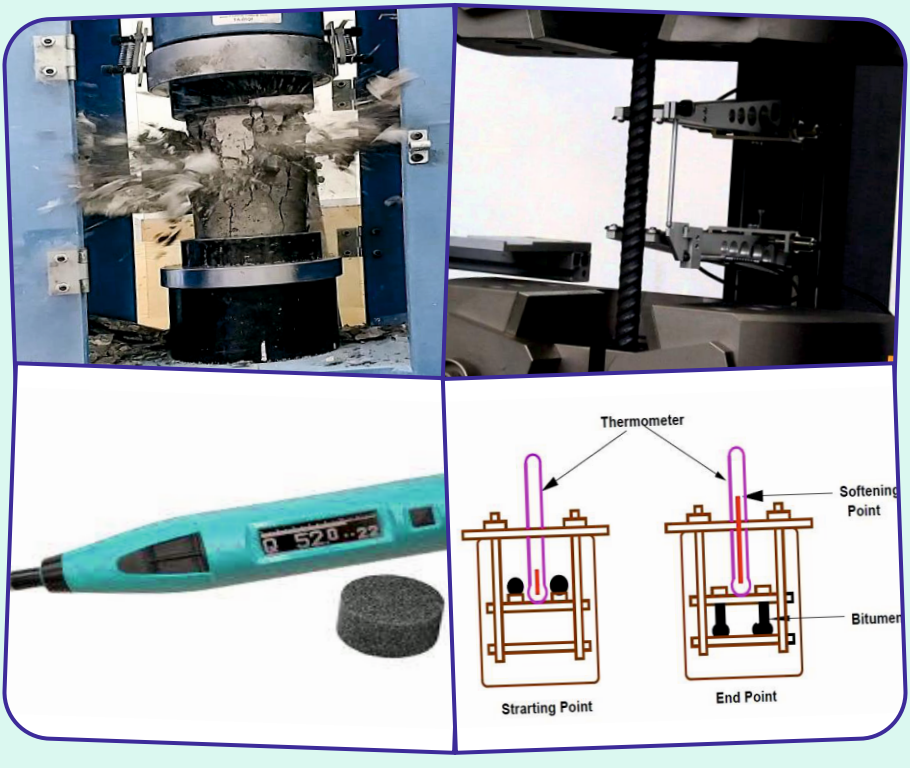
ज्ञान ज्योति से मार्गदर्शन

To Beam As A Beacon of Knowledge

HANDBOOK OF MATERIAL TESTING



November 2022

Indian Railways Institute of Civil Engineering

Pune - 411001

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FOREWORD TO SECOND EDITION

Testing of materials is an essential part of the Quality Control system for any Civil Engineering work. Awareness about relevant test procedures is necessary for the officials carrying out these tests in the lab as well as for the field engineers executing the works at site.

Seeing the importance of testing of materials, IRICEN had published "Handbook of Material Testing" in year 2006. This publication needed revision due to change in codes/standards. The tests related to Soil & Blanket materials have been deliberated in a separate book titled "Geotechnical Testing for Earthwork in Railway Projects". A need was also felt to include the tests for water quality and reinforcement also as separate chapters.

Therefore, the thoroughly revised/rewritten second edition of the "Handbook of Material Testing" has been authored by Shri R. K. Shekhawat (Senior Professor/IRICEN). For testing of Concrete & its' ingredients and Bitumen, this will serve as a ready reckoner for the officials performing tests in lab and the engineers executing the works at site.

Suggestions for further improvements, including those related to addition/deletion of any topics, may be sent to IRICEN for consideration in next revision of the book.

Pune
November, 2022

Ashok Kumar
Director General
IRICEN, Pune

PREFACE TO SECOND EDITION

The materials being used in any Civil Engineering work need to be tested, to ensure their conformance to relevant technical specifications. Proper understanding and appreciation of these tests is essential for field engineers to get these tests performed as per correct procedure and to interpret the test results. For the officials performing tests in lab also, proper appreciation of relevant test procedures is necessary to obtain reliable test results.

These tests are carried out as per relevant Indian Standard (IS Code) or Indian Railway Standard (IRS). But it is very difficult, for every field engineer to possess all these Standards/Codes and use them. To facilitate this, IRICEN had published "Handbook of Material Testing" in year 2006. But this book needed thorough revision as:

(i) Many of the relevant codes have been revised after year 2006.

(ii) For tests related to Soil and Blanket materials, separate detailed book on "Geotechnical testing for Earthwork in Railway Projects" has been published by IRICEN in year 2018.

(iii) Tests related to Reinforcement Steel and Water are to be added, as they were not included in the earlier book.

(iv) Some test procedures are to be added/elaborated as compared to the earlier book.

Therefore, to serve as a "ready reckoner" for field engineers and to serve as "laboratory manual", this revised "Handbook of Material Testing" is being published;

which covers all the tests required to be performed for Concrete & its' gradients and Bitumen. Objective of Test, Reference Standard, Equipment used for performing the Test, Observations to be recorded, Results to be presented and General Remarks (if any) have been brought out for each of the test.

The support and help rendered by Shri B. Ravi Kumar (Senior Instructor/IRICEN) and Shri Sabyasachi Roy (Senior Instructor/IRICEN), in collecting the contents, proof reading of the book and offering valuable suggestions, is appreciated.

It is felt that this book will be useful to engineers of Indian Railways. However, there is always a scope for improvement in any publication. Therefore, the suggestions for improvement are welcome from all the readers and the same may please be forwarded for incorporation in the future editions.

Pune
November, 2022

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PREFACE

While designing a structure, engineer assumes certain value of strength for each of material being used therein. When the structure is being constructed, it is the bounden duty of the field engineers to get the same validated by regular testing of material. The quality of materials used in any infrastructure does play a vital role with regard to its ultimate strength and durability in the long run. Hence, the materials need to be tested according to certain standard procedures developed by ASTM, BIS, RDSO to give a clear picture of material strength.

The "Handbook of Material Testing" is an attempt by IRICEN to bring together the standard test procedures for materials frequently used in the civil engineering infrastructure of Indian Railways. It is hoped that this will be a helpful guide to the field engineers. A list of suppliers of various testing equipments has also been provided alongwith their addresses to enable the engineers in setting up of a field laboratory, in case the need be.

Any suggestions to ameliorate the content of this handbook would be welcome.

Shiv Kumar
Director
IRICEN, Pune

ACKNOWLEDGEMENT

Strength and durability of any infrastructure is a reflection of the quality of materials used. Quality control of materials can only be ensured through certain standard test procedures designed by ASTM, BIS, RDSO and others.

The "Handbook of Material Testing" is an attempt towards this aim, of creating a collection of standard test procedures for materials, commonly used in civil engineering infrastructure on the Indian Railways. This will serve as a guide to the field engineers. The readers are advised in their interest to refer to the latest standards to avoid any omission, due to changes/amendments in the standards. The handbook also includes a list of suppliers of material testing equipments, alongwith their addresses to facilitate the setting up of a field laboratory.

The support rendered by the faculty and staff of IRICEN in this endeavour, needs mention. Shri Praveen Kumar, Professor/Computers and Shri J.M. Patekari, Chief Technical Assistant, have been of technical assistance and Shri Vijayakumaran. V, my Personal Assistant, has been extremely useful in the task of word processing.

I am thankful to Shri Shiv Kumar, Director/IRICEN for his guidance and encouragement.

R.K. Verma
Sr. Professor/Track
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Chapter - 1

TESTS ON CEMENT

The quality of cement being used is to be determined on the basis of its conformity to the performance characteristics given in the respective Indian Standard Specification for that cement. Any trade mark or any trade name indicating any special features not covered in the standard or any qualification or other special performance, characteristics sometimes claimed/ indicated on the bags or containers or in advertisements alongside the "Statutory Quality Marking" or otherwise have no relation whatsoever with the characteristics guaranteed by the Quality Marking. It is, therefore, advisable to go by the characteristics as given in the corresponding Indian Standard Specification or seek specialist advise to avoid any problem in concrete making and construction.

Following are the test typically conducted to check the performance characteristics of the cement:

(1.1) Fineness of Cement:

- (A) By 90 Micron Sieve
- (B) By Blain's Air Permeability

(1.2) Consistency of Cement

(1.3) Initial and Final Setting Time of Cement

(1.4) Soundness of Cement:

- (A) By Le-Chatelier Method
- (B) By Autoclave Method

(1.5) Strength Test of Cement

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1.1 Fineness of Cement

Fineness of the cement represents the particle size of the cement. The fineness of cement effects the rate of hydration and, therefore, the rate of gain of strength and the rate of heat evolution. The Finer cement provides a greater surface area for the hydration process and faster strength development. But increase in fineness of the cement also increases the drying shrinkage of the concrete.

(A) By 90 Micron Sieve

1. Introduction: In this method, the fineness of cement is measured by sieving it on standard sieve (i.e. 90 micron). A sample having a known proportion of material coarser than 90 micron size is taken as per the provisions of IS 3535:1986 and the relevant standard specification for the type of cement being tested. The sample shall be thoroughly mixed and sieved on the 90 micron sieve. The proportion of cement with grain sizes larger than the specified mesh size is determined.

2. Reference: IS-4031(Part-I):1996 (Reaffirmed-2021) "Method of Physical Tests for Hydraulic Cement-Determination of Fineness by Dry Sieving".

3. Apparatus required

(i) **Test Sieve:** With a firm, durable, non-corrodible, cylindrical frame of 150 mm to 200 mm nominal diameter and 40 mm to 100 mm depth, fitted with 90 micron mesh sieve cloth of woven stainless steel, or other abrasion-resisting and non-corrodible metal wire. A tray fitting beneath the sieve frame and a lid fitting above it shall be provided to avoid loss of material during sieving.

(ii) **Balance:** capable of weighing up to 10 g to the nearest 10 mg.

(iii) **Brush:** A nylon or pure bristle brush, preferably with 25 to 40 mm bristle, for cleaning the sieve.

4. Material for Checking the Sieve: A Standard reference material of known sieve residue shall be used for checking the sieve. The material shall be stored in sealed, airtight containers to avoid changes in its characteristics due to absorption or deposition from the atmosphere. The containers shall be marked with the sieve residue of the reference material.

5. Procedure

5.1 Determination of the Cement Residue

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

(ii) Fit the tray under the sieve, weigh approximately 10 g of cement to the nearest 0.01 g and place it on the sieve, being careful to avoid loss. Disperse any agglomerates. Fit the lid over the sieve. Agitate the sieve by swirling, planetary and linear movement until no more fine material passes through it. Remove and weigh the residue. Express its mass as a percentage, R1, of the quantity first placed in the sieve to the nearest 0.1 percent. Gently brush all the fine material off the base of the sieve into the tray.

(iii) Repeat the whole procedure using a fresh 10 g sample to obtain R2. Calculate R as the mean of R1 and R2 as a percentage, expressed to the nearest 0.1 percent. If the results differ by more than 1 percent absolute, carry out a third sieving and calculate the mean of the three values.

(iv) The sieving process is carried out manually. Alternatively, a sieving machine may be used provided that it can be shown to give the same results as the manual operation.

5.2 Checking the Sieve

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

(ii) Fit the tray under the sieve. Weigh approximately 10 g of the reference material to the nearest 0.01 g and place it in the sieve, being careful to avoid loss.

(iii) Carry out the sieving procedure as in Para 5.1 including the repeat determination of residue to yield two values P1 and P2, expressed to the nearest 0.1 percent. The two values of P1 and P2 for a satisfactory sieve should differ by not more than 0.3 percent. Their mean P characterizes the state of the sieve.

(iv) Given the known residue on the 90 micron mesh of the reference material, R0, calculate R0/P as the sieve factor, F, expressed to the nearest 0.01. The residue, R, determined as in Para 5.1 shall be corrected by multiplying by F, which may have a value of 1.00 ± 0.20.

(v) Check the sieve after every 100 sievings.

NOTE: Any other checking procedure, such as the optical methods described in IS 460 (Part 3):1985 may be used. All sieves will wear slowly and consequently their sieve factor, F, will slowly change.

6. Reporting of Result: Report the value of R, to the nearest 0.1 percent, as the residue on the 90 micron sieve for the cement tested.

The standard deviation of the repeatability is about 0.2 percent and of the reproducibility is about 0.3 percent.

(B) By Blain's Air Permeability

1. Introduction: In this test the fineness of cement is represented by specific surface expressed as total surface area in cm^2/g. The samples of the cement shall be taken according to the requirements of IS 3535:1986 and the relevant standard specification for the type of cement being tested. The sample shall be thoroughly mixed before testing.

2. Reference: IS-4031(Part-II):1999 (Reaffirmed-2013) "Method of Physical Tests for Hydraulic Cement-Determination of Fineness by Blain's Air Permeability Method".

3. Apparatus required:

(i) Variable Flow Type Air Permeability Apparatus (Blain's Type) and the accessories: conforming to IS 5516 (Fig. 1.1.1).

(ii) Timer: with a positive starting and stopping mechanism and 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.



Fig. 1.1.1: Blain Apparatus



Fig. 1.1.2: Pycnometer

(iii) 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.

(iv) Standard Weights.

(v) Pycnometer or other convenient means of determining the density of cement (Fig. 1.1.2).

(vi) 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.

(vii) Mercury of reagent grade or better.

(viii) Reference cement of known specific surface.

(ix) Light oil, to prevent formation of mercury amalgam on the inner surface of the cell.

(x) 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 micron).

(xi) Light grease, for ensuring an airtight joint between cell and manometer and its stopcock.

4. Test Procedure

4.1 Test Condition: 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.

4.2 Compacted Cement Bed

4.2.1 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 ρV(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 porosity, e, in the compacted bed of total volume V. The determination of ρ is described in Para 4.2.3 and that of V in Para 4.4.1.

4.2.2 Preparation of the Samples: 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.

4.2.3 Determination of Density: Determine the density of the cement using a device such as a pycnometer 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.

4.2.4 Formation of the Bed: To give a cement bed of porosity e = 0.500 weigh a quantity of cement, m_1, calculated from:

$m_1 = 0.500\ \rho V$ (g)

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 900 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.

4.3 Air Permeability Test

4.3.1Basis: The specific surface, S (in cm^2/g), is conveniently expressed as:

$$S = \frac{K}{\rho} \times \frac{\sqrt{e^3}}{(1-e)} \times \frac{\sqrt{t}}{\sqrt{0.1\eta}}$$

Where:

K is the apparatus constant,

e is the porosity of bed,

t is the measured time (s),

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

n is the viscosity of air at the test temperature taken from Table 1.1 (Pa.s).

With the specified porosity of e=0.5000 and temperatures:

(a) at 27±2^{0}C (in cm^2/g)

$$S = \frac{521.08\ K \times \sqrt{t}}{\rho}$$

(b) at 20±2^{0}C (in cm^2/g)

$$S = \frac{524.2\ K \times \sqrt{t}}{\rho}$$

Table 1.1: Density of Mercury D, Viscosity of Air (n) and $\sqrt{(0.1v)}$ as Function of Temperature

Temperature (^{0}C)	Mass Density of Mercury (g/cm^3)	Viscosity of Air (Pa.s)	$\sqrt{(0.1n)}$
16	13.56	0.00001788	0.001337
18	13.55	0.00001798	0.001341
20	13.55	0.00001808	0.001345
22	13.54	0.00001818	0.001348
24	13.54	0.00001828	0.001352
26	13.53	0.00001837	0.001355
28	13.53	0.00001847	0.001359
30	13.52	0.00001857	0.001363
32	13.52	0.00001867	0.001366
34	13.51	0.00001876	0.001370

NOTE: Intermediate value shall be obtained by Linear interpolation

4.3.2 Procedure

(i) 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.

(ii) 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 0.2s and the temperature to the nearest 10^0C.

(iii) 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 Para 4.2.4 or, where there is little cement available, by breaking up the first bed and reforming it as in Para 4.2.4. Carry out the permeability test twice on the second bed, recording the values of time and temperature as before.

4.4 Calibration of Apparatus

4.4.1 Determination of the Bed Volume

(i) 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.

a. 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.

b. 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:

$V = (m_2 - m_3)/D \quad (cm^3)$

Where, D is the density of mercury at the test temperature taken from Table 1.1

(ii) 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.

4.4.2 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 Para 4.2.2 to Para 4.2.4 and Para 4.3.2. Record the time t, and the temperature of test using the same bed; repeat twice the procedure of Para 4.3.2 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:

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

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

n_0 is the air viscosity at the mean of the three temperatures (Pa.s) (Table 1.1)

with the specified porosity of e=0.5000

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

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

4.4.3 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 1000 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.

4.5 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 Para 4.2.4. 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 Para 4.2.4 then becomes:

$m_4 = (1-e_1)\ \rho_1\ V\ (g)$

where, e_1 is the porosity determined experimentally.

4.6 Simplification of the Calculation

4.6.1 Basic Formula

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

$$S = \frac{\rho_0}{\rho} \times \frac{(1 - e_0)}{(1 - e)} \times \frac{\sqrt{e^3}}{\sqrt{e_0^3}} \times \frac{\sqrt{0.1\eta o}}{\sqrt{0.1\eta o}} \times \frac{\sqrt{t}}{\sqrt{t_0}} So$$

Where:

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

e is the porosity of the cement under test

e_0 is the porosity of the bed of reference cement

t is the measured time of cement under test(s)

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

ρ is the density of 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 1.1 (Pa.s) and

η_0 is the air viscosity at the mean of three temperatures (Table 1.1) for the reference cement (Pa.s)

4.6.2 Effect of specified Porosity

Use of the specified porosity, e=0.5000 for both the reference and test cement simplifies the formula to:

$$S = \frac{\rho_0}{\rho} \frac{\sqrt{0.1\eta o}}{\sqrt{0.1\eta o}} \text{x} \frac{\sqrt{t}}{\sqrt{t_0}} So \ (cm^2/g)$$

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

4.6.3 Effect of Density of Cement: The only remaining possibility of simplification is the elimination of density (ρ) terms. This has previously been done where the only cements for which is a value of ρ of 3.15 was assumed to supply. That assumption is known to produce errors up to 1 percent.

5. Expression of Results

(i) Where the porosity is e=0.500, the four times and temperatures resulting from the procedure of Para 4.3.2 shall be examined to check that the temperatures fall within the specified range of 27 ± 2^0C or 20 ± 2^0C. The resulting value of S, to the nearest 10 cm^2/g, shall be reported as the specific surface of the cement.

(ii) 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.

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

(iv) If, owing to a breakdown in control or for other reasons, the four temperatures do not lie within the specified range of 27 ± 2^0C or 20 ± 2^0C a value of S shall be reported, to the nearest 10 cm^2/g, as specific surface of the cement.

1.2 Consistency of Cement

1. Introduction: Consistency of cement is defined as minimum quantity of water added in cement to form uniform paste that provide sufficient viscosity and desirable strength for different type of structural work. Adding less or excess amount of water in cement causes reduction in its strength.

The samples of cement shall be taken as per IS:3535-1986 and the relevant standard specification for the type of cement being tested. The sample shall be thoroughly tested before testing. The temperature of moulding room, dry materials and water shall be maintained at 27±2^{0}C. The relative humidity of the laboratory shall be 65±5 percent.

2. Reference: IS-4031(Part-IV):1988 (Reaffirmed-2019) "Method of Physical Tests for Hydraulic Cement-Determination of Consistency of Standard Cement Paste".

3. Apparatus required:

(i) **Vicat Apparatus:** Conforming to IS 5513-1976 (Fig. 1.2.1).

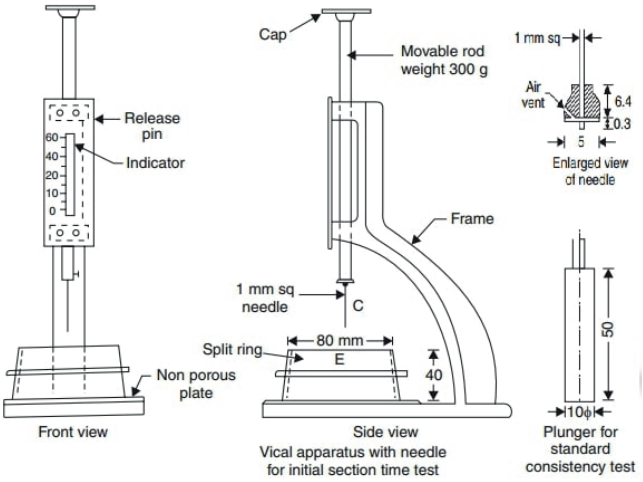




Fig. 1.2.1: Vicat Apparatus

(ii) Balance: On balance in use, the permissible variation at a load of 1000 g shall be plus or minus 1.0 g. The permissible variation on new balance shall be one-half of this value. The sensibility reciprocal shall not be greater than twice the permissible variation.

NOTE-1: The sensibility reciprocal is generally defined as the change in load required to change the position of rest of the indicating element or elements of a non-automatic indicating scale a definite amount at any load.

NOTE-2: Self-indicating balance with equivalent accuracy may also be used.

(iii) Standard Weights: The permissible variation in weights in weighing the cement shall be as prescribed in Table 1.2

Table 1.2: Permissible variation of Weights

Weight (g)	Permissible variation on weight in use, plus or minus (g)
500	0.35
300	0.30
250	0.25
200	0.20
100	0.15

50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

(iv) Gauging Trowel: confirming to IS:10086-1982 (Fig. 1.2.2)



Fig. 1.2.2: Gauging Trowel

4. Procedure

4.1 The standard consistency of a cement paste is defined as that consistency which will permit the Vicat plunger to penetrate to a point 5 to 7mm from the bottom of the Vicat mould when the cement paste is tested as described in Para 4.2 to 4.4.

4.2 Prepare a paste of weighed quantity of cement with a weighed quantity of potable or distilled water, taking care that the time of gauging is not less than 3 min., nor more than 5 min, and the gauging shall be completed before any sign of setting occurs. The gauging time shall be counted from the time of adding water to the dry cement until commencing to fill the mould. Fill the Vicat mould with this paste, the mould resting upon a non-porous plate. After completely filling the mould, smoothen the surface of the paste, making it level with the top of the mould. The mould

may be slightly shaken to expel the air.

Clean appliance shall be used for gauging. In filling the mould, the operator's hands and blade of the gauging trowel shall alone be used.

4.3 Place the test block in the mould, together with the non-porous resting plate, under the rod bearing the plunger; lower the plunger gently to touch the surface of the test block, and quickly release, allowing it to sink into the paste. This operation shall be carried out immediately after filling the mould.

4.4 Prepare trial pastes with varying percentages of water and test as described above until the amount of water necessary for making up the standard consistency as defined in 4.1 is found.

5. Interpretation and Reporting of Result: Express the amount of water as a percentage by mass of the dry cement to the first place of decimal.

1.3 Initial and Final Setting Time of Cement

1. Introduction: The initial setting time is regarded as the time elapsed between the time when the water is added to the cement, to the time that the paste starts losing its plasticity. If delayed further, cement loses its' strength. Initial setting time is an important time to know for concrete transportation, placing and curing. Initial setting time is also utilized to delay the process of hydration or hardening.

The final setting time is the time elapsed between the time when the water is added to the cement, and the time when the paste has completely lost its plasticity and has attained sufficient firmness to resist certain definite pressure. The final setting time is utilized for the safe removal of scaffolding or formwork.

The samples of cement shall be taken as per IS:3535-1986 and the relevant standard specification for the type of cement being tested. The sample shall be thoroughly tested before testing. The temperature of moulding room, dry materials and water shall be maintained at 27 ± 2^0C. The relative humidity of the laboratory shall be 65±5 percent. The moist closer or moist room shall be maintained at 27 ± 2^0C and at a relative humidity of not less than 90 percent.

2. Reference: IS-4031(Part-V):1988 (Reaffirmed-2019) "Method of Physical Tests for Hydraulic Cement-Determination of Initial and Final Setting Times".

3. Apparatus required

(i) Vicat Apparatus: conforming to IS 5513-1976 (Fig. 1.3.1).

(ii) Balance: On balance in use, the permissible variation at a load of 1000 g shall be plus or minus 1.0 g. The permissible variation on new balance shall be one-half of this value. The sensibility reciprocal shall not be greater than twice the permissible variation.

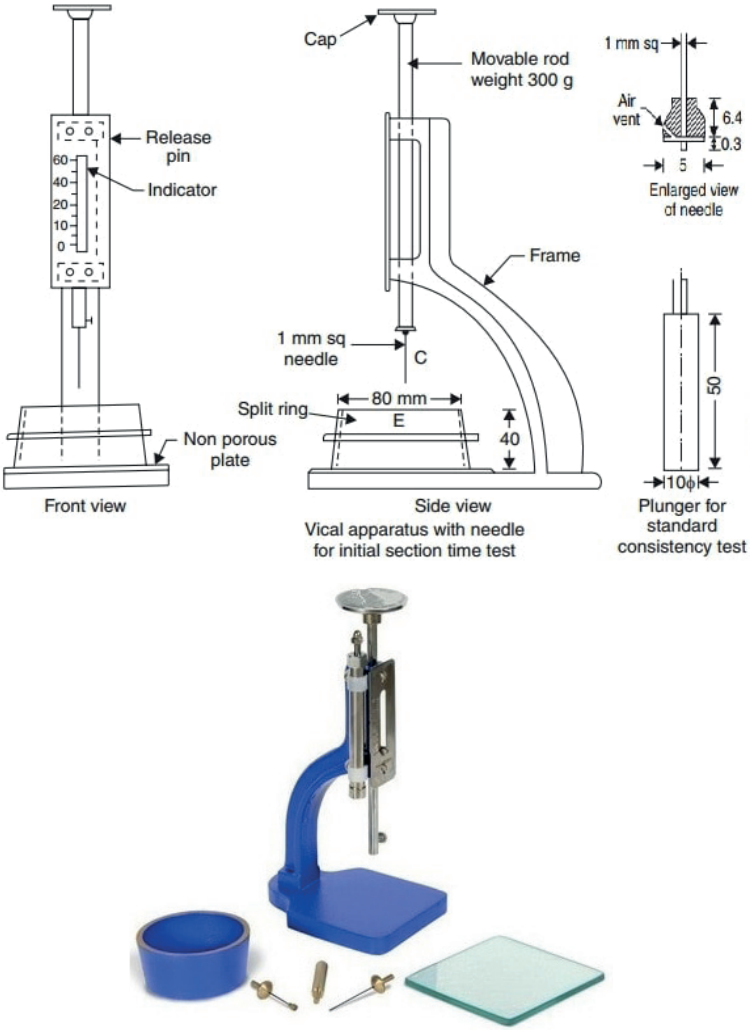


Fig. 1.3.1: Vicat Apparatus

NOTE-1: The sensibility reciprocal is generally defined as the change in load required to change the position of rest of the indicating element or elements of a non-automatic indicating scale a definite amount at

any load.

