do	Outside ±3°C	do

5.7 Expression of Results

Where the porosity is $e = 0.500$, the four times and temperatures resulting from the procedure of **5.3.2** shall be examined to check that the temperatures all fall within the specified range of 27 ± 2°C or 20 ± 2°C. The resulting value of S, to the nearest 10 cm^2/g, shall be reported as the specific surface of the cement.

A difference of 1 percent between the means of the fineness measurements carried out on two different powder beds from one and the same sample is acceptable.

The standard deviation of the repeatability is about 50 cm^2/g and of the reproducibility is about 100 cm^2/g.

Where the porosity e is not = 0.500, equation 5 shall be used and the result to the nearest 10 cm^2/g reported as the specific surface of the cement.

If, owing to a breakdown in control or for other reasons, the four temperatures do not lie within the specified range of 27 ± 2°C or 20 ± 2°C, a value of S shall be reported, to the nearest 10 cm^2/g, as specific surface of the cement.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

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Review of Indian Standards

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This Indian Standard has been developed from Doc: No. CED 2 (5171).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Dee Kay Printers, New Delhi, India