NOTE-2: Self-indicating balance with equivalent accuracy may also be used.

(iii) Standard Weights: The permissible variation in weights in weighing the cement shall be as prescribed in Table 1.3

Table 1.3: Permissible variation of Weights

Weight (g)	Permissible variation on weight in use, plus or minus (g)
500	0.35
300	0.30
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

(iv) Gauging Trowel: confirming to IS:10086-1982 (Fig. 1.3.2)



Fig. 1.3.2: Gauging Trowel

4. Procedure

4.1 Preparation of Test Block: Prepare a neat cement paste by gauging the cement with 0.85 times the water required to give a paste of standard consistency. Potable or distilled water shall be used in preparing the paste. The paste shall be gauged in the manner and under the conditions prescribed in IS:4031 (Part 4)- 1988. Start a stop-watch at the instant when water is added to the cement. Fill the Vicat mould with a cement paste gauged as above, the mould resting on a nonporous plate. Fill the mould completely and smooth off the surface of the paste making it level with the top of the mould. The cement block thus prepared in the mould is the test block.

4.1.1 Immediately after moulding, place the test block in the moist closet or moist room and allow it to remain there except when determinations of time of setting are being made.

NOTE-1: Clean appliances shall be used for gauging.

NOTE-2: All the apparatus shall be free from vibration during the test.

NOTE-3: Care shall be taken to keep the needle straight.

4.2 Determination of Initial Setting Time: Place the test block confined in the mould and resting on the non-porous plate, under the rod bearing the needle; lower the needle gently until it comes in contact with the surface of the test block and quickly release, allowing it to penetrate into the test block. In the beginning, the needle will completely pierce the test block. Repeat this procedure until the needle, when brought in contact with the test block and released as described above, fails to pierce the block beyond 5.0 ± 0.5 mm measured from the bottom of the mould.

The period elapsing between the time when water is added to the cement and the time at which the

needle fails to pierce the test block to a point 5.0 ± 0.5 mm measured from the bottom of the mould shall be the initial setting time.

4.3 Determination of Final Setting Time: Replace the needle of the Vicat apparatus by the needle with an annular attachment "F". The cement shall be considered as finally set when, upon applying the needle gently to the surface of the test block, the needle makes an impression thereon, while the attachment fails to do so.

The period elapsing between the time when water is added to the cement and the time at which the needle makes an impression on the surface of test block while the attachment fails to do so shall be the final setting time.

In the event of a scum forming on the surface of the test block, use the underside of the block for the determination.

5. Interpretation and Reporting of Result: The results of initial and final setting time shall be reported to the nearest five minutes.

1.4 Soundness of Cement

1. Introduction: One of the physical characteristics of cement is its ability to resist a change in its volume or shape as it hardens or sets. The degree of restraint it provides to any volumetric alteration characterizes its soundness. It is important that the cement after setting shall not undergo any appreciable change of volume. Lack of soundness may lead to volume expansion that may induce tensile stresses, ultimately leading to cracking, which will cause serious difficulties for the durability of structures when such cement is used. The unsoundness in cement is due to the presence of excess of free lime than that could be combined with acidic oxide at the kiln. It is also likely that too high a proportion of magnesium content or calcium sulphate content may cause unsoundness in cement.

2. Sampling and Selection of Test Specimen: The samples of the cement shall be taken in accordance with the requirements of IS:3535-1986 and the relevant standard specification for the type of cement being tested. The sample shall be thoroughly mixed before testing.

3. Temperature and Humidity: The temperature of moulding room, dry materials and water shall be maintained at 27 ± 2°C. The relative humidity of the laboratory shall be 65 ± 5 percent. The moist closet or moist room shall be maintained at 27 ± 2°C and at a relative humidity of not less than 90 percent.

4. Reference: IS-4031(Part-III):1988 (Reaffirmed-2019) "Method of physical tests for Hydraulic cement-Determination of Soundness".

(A) By Le-Chatelier Method

1. Apparatus required

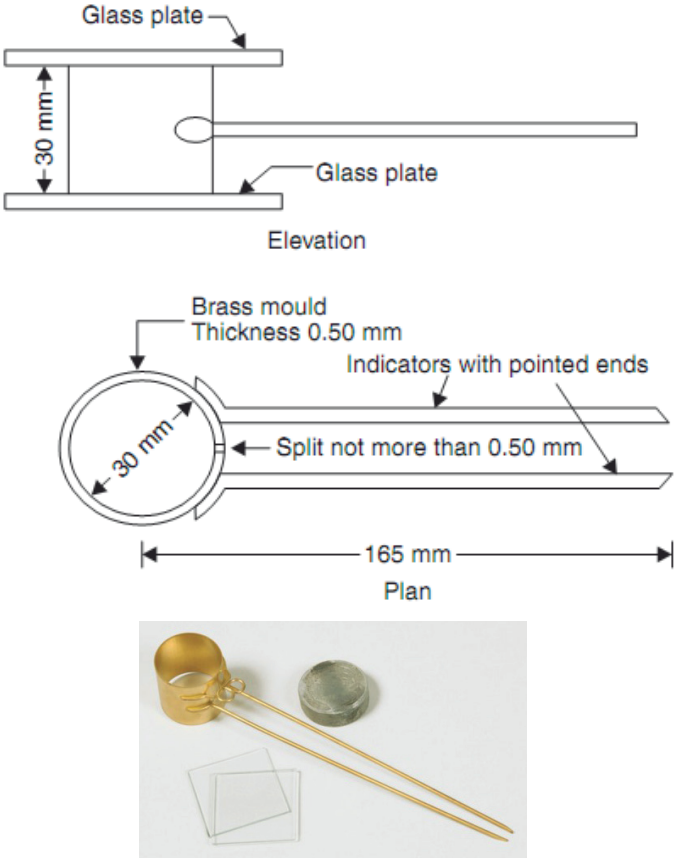


Fig. 1.4.1: Le-Chatelier Apparatus

(i) Le Chatelier Apparatus: conforming to IS 5514-1969 (Fig. 1.4.1)

(ii) Balance: On balance in use, the permissible variation at a load of 1000 g shall be plus or minus 1.0 g. The permissible variation on new balance shall be one-half of this value. The sensibility reciprocal shall not be greater than twice the permissible variation.

(iii) Weights: The permissible variation in weights in weighing the cement shall be as prescribed in Table 1.4

Table 1.4: Permissible variation of Weights

Weight (g)	Permissible variation on weight in use, plus or minus (g)
500	0.35
300	0.30
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

(iv) Water Bath: capable of containing immersed Le-Chatelier moulds with specimens and of raising their temperature from 27 ± 2°C to boiling in 27 ± 3 minutes (Fig. 1.4.2).



Fig. 1.4.2: Water Bath

NOTE-1: The sensibility reciprocal is generally defined as the change in load required to change the position of rest of the indicating element or elements of a non-automatic indicating scale a definite amount at any load.

NOTE-2: Self-indicating balance with equivalent accuracy may also be used.

2. Procedure

2.1 Place the lightly oiled mould on a lightly oiled glass sheet and fill it with cement paste formed by gauging cement with 0.78 times the water required to give a paste of standard consistency. The paste shall be gauged in the manner and under the conditions prescribed in IS:4031 (Part 4)-1988, taking care to keep the edges of the mould gently together while this operation is being performed. Cover the mould with another piece of lightly oiled glass sheet, place a small weight on this covering glass sheet and immediately submerge the whole assembly in water at a temperature of 27 ± 2^0C and keep there for 24 hours.

2.2 Measure the distance separating the indicator points to the nearest 0.5 mm. Submerge the mould again in water at the temperature prescribed above. Bring the water to boiling, with the mould kept submerged, in 25 to 30 minutes, and keep it boiling for three hours. Remove the mould from the water, allow it to cool and measure the distance between the indicator points. The difference between these two measurements indicates the expansion of the cement.

3. Interpretation and Reporting of Result: Calculate the mean of two values to the nearest 0.5 mm to represent the expansion of cement.

4. Retest: In the event of cement failing to meet the test for soundness, a retest may be made after aeration. For this purpose, spread out the cement in a layer of 75 mm thickness and store it for 7 days in an atmosphere

maintained at 27 ± 2'C and relative humidity of 50 to 80 percent. Retest this cement as described in Para 3.

(B) By Autoclave Method

1. Apparatus required

(i) Autoclave: The autoclave shall consist of a high-pressure steam boiler equipped with suitable safety device. The capacity of heating unit shall be such that with maximum load (water plus specimens) the pressure of the saturated steam in the autoclave may be raised to a gauge pressure of 2.1 MPa or to an absolute pressure of about 2.2 MPa, in 1 to 1¼ hour from the time the heat is turned on. The automatic pressure control shall be capable of maintaining the pressure at 2.1 ± 0.1 Mpa corresponding to a temperature of 215.7 ± 1.7^0C. The autoclave shall be designed to permit the pressure to drop from 2.1 MPa to less than 0.07 MPa in one hour after the heat supply has been shut off. It shall be equipped with a vent valve for allowing the escape of air during the early part of the heating period and for releasing any steam pressure remaining at the end of the one-hour cooling period. The pressure gauge shall have a nominal dial diameter of 115 mm and shall be graduated from 0 to 4.1 MPa with scale division of not more than 0.04 MPa. The error in the gauge shall not exceed plus or minus 0.02 MPa at the operating pressure of 2.1 MPa (Fig. 1.4.3).

(ii) Length Comparator: Changes in length of the test specimen shall be measured by an apparatus conforming to IS:9459-1980 (Fig. 1.4.4).

2. Preparation of Test Specimens

2.1 Preparation of Moulds: The moulds shall be thinly covered with mineral oil. After this operation, the stainless steel or non-corroding metal reference inserts with knurl heads shall be set to obtain an effective gauge length of 250 mm, care being taken to keep them clean and free from oil.



Fig. 1.4.3: Autoclave

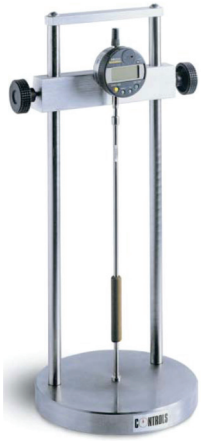


Fig. 1.4.4: Length Comparator

2.2 Mixing Cement Paste: The standard batch of cement paste shall consist of 500 g of cement, mixed with sufficient water to give a paste of standard

consistency.

2.3 Moulding Specimens: Immediately following the completion of mixing, the test specimens shall be moulded in one or two layers, each layer being compacted with the thumb or forefinger by pressing the paste 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 paste shall be cut 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 hand shall be protected by rubber gloves.

2.4 Storage of Test Specimens: After the mould has been filled, it shall be immediately placed in a moist closet or a moist room. Specimens shall remain .in the moulds in the moist room for at least 24 hours. If removed from the moulds before 24 h, they shall be kept in the moist closet or moist room until tested.

3. Procedure

3.1 At 24±½ hours after moulding, the specimens shall be removed from the moist atmosphere, measured for length, and placed in the autoclave at room temperature in a rack so that the four sides of each specimen shall be exposed to saturated steam. The autoclave shall contain enough water to maintain an atmosphere of saturated steam vapour during the entire period of test. Ordinarily, 7 to 10 percent of the volume of the autoclave shall be occupied by water.

3.2 To permit air to escape from the autoclave during the early portion of the heating period, the vent valve shall be left open until steam begins to escape. The valve shall then be closed and the temperature of the autoclave shall be raised at such a rate as will bring the gauge pressure of the steam to 2.1 MPa in 1 to 1¼ hour from the time the heat is turned on. The 2.1 ± 0.1 MPa pressure shall be maintained for 3 hours. At the end of 3 hours period, the heat supply shall be

shut off and the autoclave cooled, at a rate such that the pressure will be less than 0.1 MPa at the end of the hour, and any pressure remaining shall be slow released by partially opening the vent valve until atmospheric pressure is attained. The autoclave shall then be opened and the test specimens immediately placed in water, the temperature of which is above 90^0C. The water surrounding the bars shall then be cooled at a uniform rate by adding cold water so that the temperature of the water shall be lowered to 27 ± 2^0C in 15 min. The water surrounding the specimens shall then be maintained at 27 ± 2^0C in 15 min when the specimens shall be surface dried and their lengths measured again.

4. Safety Precautions

4.1 The pressure gauge should have a maximum capacity of 4.2 MPa. This is important because with too small a capacity there is but a little length of arc in which the gauge hand may indicate pressure above the specified maximum working pressure. The operator must be sure that the gauge hand has not passed the maximum graduation on the scale.

4.2 It is well to leave the pressure gauge tested, but in any event thermometer shall always be used together with the pressure gauge, so as to provide a means of detecting any failure of the pressure gauge to operate properly and also to indicate any unusual conditions such as that resulting from loss of water from the autoclave during the test.

4.3 The automatic control shall be maintained in proper working order at all times.

4.4 The safety valve shall be set so as to relieve the pressure at about 6 to 10 percent above the maximum of 2.1 MPa specified, that is at about 2.3 MPa. The safety valve shall be tested at least twice a year, either with a gauge testing device or by adjusting the automatic controls so as to allow the autoclave to reach a pressure of about 2.3 MPa at which pressure

the safety valve shall either open or be adjusted to open. The safety valve discharge shall be directed away from the operator.

4.5 Heavy leather work gloves shall be worn to prevent burning of the hands when removing the top of the autoclave at the end of the test. The vent valve shall be directed away from the operator. When removing the autoclave lid, the lid shall be so tilted that any steam escaping from beneath the lid may be discharged away from the operator. Care shall be taken to avoid scalding by any liquid that may have been used in the autoclave well.

4.6 It shall be remembered that for many of the autoclave pressure gauges now in use the return of the gauge hand to the initial rest or starting point does not necessarily indicate zero pressure within the autoclave; there may then still remain an appreciable pressure.

4.7 A few drops of kerosene placed in the vent valve about once a week will aid in keeping the needle clean and in good-working condition.

5. Interpretation and Reporting of Result: The difference in lengths of the test specimen before and after autoclaving shall be calculated to the nearest 0.01 percent of the effective gauge length which is the length between the innermost points of the metal inserts used as reference points and shall be reported as the autoclave expansion of the cement. A contraction (negative expansion) shall be indicated by prefixing a minus sign to the percentage expansion reported.

6. Retest: In the event of cement failing to meet the test for soundness, a retest may be made after aeration. For this purpose, spread out the cement in a layer of 75 mm thickness and store it for 7 days in an atmosphere maintained at 27 ± 2'C and relative humidity of 50 to 80 percent. Retest this cement as described in Para 2 to 3.

1.5 Strength Test of Cement

1. Introduction: The compressive strength of hardened cement is one of its most important properties. Therefore, the cement is always tested for its strength at the laboratory before being used in works. Strength tests are not made on neat cement paste because of difficulties of excessive shrinkage and subsequent cracking of neat cement.

The samples of the cement shall be taken in accordance with the requirements of IS:3535-1986 and the relevant standard specification for the type of cement being tested. The sample shall be thoroughly mixed before testing.

The temperature of moulding room, dry materials and water shall be maintained at 27 ± 2°C. The relative humidity of the laboratory shall be 65 ± 5 percent. The moist closet or moist room shall be maintained at 27 ± 2°C and at a relative humidity of not less than 90 percent.

Standard Sand to be used in the preparation of mortar cubes shall conform to IS:650-1966.

2. Reference: IS:4031 (Part-VII) : 1988 (Reaffirmed-2019) "Method of physical tests for Hydraulic Cement-Determination of Compressive Strength of Masonry Cement".

3. Apparatus required

(i) Balance: On balance in use, the permissible variation at a load of 1000 g shall be plus or minus 1.0 g. The permissible variation on new balance shall be one-half of this value. The sensibility reciprocal shall not be greater than twice the permissible variation.

NOTE-1: The sensibility reciprocal is generally defined as the change in load required to change the position of rest of the indicating element or elements of a non-automatic indicating scale a definite amount at any load.

NOTE-2: Self-indicating balance with equivalent accuracy may also be used.

(ii) Weights: The permissible variation in weights in weighing the cement shall be as prescribed in Table 1.5

Table 1.5: Permissible variation of Weights

Weight (g)	Permissible variation on weight in use, plus or minus (g)
500	0.35
300	0.30
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

(iii) Cube Moulds: of 50 mm size and accessories conforming to IS:10086-1982 (Fig. 1.5.1).



Fig. 1.5.1: Cube Mould



Fig. 1.5.2: Planetary Mixer



Fig. 1.5.3: Flow Table

(iv) Planetary Mixer: conforming to IS:10890-1984 (Fig. 1.5.2).

(v) Flow Table and Accessories: conforming to

IS:5512-1983 (Fig. 1.5.3).

(vi) Tamping Rod: conforming to 6.1 (c) of IS:10086-1982.

4. Procedure

4.1 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 the mineral' oil, petrolatum or light cup grease.

4.2 Preparation of Mortar

(i) Clean appliances shall be used for mixing. Temperature of water and that of the test room at the time when these operations are being performed shall be 27±2°C. Potable/distilled water shall be used in preparing the cubes.

(ii) The material for each set of three specimens shall be mixed separately and shall be as follows:

1.	Masonry Cement	420 g
2.	Other Cement	440 g

(iii) The amount of water used for gauging shall be such as to produce a flow of 10±5 percent with 25 drops in 15 seconds as determined in Para 4.3

4.3 Determination of Flow

4.3.1 Trial Mixing: With dry material as specified in Para 4.2, make trial mortars with different percentages of water until specified flow is obtained. Make each trial flow test with fresh mortar. The mixing shall be done mechanically by means of mixing apparatus as specified in Para 3 (iv). 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 masonry cement to the water, then start the mixer and mix at the slow speed (140±5 rev/min) for 30 second;

c) Add the entire quantity. of sand slowly over a period of 30 s, while mixing at slow speed (140±5 rev/min);

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 s of this interval, quickly scrap 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); and

g) In cases requiring further remixing, any mortar adhering to the side of the bowl shall be quickly scraped down into the batch with the scraper prior to remixing which is to be continued till a uniform mortar is obtained.

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

4.3.2 Carefully wipe the flow-table top clean and dry and place the mould at the centre. Place about 25 mm thick layer of mortar mixed in accordance with Para 4.3.1 in the mould and tamp 20 times with the tamping rod. The tamping pressure shall be just 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 excess mortar to a plain

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 completion of the mixing operation. Immediately drop the table through a height of 12.5 mm, 25 times in 15 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.

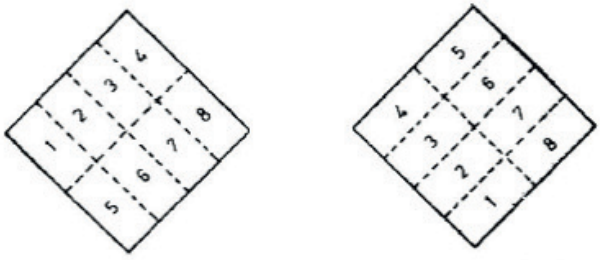
4.4 The material for moulding each batch of test specimens shall be mixed separately using the quantities of dry materials, conforming to the proportions specified in Para 4.2 and the quantity of water as determined in 4.3. Mixing of mortar shall be done mechanically as described in Para 4.3.1

4.5 Moulding of Specimens

4.5.1 Immediately following completion of the flow test, return the mortar from the flow mould to the mixing bowl. Quickly scrape down into the batch the mortar that may have collected on the side of the bowl and give the entire batch a 15 seconds mixing at medium speed (285±10 rev /min). Start moulding the specimens within a total elapsed time of not more than 2 min and 15 seconds after completion of the original mixing of the mortar batch. Place a layer of mortar about 25 mm in thickness in all the cube compartments. Tamp the mortar in each cube compartment 32 times in about 10 seconds in four rounds, each round to beat right angles to the other and consisting of eight adjoining strokes over the surface of the specimen as illustrated in Fig. 1.5.4. The tamping pressure

shall be just sufficient to ensure uniform filling of the moulds. The four rounds of tamping (32 strokes) of the mortar shall be completed in one cube before going to the next. When the tamping of the first layer in all of the cube compartments is completed, fill the compartments with the remaining mortar and then tamp as specified for the first layer. During tamping of the second layer, bring in the mortar forced out on to the tops of the moulds after each round of tamping by means of the gloved fingers and the tamper upon completion of each round and before starting the next round of tamping. On completion of the tamping, the tops of all cubes should extend slightly above the tops of the moulds. Bring in the mortar that has been forced out on to the tops of the moulds with a trowel and smooth off the cubes by drawing the flat side of the trowel, (with the leading edge slightly raised) once across the top of each cube at right angles to the length of the mould. Then for the purpose of levelling the mortar and making the mortar that protrudes above the top of the mould of more uniform thickness, draw the flat side of the trowel with the leading edge slightly raised lightly once along the length of the mould. Cut off the mortar to a plane surface flush with the top of the mould by drawing the straight edge of the trowel (held nearly perpendicular to the mould) with a sawing motion over the length of the mould.

NOTE: When a duplicate batch is to be made immediately for additional specimens the repetition of flow test may be omitted and the mortar allowed to stand in the mixing bowl for 90 seconds and then remixed for 15 seconds. at medium speed before starting the moulding of the specimens.



ROUNDS 1 AND 3 ROUNDS 2 AND 4

Fig. 1.5.4

4.6 Storage and Curing of Specimens: All test specimens, immediately after moulding and compaction, shall be kept in moulds on plane plates in a moist cabinet, maintained at a temperature of 27±2°C and the relative humidity of 90 percent or more, from 48 to 52 hours in such a manner that the upper surfaces shall be exposed to the moist air. The cubes shall then be removed from the moulds and placed in the moist cabinet for five days in such a manner as to allow free circulation of air around at least five faces of the specimens. After five days curing in moist cabinet, the cubes for 7-day compressive strength shall be removed for testing whereas the cubes for 28-day compressive strength test shall be immersed in clean water for another twenty-one days in storage tanks of non-corrosive materials.

4.7 Testing

(i) Test not less than three cubes for compressive strength for each of the curing periods of 7 and 28 days as indicated in Para 4.6, the periods being reckoned from the completion of moulding and compaction.

(ii) Testing of the cube specimens shall be carried out immediately after their removal from the moist cabinet for 7-day specimens, and from storage water for all other specimens. If more

than one specimen at a time is removed from the moist cabinet for 7-day tests, these cubes shall be covered with a damp cloth until the time of testing. If more than one specimen at a time is removed from storage water for testing, these cubes shall be placed in a pan of water at a temperature of 27±2^{O}C and of sufficient depth to completely immerse each cube until the time of testing.

(iii) The cubes shall be tested on their sides without any packing between the cube and the steel plattens of the electrically operated testing machine. One of the plattens shall be carried on a base and shall be self-adjusting. An initial loading up to one-half of the expected maximum load for specimens having expected maximum loads of more than 13,500 N may be applied at any convenient rate. Apply no initial loading to specimens having expected maximum loads of less than 13,500 N. Adjust the rate of load without interruption so that the breaking strength of the cube is reached in not less than 20 seconds and not more than 80 seconds. Make no adjustment in the control of the testing machine while a specimen is yielding rapidly immediately before failure.

5. **Interpretation and Reporting of Result:** The measured compressive strength of the cubes shall be calculated by dividing the maximum load applied to the cubes during the test by the cross-sectional area, calculated from the mean dimensions of the section and shall be expressed to the nearest 0.5 N/mm2. In determining the compressive strength, do not consider specimens that are manifestly faulty, or that give strengths differing by more than 10 percent from the average value of all test specimens. After discarding specimens or strength values, if less than two strength values are left for determining the compressive strength at any given period, a retest shall be made.

Chapter - 2

TESTS ON AGGREGATES

Aggregates shall be naturally occurring (crushed or uncrushed) stones, gravel, sand or combination thereof or produced from other than natural sources. They shall be hard, dense, strong, durable, clear and free from veins; and free form injurious amounts of disintegrated pieces, alkali, fee lime, vegetable matter and other deleterious substances as well as adherent coating.

Following are the tests typically conducted on the aggregates :

(2.1) Sieve Analysis

(2.2) Water Absorption

(2.3) Aggregate Abrasion Value

(2.4) Aggregate Impact Value

(2.5) Aggregate Crushing Value

(2.6) Silt Content

(2.7) Bulking of Sand

(2.8) Flakiness Index and Elongation Index

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2.1 Sieve Analysis

Aggregates are important components for making concrete and properties of concrete are substantially affected by various characteristics of the aggregates used, with Size and Gradation of aggregates needing one of these characteristics. Sieve analysis is the most common method to determine the Size and Gradation of Coarse and Fine aggregates.

1. **Introduction:** This method covers the procedure for determination of particle size distribution of fine, coarse and all-in-aggregates by sieving or screening.

2. Reference: IS-2386(Part-I):1963 (Reaffirmed- 2021) "Method of Tests for Aggregates for Concrete. Part-I: Particle Size and Shape".

3. Apparatus required

3.1 Sieves: Sieves of the sizes given in Table-2.1, conforming to IS: 460-1962 Specification for Test Sieves (Revised).

Table 2.1: IS Sieves for Sieve Analysis

Type	Sieve Designation
Square hole, perforated plate	80mm, 63mm, 50mm, 40mm, 31.5mm, 25mm, 20mm, 16mm, 12.5mm, 10mm, 6.3mm, 4.75mm
Fine mesh, wire cloth	3.35mm, 2.36mm, 1.18mm, 600 micron, 300 micron, 150 micron, 75 micron

3.2 Balance: Readable and accurate to 0.1 percent of the weight of the test sample.

4. Sample: The weight of sample for Coarse and Fine aggregates shall not be less than the weight given in Table-2.2. The weight of sample for all-in-aggregates shall not be less than the weight given in Table-2.3. The sample shall be prepared from the larger sample either by quartering or by means or a sample divider.

5. Procedure:

5.1 The sample shall be air dried, at room temperature or by heating at a temperature of 100°-110°C, before weighing and sieving. The sample shall then be weighed and sieved successively on the appropriate sieves starting with the largest.

Table 2.2: Minimum Weight for Sampling (Coarse and fine Aggregates)

Maximum Size present in substantial proportion (mm)	Minimum weight of Sample for testing (kg)
63	100
50	100
40	50
25	50
20	25
16	25
12.5	12
10	6
6.3	3

Table 2.3: Minimum Weight for Sampling (All-in-aggregates)

Maximum Size present in substantial proportion (mm)	Minimum weight of Sample for testing (kg)
63	50
50	35
40 or 31.5	15
25	5
20 or 16	2

12.5	1
10	0.5
6.3	0.2
4.75	0.2
2.36	0.1

5.2 Each sieve shall be shaken separately until not more than a trace passes, but for a period of not less than two minutes. Material shall not be forced through the sieve by hand pressure, but on sieves coarser than 20mm, placing of particles is permitted. Lumps of fine material, if present, may be broken by gentle pressure with fingers against the side of the sieve. Light brushing may be done on the underside of the sieve, with a soft brush, to clear the sieve openings. Light brushing with a fine camel hair brush may be used on the 150 micron and 75 micron IS Sieves to prevent aggregation of powder and blinding of apertures.

5.3 On completion of sieving, the material retained on each sieve, together with any material cleaned from the mesh, shall be weighed.

5.4 In order to prevent binding of the sieve apertures by over-loading, the weight of the aggregate retained on the sieve at completion of the operation shall not be greater than the value given Table-2.4. This normally requires several operations on each sieve.

Table 2.4: Maximum Weight retained after Sieving

Coarse Aggregate			Fine Aggregate	
IS Sieve	Maximum Weight (kg) for		IS Sieve	Maximum weight (g) for 20 cm dia sieve
	45 cm dia. sieve	45 cm dia. sieve		
50 mm	10	4.5	2.36 mm	200
40 mm	8	3.5	1.18 mm	100
31.5 or 25 mm	6	2.5	600 micron	75
20 mm	4	2.0	300 micron	50
16 or 12.5 mm	3	1.5	150 micron	40
10 mm	2	1.0	75 micron	25
6.3 mm	1.5	0.75		
4.75 mm	1.0	0.50		
3.35 mm	-	0.30		

6. Reporting of Result: The results shall be calculated and reported as:

(a) the cumulative percentage by weight of the total sample passing each of the sieves, to the nearest whole number; or

(b) the percentage by weight of the total sample passing one sieve and retained on the next smaller sieve, to the nearest 0.1 percent.

6.1 Graphical Method of Recording Results: The results of sieve analysis may be recorded graphically on the chart for recording sieve analysis shown in Fig. 2.1.

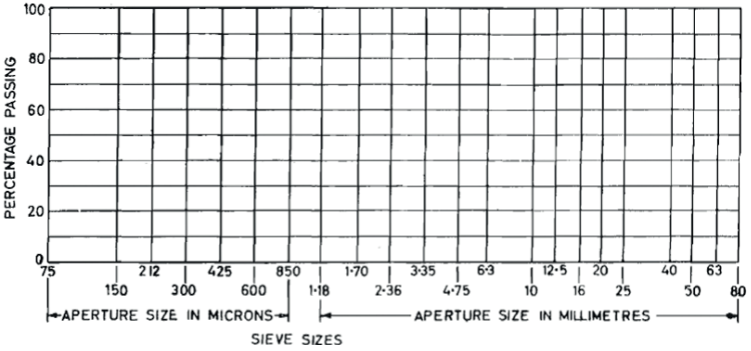


Fig. 2.1: Chart of Sieve Analysis Results

2.2 Water Absorption

1. Introduction: All aggregates have pores, which can get filled with water. The water absorption of the aggregates, along with the moisture content of the aggregates, are important factors to consider when proportioning the concrete mix, particularly the amount of water required.

2. Reference: IS-2386(Part-III):1963 (Reaffirmed-2021) "Method of Tests for Aggregates for Concrete. Part-III: Specific Gravity, Density, Voids, Absorption and Bulking".

(A) Method-I: Aggregate larger than 10mm

3. Apparatus required

(a) Balance: Of capacity not less than 3 kg, accurate to 0.5 g and of such a type and shape as to permit the basket containing the sample to be suspended from the beam and weighed in water.

(b) Oven: A well ventilated oven, thermostatically controlled, to maintain a temperature of 100 to 110^0C.

(c) Wire basket of not more than 6.3 mm mesh or a perforated container of convenient size, preferably chromium plated and polished, with wire hangers not thicker than one millimeter for suspending it from the balance.

(d) A stout watertight container in which the basket may be freely suspended.

(e) Two dry soft absorbent cloths each not less than 75 X 45 cm.

(f) A shallow tray of area not less than 650 cm^2.

(g) An airtight container of capacity similar to that of the basket.

4. Sample: A sample of not less than 2000 g shall be tested. Aggregates which have been artificially heated shall not normally be used. If such material is used, the fact shall be stated in the report. Two tests shall be made, and it is recommended that the two samples are not tested concurrently.

5. Procedure:

(a) The sample shall be thoroughly washed to remove finer particles and dust, drained and then placed in the wire basket and immersed in distilled water at a temperature between 22°C and 32°C with a cover of at least 5 cm of water above the top of the basket.

(b) The entrapped air shall be removed from the sample by lifting the basket 25mm above the base of the tank and allowing it to drop 25 times at the rate of about one drop per second. The basket and aggregate shall remain completely immersed during the operation and for a period of 24 ± 1/2 hours afterwards.

(c) The basket and the aggregate shall then be removed from the water and allowed to drain for a few minutes, after which the, aggregate shall be gently emptied from the basket on to one of the dry clothes.

(d) The aggregate placed on the dry cloth shall be gently surface dried with the cloth, transferring it to the second dry cloth when the first will remove no further moisture. It shall then be spread out not more than one stone deep on the second cloth, and lest exposed to the atmosphere away from direct sunlight or any other source of heat for not less than 10 minutes, or until it appears to be completely surface dry (which with some aggregates may take an hour or more). The aggregate shall be turned over at least once during this period and a gentle current of unheated air may be used after the first ten minutes to accelerate the drying of difficult aggregates. The

aggregate shall then be weighed (weight B).

(e) The aggregate shall then be placed in the oven in the shallow tray, at a temperature of 100 to 110^0C and maintained at this temperature for 24 ± 1/2 hours. It shall then be removed from the oven, cooled in the airtight container and weighed (weight C).

6. Calculations: Water absorption shall be calculated as follows:

Water absorption (percent of dry weight) = 100 (B-C)/C

7. Reporting of Result: The individual and mean results shall be reported. The size of the aggregate tested shall be stated.

(B) <u>Method-II: Aggregate between 40mm and 10mm</u>

3. Apparatus required

(a) <u>Balance:</u> Of capacity not less than 3 kg and accurate to 0.5 g, and of such a type as to permit the weighing of the vessel containing the aggregate and water.

(b) <u>Oven:</u> A well ventilated oven, thermostatically controlled to maintain a temperature of 100 to 110^0C.

(c) <u>Glass Vessel or Jar:</u> A wide mouthed glass vessel such as a jar of about 1.5 liters capacity, with a flat ground lip and a plane ground disc of plate glass to cover it, giving a virtually watertight fit.

(d) Two dry soft absorbent cloths, each not less than 75 X 45 cm.

(e) A shallow tray of area not less than 325 cm^2.

(f) An airtight container large enough to take the sample.

4. Sample: A sample of about one kilogram shall be tested. Aggregates which have been artificially heated shall not normally be used. If such material is used, the fact shall be stated in the report. Two tests shall be made,

and it is recommended that the two samples are not tested concurrently.

5. Procedure

(a) The sample shall be screened on a 10mm IS sieve, thoroughly washed to remove fine particles of dust, and immersed in distilled water in the glass vessel; it shall remain immersed at a temperature of 22 to 32°C for 24 ± 1/2 hours. Soon after immersion and again at the end of the soaking period, air entrapped in or bubbles on the surface of the aggregate shall be removed by gentle agitation. This may be achieved by rapid clockwise and anti-clockwise rotation of the vessel between the operator's hands.

(b) The vessel shall be overfilled by adding distilled water and the plane ground-glass disc slid over the mouth so as to ensure that no air is trapped in the vessel.

(c) The aggregate shall be placed on the dry cloth shall be gently surface dried with the cloth, transferring it to a second dry cloth when the first will remove no further moisture. It shall then be spread out not more than one stone deep on the second cloth, and left exposed to the atmosphere away from direct sunlight or any other source of heat for not less than 10 minutes or until it appears to be completely surface dry (which with some aggregates may take an hour or more) The aggregate shall be turned over at least once during this period and a gentle current of unheated air may be used after the first ten minutes to accelerate the drying of difficult aggregates. The aggregate shall then be weighed (weight B).

(d) The aggregate shall be placed in the oven in the shallow tray, at a temperature of 100 to 110^0C for 24 ± 1/2 hours. It shall then be cooled in airtight container and weighed (weight C).

6. Calculations: Water absorption shall be calculated as follows:

Water absorption (percent of dry weight) = 100 (C-D)/D

7. Reporting of Result: The individual and mean results shall be reported. The size of the aggregate tested shall be stated.

(C) Method-III: Aggregate smaller than 10mm

3. Apparatus required

(a) Balance: Of capacity not less than 3 kg and accurate to 0.5 g, and of such a type as to permit the weighing of the vessel containing the aggregate and water.

(b) Oven: A well ventilated oven, thermostatically controlled, to maintain a temperature of 100 to 110^0C.

(c) Vessel: Any form of vessel capable of holding 0.5 to 1 kg of material up to 10 mm in size and capable of being filled with water to a constant volume with an accuracy of ± 0.5 ml. Either of the two following vessels is suitable:

(i) A glass vessel, referred to later as a pycnometer, of about one liter capacity having a metal conical screw top with a 6-mm diameter hole at its apex. The screw top shall be watertight when it is screwed on to the jar, and, if necessary, a rubber or fibre washer shall be inserted in the joint. If such a washer is used, a mark shall be made on the jar to correspond with a mark on the screw top so that the screw is tightened to the same position every time and the volume contained by the jar is constant throughout the test. A suitable vessel can be made from a 1kg fruit preserving jar in which the glass lid normally used is replaced by a sheet metal cone as shown in Fig. 2.2.1; or

(ii) A wide-mouthed glass vessel, such as a gas jar, of about 1.25 litres capacity, with a flat ground lip and a plane ground disc of plate glass to cover it giving a virtually watertight fit.

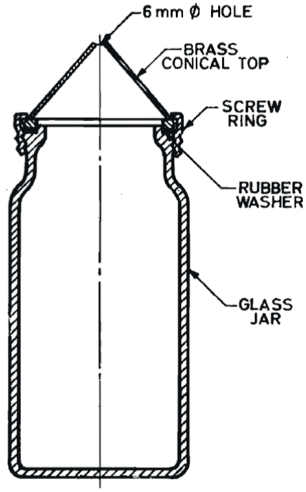


Fig. 2.2.1: Pycnometer

(d) A means of supplying a current of warm air, such as a hair drier.

(e) A tray of area not less than 325 cm^2.

(f) An airtight container large enough to take the sample.

(g) Filter papers and funnel.

4. Procedure

(a) A sample of about 1 kg for 10mm to 4.75 mm or 500 g if finer than 4.75 mm, shall be placed in the tray and covered with distilled water at a temperature of 22 to 32°C. The air entrapped in or bubbles on the surface of the aggregate shall be removed by gentle agitation with a rod. The sample shall remain immersed for 24 ± 1/2 hours.

(b) The water shall then be carefully drained from the sample, by decantation through a filter paper, any material retained being returned to the sample. The aggregate including any solid matter retained on the filter paper shall be exposed to a gentle current of warm air to evaporate surface moisture and shall

be stirred at frequent intervals to ensure uniform drying until no free surface moisture can be seen and the material just attains a 'free-running' condition. Care shall be taken to ensure that this stage is not passed. The saturated and surface-dry sample shall be weighed (weight A).

(c) The water shall then be carefully drained from the sample by decantation through a filter paper and any material retained returned to the sample. The sample shall be placed in the oven in the tray at a temperature of 100 to 110°C for 24 ± 1/2 hours, during which period it shall be stirred occasionally to facilitate drying. It shall be cooled in the air-tight container and weighed (weight D).

(d) Two tests shall be made.

5. Calculations: Water absorption shall be calculated as follows:

Water absorption (percent of dry weight) = 100 (A-D)/D

6. Reporting of Result: The individual and mean results shall be reported and the grading of the aggregate shall be stated.

2.3 Aggregate Abrasion Value

1. Introduction: The Los Angeles Abrasion Machine is used to determine the Aggregate Abrasion Value (AAV) by testing the measure of the resistance of aggregates to surface wear by abrasion.

As per IS:383, the Abrasion Value for the Aggregate to be used for concrete for wearing surfaces (such as runways, roads, pavements, tunnel lining carrying water, spillways and silting basins) shall be 30 percent maximum. For aggregates to be used in concrete other than wearing surfaces, the Abrasion Value shall be 50 percent maximum.

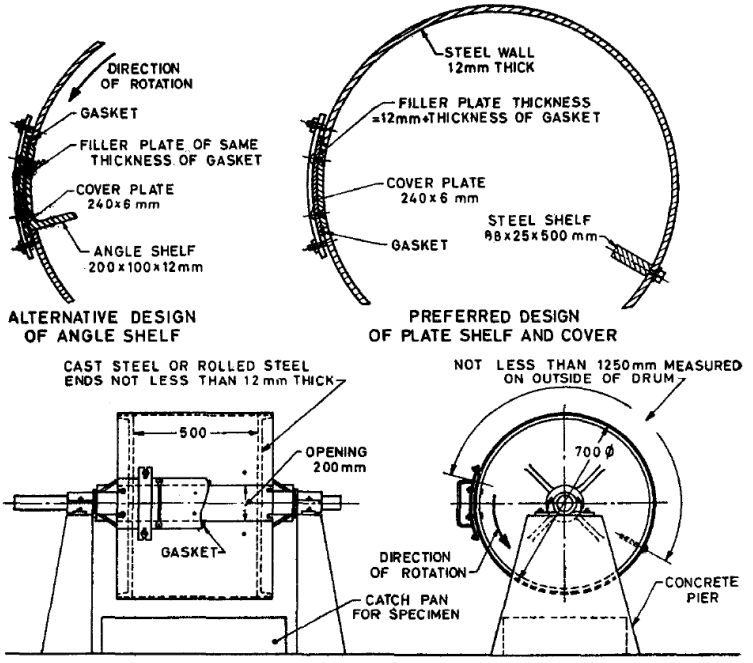
2. Reference: IS-2386(Part-IV):1963 (Reaffirmed-2021) "Method of Tests for Aggregates for Concrete. Part-IV: Mechanical Properties".

3. Apparatus required

3.1 Los Angeles Machine: The machine shall consist of a hollow steel cylinder, closed at both ends, having an inside diameter of 700 mm and an inside length of 500 mm. The cylinder shall be mounted on stub shafts attached to the ends of the cylinders but not entering it, and shall be mounted in such a manner that it may be rotated about its axis in a horizontal position. An opening in the cylinder shall be provided for the introduction of the test sample. The opening shall be closed dust-tight with a removable cover bolted in place. The cover shall be so designed as to maintain the cylindrical contour of the interior surface unless the shelf is so located that the charge will not fall on the cover, or come in contact with it during the test. A removable steel shelf, projecting radially 88 mm into the cylinder and extending its full length, shall be mounted along one element of the interior surface of the cylinder. The shelf shall be of such thickness and so mounted, by bolts or other approved means, as to be firm and rigid. The position of the shelf shall be such that the distance from the shelf to the opening,

measured along the circumference of the cylinder in the direction of rotation, shall be not less than 1250 mm (Fig. 2.3.1 and 2.3.2)

3.2 Abrasive Charge: The abrasive charge shall consist of cast iron spheres or steel spheres approximately 48 mm in diameter and each weighing between 390 and 445 g (Fig. 2.3.3). The abrasive charge, depending upon the grading of the sample as described in Table 2.6 shall be as given in Table 2.5.



NOTE 1 — Shaft bearing will be mounted on concrete piers or other rigid supports.

NOTE 2 — Suggested horse power for motor is not less than one.

Fig. 2.3.1: Los Angeles Abrasion Testing Machine





Fig. 2.3.2 **Fig. 2.3.3**

Table 2.5: Weight of Charge

Grading	Number of Spheres	Weight of Charge (g)
A	12	5000 ± 25
B	11	4584 ± 25
C	8	3300 ± 25
D	6	2500 ± 25
E	12	5000 ± 25
F	12	5000 ± 25
G	13	5000 ± 25

4. Sample: The test sample shall consist of clean aggregate which has been dried in an oven at 105 to 110°C to substantially constant weight and shall conform to one of the gradings shown in Table 2.6. The grading used shall be those most nearly representing the aggregate furnished for the work.

Table 2.6: Grading of Test Samples

Sieve Size (Square Hole)		Weight in g of Test Sample for Grade						
Passing (mm)	Retained on (mm)	A	B	C	D	E	F	G
80	63					2500*		
63	50					2500*		
50	40					3000*	5000*	
40	25	1250					5000*	5000*
25	20	1250						5000*
20	12.5	1250	2500					
12.5	10	1250	2500					
10	6.3			2500				
6.3	4.75			2500				
4.75	2.36				5000			
* Tolerance of ± 2% permitted								

5. Procedure

(a) The test sample and the abrasive charge shall be placed in the testing machine and the machine rotated at a speed of 20 to 33 rev/min. For gradings A, B, C and D, the machine shall be rotated for 500 revolutions; for gradings E, F and G, it shall be rotated for 1000 revolutions. At the completion of the test, the material shall be discharged from the machine and a preliminary separation of the sample made on a sieve coarser than the l.70 mm IS Sieve. The finer portion shall then be sieved on a 1.70-mm IS Sieve

(b) The material coarser than the 1.70mm IS Sieve shall be washed dried in an oven at 105 to 110°C to a substantially constant weight, and accurately weighed to the nearest gram.

6. Reporting of Result: The difference between the original weight and the final weight of the test sample shall be expressed as a percentage of the original weight of the test sample. This value shall be reported as the percentage of wear.

2.4 Aggregate Impact Value

1. Introduction: The aggregate impact value is a measure of resistance to sudden impact or shock, which may differ from its resistance to gradually applied compressive load. Thus, it indicates the toughness property of the ballast. This test can be used as alternate to Aggregate Crushing Value test.

As per IS:383, the Impact Value for the Aggregate to be used for concrete for wearing surfaces (such as runways, roads, pavements, tunnel lining carrying water, spillways and silting basins) shall be 30 percent maximum. For aggregates to be used in concrete other than wearing surfaces, the Crushing Value shall be 45 percent maximum.

2. Reference: IS-2386(Part-IV):1963 (Reaffirmed-2021) "Method of Tests for Aggregates for Concrete. Part-IV: Mechanical Properties".

3. Apparatus required

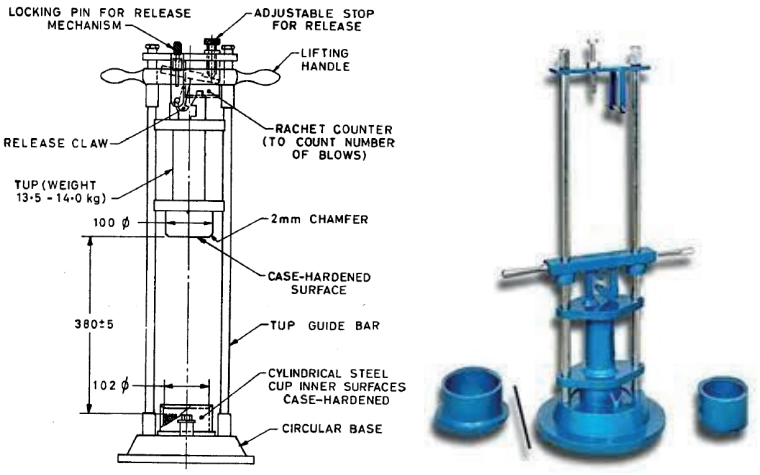


Fig. 2.4.1: Aggregate Impact Testing Machine

3.1 Aggregate Impact Testing Machine (Fig. 2.4.1) complying with the following:

(1) Total weight between 45 kg and 60 kg.

(2) Having a metal base weighing between 22 and 30kg with a plane lower surface of not less than 30m diameter, and supported on a level, plane and firm floor at least 45 cm thick. The machine shall be prevented from rocking either by fixing it to the floor or by supporting it on a level and plane metal plate cast into the surface of the floor.

(3) A cylindrical steel cup of internal diameter 102mm, internal depth 50mm and thickness not less than 6.3mm, with its inner surface case hardened, fastened at the centre of the base and easily removed for emptying.

(4) A metal hammer weighing 13.5 to 14.0kg, the lower end of which shall be cylindrical in shape, 100mm in diameter and 5cm long, with a 2mm chamfer at the lower edge, and case-hardened. It shall slide freely between vertical guides and be concentric with the cup.

(5) Means for raising the hammer and allowing it to fall freely between the vertical guides from a height of 380 ± 5.0mm on to the test sample in the cup, and means for adjusting the height of fall within 5mm.

NOTE: Some means for automatically recording the number of blows is desirable.

3.2 Sieves: The IS Sieves of sizes 12.5, 10 and 2.36mm.

3.3 Measure: A cylindrical metal measure, tared to the nearest gram, of sufficient rigidity to retain its form under rough usage, and of internal diameter 75mm and internal Depth 50mm.

3.4 Tamping Rod: A straight metal tamping rod of circular cross-section, 10mm in diameter and 230mm long, rounded at one end.

3.5 Balance: A balance of capacity not less than 500g, readable and accurate to 0.1 g.

3.6 Oven: A well-ventilated oven, thermostatically controlled to maintain a temperature of 100 to 110°C.

4. Preparation of Test Sample

4.1 The test sample shall consist of aggregate passing 12.5mm IS Sieve and retained on 10mm IS Sieve. It shall be dried in oven for four hours at a temperature of 100 to 110°C and cooled.

4.2 The measure shall be filled about one-third full with the aggregate and tamped with 25 strokes of the rounded end of the tamping rod. Further similar quantity of aggregate shall be added and a further tamping of 25 strokes given. The measure shall finally be filled to overflowing, tamped 25 times and the surplus aggregate struck off, using the tamping rod as a straight-edge. The net weight of aggregate in the measure shall be determined to the nearest gram (Weight A).

5. Test Procedure

5.1 The impact machine shall rest without wedging or packing upon the level plate, block or floor; so that it is rigid and the hammer guide columns are vertical.

5.2 The cup shall be fixed firmly in position on the base of the machine with the whole of the test sample placed in it.

5.3 The hammer shall be raised until its lower face is 380mm above the upper surface of the aggregate in the cup, and allowed to fall freely on to the aggregate. The test sample shall be subjected to a total of 15 such blows each being delivered at an interval of not less than one second.

5.4 The crushed aggregate shall then be removed from the cup and the whole of it sieved on the 2.36mm IS Sieve until no further significant amount passes in one minute. The fraction passing the sieve shall

be weighed to an accuracy of 0.1g (Weight B). The fraction retained on the sieve shall also be weighed (Weight C) and, if the total weight (B+C) is less than the initial weight (Weight A) by more than one gram, the result shall be discarded and a fresh test made. Two tests shall be made.

6. Calculations: The ratio of the weight of fines formed to the total sample weight in each test shall be expressed, as a percentage, the result being recorded to the first decimal place:

Aggregate impact value = (B / A) X 100

Where:

B = Weight of fraction passing 2.36 mm IS Sieve, and

A = Weight of oven-dried sample

7. Reporting of Result: The mean of the two results shall be reported to the nearest whole number as the aggregate impact value of the tested material.

2.5 Aggregate Crushing Value

1. Introduction: Aggregate Crushing value of aggregates indicates its strength and it is a numerical index of the strength of the coarse aggregate used in concreting, construction of roads, railway ballast and pavements etc.

As per IS:383, the Crushing Value of the Aggregate to be used for concrete for wearing surfaces (such as runways, roads, pavements, tunnel lining carrying water, spillways and silting basins) shall be 30 percent maximum. For aggregates to be used in concrete other than wearing surfaces, in case the Crushing Value of the Aggregate to be used exceeds 30 percent, then the test for "ten percent fines" should be conducted and the minimum test load for the ten percent fines should be 50 kN.

2. Reference: IS-2386(Part-IV):1963 (Reaffirmed-2021) "Methods of Test for Aggregates for Concrete. Part-IV: Mechanical Properties".

3. Apparatus required

3.1 A 15cm diameter open-ended steel cylinder, with plunger and base-plate, of the general form and dimensions shown in Fig. 2.5.1 and Table 2.7. The surfaces in contact with the aggregate shall be machined and case hardened or otherwise treated so as to have a diamond pyramid hardness number (VH) of not less than 650 VH.

3.2 A straight metal tamping rod of circular cross-section 16mm in diameter and 45 to 60cm long, rounded at one end.

3.3 A balance of capacity 3kg, readable and accurate to one gram.

3.4 IS Sieves of sizes 12.5, 10 and 2.36mm.

3.5 A compression testing machine capable of applying a load of 46 tonnes and to give a uniform rate of loading so that the maximum load is reached in 10 minutes. The machine may be used with or without a spherical seating.

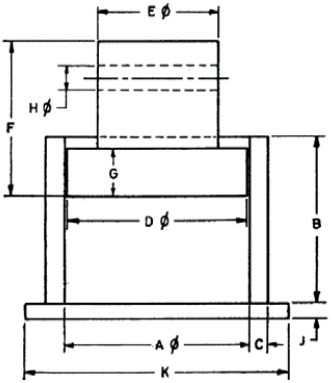




Fig. 2.5.1: Aggregate Crushing Value Test Cylinder

Table 2.7: Principal Dimensions of Test Cylinder

Letter	Dimension for	150mm Cylinder	75mm Cylinder
	Cylinder	mm	mm
A	Internal Diameter	152.0 ± 0.5	77 ± 0.5
B	Height	130 to 140	70 to 80≥
C	Wall Thickness	≥ 16	≥ 8
	Plunger		
D	Diameter of Piston	150 ± 0.5	75 ± 0.5
E	Diameter of Stem	100 to 150	50 to 75
F	Height	100 to 115	65 to 75
G	Depth of Piston	≥ 25	≥ 20
H	Diameter of Hole (nominal)	20	10
	Base Plate		
J	Thickness (nominal)	6.3	6.3
K	Side length of Square	200 to 230	110 to 115

3.6 Cylindrical metal measure of sufficient rigidity to retain its form under rough usage and of internal

diameter 11.5cm and internal height 18.0cm.

4. Preparation of Test Sample: The material for the standard test shall consist of aggregate passing 12.5mm IS Sieve and retained on 10mm IS Sieve. For other sizes, the material shall be separated on the appropriate sieves given in Table 2.8.

4.1 The aggregate shall be tested in a surface-dry condition. If dried by heating, the period of drying shall not exceed four hours, the temperature shall be 100 to 110°C and the aggregate shall be cooled to room temperature before testing.

4.2 The quantity of aggregate shall be such that the depth of material in the cylinder, after tamping, shall be 10 cm.

Table 2.8: For non-standard Size of Aggregates

Nominal Sizes (IS Sieves)		Diameter of Cylinder to be used (cm)	Size of IS Sieve for separating fines
Passing through	Retained on		
mm	mm		
25	20	15.0	4.75 mm
20	12.5	15.0	3.35 mm
10	6.3	15.0 or 7.5	1.70 mm
6.3	4.75	15.0 or 7.5	1.18 mm
4.75	3.35	15.0 or 7.5	850 microns
3.35	2.36	15.0 or 7.5	600 microns
Note: About 6.5 kg of natural aggregate is required to provide the two test samples for the 15 cm cylinder, or about 1 kg for 7.5 cm cylinder.			

4.3 The cylindrical measure may be filled in three layers of approximately equal depth, each layer being tamped 25 times with the rounded end of the tamping rod and finally levelled off, using the tamping rod as a straight-edge.

4.4 The weight of material comprising the test sample shall be determined (Weight A) and the same weight of sample shall be taken for the repeat test.

5. Test Procedure: The cylinder of the test apparatus shall be put in position on the base-plate and the test sample added in thirds, each third being subjected to 25 strokes from the tamping rod. The surface of the aggregate shall be carefully levelled and the plunger inserted so that it rests horizontally on this surface, care being taken to ensure that the plunger does not jam in the cylinder.

5.1 The apparatus, with the test sample and plunger in position, shall then be placed between the platens of the testing machine and loaded at as uniform a rate as possible so that the total load is reached in 10 minutes. The total load shall be 40 tonnes.

5.2 The load shall be released and the whole of the material removed from the cylinder and sieved on a 2.36mm IS Sieve for the standard test, or the appropriate sieve given in Table 2.8. The fraction passing the sieve shall be weighed (Weight B). In all of these operations, care shall be taken to avoid loss of the fines. Two tests shall be made.

6. Calculations: The ratio of the weight of fines formed to the total sample weight in each test shall be expressed, as a percentage, the result being recorded to the first decimal place:

Aggregate crushing value = (B / A) x 100

Where:

B = Weight of fraction passing the appropriate sieve, and

A = Weight of surface-dry sample.

7. Reporting of Results: The mean of the two results shall be reported to the nearest whole number as the "aggregate crushing value" of the size of material tested, which shall be stated.

Note-1: Aggregate larger than 12.5mm - In general, the

larger sizes of aggregate will give a higher aggregate crushing value, but the relationship between the values obtained with different sizes will vary from one aggregate to another. Particular care shall be taken with larger sizes of aggregate to ensure that the plunger does not jam in the cylinder. However, for such aggregate, a 7.5cm diameter cylinder may be used, and this has been found to give slightly higher results than the standard cylinder, so that the errors are compensating.

Note-2: Aggregate smaller than 10mm - In general, the smaller sizes of aggregate will give a lower aggregate crushing value, but the relationship between the values obtained with different sizes will vary from one aggregate to another.

Note-3: For testing aggregate smaller than 10 mm

(a) The form and dimensions of the 7.5cm cylinder shall be as shown in Fig. 2.5.1 and Table 2.7, and the surfaces shall be as for the standard cylinder.

(b) The tamping rod shall be 8mm in diameter and 30cm long, rounded at one end.

(c) The balance shall be of capacity 500g, readable and accurate to 0.2g.

(d) The IS Sieves shall be as given in Table 2.8.

(e) The compression testing machine shall be capable of applying a load of 10 tonnes uniformly in 10 minutes.

(f) The metal measure shall be 6cm in diameter and 9cm in height.

(g) The depth of material in the 7.5cm cylinder shall be 5cm after tamping.

(h) The total load applied in 10 minutes shall be 10 tonnes.

2.6 Silt Content

Silt is granular material of a size between sand and clay, with mineral origin of mainly quartz and feldspar. Excessive quantity of silt, with fine aggregates in concrete, not only reduces the bonding of cement and fine aggregates but also affects the strength and durability of concrete adversely.

1. Introduction: This method covers the procedure for determination of Clay, Fine Silt and Fine Dust in fine aggregates, by using Sedimentation Method, which is a Gravimetric Method. Gravimetric Method is a method of quantitative chemical analysis in which the constituent sought is converted into a substance (of known composition) that can be separated from the sample and weighed. The steps commonly followed in gravimetric analysis are (1) Preparation of a solution containing a known weight of the sample, (2) Separation of the desired constituent, (3) Weighing the isolated constituent, and (4) Computation of the amount of the particular constituent in the sample from the observed weight of the isolated substance.

2. Reference: IS-2386 (Part-II) : 1963 (Reaffirmed-2021) "Method of Tests for Aggregates for Concrete. Part-II: Estimation of Deleterious Materials and Organic Impurities".

3. Apparatus required

3.1 A watertight screw-topped glass jar of dimensions similar to a 1kg fruit preserving jar.

3.2 A device for rotating the jar about its long axis, with this axis horizontal, at a speed of 80±20 rev/min.

3.3 A sedimentation pipette of the Andreason type of approximately 25ml capacity and of the general form indicated in Fig. 2.6.1. This consists mainly of a pipette fitted at the top with a two-way tap and held

rigidly in a clamp which can be raised or lowered as required, and which is fitted with a scale from which the changes in height of the pipette can be read.

The volume of the pipette A, including the connecting bore of the tap B, is determined by filling with distilled water; by reversing the tap, the water is run out into a bottle, weighed and the volume calculated.

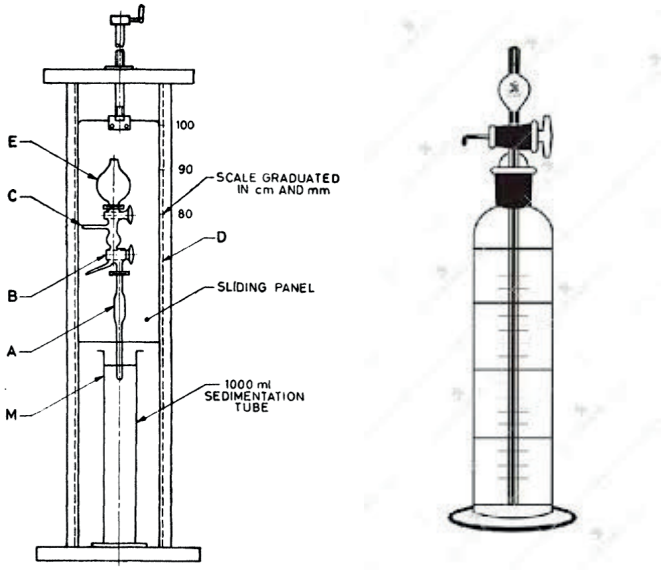


Fig. 2.7.1: Andreason type Pipette

3.4 A 1000 ml measuring cylinder.

3.5 A scale or balance of capacity not less than 10kg, readable and accurate to one gram.

3.6 A scale or balance of capacity not less than 250g, readable and accurate to 0.001g.

3.7 A well-ventilated oven, thermostatically controlled, to maintain a temperature of 100 to 110°C.

3.8 Chemicals: A solution containing 8g of sodium oxalate per litre of distilled water shall be taken. For use, this stock solution is diluted with distilled water

to one tenth (that is 100ml diluted with distilled water to one litre).

4. Test Sample: The sample for test shall be prepared from the main sample taking particular care that the test sample contains a correct proportion of the finer material. The amount of sample taken for test shall be in accordance with Table 2.9.

Table 2.9: Weight of Sample

Maximum Size present in substantial proportion	Approximate Weight of Sample for Test
(mm)	(kg)
63 to 25	6
20 to 12.5	1
10 to 6.3	0.5
4.75 or smaller	0.3

All-in aggregates shall be separated into fine and coarse fractions by sieving on a 4.75mm IS Sieve and the two samples so obtained shall be tested separately.

5. Test Procedure

5.1 Method for Fine Aggregate: Approximately 300g of the sample in the air-dry condition, passing the 4.75mm IS Sieve, shall be weighed and placed in the screw-topped glass jar, together with 300ml of the diluted sodium oxalate solution. The Tubber washer and cap shall be fixed, care being taken to ensure watertightness. The jar shall then be rotated about its long axis, with this axis horizontal, at a speed of 80±20 rev/min for a period of 15 minutes.

5.1.1 At the end of 15 minutes, the suspension shall be poured into the 1000 ml measuring cylinder and the residue washed by gentle swirling and decantation of successive 150 ml

portions of sodium oxalate solution, the washings being added to the cylinder until the volume is made up to 1000 ml. The determination shall be completed as described in Para 5.3.

5.2 Method for Coarse Aggregate: The weighed sample shall be placed in a suitable container, covered with a measured volume of sodium oxalate solution (0.8 g per litre), agitated vigorously to remove all adherent fine material and the liquid suspension transferred to the 1000 ml measuring cylinder. This process shall be repeated as necessary until all clayey material has been transferred to the cylinder. The volume shall be made up to 1000 ml with sodium oxalate solution and the determination completed as described in Para 5.3.

5.3 The suspension in the measuring cylinder shall be thoroughly mixed by inversion and the tube and contents immediately placed in position under the pipette. The pipette A shall then be gently lowered until the tip touches the surface of the liquid, and then lowered a further 10cm into the liquid. Three minutes after placing the tube in position, the pipette A and the bore of tap B shall be filled by applying gentle suction at C. A small surplus may be drawn up into the bulb between tap B and tube C, but this shall be allowed to run away and any solid matter shall be washed out with distilled water from E. The pipette shall then be removed from the measuring cylinder and its contents run into a weighed container, any adherent solids being washed into the container by distilled water from E through the tap B.

The contents of the container shall be dried at 100 to 110°C to constant weight, cooled and weighed.

6. Calculations: The proportion of fine silt and clay or fine dust shall then be calculated from the following formula:

Percentage of clay and fine silt or fine dust =

$$\frac{100}{W_1}\left(\frac{1000W_2}{V}\right) - 0.8$$

Where:

W1 = Weight in g of the original sample,

W2 = Weight in g of the dried residue,

V = Volume in ml of the pipette, and

0.8 = Weight in g of sodium oxalate in one litre of the diluted solution.

7. Reporting of Result: The clay, fine silt and fine dust content shall be reported to the nearest 0.1 percent.

2.7 Bulking of Sand

The free moisture content in the fine aggregate or sand results in bulking of its volume. The phenomenon of increase in sand volume due to the addition of water or increase of moisture content is termed as Bulking of Sand. Bulking of Sand depends on the size of the particles in the sand and also on the quantity of moisture content in sand. The volume of sand can increase from 20% to 40% due to an increase of 5 to 8% of moisture content. When concrete mix is designed, bulking of sand needs to be considered. If this is not done, the concrete designed will have an insufficient amount of sand resulting in a harsh mix.

1. Introduction: This method covers the the field method for determining the necessary adjustment for the bulking of fine aggregates.

2. Reference: IS-2386(Part-III):1963 (Reaffirmed-2021) "Method of Tests for Aggregates for Concrete. Part-III: Specific Gravity, Density, Voids, Absorption and Bulking".

3. Test Procedure

3.1 The procedure to be adopted may be varied, but two methods are suggested in Para 3.2 and Para 3.3. Both depend on the fact that the volume of inundated sand is the same as if the sand were dry.

3.2 Put sufficient quantity of the sand loosely into a container. Until it is about two-thirds full. Level off the top of the sand and pushing a steel rule vertically down through the sand at the middle to the bottom, measure the height. Suppose this is h cm.

3.2.1 Empty the sand out of the container into another container where none of it will be lost. Half fill the first container with water. Put back about half the sand and rod it with a steel rod, about 6mm in diameter, so that its volume is

reduced to a minimum. Then add the remainder of the sand and rod it in the same way. Smooth and level the top surface of the inundated sand and measure its depth at the middle with the steel rule. Suppose this is h' cm.

3.2.2 The percentage of bulking of the sand due to moisture shall be calculated from the formula:

$$\text{Percentage bulking} = \left(\frac{h}{h'}-1\right) \text{x } 100$$

3.3 In a 250ml measuring cylinder, pour the damp sand (consolidated by shaking) until it reaches the 200ml mark. Then fill the cylinder with water and stir the sand well. The water shall be sufficient to submerge the sand completely. It will be seen that the sand surface is now below its original level. Suppose the surface is at the mark y ml.

The percentage of bulking of the sand due to moisture shall be calculated from the formula:

$$\text{Percentage bulking} = \left(\frac{200}{y}-1\right) \text{x } 100$$

4. Reporting of Result: Report the percentage bulking of the sand to the nearest whole number.

2.8 Flakiness Index and Elongation Index

"Flakiness Index" of the coarse aggregates is the percentage by weight of particles in it, whose least dimension (i.e. thickness) is less than three-fifths of its mean dimension. "Elongation Index" of the coarse aggregates is the percentage by weight of particles in it, whose largest dimension (i.e. length) is greater than one and four-fifths times its mean dimension. Flaky and elongated aggregates may have adverse effects on concrete and bituminous mix. For instance, flaky and elongated particles tend to lower the workability of concrete mix which may impair the long-term durability. For bituminous mix, flaky particles are liable to break up and disintegrate during the pavement rolling process.

Flakiness and Elongation Index should be determined using same sample. After carrying out the Flakiness Index test, the flaky material shall be removed from the sample and the remaining material shall be used for Elongation Index test. As per IS:3838, the combined Flakiness and Elongation Index shall not exceed 40 percent; but the Engineer in-charge may relax this limit keeping in view the requirement & availability of aggregates and performance based on test on concrete.

1. Introduction: This method covers the procedure for determination of Flakiness Index and Elongation Index of coarse aggregates.

2. Reference: IS-2386(Part-I):1963 (Reaffirmed- 2021) "Method of Tests for Aggregates for Concrete. Part-I: Particle Size and Shape".

(A) <u>Determination of Flakiness Index</u>

3. Apparatus required

3.1 <u>Balance:</u> The balance shall be of sufficient capacity and sensitivity and shall have an accuracy of 0.1 percent of the weight of the test sample.

3.2 Metal Gauge: The metal gauge (called as Flakiness Index gauge or Thickness gauge also) shall be of the pattern shown in Fig. 2.8.1 and Fig. 2.8.2.

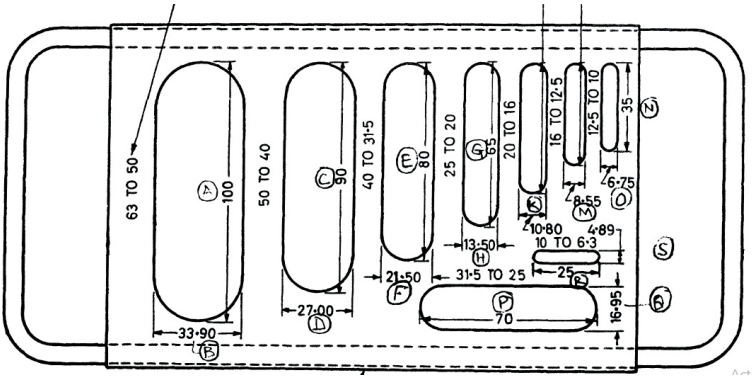


Fig. 2.8.1: Flakiness Index Gauge

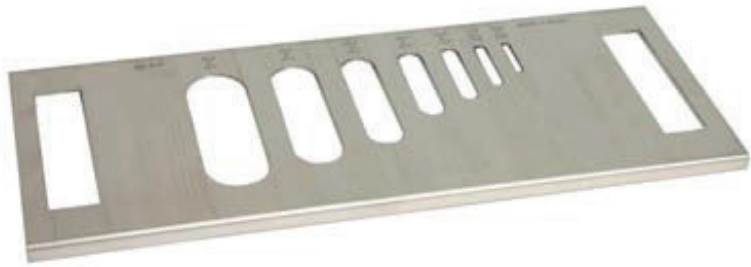


Fig. 2.8.2: Flakiness Index Gauge

3.3 Sieves: IS Sieves of sizes shown in Table 2.10.

Table 2.10: Dimension of Thickness and Length Gauges

Size of Aggregates		Thickness Gauge (*) (mm)	Length Gauge ($) (mm)
Passing through IS Sieve	Retained on IS Sieve		
63 mm	50 mm	33.90	-
50 mm	40 mm	27.00	81.0
40 mm	25 mm	19.50	58.5
31.5 mm	25 mm	16.95	-
25 mm	20 mm	13.50	40.5
20 mm	16 mm	10.80	32.4
16 mm	12.5 mm	8.55	25.6
12.5 mm	10 mm	6.75	20.2
10 mm	6.3 mm	4.89	14.7
(*) This dimension is equal to 0.60 times the mean sieve size. ($) This dimension is equal to 1.80 times the mean sieve size.			

4. Sample: A quantity of aggregate shall be taken sufficient to provide the minimum number of 200 pieces of any fraction to be tested.

5. Procedure:

5.1 Sieving: The sample shall be with the sieves specified in Table 2.10.

5.2 Separation of Flaky Material: Each fraction shall be gauged in turn for thickness on a metal gauge of the pattern shown in Fig. 2.8.1 and Fig. 2.8.2 or in bulk on sieves having elongated slots. The width of the slot used in the gauge or sieve shall be of the dimensions specified in Col.3 of Table 2.10 for the appropriate size of material.

5.3 Weighing of Flaky Material: The total amount passing the gauge shall be weighed to an accuracy of at least 0.1 percent of the weight of the test sample.

6. Reporting of Result: The flakiness index is the total weight of the material passing the various thickness gauges or sieves, expressed as a percentage of the total weight of the sample gauged.

(B) Determination of Elongation Index

3. Apparatus required

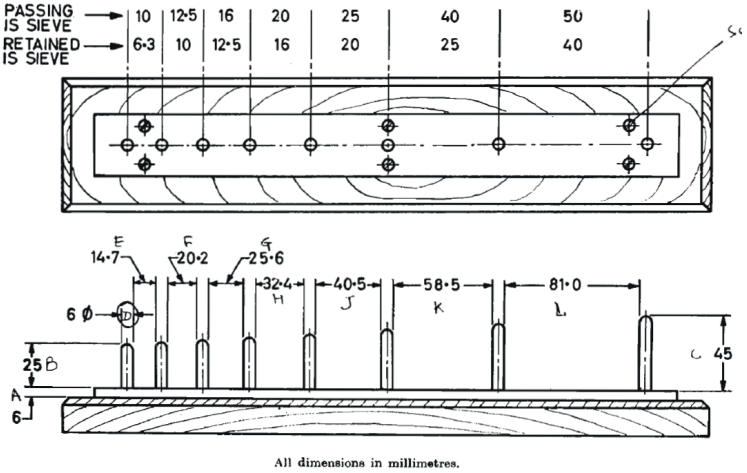


Fig. 2.8.3: Elongation Index Gauge

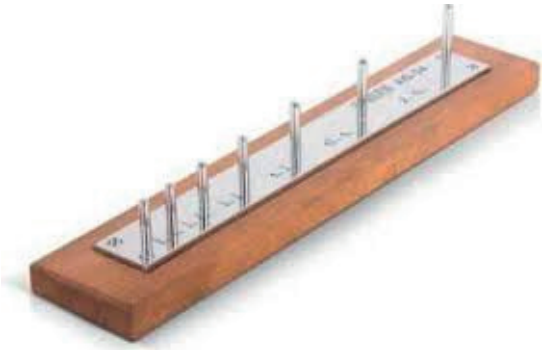


Fig. 2.8.4: Elongation Index Gauge

3.1 Balance: The balance shall be of sufficient capacity and sensitivity and shall have an accuracy of 0.1 percent of the weight of the test sample.

3.2 Metal Gauge: The metal gauge (called as Elongation Index gauge or Length gauge also) shall be of the pattern shown in Fig. 2.8.3 and Fig. 2.8.4.

3.3 Sieves: IS Sieves of sizes shown in Table 2.10.

4. Sample: A quantity of aggregate shall be taken sufficient to provide the minimum number of 200 pieces of any fraction to be tested.

5. Procedure:

5.1 Sieving: The sample shall be with the sieves specified in Table 2.10.

5.2 Separation of Elongated Material: Each fraction shall be gauged individually for length on a metal length gauge of the pattern shown in Fig. 2.8.3 and Fig. 2.8.4. The gauge length used shall be that specified in Col. 4 of Table 2.10 for the appropriate size of material.

5.3 Weighing of Elongated Material: The total amount retained by the length gauge shall be weighed to an accuracy of at least 0.1 percent of the weight of the test sample.

6. Reporting of Result: The elongation index is the total weight of the material passing the various length gauges, expressed as a percentage of the total weight of the sample gauged.

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Chapter - 3

TESTS ON REINFORCEMENT STEEL

The reinforcement shall be free from loose mill scales, loose rust and coats of paint, oil, mud or any other substances which may destroy or reduce bond. Sand blasting or other treatment is recommended to clean reinforcement.

The modulus of elasticity of steel shall be taken as 200 kN/mm^2. The characteristic yield strength of different steel shall be assumed as the minimum yield stress/0.2 percent proof stress specified in the relevant Indian Standard.

Following test are typically conducted on the reinforcement steel:

(3.1) Tensile Test

(3.2) Bend Test

(3.3) Re-bend Test

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3.1 Tensile Test

1. Introduction: The tension test subjects a specimen of the material under examination to a measured load sufficient to cause rupture. This chapter describes the method for tensile testing of metallic materials and defines the mechanical properties which can be determined at room temperature.

2. Reference: IS-1608 (Part 1): 2022 "Metallic Materials- Tensile Testing, Part-1: Method of Test at Room Temperature".

3. Definitions

3.1 Gauge length (L): length of the parallel portion of the test piece on which elongation is measured at any moment during the test.

3.1.1 Original gauge length (Lo): Length between gauge length marks on the test piece measured at room temperature before the test.

3.1.2 Final gauge length after fracture (Lu): Length between gauge length marks on the test piece measured after rupture, at room temperature, the two pieces having been carefully fitted back together so that their axes lie in a straight line.

3.2 Parallel length (Lc): Length of parallel reduced section of the test piece. The concept of parallel length is replaced by the concept of distance between grips for non-machined test pieces.

3.3 Elongation: Increase in the original gauge length (Lo) at any moment during the test.

3.4 Percentage elongation: Elongation expressed as a percentage of the original gauge length (Lo).

3.4.1 Percentage permanent elongation: Increase in the original gauge length of a test piece after removal of a specified stress, expressed as a percentage of the original gauge length.

3.4.2 Percentage elongation after fracture (A): Permanent elongation of the gauge length after fracture (Lu - Lo), expressed as a percentage of the original gauge length.

3.5 Extensometer gauge length (Le): Initial gauge length of the extensometer used for measurement of extension.

3.6 Extension: Increase in the extensometer gauge length (Le) at any moment during the test.

3.6.1 Percentage extension strain (e): Extension expressed as a percentage of the extensometer gauge length.

3.6.2 Percentage permanent extension: Increase in the extensometer gauge length, after removal of a specified stress from the test piece, expressed as a percentage of the extensometer gauge length

3.6.3 Percentage total extension at maximum force (Agt): Total extension (elastic extension plus plastic extension) at maximum force, expressed as a percentage of the extensometer gauge length.

3.6.4 Percentage plastic extension at maximum force (Ag): Plastic extension at maximum force, expressed as a percentage of the extensometer gauge length.

3.6.5 Percentage total extension at fracture (At): Total extension (elastic extension plus plastic extension) at the moment of fracture, expressed as a percentage of the extensometer gauge length.

3.7 Testing rate: Rate used during the test.

3.7.1 Strain rate (eLe): Increase of strain, measured with an extensometer, in extensometer gauge length, per time.

3.7.2 Estimated strain rate over the parallel

length (eLc): Value of the increase of strain over the parallel length of the test piece per time based on the crosshead separation rate and the parallel length of the test piece.

3.7.3 Stress rate (R): Increase of stress per time

3.8 Percentage reduction of area (Z): Maximum change in cross-sectional area which has occurred during the test (S_o - S_u), expressed as a percentage of the original cross sectional area So :

$$Z = [(S_o - S_u) / S_o] \times 100$$

3.9 Maximum force (F_m): Highest force that the test piece withstands during the test.

3.10 Stress (R): At any moment during the test, force divided by the original cross-sectional; area (S_o) of the test piece.

3.10.1 Tensile strength (R_m): Stress corresponding to the maximum force.

3.10.2 Yield strength: When the metallic material exhibits a yield phenomenon, stress corresponding to the point reached during the test at which plastic deformation occurs without any increase in the force.

3.10.2.1 Upper yield strength (R_{eH}): Maximum value of stress prior to the first decrease in force.

3.10.2.2 Lower yield strength (R_{eL}): Lowest value of stress during plastic yielding, ignoring any initial transient effects.

3.10.3 Proof strength, plastic extension (R_p): Stress at which the plastic extension is equal to a specified percentage of the extensometer gauge length.

3.10.4 Proof strength, total extension (R_t): Stress at which total extension (elastic plus plastic extension) is equal to a specified percentage of the extensometer gauge length.

3.10.5 Permanent set strength (R_r): Stress at which, after removal of force, a specified permanent elongation or extension expressed respectively as a percentage of original gauge length or extensometer gauge length, has been exceeded.

3.11 Fracture: Phenomenon which is deemed to occur when total separation of the test piece occurs.

3.12 Modulus of elasticity (E): Quotient of change of stress (ΔR) and change of percentage extension (Δe) in the range of evaluation, multiplied by 100%.

$$E = (\Delta R / \Delta e) \times 100\ \%$$

3.13 Coefficient of determination (R^2): Additional result of the linear regression which describes the quality of stress-strain curve in the evaluation range.

3.14 Standard deviation of the slope (S_m): Additional result of the linear regression which describes the difference of the stress values from the best fit line for the given extension values in the evaluation range.

4. Symbols

Table 3.1: Symbols and Designation

Symbol	Unit	Designation
Test piece		
a_0, T^a	mm	Original thickness of flat test piece or wall thickness of a tube
b_0	mm	Original width of the parallel length of a flat test piece or average width of the longitudinal strip taken from a tube or width of flat wire
d_0	mm	Original diameter of the parallel length of a circular test piece, or diameter of round wire or internal diameter of a tube
D_0	mm	Original external diameter of a tuber

L_0	mm	Original gauge length
L_0'	mm	Initial gauge length for determination of A_{wn}
L_c	mm	Parallel length
L_e	mm	Extensometer gauge length
L_t	mm	Total length of test piece
L_u	mm	Final gauge length after fracture
L_u'	mm	Final gauge length after fracture for determination of A_{wn}
S_0	mm^2	Original cross-sectional area of the parallel length
S_u	mm^2	Minimum cross-sectional area after fracture
k	-	Coefficient of proportionality
Z	%	Percentage reduction of area
Elongation		
A	%	Percentage elongation after fracture
A_{wn}	%	Percentage elongation without necking
Extension		
e	%	Extension
A_e	%	Percentage yield point extension
A_g	%	Percentage plastic extension at maximum force F_m
A_{gt}	%	Percentage total extension at maximum force F_m
A_t	%	Percentage total extension at fracture
ΔL_m	mm	Extension at maximum force
ΔL_f	mm	Extension at fracture
Rates		
e_{Le}	s^{-1}	Strain rate

e_{Lc}	s^{-1}	Estimated strain rate over the parallel length
R	MPa $^{s-1}$	Stress rate
v_c	MPa s^{-1}	Crosshead separation rate
Force		
F_m	N	Maximum force
Yield strength – Proof strength – Tensile strength		
R	MPa	Stress
R_{eH}	MPa	Upper yield strength
R_{eL}	MPa	Lower yield strength
R_m	MPa	Tensile strength
R_p	MPa	Proof strength, plastic extension
R_r	MPa	Specified permanent set strength
R_t	MPa	Proof strength, total extension
Modulus of elasticity – slope of stress-percentage extension curve		
E	GPa	Modulus of elasticity
m	MPa	Slope of the stress-percentage extension curve at a given moment of the test
m_E	MPa	Slope of the elastic part of the stress-percentage extension curve
R_1	MPa	Lower stress value
R_2	MPa	Upper stress value
e_1	%	Lower strain value
e_2	%	Upper strain value
R^2	-	Coefficient of determination
S_m	MPa	Standard deviation of the slope
$S_{m(rel)}$	%	Relative standard deviation of the slope

5. Test pieces

5.1 Shape and dimension

5.1.1 General: The shape and dimensions of the test pieces may be constrained by the shape and dimensions of the metallic product from which the test pieces are taken. The cross-section of the test pieces may be circular, square, rectangular, annular or, in special cases, some other uniform cross-section.

Preferred test pieces have a direct relationship between the original gauge length (L0) and cross-sectional area (S0), expressed by the formula $L0 = k\sqrt{S0}$, where k is a coefficient of proportionality, and are called proportional test pieces. The internationally adopted value for k is 5.65. The original gauge length shall be not less than 15mm. When the cross-sectional area of the test piece is too small for this requirement to be me with, a higher value of k (preferably 11.3) or a non-proportional test piece may be used, wherein the original gauge length is independent of the original cross-sectional area.

The test piece is usually obtained by machining a sample from the product or a pressed blank or casting. However, products of uniform cross-section (sections, bars, wires, etc.) and also as-cast test pieces (i.e. for cast iron and non-ferrous alloys) may be tested without being machined.

5.1.2 Machined test pieces: Machined test pieces shall incorporate a transition radius between the gripped ends and the parallel length if these have different dimensions.

The gripped ends may be of any shape to suit the grips of the testing machine. The axis of the test piece shall coincide with the axis of application of the force.

The parallel length, Le, or, in the case where the

test piece has no transition radii, the free length between the grips, shall always be greater than the original gauge length, L_0.

5.1.3 Unmachined test pieces: If the test piece consists of an unmachined length of the product or of an unmachined test bar, the free length between the grips shall be sufficient for gauge marks to be at a reasonable distance from the grips.

As-cast test piece shall incorporate a transition radius between the gripped ends and the parallel length. The gripped ends may be of any shape to suit the grips of the testing machine provided that they enable the center of the test piece to coincide with the axis of application of force. The parallel length, Lc, shall always be greater than the original gauge length, L_0.

5.2 Types: The main types of test pieces are defined in the Annexes-B to E of the IS 1608 (Part- 1):2022, according to the shape and type of the product, as shown in Table 3.2.

Table 3.2: Main types of Test Pieces

Dimensions in mm

Type of product				**Corresponding Annex**
Sheets – Plates - Flats	**Wire – Bars - Sections**			
				
Thickness a	**Diameter or Side**			
0.1 ≤ a < 3	-			B
-	< 4			C
a ≥ 3	≥ 4			D
Tubes				E

6. Determination of original cross-sectional area: The relevant dimensions of the test piece should be measured at sufficient cross-sections perpendicular to the longitudinal axis in the central region of the parallel length of the test piece. A minimum of three cross-sections is recommended. The original cross-sectional area (S_0) is the average cross-sectional area.

7. Original gauge length and extensometer gauge length

7.1 Choice of the original gauge length: For proportional test pieces, if the original gauge length is not equivalent to $5.65\sqrt{S_0}$, where S_0 is the original cross-sectional area of the parallel length, the symbol A should be supplemented by a subscription indicating the coefficient of proportionality used (e.g. $A_{11.3}$ indicates a percentage elongation of the gauge length $L_0 = 11.3\sqrt{S_0}$).

For non-proportional test pieces, the symbol A should be supplemented by a subscript indicating the original gauge length used, expressed in mm (e.g. A_{80} indicates percentage elongation of a gauge length L_0 of 80mm).

7.2 Marking the original gauge length: For the manual determination of the elongation after fracture A, each end of the original gauge length, L_0, shall be marked to an accuracy of ±1 %.

For proportional test pieces, the calculated value of the original gauge length may be rounded to the nearest multiple of 5mm, provided that the difference between the calculated and marked gauge length is less than 10% of L_0.

If the parallel length, Lc, is much greater than the original gauge length, as for instance, with unmachined test pieces, a series of overlapping gauge lengths may be marked.

7.3 Choice of the extensometer gauge length: For measurement of yield and proof strength

parameters, Le should span as much of the parallel length of the test piece as possible. Ideally, as a minimum, Le should be greater than 0.50L0 but less than approximately 0.90Lc. This should ensure that the extensometer detects all yielding events that occur in the test piece. Further, for measurement of parameters "at" or "after reaching" maximum force, Le should be approximately" equal to L0.

8. Conditions of testing

8.1 Setting the force zero point: The force-measuring system shall be set to zero after the testing loading train has been assembled, but before the test piece is actually gripped at both ends. Once the force zero point has been set, the force measuring system shall not be changed in any way during the test.

8.2 Method of gripping: The test pieces shall be gripped by suitable means, such as wedges, screwed grips, parallel jaw faces, or shouldered holders. Every endeavour should be mad e to ensure that test pieces are held in such a way that the force is applied as axially as possible, in order to minimize bending. In order to ensure the alignment of the test piece and grip arrangement, a preliminary force may be applied provided it does not exceed a value corresponding to 5 % of the specified or expected yield strength. A correction of the extension should be carried out to take into account the effect of the preliminary force.

8.3 Testing rates

8.3.1 General information: Unless otherwise agreed, the choice of method (A1, A2 or B) and test rates are at the discretion of the producer or the test laboratory assigned by the producer, provided that these meet the requirements of this document.

The difference between Method A and Method B is that the necessary testing speed of Method A is defined at the point of interest (e.g. $R_{p0.2}$), where

the property has to be determined, whereas, in Method B, the necessary testing speed is set in the elastic range before the property (e.g. $R_{p0.2}$) has to be determined.

8.3.2 Testing rate based on strain rate (Method A)

8.3.2.1 General: Method A is intended to minimize the variation of the test rates during the moment when strain rate sensitive parameters are determined and to minimize the measurement uncertainty of the test results. Two different types of strain rate control are described in this subclause.

- Method A1 closed loop involves the control of the strain rate itself, e_{Le} that is based on the feedback obtained from an extensometer.
- Method A2 open loop involves the control of the estimated strain rate over the parallel length, e_{Lc}, which is achieved by using the crosshead separation rate calculated by multiplying the required strain rate by the parallel length.

The testing rate shall conform to the following requirements:

(a) Unless otherwise specified, any convenient speed of testing may be used up to a stress equivalent to half of the expected yield strength. Above this range and for the determination of R_{eH}, R_p or R_t, the specified strain rate, eLe (or for Method A2 the crosshead separation rate vc), shall be applied.

In this range, to eliminate the influence of the compliance of the tensile testing machine,

the use of an extensometer measuring the extension of the test piece is necessary to have accurate control over the strain rate. For testing machines unable to control by strain rate, method A2 may be used.

(b) During discontinuous yielding, the estimated strain rate over the parallel length, e_{Lc} should be applied. In this range, it is impossible to control the strain rate using the extensometer clamped on to the test piece because local yielding can occur outside the extensometer gauge length. The required estimated strain rate over the parallel length may be maintained in this range sufficiently accurately using a constant crosshead separation rate, v_c (open loop).

$v_c = L_c \, e_{Lc}$

where:

e_{Lc} is the estimated strain rate over the parallel length

Lc is the parallel length

(c) In the range following R_p or R_t or end of yielding, e_{Le} or e_{Lc} can be used. The use of e_{Lc} is recommended to avoid any control problems which may arise if necking occurs outside the extensometer gauge length.

The strain rates specified in Para 8.3.2.2 to 8.3.2.4 shall be maintained during the determination of the relevant material property.

During switching to another strain rate or to another control mode, no discontinuities in the stress strain curve should be introduced which distort the values of R_m, A_g or A_{gt}). This effect can be reduced by a suitable gradual switch between the rates.

The testing rate used should be documented.

8.3.2.2 Strain rate for the determination of the upper yield strength, R_{eH}, or proof strength properties, R_p and R_t: The strain rate, e_{LE}, shall be kept as constant as possible up to and including the determination of R_{eH} or R_p or R_t. During the determination of these material properties, the strain rate, e_{Le}, shall be in one of the two following specified ranges.

Range 1: e_{Le} = 0.00007 s^{-1}, with a relative tolerance of ±20%.

Range 2: e_{Le} = 0.00025 s^{-1}, with a relative tolerance of ±20% (recommended, unless otherwise specified).

If the testing machine is not able to control the strain rate directly, Method A2 shall be used.

8.3.2.3 Strain rate for determination of the lower yield strength, R_{eL}, and percentage yield point extension, A_e: Following the detection of the upper yield strength, the estimated strain rate over the parallel length, eLc, shall be maintained in one of the following specified ranges until discontinuous yielding has ended:

Range 2: e_{Lc} = 0.00025 s^{-1}, with a relative tolerance of ±20% (recommended, when ReL is determined).

Range 3: 0.002 s^{-1}, with a relative tolerance of ±20%.

8.3.2.4 Strain rate for the determination of the tensile strength, Rm, percentage elongation after fracture, A, percentage

total extension at the maximum force, A_{gt}, percentage plastic extension at maximum force, A_g, and percentage reduction area, Z: After determination of the required yield/proof strength properties, the estimated strain rate over the parallel length, e_{Lc}, shall be changed to one of the following specified ranges.

Range 2: e_{Lc} = 0.00025 s^{-1}, with a relative tolerance of ±20% .

Range 3: 0.002 s^{-1}, with a relative tolerance of ±20%.

Range 4: 0.0067 s^{-1}, with a relative tolerance of ±20% (0.4 min^{-1}, with a relative tolerance of ±20%) (recommended, unless otherwise specified).

If the purpose of the tensile test is only to determine the tensile strength, then an estimated strain rate over the parallel length of the test piece according to range 3 or 4 may be applied throughout the entire test.

8.3.3 Testing rate based on stress rate (Method B)

8.3.3.1 General: The testing rates shall conform to the following requirements depending on the nature of the material. Unless otherwise specified, any convenient speed of testing may be used up to a stress equivalent to half of the specified yield strength. The testing rates above this point are specified below.

It is not the intent of Method B to maintain constant stress rate or to

control stress rate with closed loop force control while determining yield properties, but only to set the crosshead speed to achieve the target stress rate in the elastic region (see Table 3.3). When a specimen being tested begins to yield, the stressing rate decreases and can even become negative the case of a specimen with discontinuous yielding. The attempt to maintain a constant stressing rate through the yielding process requires the testing machine to operate at extremely high speeds and, in most cases, this is neither practical nor desirable.

Table 3.3: Stress Rate

Modulus of elasticity of the material, E (MPa)	Stress rate, (MPa s-1)	
	Min.	Max.
< 150000	2	20
≥ 150000	6	60

8.3.3.2 Yield and proof strengths

8.3.3.2.1 Upper yield strength, R_{eH}: The rate of separation of the crossheads of the machine shall be kept as constant as possible and within the limits corresponding to the stress rates in Table 3.3.

8.3.3.2.2 Upper and lower yield strengths, R_{eH} and R_{eL}: If only the lower yield strength is being determined, the strain rate during yield of the parallel length of the test piece shall be between 0.00025 s^{-1} and 0.0025 s^{-1}. The strain rate

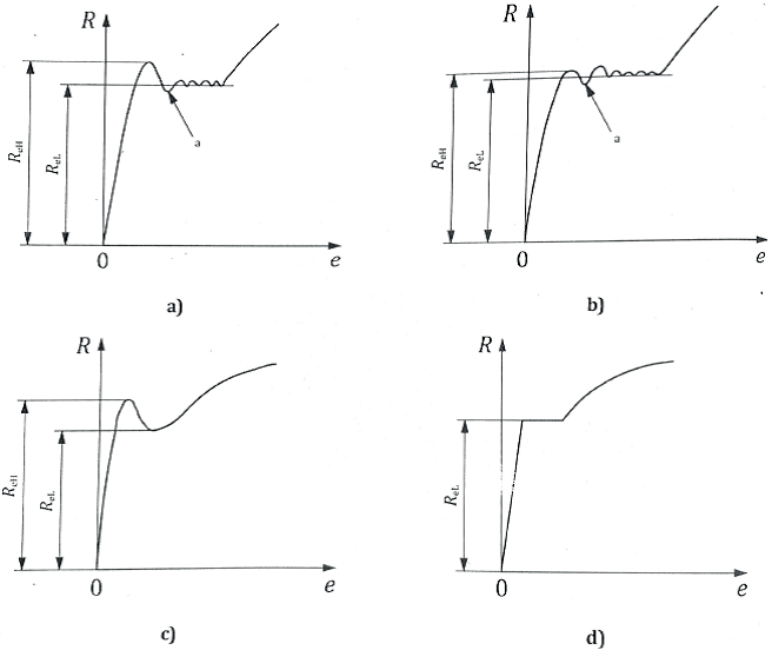
within the parallel length shall be kept as constant as possible. If this rate cannot be regulated directly, it shall be fixed by regulating the stress rate just before yield begins, the controls of the machine not being further adjusted until completion of yield.

In no case shall the stress rate in the elastic range exceed the maximum rates given in Table 3.3

8.3.3.2.3 Upper and lower yield strengths, R_{eH} and R_{eL}: If both the upper and lower yield strengths are determined during the same test, the conditions for determining the lower yield strength shall be complied with.

8.3.3.2.4 Proof strength (plastic extension) and proof strength (total extension), R_p and R_t: The crosshead separation rate of the machine shall be kept as constant as possible and within the limits corresponding to the stress rates in Table 3.3 for the elastic range. This crosshead separation rate shall be maintained up to the proof strength (plastic extension or total extension). In any case, the strain rate shall not exceed 0.0025 s-1.

8.3.3.2.5 Rate of separation: If the testing machine is not capable of measuring or controlling the strain rate, a crosshead separation rate equivalent to the stress rate given in Table 3.3 shall be used until completion of yield.



Key:

e	Percentage extension	R	Stress
R_{eH}	Upper yield strength	R_{eL}	Lower yield strength
a	Initial transient effect		

Fig. 3.1.1: Example of upper and lower yield strengths for different types of curves

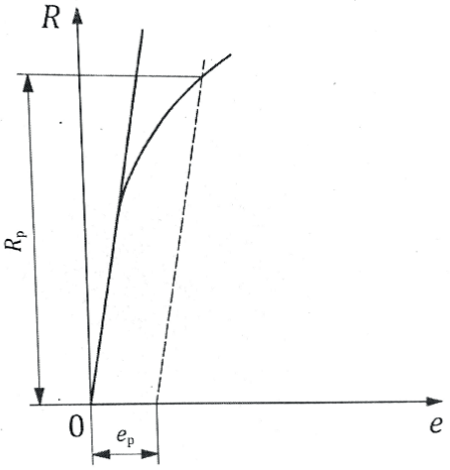
9. Determination of the upper yield strength: R_{eH} may be determined from the force-extension curve or peak load indicator and is defined as the maximum value of stress prior to the first decrease in force. The value is calculated by dividing this force by the original cross-sectional area of the test piece, S_o (see Fig. 3.1.1).

10. Determination of the lower yield strength: R_{eL} is determined from the force-extension curve and is defined as the lowest value of stress during plastic yielding, ignoring any initial transient effects. The value is calculated

by dividing this force by the original cross-sectional area of the test piece, S_o (See Figure 3.1.1).

In case of materials having yield phenomena and when Ae is not to be determined: for productivity of testing, R_{eL} may be reported as the lowest stress within the first 0.25% strain after R_{eH}, not taking into account any initial transient effect. After determining R_{eL} by this procedure, the test rate may be increased as per Para 8.3.2.4 or Para 8.3.3.3. Use of this shorter procedure should be recorded on the test report.

11. Determination of proof strength, plastic extension



Key

e Percentage extension

e_p Specified percentage plastic extension

R Stress

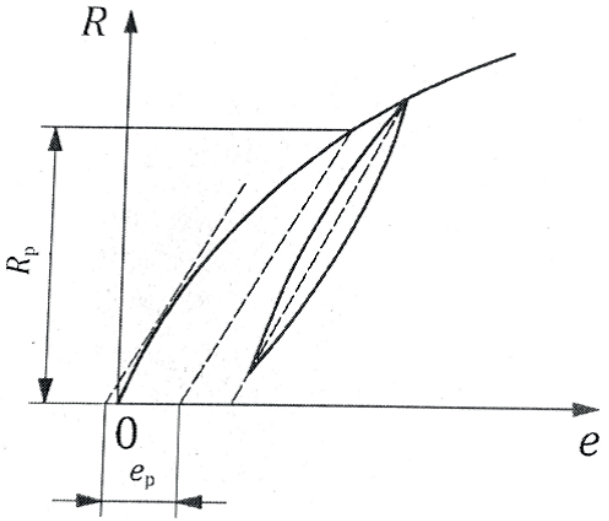
R_p Proof strength plastic extension

Fig. 3.1.2: Proof strength, plastic extension, Rp

11.1 R_p is determined from the force-extension curve by drawing a line parallel to the linear portion of the curve and at a distance from it equivalent

to the prescribed plastic percentage extension, e.g. 0.2%. The point at which this line intersects the curve gives the force corresponding to the desired proof strength plastic extension. The latter is obtained by dividing this force by the original cross-sectional area of the test piece, S_o (see Fig. 3.1.2).

If the straight portion of the force-extension curve is not clearly defined, thereby preventing drawing the parallel line with sufficient precision, the following procedure is recommended (see Fig. 3.1.3):



Key

e Percentage extension

e_p Specified percentage plastic extension

R Stress

R_p Proof strength, plastic extension

Fig. 3.1.3: Proof strength, plastic extension, Rp, alternative procedure

When the presumed proof strength has been exceeded, the force is reduced to a value equal to about 10% of the force obtained. The force is then increased again until it exceeds the value obtained originally. To determine the desired proof strength, a line is drawn through the hysteresis loop. A line is then drawn parallel to this line, at a distance from the corrected origin of the curve, measured along the abscissa, equal to the prescribed plastic percentage extension. The intersection of this parallel line and the force-extension curve gives the force corresponding to the proof strength. The value is calculated by dividing this force by the original cross-sectional area of the test piece, So (see Fig. 3.1.3).

NOTE: Several methods can be used to define the corrected origin of the force-extension curve. One of these is to construct a line parallel to that determined by the hysteresis loop so that it is tangential to the force-extension curve. The point where this line crosses the abscissa is the corrected origin of the force-extension curve (see Fig. 3.1.3).

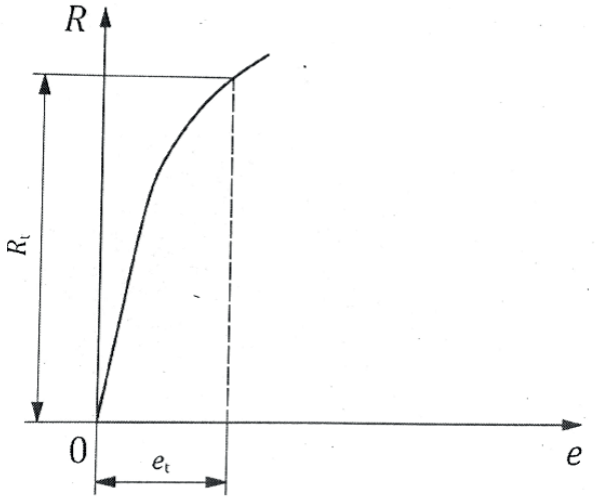
Care should be taken to ensure that the hysteresis is performed after the final proof strength has passed, but at as low an extension as possible, as performing it at excessive extensions will have an adverse effect on the slope obtained.

11.2 The property may be obtained without plotting the force-extension curve by using automatic devices (microprocessor, etc.).

12. Determination of proof strength, total extension

12.1 R_t is determined on the force-extension curve, taking Para 8.2 into consideration, by drawing a line parallel to the ordinate axis (force axis) and at a distance from this equivalent to the prescribed total percentage extension. The point at which this line intersects the curve gives the force corresponding to

the desired proof strength. The value is calculated by dividing this force by the original cross-sectional area of the test piece, S_o (see Fig. 3.1.4).



Key

e Percentage extension

e_t Percentage total extension

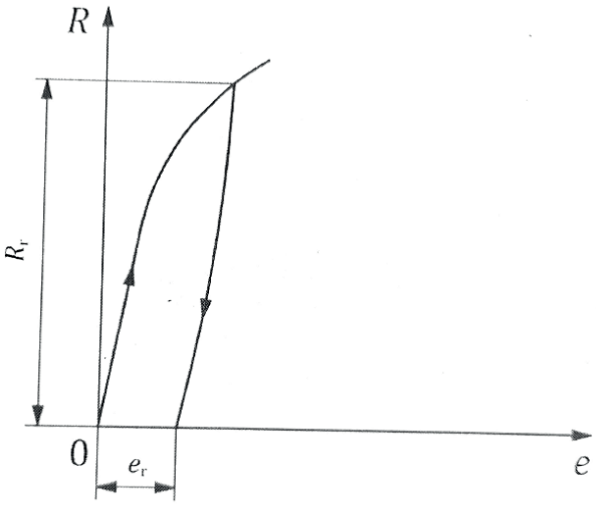
R Stress

R_t Proof strength, total extension

Fig. 3.1.4: Proof strength, total extension, Rt

12.2 The property may be obtained without plotting the force-extension curve by using automatic devices.

13. Method of verification of permanent set strength: The test piece is subjected to a force corresponding to the specified stress for 10 s to 12 s. This force is obtained by multiplying the specified stress by the original cross-sectional area of the test piece, So. After removing the force, it is then confirmed that the permanent set extension or elongation is not more than the percentage specified for the original gauge length; see Fig. 3.1.5.



Key

e Percentage elongation or percentage extension

e_r Percentage permanent set extension or elongation

R Stress

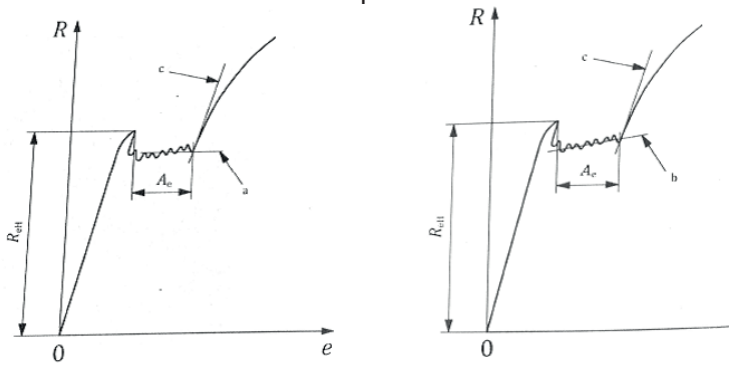
R_r Specified permanent set strength

Fig. 3.1.5: Permanent set strength, Rr

Note: This is a pass/fail test, which is not normally performed as a part of the standard tensile test. The stress applied to the test piece and the permissible set extension or elongation are specified either by the product specification or the requester of the test. Example: Reporting "$R_{r0.5}$ = 750 MPa Pass" indicates that a stress of 750 MPa was applied to the test piece and the resulting permanent set was less than or equal to 0.5%.

14. Determination of the percentage yield point extension: For materials that exhibit discontinuous yielding, A_e is determined from the force-extension curve by subtracting the extension ReH from the extension at

the start of uniform work-hardening. The extension at the start of uniform work-hardening is defined by the intersection of a horizontal line through the last local minimum point, or a regression line through the range of yielding, prior to uniform work-hardening and a line corresponding to the highest slope of the curve occurring at the start of uniform work-hardening (see Fig. 3.1.6). It is expressed as a percentage of the extensometer gauge length, L_e. The method used (see Fig. 3.1.6a or b) should be documented in the test report.



(a) Horizontal line method (b) Regression method

Key

A_e Percentage yield point extension

e Percentage extension

R Stress

R_{eH} Upper yield strength

a Horizontal line through the last local minimum, prior to uniform work-hardening

b Regression line through the range of yielding, prior to uniform work-hardening

c Line corresponding to the highest slope of the curve occurring at the start of uniform work-hardening

Fig. 3.1.6: Different evaluation methods for percentage yield point extension, Ae

15. Determination of the percentage plastic extension at maximum force: The method consists of determining the extension at maximum force on the force-extension curve obtained with an extensometer and subtracting the elastic strain. Calculate the percentage plastic extension at maximum force, A_g, from the following formula:

$$A_g = [(\Delta L_m / L_e) - (R_m / m_E)] \times 100$$

Where:

ΔL_m is the extension at maximum force

L_e is the extensometer gauge length

R_m is the tensile strength

m_E is slope of elastic part of stress-percentage extension curve

Key

A Percentage elongation after fracture (determined from the extensometer signal or directly from the test piece)

A_g Percentage plastic extension at maximum force

A_{gt} Percentage total extension at maximum force

A_t Percentage total extension at fracture

e Percentage extension

m_E Slope of the elastic part of the stress-percentage elongation curve

R Stress

R_m Tensile strength

Δe Plateau extent (for determination of A_g, see Para 15, for determination of A_{gt}, see Para 16)

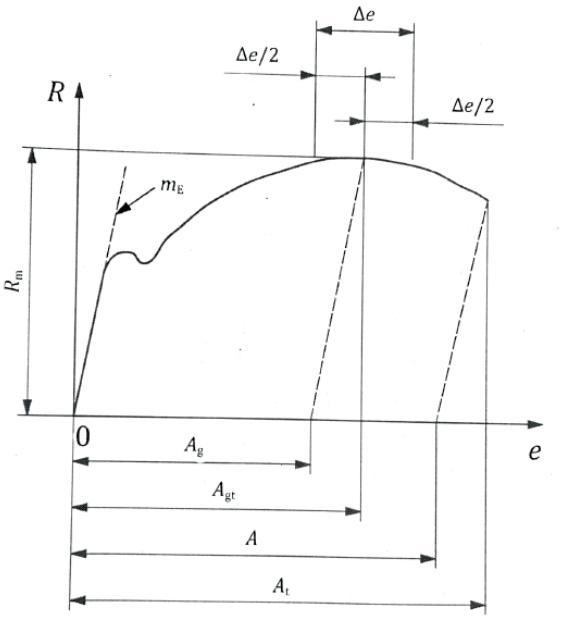


Fig. 3.1.7: Definition of extension

NOTE: For materials which exhibit a plateau at maximum force, the percentage plastic extension at maximum force is the extension at the mid-point of the plateau (see Fig. 3.1.7).

16. Determination of the percentage total extension at maximum force: The method consists of determining the extension at maximum force on the force-extension curve obtained with an extensometer. Calculate the percentage total extension at maximum force, Agt, from the following formula:

$A_{gt} = (\Delta L_m / L_e) \times 100$

Where:

ΔL_m is the extension at maximum force

L_e is the extensometer gauge length

NOTE: For materials which exhibit a plateau at maximum force, the percentage plastic extension at maximum force is the extension at the mid-point of the plateau (see Fig. 3.1.7).

17. Determination of the percentage total extension at fracture: The method consists of determining the extension at fracture on the force-extension curve obtained with an extensometer. Calculate the percentage total elongation at fracture, At, from the following formula:

$A_t = (\Delta L_f / L_e) \times 100$

Where:

ΔL_f is the extension at fracture

L_e is the extensometer gauge length

18. Determination of the percentage elongation after fracture

18.1 Percentage elongation after fracture shall be determined in accordance with the definition given in Para 3.4.2. For this purpose, the broken pieces of the test piece shall be carefully fitted back together so that their axes lie in a straight line. Special precautions shall be taken to ensure proper contact between the broken parts of the test piece when measuring the final gauge length. This is particularly important for test pieces of small cross-section and test pieces having low elongation values.

Calculate the percentage elongation after fracture, A, from the following formula:

$A = [(L_u - L_o) / L_o] \times 100$

Where:

L_u is the final gauge length after fracture

L_o is the original gauge length

Elongation after fracture, $L_u - L_o$, shall be determined to the nearest 0.25 mm or better using a measuring device with sufficient resolution.

If the specified minimum percentage elongation is less than 5%, it is recommended that special precautions be taken (as given in App. H of the IS:1608, Part 1). The result of this determination is valid only if the distance between the fracture and the nearest gauge mark is not less than $L_0/3$. However, the percentage elongation after fracture can be regarded as valid, irrespective of the position of the fracture, if the percentage elongation after fracture is equal to or greater than the specified value. To avoid having to reject test pieces where the distance between the fracture and the next gauge mark is less than $L_0/3$, the method described in Annex. I of IS:1608 (Part 1) may be used by agreement.

18.2 When extension at fracture is measured using an extensometer, it is not necessary to mark the gauge lengths. The elongation is measured as the total extension at fracture, and it is therefore necessary to deduct the elastic extension in order to obtain percentage elongation after fracture. To obtain comparable values with the manual method, additional adjustments can be applied (e.g. high enough dynamic and frequency bandwidth of the extensometer) (see A 2.2 of IS:1608, Part 1).

The result of this determination is valid only if fracture and localized extension (necking) occur within the extensometer gauge length, L_e. The percentage elongation after fracture can be regarded as valid regardless of the position of the fracture cross-section if the percentage elongation after fracture is equal to or greater than the specified value. If the product standard specifies the determination of percentage elongation after fracture for a given gauge length, the extensometer gauge length should be equal to this length.

19. determination of percentage reduction of area: Percentage reduction of area shall be determined in accordance with the definition given in Para 3.8. If

necessary, the broken pieces of the test piece shall be carefully fitted back together so that their axes lie in a straight line.

For round test pieces, the measurements at the minimum reduced section should be made in 2 planes at 90° to each other and the average used for the calculation of Z. Care should be taken to ensure that the fracture surfaces are not displaced when making the readings. Calculate the percentage reduction of area, Z, from the following formula:

$$Z = [(S_o - S_u) / S_o] \times 100$$

Where:

S_0 is the original cross-sectional area of the parallel length

S_u is the minimum cross-sectional area after fracture

It is recommended to measure Su to an accuracy of ±2%. Measuring Su with an accuracy of ±2% on small diameter round test pieces, or test pieces with other cross-sectional geometries, may not be possible.

20. Test report: The test report shall contain at least the following information, unless otherwise agreed by the parties concerned:

(a) Reference to this document, extended with the test condition information specified in Para 8.3.4;

(b) Identification of the test piece;

(c) Specified material, if known;

(d) Type of test piece;

(e) Location and direction of sampling of test pieces, if known;

(f) Testing control mode(s) and testing range(s) (see Para 8.3.1) if different from the recommended methods and values given in Para 8.3.2 and Para 8.3.3;

(g) Test results: Results should be rounded to the following precisions or better, if not otherwise specified in product standards: strength values, in MPa, to the nearest whole number;

- Percentage yield point extension values, Ae, to the nearest 0.1%;
- All other percentage extension and elongation values to the nearest 0.5%;
- Percentage reduction of area, Z, to the nearest 1%.

3.2 Bend Test

1. Introduction: The steel bars are bent to form a specific shape such as shear stirrups, L-bars, and other shapes, prior to installation into concrete structure. Therefore, steel should have sufficient ductility to enable the bending of reinforcement bars without affecting the steel strength. Hence, the bending test of reinforcement steel is performed to verify the steel ductility and to ensure that no fracture or cracks occur during the bending.

This chapter specifies a method for determining the ability of metallic materials to undergo plastic deformation in bending. It applies, to test pieces taken from metallic products as specified in the relevant product standard. It is not applicable to certain materials and/or products, for example tubes in full section or welded joints, for which other standards exist.

2. Reference: IS-1599: 2019 "Metallic Materials – Bend Test".

3. Principle: The bend test consists of submitting a test piece of round, square, rectangular, or polygonal cross section to plastic deformation by bending, without changing the direction of loading, until a specified angle of bend is reached.

The axes of the two legs of the test piece remain in a plane perpendicular to the axis of bending. In the case of 180O bend, the two lateral surfaces may, depending on the requirements of the product standard, lie flat against each other or may be parallel at a specified distance, an insert being used to control this distance.

4. Symbols and Designations

Symbol	Designation	Unit
a	Thickness or Diameter of Test piece (or diameter of the inscribed circle for pieces of polygon cross-section)	mm
b	Width of the Test piece	mm
L	Length of Test Piece	mm
l	Distance between supports	mm
D	Diameter of Mandrel	Mm
α	Angle of Bend	Degree
r	Internal radius of bend portion of test piece after bending	mm
f	Displacement of the former	Mm
c	Distance between the plane including the horizontal axis of supports and the central axis of the rounded portion of the former before test	mm
p	Distance between the vertical planes including the central axis of supports and the vertical plane including the central axis of the former	mm
R	Radius of the supports	mm

5. Test equipment

5.1 General: The bend test shall be carried out in testing machines or presses equipped with the following devices:

(a) Bending device with two supports and a former, as shown in Fig. 3.2.1

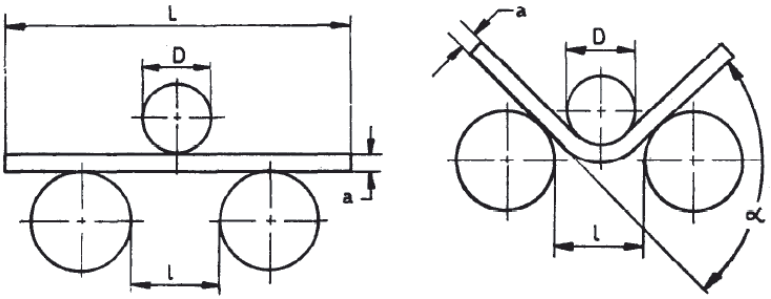


Fig. 3.2.1: Bending device with two supports and s former

(b) Bending device with a V-block and a former, as shown in Fig. 3.2.2.

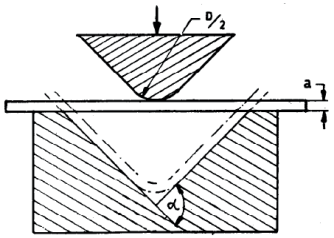


Fig. 3.2.2: Bending device with a V-block and a former

(c) Bending device with a clamp and a former, as shown in Fig. 3.2.3

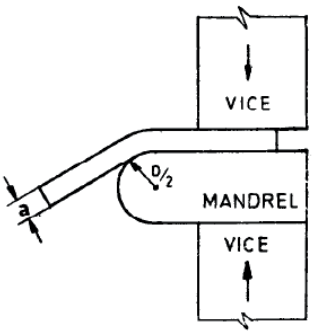


Fig. 3.2.3: Bending device with a clamp

5.2 Bending device with supports and a former

5.2.1 The length of the supports and the width of the former shall be greater than the width or diameter of the test piece. The diameter of the former is determined by the product standard. The test piece supports and former shall be of sufficient hardness (Fig. 3.2.1)

5.2.2 Unless otherwise specified, the distance between the supports, "l", shall be:

$$l = (D + 3a) \pm a/2$$

and shall not change during the bend test

NOTE: When the distance between the supports (l) is specified smaller than or equal to D + 2a, it can result in clamping during the test and stretch forming of the test piece.

5.3 Bending device with a V-block: The tapered surfaces of the V-block shall form an angle of $180^0 - \alpha$ (see Fig. 3.2.2). The angle α is specified in the relevant standard. The edges of the V-block shall be rounded to a radius between 1 and 10 times the thickness of the test piece and shall be of sufficient hardness.

5.4 Bending device with a clamp: The device consists of a clamp and a former of sufficient hardness; it may be equipped with a lever for applying force to the test piece (Fig. 3.2.3). Because the position of the left face of the clamp could influence the test results, the left face of the clamp (as shown in Fig. 3.2.3) should not reach up to or beyond the vertical line through the center of the circular former shape.

6. Test Piece

6.1 General: Round, square, rectangular or polygonal cross section test pieces shall be used in the test. Any areas of the material affected by shearing or flame cutting and similar operations during the cutting of test pieces shall be removed. However, testing a

test piece, the affected parts of which have not been removed, is acceptable provided the resultant bend is satisfactory.

6.2 Edges of rectangular test pieces: The edges of rectangular test pieces shall be rounded to a radius not exceeding the following values:

- 3mm, when the thickness of test pieces is 50mm or greater;
- 1.5mm, when the thickness of test pieces is less than 50mm and more than or equal to 10mm (inclusive);
- 1mm, when the thickness is less than 10mm.

The rounding shall be made so that no transverse burrs, scratches or marks are formed which might adversely affect the test result. However, testing a test piece, the edges of which have not been rounded, is acceptable provided the result is satisfactory.

6.3 Width of the test piece: Unless otherwise specified in the relevant standard, the width of the test piece shall be as follows:

(a) The same as the product width, if the later is equal to or less than 20mm;

(b) When the width of a product is more than 20mm:

(i) 20 ± 5 mm for products of thickness less than 3mm,

(ii) Between 20 and 50mm for products of thickness equal to or greater than 3mm.

6.4 Thickness of the test piece:

6.4.1 The thickness of the test pieces from sheets, strips and sections shall be equal to the thickness of the product to be tested. If the thickness of the product is greater than 25mm, it may be reduced by machining one surface to not less than 25mm. During

bending, the unmachined side shall be the tension-side surface of the test piece.

6.4.2 Test pieces of round or polygonal cross-section shall have a cross-section equal to that of the product, if the diameter (for a round cross section) or the inscribed circle diameter (for polygonal cross-section) does not exceed 30mm. When the diameter or the inscribed circle diameter, of the test piece exceeds 30mm and including 50mm, it may be reduced to not less than 25mm. When the diameter or the inscribed circle diameter, of the test piece exceeds 50mm, it shall be reduced to not less than 25mm (refer Fig. 3.2.4). During bending, the unmachined side shall be the tension-side surface of the test piece.

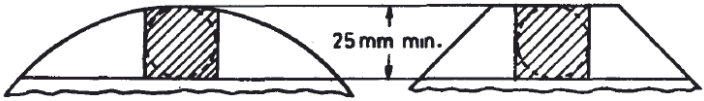


Fig. 3.2.4: Diameter and the inscribed circle diameter

6.5 Test pieces from forgings, castings and semi-finished products: In the case of forgings, castings and semifinished products, the dimensions of the test piece and sampling shall be as specified in the relevant standard or by agreement.

6.6 Agreement for test pieces of greater thickness and width: By agreement, test pieces of a greater thickness and width than those specified in Para 6.3 and 6.4 may be subjected to the bend test.

6.7 Length of test piece: The length of the test piece depends on the thickness of the test piece and the test equipment used.

7. Procedure

7.1 In general, tests are carried out at ambient temperature between 10^0C and 35^oC. Tests carried out under controlled conditions shall be made at a temperature of 23 ± 5°C.

7.2 The bend test is carried out using one of the following methods specified in the relevant standard:

(a) A specified angle of bend is achieved under the force and for the given conditions (see Fig. 3.2.1, 3.2.2 and 3.2.3).

(b) The legs of the test piece are parallel to each other at a specified distance apart while under the force (see Fig. 3.2.6).

(c) The legs of the piece are in direct contact while under an appropriate force (see Fig. 3.2.7)

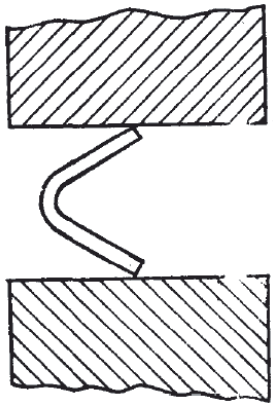


Fig. 3.2.5: Bending the legs of the test piece

7.3 In the bend test to a specified angle of bend, the test piece shall be placed on the supports (see Fig. 3.2.1) or on the V-block (see Fig. 3.2.2) and bend it in the middle between supports by the action of a force. The angle of bend, α, can be calculated from the measurement of the displacement of the former as given in Annex. A.

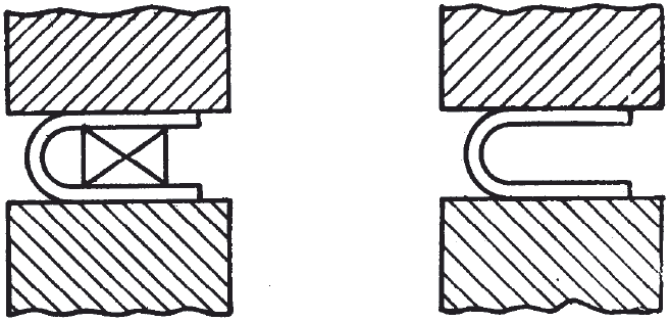


Fig. 3.2.6: Legs of the test piece parallel to each other

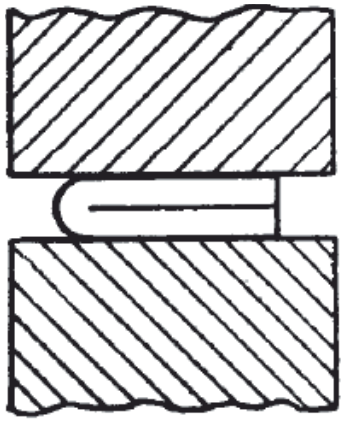


Fig. 3.2.7: Legs of test piece in direct contact

For the three methods (Fig. 3.2.1, 3.2.2 and 3.2.3), the bending force shall be applied slowly so as to permit free plastic flow of the material.

In case of dispute, a testing rate of (1 ± 0.2) mm/s shall be used.

If it is not possible to bend the test piece directly to the specified angle in the manner described above, the bend shall be completed by pressing directly on the ends of the legs of the test piece (see Fig. 3.2.5).

In a bend test requiring parallel legs, the test piece

may be bent first, as indicated in Fig. 3.2.5, and then placed between the parallel plates of the press (see Fig. 3.2.6), where it is further formed by application of a force to obtain parallelism of the legs. The test may be carried out with or without an insert. The thickness of the insert shall be as defined in the relevant standard or by agreement.

An alternate method of test is that of bending over a former (see Para 5.4).

7.4 If specified, the test piece, after its preliminary bending, shall be further bent between the parallel plates of the press, by application of a force, to obtain direct contact between the legs of the test piece (see Fig. 3.2.7).

8. Interpretation of Result

8.1 The interpretation of the bend test is carried out according to the requirements of the product standards. When these requirements are not specified, absence of cracks visible without the use of magnifying aids is considered as the evidence that the test piece withstood the bend test.

8.2 The angle of bend, specified in product standards, is always considered as a minimum. If the internal radius of a bend is specified, it is considered as a maximum.

9. Test report: The test report shall include the following information:

(a) Reference to this standard;

(b) Identification of the test piece (type of material, cast number, direction of the test piece axis relative to a product, etc.);

(c) Shape and dimensions of the test piece;

(d) Test method;

(e) Any deviation from this standard; and

(f) Test result.

Annex. A

[Determination of the bend angle from the measurement of the displacement of the former]

$\sin \alpha/2 = [(p \times c) \times + \{W \times (f - c)\}] / [p^2 + (f - c)^2]$

$\cos \alpha/2 = [(W \times p) - \{c \times (f - c)\}] / [p^2 + (f - c)^2]$

where:

$W = [p^2 + (f - c)^2 - c^2]^{0.5}$

$c = R + a + D/2$

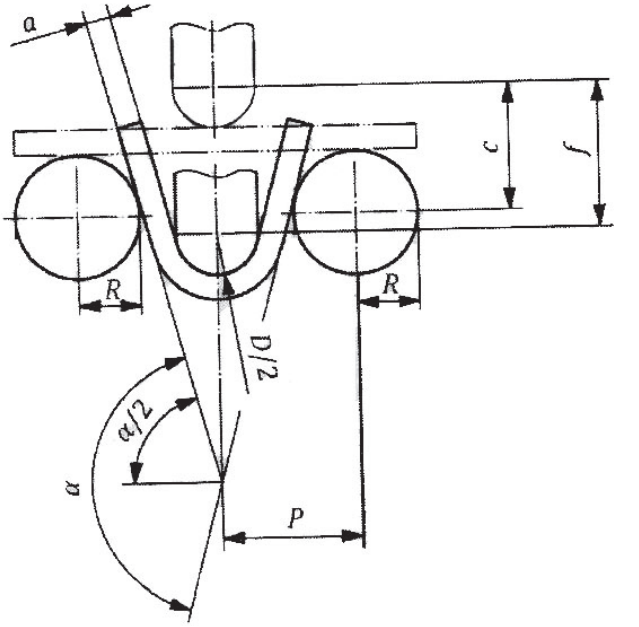


Fig. 3.2.8: Values for the calculation of the bend angle, α

3.3 Re-bend Test

The purpose of re-bend test is to measure the effect of strain ageing on steel. Strain ageing has embrittlement effect which takes place after cold deformation by diffusion of nitrogen in steel.

1. Introduction: This chapter describes the procedure for conducting the re-bend test on reinforcement steel bars.

2. Reference: IS-1786:2008 (Reaffirmed-2018) "High Strength Deformed Steel Bars and Wires for Concrete Reinforcement - Specification".

3. Preparation of Test Sample: The test pieces shall be full sections of the bars and shall be subjected to test without any further modifications. No reduction in size by machining or otherwise shall be permissible, except in case of bars of size 28mm and above. Before the test pieces are selected, the manufacturer or supplier shall furnish the purchaser or his authorized representative with copies of the mill records giving the mass of bars in each bundle/cast with sizes as well as the identification marks.

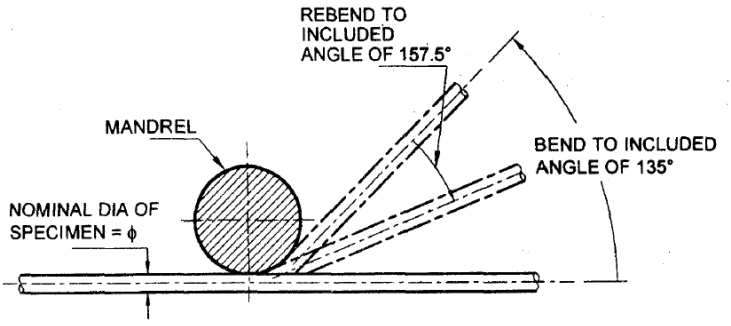


Fig. 3.3.1: Re-bend Test

Table 3.4: Diameter of Mandrel

SN	Nominal Size of Specimen	For Fe415 & Fe500	For Fe415D & Fe500D	For Fe550 & Fe600	For Fe550D & Fe600D
1	Upto and including 10mm	5 Ø	4 Ø	7 Ø	6 Ø
2	Over 10mm	7 Ø	6 Ø	8 Ø	7 Ø

Where Ø is the nominal size of test piece, in mm.

4. Procedure: Tine test piece snail be bent to an included angle of 135^0 (see Fig. 3.3.1) using a mandrel of appropriate diameter (see Table 3.4). The bent piece shall be aged by keeping in boiling water (100^0C) for 30 min and then allowed to cool. The piece shall then be bent back to have an included angle of $157½^0$. The specimen shall be considered to have passed the test if there is no rupture or cracks visible to a person of normal or corrected vision on the re-bent portion.

5. Retest: Should any one of the test pieces first selected fail to pass the test, two further samples shall be selected for testing in respect of each failure. Should the test pieces from both these additional samples pass, the material represented by the test samples shall be deemed to comply with the requirements of the test. Should the test piece from either of these additional samples fail, the material presented by the samples shall be considered as not having complied with this standard.

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Chapter - 4

TESTS ON WATER

Water used for mixing and curing shall be clean and free from injurious amounts of oils, acids, alkalis, salts, sugar, organic materials or other substances that may be deleterious to concrete or steel.

Potable water is generally considered satisfactory for mixing concrete. However, in case of doubt regarding development of strength, the suitability of water for making concrete shall be ascertained by the compressive strength and initial setting time tests as given below:

(i) Average 28 days compressive strength of at least three 150mm concrete cubes prepared with water proposed to be used shall not be less than 90 percent of the average of strength of three similar concrete cubes prepared with distilled water. The cubes shall be prepared, cured and tested as per provisions of lS:516.

(ii) The initial setting time of test blocks made with the appropriate cement and the water proposed to be used shall not be less than 30 min and shall not differ by ± 30 min from the initial setting time of control test block prepared with the same cement and distilled water. The test blocks shall be prepared and tested as per provisions of IS:4031(Part-15).

Following laboratory tests are typically conducted on the water to confirm its suitability for use in mixing concrete:

(4.1) Total Suspended Solids (Matter) in Water

(4.2) Total Solids (Matter) in Water

(4.3) Acidity of Water

(4.4) Alkalinity of Water

(4.5) pH Value of Water

(4.6) Chlorides in Water

(4.7) Sulphates in Water

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4.1 Total Suspended Solids (Matter) in Water

1. Introduction: Total Suspended Solids (TSS) is suspended particles (by dry weight) in water that is not dissolved and can be trapped by a filter by using a filtration system. Presence of impurities in water for concrete mix leads to decrease in structural properties of concrete such as strength and durability to a large extent. It is found that high content of suspended particles does not affect the strength of the concrete, but affect other properties of the same. The IS:456-2000 prescribes an allowable limit of 2000 mg/liter for suspended particles in water. Before use of water in concrete, the muddy water should undergo settlement in the basin. This chapter describes a gravimetric method for the determination of non-filterable residue.

2. Reference: IS-3025(Part-17):2022 "Methods of Sampling and Test (Physical and Chemical) for Water and Wastewater. Part-17: Non-Filterable Residue (Total Suspended Solids at 103^0C-105^0C)".

3. Principle: The non-filterable residue is determined by passing the sample through a weighed filter and drying the filter at 103°C-105°C in an oven. The increase in the filter weight represents Total Suspended Solids (TSS).

4. Apparatus required

4.1 Hot Plate/Block, to maintain < 100^oC temperature (Fig. 4.1.1).



Fig. 4.1.1



Fig. 4.1.2

4.2 Wide-Bore Pipettes, Class B glass, mechanical or electronic (Fig. 4.1.2).

4.3 Graduated Cylinders, Class A.

4.4 Evaporating Dish, 90 mm, 100 ml capacity made of platinum, porcelain, silica or borosilicate glass. Platinum is suitable for all tests; porcelain, silica and glass may be used for samples with a pH value less than 9.0.

4.5 Drying Oven, with thermostatic control for maintaining temperature with 103°C - 105°C.

4.6 Muffle Furnace, capable of operation at 550°C (Fig. 4.1.3).





Fig. 4.1.3 **Fig. 4.1.4**

4.7 Filter, anyone of the following may be used.

4.7.1 Glass Fibre Filter Disc, (Whatman GF/C or equivalent) 22mm to 125mm diameter, ≤ 2 µm nominal pore size without organic binder.

4.7.2 Paper, acid washed, ash-less hard filter finish; filter paper sufficiently retentive for fine particles (Pore size 2 µm to 2.5 µm equivalent to Whatman filter No. 542).

4.7.3 Sintered Disc, G-5 or its equivalent with pore size 1 µm to 2 µm.

4.7.4 Membrane Filter, 0.45 µm membrane.

4.7.5 Gooch Crucible, 30 ml capacity with 2.1cm,

2.4cm or 5.5cm diameter (pore size 1.2 µm) glass fibre filter disc. (Whatman GF/C or equivalent).

4.7.6 Crucible, Porous-bottom silica, sintered glass, porcelain, stainless steel or Alundum crucible with a maximum pore size of 5 µm.

4.7.7 Glass Fibre Filter Disc, (Whatman GF/C or equivalent) 2.1cm to 5.5cm in diameter, pore size 1.2 µm.

4.8 Filtering Apparatus, depending on type of filter used.

4.9 Desiccator, provided with a colour indicating desiccant (Fig. 4.1.4).

4.10 Analytical Balance, of 200 g capacity and capable of weighing to nearest 0.1 mg.

4.11 Magnetic Stirrer, with Teflon coated stirring bars.

5. Procedure

5.1 Preparation of Glass Fibre Filter Disc: Place the glass fibre filter on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the dish with three successive 20 ml volumes of reagent grade water. Remove all traces of water by continuing suction. Remove filter from membrane filter apparatus (or both crucible and filter, if Gooch crucible is used) and dry in an oven at 103°C - 105°C for 1 hour. Transfer to a desiccator and weigh after half an hour. Repeat the drying cycle until a constant mass is obtained (mass loss is less than 0.5 mg in successive weighing). Weigh immediately before use. After weighing, handle the filter or crucible filter with forceps or tongs only.

5.2 Sample Volume: In potable waters non-filterable residue is usually small. Relatively large volume of water is passed through filter so as to obtain between 2.5 mg to 200 mg dried residue. For deciding volume to be taken, turbidity values may be taken into

consideration. If turbidity values of a sample is less than 50 units, filter 1 sample and if turbidity value exceeds 50 units, filter sufficient sample so that non-filterable residue is 50 mg to 100 mg.

5.3 Stir volume of sample with a magnetic stirrer or shake it vigorously. Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fitted support.

5.4 Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected according to Para 5.2 to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through.

5.5 With suction on, wash the graduated cylinder filter non-filterable residue with portions of > 10 ml reagent grade water allowing complete drainage between washings. Remove all traces of water by continuing to apply vacuum after the wash water has passed through.

5.6 After filtration, transfer the filter along with contents to an oven maintained at either 103°C - 105°C for at least 1 hour. Cool in a desiccator and weigh. Repeat the drying cycle till constant mass is obtained. Alternatively, remove crucible and filter from crucible adapter, wipe dry from outside with filter paper and dry at 103°C-105°C in an oven. Cool in a desiccator and weigh. Repeat the drying cycle to constant mass till the difference in the successive mass is less than 0.5 mg.

6. Calculation: Calculate the total suspended solids from the following equation:

Total suspended solids, mg/l = [(A – B) / V] x 1000

Where:

A = final weight of (filter + dish), in mg;

B = weight of dish, in mg; and

V = volume of the sample, in ml.

7. Report: Report in whole numbers for less than 100 mg/l and to three significant figures for higher values. Report the temperature of determination.

8. Precision and Accuracy: Precision of the method is about 5 percent. Accuracy cannot be estimated because the non-filterable residue as determined by this method is a quantity define by the procedure followed.

4.2 Total Solids (Matter) in Water

1. Introduction: The water used for concrete mixing should be reasonably free from impurities such as suspended solids, organic solids and dissolved salts (inorganic solids), which may adversely affect the properties of the concrete, especially the setting, hardening, strength, durability, pit value, etc. This chapter describes a gravimetric method for the determination of volatile and fixed portions of total, filterable and non-filterable solids (residues) in water. The IS:456-2000 prescribes an allowable limit of 200 mg/liter for organic solids and 3000 mg/liter for inorganic solids in water.

2. Reference: IS-3025(Part-18):2022 "Methods of Sampling and Test (Physical and Chemical) for Water and Wastewater. Part-18: Volatile and Fixed Solids (Total, Filterable and Non-filterable) at 550^0C".

3. Terminology

3.1 "Total solids" is the material left in a vessel after evaporation of a sample of water and its subsequent drying in an oven at a definite temperature. Total solids include "total suspended solids" the portion of total solids retained by a filter and "total dissolved solids" the portion that passes through the filter.

3.2 Total Fixed Solids: The dish with residue after completion of test for total residue is heated in a muffle furnace at (550 ± 50)°C for 1 hour. The solid portion that is volatilised during ignition is called volatile solids. It will be mostly organic matter. The balance matter will be inorganic matter.

4. Apparatus required

4.1 Wide-Bore Pipettes, Class B glass, mechanical or electronic.

4.2 Graduated Cylinders, Class A.

4.3 Drying Oven, with thermostatic control for maintaining temperature with 103°C - 105°C.

4.4 Filter, anyone of the following may be used:

4.4.1 Glass Fibre Filter Disc, (Whatman GF/C or equivalent) from 22 mm to 125 mm diameter, </– 2um nominal pore size without organic binder.

4.4.2 Paper, acid washed, ashless hard filter finish; filter paper sufficiently retentive for fine particles (Pore size 2 - 2.5 µm equivalent to Whatman filter No. 542).

4.4.3 Gooch Crucible, 25 ml to 40 ml capacity with Gooch crucible adapter (Fig. 4.2.1).

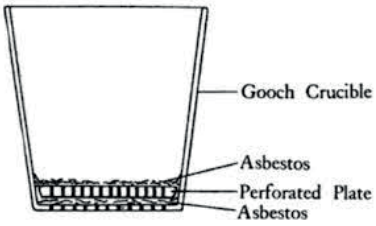


Fig. 4.2.1



Fig. 4.2.2

4.4.4 Sintered Disc, G-5 or its equivalent with pore size from 1 µm to 2 µm (Fig. 4.2.2).

4.4.5 Membrane Filter, 0.45 µm membrane.

4.5 Evaporating Dish, 90 mm, 100 ml capacity made of platinum, porcelain, silica or borosilicate glass. Platinum is suitable for all tests; porcelain, silica and glass may be used for samples with a pH value less than 9.0.

4.6 Desiccator, provided with a colour indicating desiccant.

4.7 Muffle Furnace, capable of operation at 550°C.

4.8 Analytical Balance, of 200 g capacity and capable of weighing to nearest 0.1 mg.

5. Sample: Preservation of sample is not practical. Refrigeration or chilling to 4°C is recommended.

6. Procedure

6.1 Heat the clean evaporating dish to 550°C for 1 hour. Cool, desiccate, weigh and store in desiccator until ready for use.

6.2 Select volume of the sample which has residue between 25 mg and 250 mg, preferably between 100 mg and 200 mg. This volume may be estimated from values of specific conductance. To obtain a measurable residue, successive portions of sample may be added to the sample dish.

6.3 Pipette this volume in a weighed evaporating dish on steam-bath. Evaporation may also be performed in a drying oven. The temperature shall be lowered to approximately 98°C to prevent boiling and splattering of the sample. After complete evaporation of water from the residue, transfer the dish to an oven at 103°C - 105°C or 179°C - 181°C and dry to constant mass, that is, till the difference in the successive weighing is less than 0.5 mg. Drying for a long duration (usually 1 hour to 2 hour) is done to eliminate necessity of checking for constant mass. The time for drying to constant mass with a given type of sample when a number of samples of nearly same type are to be analyzed, has to be determined by trial.

6.4 Weigh the dish as soon as it has cooled avoiding residue to stay for long time as some residues are hygroscopic and may absorb water from desiccant that is absolutely dry.

6.5 After weighing, ignite the dish in a muffle furnace at (550 ± 50)°C for 1 hour. After ignition, allow the vessel to partially cool in air and transfer to desiccator, cool and weigh.

7. Calculations: Calculate the fixed residue and volatile residue as follows (total filterable or non-filterable):

Volatile solids, mg/1 = [(A – B) / V] x 1000

Fixed solids, mg/1 = [(B – C) / V] x 1000

Where:

A = final weight of residue + dish/filter before ignition, in mg;

B = final weight of residue + dish/filter after ignition, in mg;

C = weight of dish/filter, in mg; and

V = volume in ml, of the sample.

8. Report: Report to the nearest whole number for values up to 100 mg/l and to three significant figures for higher values. Report the temperature of determination.

4.3 Acidity of Water

1. Introduction: The water that consists of industrial waste is not suitable for concrete construction. The industrial water consists of detrimental acids or alkalis that depend on the waste product of the respective industry. In terms of pH value, the water that has a pH value greater than 6 can be employed for the concrete construction. But the pH value will not give a proper and adequate measure about the acid content in the water. The acid content in water can be gauged accurately based on total acidity. As per IS:456-2000, to neutralize 100 ml sample of water, using phenolphthalein as indicator, it should not require more than 5 ml of 0.02 normal NaOH (Caustic soda).

2. Reference: IS-3025(Part-22):1986 (Reaffirmed-2019) "Method of Sampling and Test (Physical and Chemical) for Water and Wastewater. Part-22: Acidity".

3. Principle and Theory: Acidity of water is its quantitative capacity to react with a strong base to a designated pH. It may be defined as equivalent concentration of hydrogen ions in mg/l. The equation in its simplest form is as follows:

$$H + NaOH = H2O + Na$$

4. Sample: The test sample used should be free from turbidity or filtered through 0.45 μm membrane filter.

5. Apparatus

5.1 pH Meter (Fig. 4.3.1)

5.2 Burette – 50 ml capacity.

5.3 Magnetic Stirring Device



Fig. 4.3.1: pH Meter

6. Reagents

6.1 Distilled Water : pH should not be less than 6.0. If the pH is less than 6.0, it shall be freshly boiled for 15 minutes and cooled to room temperature. Deionized water may be used provided that it has a conductance of less than 2 µS/m and a pH more than 6.0.

6.2 Potassium Acid Phthalate : 0.02 N. Dissolve 4.0846 g of potassium acid phthalate salt ($KHC_8H_4O_4$) (dried at 120°C for 2 hours) in carbon dioxide free distilled water and dilute to 1 litre.

6.3 Sodium Hydroxide Solution : 15 N.

6.3.1 Sodium hydroxide solution : 1 N. Dilute 67 ml of 15 N sodium hydroxide solution (Para 6.3) to one litre with distilled water.

6.3.2 Sodium hydroxide solution : 0.02 N. Dilute 20 ml of 1 N sodium hydroxide solution (Para 6.3.1) to one litre and standardize using standard potassium acid phthalate (Para 6.2).

6.4 Phenolphthalein Indicator : Dissolve 0.5 g of phenolphthalein in 100 ml, 1: 1 (v/v) alcohol water mixture and add 0.02 N sodium hydroxide solution

drop by drop till very faint pink colour is observed.

6.5 Methyl Orange Indicator : Dissolve 0.5 g of methyl orange in distilled water and make up to 100 ml in a volumetric flask.

7. Procedure

7.1 Indicator Method: Pipette 20 ml or a suitable portion of sample into a 100 ml beaker. The sample size shall be so selected so that not more than 20 ml of titrant is needed for the titration. Determine the pH of water. If pH is less than 3.7, add two drops of methyl orange indicator into the first sample beaker and titrate with standard 0.02 N sodium hydroxide solution until the colour changes to the faint orange characteristic of pH 3.7. Record the volume of sodium hydroxide used. To the second sample beaker, add 2 to 3 drops of phenolphthalein indicator and titrate with 0.02 N sodium hydroxide solution to the appearance of faint pink colour characteristics of pH 8.3. Record the volume used.

7.2 Potentiometric Method: Pipette 20 ml or a suitable portion of sample into a 100 ml beaker. Titrate with standard sodium hydroxide solution to pH 3.7 and pH 8.3. Record the volume of standard sodium hydroxide used. No indicator is required.

8. Calculation: Calculate acidity in the sample as follows:

Acidity at pH 3.7, as mg/l $CaCO_3$ = (A x N X 50000) / V

Acidity at pH 8.3, as mg/l $CaCO_3$ = (B x N X 50000) / V

Where:

A = Volume in ml of standard sodium hydroxide used to titrate to pH 3.7,

N = Normality of standard sodium hydroxide,

V = Volume in ml of sample taken for test, and

B = Volume in ml of standard sodium hydroxide used to titrate to pH 8.3.

4.4 Alkalinity of Water

1. Introduction: The water that consists of industrial waste is not suitable for concrete construction. The industrial water consists of detrimental acids or alkalis that depend on the waste product of the respective industry. In terms of pH value, the water that has a pH value greater than 6 can be employed for the concrete construction. But the pH value will not give a proper and adequate measure about the alkalinity of the water. As per IS:456-2000, to neutralize 100 ml sample of water, using neutral indicator, it should not require more than 25 ml of 0.02 normal H_2SO_4.

2. Reference: IS-3025(Part-23):1986 (Reaffirmed-2019) "Method of Sampling and Test (Physical and Chemical) for Water and Wastewater. Part-22: Alkalinity".

3. Principle and Theory: Alkalinity of water is the capacity of that water to accept protons. It may be defined as the quantitative capacity of an aqueous medium to react with hydrogen Icons to pH 8.3 (phenolphthalein alkalinity) and then to pH 3.7 (total alkalinity or methyl orange alkalinity). The equation in its simplest form is as follows:

$CO_3^{--} + H^+ = HCO_3^{--}$ (pH 8.3)

From pH 8.3 to 3.7, the following reaction may occur:

$HCO_3^- + H^+ = H_2CO_3$

4. Sample: The sample used for analysis should be either free from turbidity or should be allowed to settle prior to analysis.

5. Apparatus

5.1 pH Meter (Fig. 4.3.1)

5.2 Burette – 50 ml capacity.

5.3 Magnetic Stirring Device

6. Reagents

6.1 Distilled Water: Distilled water used should have pH not less than 6.0. If the water has pH less than 6.0, it shall be freshly boiled for 15 minutes and cooled to room temperature. Deionized water may be used provided that it has a conductance of less than 2 μs/cm and a pH more than 6.0.

6.2 Sulphuric Acid: Dilute 5.6 ml of concentrated sulphuric acid (relative density 1.84) to one litre with distilled water.

6.3 Standard Solution of Sulphuric Acid: 0.02 N.

6.4 Phenolphthalein Indicator: Dissolve 0.5 g of phenolphthalein in 100 ml, 1: 1 (v/v), alcohol water mixture.

6.5 Mixed Indicator Solution: Dissolve 0.02 g methyl red and 0.01 g bromocresol green in 100 ml, 95 percent, ethyl or isopropyl alcohol.

7. Procedure

7.1 Indicator Method: Pipette 20 ml or a suitable portion of sample into 100 ml beaker. If the pH of the sample is over 8.3, then add 2 to 3 drops of phenolphthalein indicator and titrate with standard sulphuric acid solution till the pink colour observed by indicator just disappears (equivalence of pH 8.3). Record the volume of standard sulphuric acid solution used. Add 2 to 3 drops of mixed indicator to the solution in which the phenolphthalein alkalinity has been determined. Titrate with the standard acid to light pink colour (equivalence of pH 3.7). Record the volume of standard acid used after phenolphthalein alkalinity.

7.2 Potentiometer Method: Pipette 20 ml or a suitable portion of sample into a 100 ml beaker and titrate with standard sulphuric acid to pH 8.3 and then to pH 3.7, using a potentiometer. No indicator is required.

8. Calculation: Calculate alkalinity in the sample as follows:

Phenolphthalein alkalinity (as mg/l of $CaCO_3$) =

(A x N x 50000) / V

Total alkalinity (as mg/l of $CaCO_3$) =

[(A + B) x N x 50000] / V

Where:

A = ml of standard sulphuric acid used to titrate to pH 8.3,

B = ml of standard sulphuric acid used to titrate from pH 8.3 to pH 3.7,

N = Normality of acid used, and

V = Volume in ml of sample taken for test.

4.5 pH Value of Water

1. Introduction: The pH value of the mixing water has a significant impact on the durability of concrete. Concrete uses cement as a binding agent, which has a pH of 11, and if this pH varies too much then it loses its binding properties as it is broken down. This will create more cracks and pores in the concrete speeding up the process exponentially leading to more problems and a swifter deterioration. If the pH of the concrete drops below 7 there will be noticeable degradation and surface damage as the cement loses its ability to bind the concrete together. From the research it has been observed that the pH value of water had little effect on 28 days compressive strength of concrete, the 90 days strength varies greatly with pH values of mixing water. Chloride ion penetration and Thermal conductivity also vary with change in Ph values of mixing water. As per IS:456-2000, the pH value of water shall not be less than 6. This chapter describes electrometric and colorimetric methods for the determination of pH value. Both methods are applicable to all types of water and waste water.

2. Reference: IS-3025(Part-11): 2002 "Method of Sampling and Test (Physical and Chemical) for Water and Wastewater. Part-11: pH Value".

3. Principle: The determination of pH value is based on measuring the potential difference of an electrochemical cell using a suitable pH meter. The pH of sample also depends on the temperature because of dissociation equilibrium. Therefore, the temperature of sample is always stated together with the pH measurements.

4. Reagents: Standard pH buffer solutions from available tablets or powder, or known amount of chemicals may be used for the preparation. Procedures for the preparation of some standard pH buffer solutions are given below and Table-4.1 shows the pH value of these buffers at different temperatures.

4.1 Distilled or Deionised water: with conductivity < 0.2 mS/m.

4.2 Buffer solutions: Preferably certified buffers with stated measurement inaccuracy for calibrating pH meters. Follow the manufacturer's instructions regarding storage and stability.

4.3 Electrolytes for liquid-filled reference electrodes: Use the electrolyte solutions recommended by the manufacturer.

4.4 Potassium chloride solution (KCl): = 3 mol/l. To prepare the potassium chloride solution as electrolyte for reference electrodes, use as suitable amount of solid potassium chloride and dissolve it in water.

5. Apparatus

5.1 Sampling bottle: Sealable, flat-bottomed, and made of polyethylene or glass. The type of stopper used shall allow the exclusion of all air from the sample bottle.

5.2 Temperature measurement device: Capable of measurement with a total uncertainty not greater than 0.5°C. The temperature sensor (Para 5.2.2) is preferred.

5.2.1 Thermometer with a 0.5°C scale.

5.2.2 Temperature sensor, separate or integrated into the pH electrode.

Temperature measurement deviations due to the device shall be corrected against a calibrated thermometer.

5.3 pH meter, providing the following means for adjustment:

(a) Zero point of the pH electrode (or offset voltage);

(b) Slope of the pH electrode;

(c) Temperature of the pH electrode;

(d) Input resistance > 1012 Ω.

Moreover, it shall be possible to change the display of the pH meter to give readings of either the pH value or the voltage. The resolution of the pH value reading on the pH meter shall be 0.01 or better.

5.4 Glass electrode and reference electrode: The chain zero-point of glass electrodes should not deviate by more than ΔpH = 0.5 (manufacturer's declared value) from the nominal pH electrode value. The value of the practical slope shall be at least 95% of the theoretical slope.

Use electrodes with electrolyte solutions and a flow rate of 0.1 ml/day to 2 ml/day as reference electrodes.

For reference electrodes with an electrolyte solution, ensure that an excess hydrostatic pressure is generated by setting the filling level of the electrolyte in the reference electrode to be higher than that of the buffer solution or the measuring solution, as appropriate. It is also possible to use pressurized reference electrodes. In limited applications, reference electrodes with a solidified electrolyte (electrolyte gel or a polymerizate of an electrolyte) may also be used.

For samples with low conductivity, electrodes with high electrolyte discharge should be used. If the conductivity is > 30 mS/m, it is also possible to use an electrolyte gel or polymerizate in the reference electrodes. In general, ensure that for electrolyte gels or polymerizates, the exchange within the diaphragm is not be caused by the discharge of the electrolyte, but by diffusion of the ions involved.

5.5 Stirrer or agitator, operating with a minimum exchange of gas between the test portion and air.

6. Sampling: The pH value may change rapidly as a result of chemical, physical or biological processes in the water sample. For this reason, whenever applicable, it is advisable to measure the pH value immediately at

the sampling point. If this is not possible, take a water sample in a sampling bottle (Para 5.1). When filling the sampling bottle, avoid gas exchange, e.g. release of carbon dioxide, between the sample and the ambient air. Fill the bottle completely and stopper it, bubble-free, e.g. with a solid stopper. The sampling bottle is preferably filled by flushing to overflowing from a water sampler via a flexible tube extending to the bottom of the bottle.

Samples should be kept cool (2°C to 8°C) and in the dark during transport and storage.

In the laboratory, measure the pH value as soon as possible. When the samples are measured in the laboratory, check possible influences of transport and storage on the pH value of the samples to be analyzed.

7. Procedure

7.1 Preparation: Ensure the functionality of the pH electrode by periodic maintenance and testing (Para 7.2). Prepare the calibration buffer solutions. For devices with automatic buffer identification, follow the manufacturer's calibration instructions. Choose the buffer solutions so that the expected measurement of the sample lies between the values of the two buffers. When using a pH electrode without an internal temperature sensor, immerse a temperature sensor in the test solution.

For measurement, prepare the glass and either the reference electrode or the mono-rod pH electrode, following the manufacturer's instructions. Turn on the measuring device; for devices with automatic buffer identification, activate the stored data of the buffer solutions prepared for calibration.

Measure the temperature of the buffer and of the sample solutions. If possible, buffer and sample should have the same temperature.

If there is no temperature sensor, adjust the device to the measuring temperature.

Take the pH values of the buffer solutions from the respective certificates, depending on the existing temperature or use automatic buffer recognition.

7.2 Calibration and adjustment of the measuring equipment: Calibrate the pH electrode at two points using buffer solutions of the expected range of pH values (two-point calibration), following the manufacturer's instructions. Afterwards, adjust the devices manually, based on the data determined. For automatic measuring devices, ensure that the prepared buffer solutions correspond to the data of the buffer solutions stored in the software of the measuring device.

Immerse the pH electrode and the temperature sensor in the first buffer, usually the one at pH 7, which is used for adjusting the zero point. Subsequently stir to avoid the enrichment of potassium chloride caused by leaking reference electrolytes near the glass electrode. Turn off the stirrer and start the calibration procedure on the measuring device. Automatic devices independently identify the stability of the measurement, store this value and adjust the zero point. When using devices with manual adjustment, initially adjust the zero point at pH 7, unless otherwise specified in the manufacturer's instructions.

Thoroughly rinse the pH electrode and the temperature sensor before, between, and after the measurements using water. Immerse the pH electrode in the second buffer solution and stir. Turn off the stirrer and start the calibration procedure for the second buffer on the measuring device. Automatic devices independently identify the stability of the measurement, store this value, and adjust the slope. For devices with manual adjustment, adjust the slope so that the pH value of the second buffer is reached.

Check the result of the adjustment of the pH electrode on two new samples of the buffer solutions used. The calibration should be checked by measuring

an independent calibration checking solution buffer instead of the buffer solutions used. The measurements shall not deviate by more than 0.03 from the relevant set point. Otherwise, repeat the procedure, and replace the pH electrode if necessary.

As a result of the calibration, record the zero point and the slope of the pH electrode together with the measuring temperature. If information is required either on the condition of the pH electrode for a broad pH range or on the quality of the buffer solutions, calibrate the pH electrode at more than two points, usually at five points (multi-point calibration).

7.3 Measurement of the samples: Whenever possible, measure the samples under the same conditions as during calibration. Preferably, determine the pH value in the sampling bottle.

When changing solutions, rinse the pH electrode and the measuring vessel with distilled or deionized water and then, if possible, with the next solution to be measured. Repeat the procedure with other subsamples, if appropriate.

The mass concentrations of solids in liquid sludge should be < 5 %.

8. Expression of results: In general, the value for the quantity pH is expressed to one decimal place. Only if the composition of the unknown solution is similar to the composition of the buffer solutions and the quality of calibration justifies it, is it reasonable to report a second decimal place. If the second decimal place is required although the conditions mentioned are not met, the reasons for taking this decision should be stated in the test report. Report also the measuring temperature.

9. Test report: The test report shall contain at least the following information:

(a) All the information required for the complete identification of the sample;

(b) The sampling method used (see Para 6);

(c) The test method used, together with reference to this International Standard;

(d) All operating details not specified in this Standard, or regarded as optional, together with details of any incident that may have influenced the result(s);

(e) The measuring conditions;

(f) The test result(s) obtained.

4.6 Chlorides in Water

1. Introduction: The presence of chlorides in the concrete is a serious cause of corrosion of the steel reinforcement. This can lead to cracking of concrete due to the expansive nature of the corrosion, as well as failures of structural steel due to loss of the steel integrity. The severity of the damage depends on the concentration of the chlorides present in the concrete, as well as the variation of the chloride from one point to another. The contamination effects of chloride salt on the compressive strength of concrete worsen with concrete age and thus should be prevented at all cost. As per IS:456-2000, the maximum permissible limit of chloride in water is 2000 mg/l for plain concrete and 500 mg/l for reinforced concrete.

This chapter describes four methods for the determination of chlorides. The argentometric method is suitable for use in relatively clear waters when 0.15 to 10 mg of chloride is present in the portion titrated. The end point of mercuric nitrate method is easier to detect. Potentiometric method is suitable for coloured or turbid samples. The ferricyanide method is an automated technique. In case of any difference of opinion, the argentometric method shall be the referee method.

2. Reference: IS-3025(Part-32):1988 (Reaffirmed-2019) "Method of Sampling and Test (Physical and Chemical) for Water and Wastewater. Part-2: Chloride".

(A) Argentometric Method

3. Principle: In a neutral or slightly alkaline solution, potassium chromate can indicate the end point of the silver nitrate titration of chloride. Silver chloride is precipitated before red silver chromate is formed.

4. Apparatus

4.1 Erlenmeyer flask: 250 ml (Fig. 4.6.1).

4.2 Burette: 50 ml (Fig. 4.6.2).

5. Reagents

5.1 Potassium chromate indicator solution: Dissolve 50 g of potassium chromate in a little distilled water. Add silver nitrate solution until a definite red precipitate is formed. Let it stand for 12 hour, filter and dilute to 1 litre with distilled water.

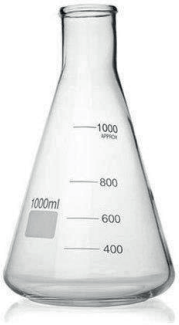


Fig. 4.6.1: Erlenmeyer flask

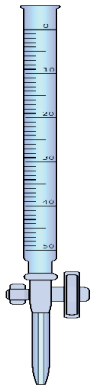


Fig. 4.6.2: Burette

5.2 Standard silver nitrate titrant: 0.014 1 N. Dissolve 2.395 g of silver nitrate in distilled water and dilute to 1 litre. Standardize against 0.014 1 N sodium chloride solution as prescribed in Para 6. 1.00 ml = 500 µg of chloride. Store in a brown bottle.

5.3 Standard sodium chloride solution: 0.014 1 N. Dissolve 824.00 mg of sodium chloride (dried at 140°C) in distilled water and dilute to 1 litre. 1.00 ml = 500 µg of chloride.

5.4 Special reagents for removal of interferences

5.4.1 Aluminium hydroxide suspension: Dissolve 1.25 g of aluminium potassium sulphate or aluminium ammonium sulphate [AlK $(SO_4)^2$ $.12H_2O$ or Al NH4 $(SO_4)^2$. $12H_2O$] in 1 litre of distilled water. Warm to 60°C and add 55 ml of

concentrated ammonium hydroxide slowly with stirring. Let it stand for 1 hour, transfer to a large bottle and wash precipitate by successive additions, with thorough mixing and decanting with distilled water, until free from chloride. When freshly prepared, the suspension occupies a volume of about 1 litre.

5.4.2 Phenolphthalein indicator solution

5.4.3 Sodium hydroxide - 1 N.

5.4.4 Sulphuric acid - 1 N.

5.4.5 Hydrogen peroxide - 30 percent.

6. Procedure: Use 100 ml sample or a suitable portion diluted to 100 ml. If the sample is highly coloured, add 3 ml of aluminium hydroxide suspension, mix, let settle and filter. If sulphide, sulphite or thiosulphate is present, add 1 ml of hydrogen peroxide and stir for 1 minute. Directly titrate the samples in the pH range 7 to 10. Adjust sample pH to 7-10 with sulphuric acid or sodium hydroxide if it is not in the range. Add 1.0 ml of potassium chromate indicator solution. Titrate with standard silver nitrate solution to a pinkish yellow end point. Standardize silver nitrate solution and establish reagent blank value by titration method.

7. Calculation

Chloride, mg/l = [(V1 - V2) x N x 35450] / V3

Where:

V1= Volume in ml of silver nitrate used by the sample,

V2= Volume in ml of silver nitrate used in the blank titration,

V3= Volume in ml of sample taken for titration, and

N = Normality of silver nitrate solution.

(B) <u>Mercuric Nitrate Method</u>

3. Principle: Chloride can be titrated with mercuric nitrate because of the formation of soluble, slightly dissociated

mercuric chloride. In the pH range 2.3 to 2.8, diphenyl carbazone indicates the end point by the formation of a purple complex with excess mercuric ions.

4. Apparatus

4.1 Erlenmeyer flask: 250 ml capacity.

4.2 Micro-burette: 5 ml with 0.01 ml graduation intervals.

5. Reagents

5.1 Standard sodium chloride solution: See Para (A)5.3 above.

5.2 Nitric acid: 0.1 N.

5.3 Sodium hydroxide: 0.1 N.

5.4 Reagents for chloride concentrations below 100 mg/l

5.4.1 Indicator-acidifier reagent: The nitric acid concentration of this reagent is an important factor in the success of the determination and can be varied as indicated in (a) or (b) to suit the alkalinity range of the sample. Reagent (a) contains -sufficient nitric acid to neutralize a total alkalinity of 150 mg as $CaCO_3$/l to the proper pH in a 100 ml sample. Adjust amount of nitric acid to accommodate samples of alkalinity different from 150 mg/l.

(a) Dissolve, in the order named, 250 mg s-diphenylcarbazone, 4.0 ml concentration nitric acid and 30 mg xylene cyanol FF in 100 ml 95 percent ethyl alcohol or isopropyl alcohol. Store in a dark bottle in a refrigerator. This reagent is not stable indefinitely. Deterioration causes a slow end point and high results.

(b) Because pH control is critical, adjust pH of highly alkaline or acid samples to 2.5 ± 0 1 with 0.1 N nitric acid or sodium hydroxide

not with sodium carbonate (Na_2CO_3). Use a pH meter with a non-chloride type of reference electrode for pH adjustment. If only the usual chloride-type reference electrode is available for pH adjustment, determine amount of acid or alkali required to obtain a pH of 2.5 ± 0.1 and discard this sample portion. Treat a separate sample portion with the determined amount of acid or alkali and continue analysis. Under these circumstances, omit nitric acid, from indicator reagent.

5.4.2 Standard mercuric nitrate titrant: 0.014 1 N. Dissolve 2.3 g mercuric nitrate [Hg $(NO_3)^2$ or 2.5 g Hg $(NO_3)^2$. H_20] in 100 ml distilled water containing 0.25 ml concentrated nitric acid. Dilute to just under 1 litre. Make a preliminary standardization by following the procedure described in Para 6.1. Use replicates containing 5.00 ml standard Sodium chloride solution and 10 mg sodium bicarbonate ($NaHCO_3$) diluted to 100 ml with distilled water. Adjust titrant to 0.014 1 N and make a final standardization; 1.00 ml = 500 µg Cl-. Store away from light in a dark bottle.

5.5 Reagent for chloride concentration greater than 100 mg/l

5.5.1 Mixed indicator reagent: Dissolve 0.50g diphenylcarbazone powder and 0.05g bromophenol blue powder in 75 ml 95 percent ethyl or isopropyl alcohol and dilute to 100 ml with the same alcohol.

5.5.2 Strong standard mercuric nitrate titrant: 0.141 N. Dissolve 25 g mercuric nitrate [Hg $(NO_3)^2$. H_2O] in 900 ml distilled water containing 5.0 ml concentrated nitric acid. Dilute to just under 1 litre and standardize by following the procedure described in Para 6.2. Use replicates

containing 25.00 ml standard sodium chloride solution and 25 ml distilled water. Adjust titrant to 0.141 N and make a final standardization; 1.00 ml = 5.00 mg Cl.

6. Procedure:

6.1 Titration of chloride concentration less than 100 mg/l: Use a 100 ml sample or small portions so that the chloride content is less than 10 mg. Add 1.0 ml indicator-acidifier reagent. For highly alkaline or acid waters, adjust pH to about 8 before adding indicator-acidifier reagent. Titrate with 0.014 1 N mercuric nitrate to a definite purple end point. The solution turns from green blue to blue a few drops before the end point. Determine the blank by titrating 100 ml distilled water containing 10 mg of sodium bicarbonate.

6.2 Titration of chloride concentrations greater than 100 mg/l: Use a sample portion requiring less than 5 ml titrant to reach the end point. Measure into a 160 ml beaker. Add approximately 0.5 ml mixed indicator reagent and mix well. The colour should be purple. Add 0.1 N nitric acid dropwise until the colour just turns yellow. Titrate with 0.141 N mercuric nitrate to first permanent dark purple. Titrate a distilled water blank using the same procedure.

7. Calculation

Chloride, mg/l = [(V1 - V2) x N x 35450] / V3

Where:

V1 = Volume in ml of titrant used for sample,

V2 = Volume in ml of titrant used for blank,

V3 = Volume in ml of the sample taken for test, and

N = Normality of the sample taken for test.

(C) Potentiometric Method

3. Principle: Chloride is determined by potentiometric titration with silver nitrate solution with a glass and silver-

silver chloride electrode system. The end point of the titration is that instrument reading at which the greatest change in voltage has occurred for a small and constant increment of silver nitrate.

4. Apparatus

4.1 Glass and silver-silver chloride electrodes

4.2 Electronic voltmeter

4.3 Mechanical stirrer

5. Reagents

5.1 Standard sodium chloride solution - 0.014 1 N.

5.2 Nitric acid - concentrated.

5.3 Standard silver nitrate titrant - 0.014 1 N.

5.4 Pretreatment reagents

5.4.1 Sulphuric acid - 1:1.

5.4.2 Hydrogen peroxide - 30 percent.

5.4.3 Sodium hydroxide - 1 N.

6. Procedure:

6.1 Standardization: Place 10.0 ml of standard sodium chloride solution in a 260 ml beaker; dilute to about 100 ml and add 2.0 ml concentrated nitric acid. Immerse stirrer and electrodes. Set instrument to desired range of millivolts or pH units. Start stirrer. Add standard silver nitrate titrant, recording scale reading after each addition. At the start large increments of silver nitrate may be added, then as the end point is approached, add small and equal increments at longer intervals so that the exact end point can be determined. Determine the volume of silver nitrate used at the point at which there is the greatest change in instrument reading per unit addition of silver nitrate. Plot a differential titration curve if the exact end point cannot be determined by inspecting the data. Plot change in instrument reading for equal increments of silver nitrate against

volume of silver nitrate added, using average of burette readings before and after each addition.

6.2 Sample analysis: Pipette 100 ml of sample or a portion containing not more than 10 mg of chloride, into a 250 ml beaker. In the absence of interfering substances, proceed as above.

In the presence of organic compounds, sulphite or other interferences, acidify sample with sulphuric acid using litmus paper. Boil for 5 minutes to remove volatile compounds. Add more sulphuric acid, if necessary, to keep solution acidic. Add 3 ml of hydrogen peroxide and boil for 15 minutes, adding chloride free distilled water to keep the volume above 60 ml. Dilute to 100 ml, add sodium hydroxide solution dropwise until alkaline to litmus, then 10 drops in excess. Boil for 5 minutes, filter into a 250 ml beaker, and wash precipitate and paper several times with hot water. Add concentrated nitric acid dropwise until acidic to litmus paper, then 2.0 ml in excess. Cool and dilute to 100 ml, if necessary. Immerse stirrer and electrodes and start stirrer. Make necessary adjustments according to manufacturer's instructions and set selection switch to appropriate setting for measuring the difference of potential between electrodes. Complete determination as detailed in Para 6.1. If an end point reading has been established from previous determinations for similar samples and conditions. use this predetermined end point. For the most accurate work, make a blank titration by carrying chloride free distilled water through the procedure.

7. Calculation

Chloride (as Cl) = [(V1 - V2) x N x 35450] / V

Where:

V1 = Volume in ml of silver nitrate titrant used in sample,

V2 = Volume in ml of silver nitrate used in blank,

V3 = Volume in ml of sample used in the test, and

N = Normality of titrant.

(D) Automated Ferricyanide Method

3. Principle: Thiocyanate ion is liberated from mercuric thiocyanate by the formation of soluble mercuric chloride. In the presence of ferric ion, free thiocyanate ion forms highly coloured ferric thiocyanate, of which the intensity is proportional to the chloride concentration.

4. Apparatus

4.1 Automated analytical equipment: The required continuous flow analytical instrument consists of the interchangeable components as shown in Fig. 4.6.3.

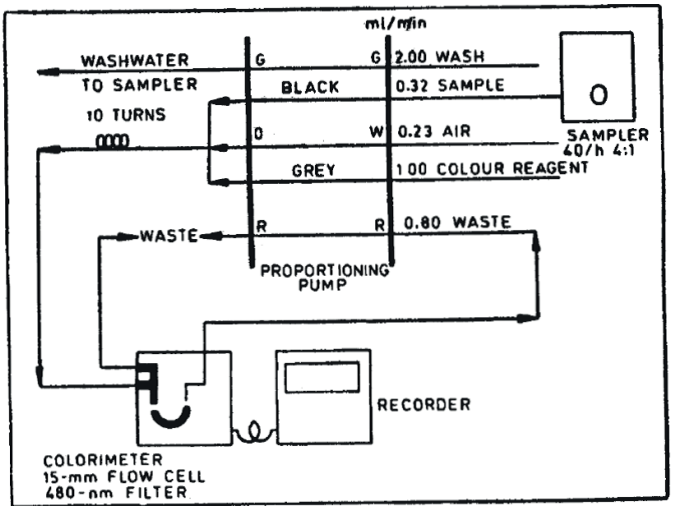


Fig. 4.6.3: Automated analytical equipment

4.2 Filters: 480 nm.

5. Reagents

5.1 Stock mercuric thiocyanate solution: Dissolve 4.17 g of mercuric thiocyanate in about 500 ml of methanol, dilute to 1000 ml with methanol, mix and filter through filter paper.

5.2 Stock ferric nitrate solution: Dissolve 202 g of ferric

nitrate [Fe $(NO_3)^3$. g H_2O] in about 500 ml of distilled water, then carefully add 21 ml of concentrated nitric acid. Dilute to 1000 ml with distilled water and mix. Filter through paper and store in coloured bottle.

5.3 Colour reagent: Add 150 ml stock mercuric thiocyanate solution to 150 ml of stock ferric nitrate solution. Mix and dilute. to 1000 ml with distilled water. Add 0.5 ml of polyoxymethylene 23 lauryl ether.

5.4 Stock chloride solution: Dissolve 1.648 2 g sodium chloride, dried at 140°C in distilled water and dilute to 1000 ml, 1.00 ml = 1.00 mg of chloride.

5.5 Standard chloride solutions: Prepare chloride standards in the desired concentration range, such as 1 to 200 mg/l, using stock chloride solution.

6. Procedure: Set up manifold as shown in Fig. 4.6.3 and follow general procedure prescribed by the manufacturer.

7. Calculation: Prepare standard curves by plotting peak heights of standards processed through the manifold against chloride concentrations in standards. Compute sample chloride concentration by comparing sample peak height with standard curve.

4.7 Sulphates in Water

1. Introduction: Sulphates are chemical salts, which dissolve in water forming solution. Solid sulphates do not react with concrete severely, but when in water solution they penetrate into the porous mass of concrete and react with the hydrated products of cement. Magnesium sulphate is more reactive and causes maximum damage to the concrete than others. Magnesium sulphate decomposes the hydrated calcium silicates as well as $Ca(OH)_2$ and hydrated tricalcium aluminate (C_3A). Eventually it forms hydrated magnesium silicate, which has no binding properties. The sulphate attack or reaction is indicated by the characteristic whitish appearance on the surface. In the hardened concrete calcium aluminate hydrate (C_3A) can react with sulphate salt from outside through pores. The product of reaction is calcium sulpho-aluminate, forming within frame work of hydrated cement paste. Due to this reaction the volume of solid phase increases upto 227%, causing gradual disintegration of concrete. The extent of sulphate attack depends on the concentration of sulphate solution and permeability of concrete. As per IS:456-2000, the Sulphate content in the water used for concrete mixing has to be limited to 400 mg/l.

2. Reference: IS-3025(Part-24/Sec-1):2022 "Method of Sampling and Test (Physical and Chemical) for Water and Wastewater. Part-24: Sulphates. Section-1: Gravimetric and turbidity methods".

(A) <u>Gravimetric Method</u>

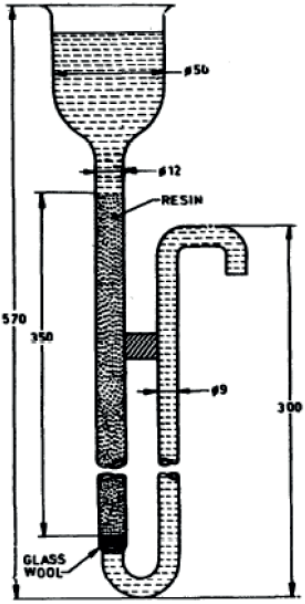
3. Applicability: This method is applicable for all the waters having sulphate concentrations above 10 mg/l; however, it is a time-consuming method.

4. Principle: Sulphate is precipitated in hydrochloric acid medium as barium sulphate by the addition of barium chloride solution. The precipitation is carried out near boiling temperature and after a period of digestion, the precipitate is filtered and washed with distilled water until

free of chlorides. It is then ignited or dried and weighed as barium sulphate ($BaSO_4$). The reaction in its simplest form is:

5. Sampling and Storage: Sampling and storage shall be done as prescribed in IS:3025 (Part 1). Highly polluted or contaminated samples should be stored at low temperature or treated with formaldehyde. Sulphite may be oxidized to sulphate by dissolved oxygen above pH 8.0; samples containing sulphite should have their pH adjusted below this value.

6. Apparatus



(All dimensions are in mm)

Fig. 4.7.1: Ion exchange Column

6.1 Steam Bath

6.2 Drying Oven, equipped with thermostatic control.

6.3 Muffle Furnace, with heat indicator.

6.4 Desiccator

6.5 Analytical Balance, with least count of 0.1 mg.

6.6 Filter Paper, acid washed, ash-less hard finish filter paper sufficiently retentive for fine precipitates (preferably Whatman No. 42/equivalent).

6.7 Crucible, porous bottom silica or porcelain crucible with a maximum pore size of 5 microns.

6.8 Ion-Exchange Column, see Fig. 4.7.1 for details. The exchange column should be regenerated by passing hydrochloric acid solution after five or six samples have passed through the column, followed by washing with distilled water.

7. Reagents

7.1 Methyl Red Indicator: Dissolve 100 mg methyl red sodium salt in distilled water and dilute to 100 ml.

7.2 Hydrochloric Acid Solution (1:4): Dilute one volume of concentrated hydrochloric acid with four volumes of distilled water.

7.3 Barium Chloride Solution: Dissolve 100 g of barium chloride ($BaCl_2$. $2H_2O$) in 1 litre distilled water. Filter through a membrane filter or hard finish filter paper (1 ml of this reagent is capable of precipitating approximately 40 mg SO_4^{2-}).

7.4 Silver Nitrate : Nitric Acid Reagent: Dissolve 8.5 g of silver nitrate and 0.5 ml of nitric acid in 500 ml distilled water.

7.5 Ion Exchange Resin: Strong cation exchange resin, amberlite IR-120 or equivalent.

8. Sample preparation

8.1 The sample used for analysis should either be free from turbidity or filtered through 0.45 µm filter.

8.2 If the total cation concentration in the sample is more than 250 mg/l or if the total heavy metal ion concentration is more than 10 mg/l, pass the sample through a cation removing ion exchange column.

8.3 If the silica concentration exceeds 25 mg/l, evaporate the sample nearly to dryness in a platinum dish on a steam bath. Add 2 ml hydrochloric acid solution (see Para 7.2), tilt the dish and rotate it until the acid comes in contact with the residue; continue the evaporation to dryness. Complete the drying in an oven at 180°C and if organic matter is present, char over the flame of a burner. Moisten the residue with 2 ml distilled water and 2 ml hydrochloric acid solution (see Para 7.2) and evaporate to dryness on steam bath. Add 5 ml hydrochloric acid solution (see Para 4.6.2), take up the soluble residue in hot water and filter. Wash the insoluble silica with several small portions of hot distilled water. Combine the filtrate and washings.

9. Procedure

9.1 Adjust the clarified sample, treated if necessary to remove interfering agents, to contain approximately 100 mg of sulphate ion in 500 ml volume.

9.2 Add 2 to 3 drops of methyl red indicator solution (see Para 7.1). Add hydrochloric acid solution (see Para 7.2) drop by drop till an orange red colour appears. Lower concentrations of sulphate ion may be tolerated if it is impracticable to concentrate the sample to the optimum level, but in such cases it is better to fix the total volume at 150 ml after concentration on hot plate.

9.3 Heat the solution to boiling, while stirring gently, add warm barium chloride solution (see Para 7.3) slowly until precipitation appears to be complete, then add about 2 ml in excess. Digest the precipitate at 80-90 °C for at least 2 hour.

9.4 Filtration: Filter the precipitate through filter paper and wash the precipitate with small portion of warm distilled water until the washings are free of chloride ions as indicated by testing with silver nitrate-nitric acid reagent (see Para 7.4).

9.5 Transfer the filter paper along with the precipitate in the crucible and dry the precipitate and ignite at 800 °C for 1 hour.

9.6 Cool in a desiccator and weigh.

10. Calculation: The concentration of sulphates in the sample from the equation:

Sulphate concentration as mg/l BaSO4 = (m × 411.5) / V

Where:

m = Mass of $BaSO_4$, in mg; and

V = volume of sample, in ml.

(B) <u>Turbidity Method</u>

3. Applicability and Range

3.1 This method can be applied for all concentration ranges of sulphate; however readings are accurate for sample aliquots containing not more than 40 mg/l of SO_4^{2-}.

3.2 The minimum detectable limit is approximately 1 mg/l SO_4^{2-}.

4. Principle: Sulphate ion is converted into barium sulphate suspension under controlled conditions. This suspension can be measured by using a nephelometer or spectrophotometer and light absorbance is measured at different concentrations; sample concentrations may be evaluated from standard curve.

5. Sampling and Storage: Sampling and storage shall be done as prescribed in IS:3025 (Part 1). The bottles shall be capped tightly as soon as the sample is collected.

6. Apparatus

6.1 Magnetic Stirrer, constant stirring speed so that it can be held constant just below splashing. Use identical sizes and shapes of magnetic stirrer bars.



Fig. 4.7.1: Nephelometer



Fig. 4.7.2: Spectrophotometer

6.2 Photometer (use one of the following, given in order of preference):

(a) Nephelometer (Fig. 4.7.1)

(b) Spectrophotometer (Fig. 4.7.2), for use at 420 nm with light path of up to 5 cm.

6.3 Stopwatch, to be used if magnetic stirrer is not equipped with an accurate timer.

6.4 Measuring Spoon, capacity from 0.2 ml to 0.3 ml.

7. Reagents

7.1 Buffer Solution: Dissolve 30 g magnesium chloride ($MgCl_2$. $6H_2O$), 5g Sodium acetate ($CH_3COONa.3H_2O$), 1 g Potassium nitrate (KNO_3) and 20 ml acetic acid (CH_3COOH) in distilled water in a 500 ml volumetric flask and make up volume up to 1000 ml.

7.2 Standard Sulphate Solution: Dissolve 0.1479 g anhydrous Na_2SO_4 in distilled water and dilute to 1000 ml (1 ml = 100 µg SO_4^{2-}).

7.3 Barium Chloride : crystals, 20 to 30 mesh.

8. Procedure

8.1 Preparation of Calibration Curve: Take 1.0 ml, 5.0 ml, 10.0 ml, 20.0 ml and 40.0 ml of the standard sulphate solution (see Para 7.2) into 250 ml conical flasks and make up to the 100 ml mark with distilled water. Take 100 ml distilled water in a 250 ml flask for

preparing distilled water blank. The concentration of the standards corresponding to 1.0 ml, 5.0 ml, 10.0 ml, 20.0 ml and 40.0 ml standard sulphate solution (1 ml = 100 µg SO_4^{2-}) are 100 µg (1 mg/l), 500 µg (5 mg/l), 1000 µg (10 mg/l), 2000 µg (20 mg/l) and 4000 µg (40 mg/l) respectively. Add 10 ml of buffer solution (see Para 7.1) and mix using magnetic stirrer. While stirring, add a spoonful of $BaCl_2$ crystals (see Para 7.3) and begin timing immediately. Stir for 30 seconds at constant speed. After stirring has ended, pour the solution into absorbance cell. Measure turbidity at 420 nm against distilled water in 30 seconds interval for 5 minutes. Record the maximum reading obtained in the 5 min period. The corrected absorbance readings of the standard solutions are obtained by subtracting the absorbance readings of the standard solutions from that of distilled water blank. Plot the corrected absorbance readings of the standard solutions against concentrations.

8.2 Sample Analysis

8.2.1 Measure 100 ml sample, or suitable portion made up to 100 ml into a 250 ml conical flask. Prepare a distilled water blank by taking 100 ml distilled water in another 250 ml conical flask. Add 10 ml buffer solution (see Para 7.1) and mix using magnetic stirrer. Follow the next procedures as done for standard solutions. Measure the absorbance reading of sample and distilled water blank against distilled water as reference. The corrected absorbance reading of sample is obtained by subtracting absorbance reading of distilled water blank from that of sample.

8.2.2 For samples having turbidity or colour, prepare a sample blank by taking 100 ml of the turbid or colured sample in a 250 ml conical flask. Add 10 ml of buffer solution (see Para 7.1) and take absorbance reading at 420 nm (without

adding BaCl2) against distilled water as reference. The corrected absorbance reading of sample is obtained by subtracting the absorbance reading of distilled water blank and sample blank from the observed absorbance reading of sample.

9. Calculation: The concentration of sulphates in the sample from the equation:

Sulphate concentration (as SO_4^{2-}) = (A × 1000) / (V x k)

Where:

A = Corrected absorbance reading of sample;

k = Slope from calibration curve; and

V = Volume of sample taken (in ml).

If the sulphate content of sample is found to be greater than 10 mg/L, then the sample analysis is repeated using buffer solution A. A distilled water blank is prepared by using buffer solution A. The corrected absorbance reading of sample is obtained by subtracting absorbance reading of distilled water blank from absorbance reading of sample.

Chapter - 5

TESTS ON FRESH CONCRETE

Workability is the most commenely tested parameter for the fresh Concrete. It can be checked by three different methods, as listed below:

(5.1) Workability of Concrete:

(A) Slump Test

(B) Compacting Factor Test

(C) Vee-Bee Consistometer Test

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5.1 Workability of Concrete

1. General: Workability of Concrete is a broad term describing how easily freshly mixed concrete can be mixed, placed, consolidated, and finished with minimal loss of homogeneity. It is a property that directly impacts strength, quality, appearance, and even the cost of labour for placement and finishing operations. A concrete mix with good workability brings many attributes together and results in a quality product with long service life.

Another similar term used to describe freshly prepared concrete is "consistency" which is the ease with which it will flow. It is a measure of fluidity or mobility of concrete.

The consistency of the concrete can be determined by many methods, out of which three most commonly used methods (Slump Test, Compacting Factor Test and Vee-Bee Consistometer Test) are covered in this chapter. It is to be noted that all these three tests are not applicable for foam concrete, no fines concrete, or where the nominal maximum aggregate size exceeds 40mm. If the concrete contains coarse aggregate with size larger than 40mm, the concrete shall be wet-sieved through 40mm screen to exclude aggregate particles bigger than 40mm.

2. Reference: All these tests are conducted as per IS-1199 (Part-2):2018 "Fresh Concrete – Methods of Sampling, Testing and Analysis; Part-2 – Determination of Consistency of Fresh Concrete".

3. Sampling: Samples for all these three tests shall be obtained as per IS 1199(Part 1). Each sample shall be remixed before carrying out the tests. The sampling is to be done as below:

3.1 Terminology

3.1.1 Batch: Quantity of concrete, mixed in one cycle of operations of a batch mixer or the quantity of concrete conveyed ready mixed in a vehicle, or the quantity of concrete discharged

over 1 min from a continuous mixer.

3.1.2 Composite Sample: Quantity of concrete consisting of a number of increments, distributed through a batch or mass of concrete, which are thoroughly mixed together.

3.1.3 Spot Sample: Quantity of concrete taken from a part of a batch or mass of concrete, consisting of one or more increments that are thoroughly mixed together.

3.1.4 Increment: Quantity of concrete taken by the single operation of a scoop.

3.2 Taking a composite sample: Concrete is sampled from a stream of moving concrete or from a pile, in a series of increments. These increments are then thoroughly mixed together.

3.3 Taking a Spot Sample: Concrete is sampled from a stream of moving concrete or from a pile, at a single point. Spot samples are not representative of the batch and should not be used to cast strength specimens.

3.4 Apparatus

3.4.1 Scoop, made from non-absorbent material not readily attacked by cement paste, with a size suitable for taking increments of concrete.

3.4.2 Containers, one or more containers, made from non-absorbent material (preferably made of metal) not readily attacked by cement paste, for receiving, transporting and remixing the concrete samples.

3.4.3 Thermometer, (when required), to measure the temperature of fresh concrete to an accuracy of ±1°C.

3.5 Procedure: Whether a composite sample or spot sample is to be taken will depend on intended use of the sample. Spot samples are not representative of the batch and should not be used to make strength

specimens.

For samples to be used for strength test, a minimum quantity of 0.02 m3 will be essential. For other tests such as air content, temperature and determination of consistency, smaller size samples may suffice. The size of samples shall also depend upon the maximum size of aggregate.

3.5.1 Obtaining a Composite Sample: Ensure that the apparatus is clean and dampen it with a moist, but not wet, cloth prior to use. Using the scoop, take the required number of increments uniformly distributed throughout the batch. When sampling from a stationary batch mixer or ready-mixed concrete truck, disregard the very first and the very last of the discharge (about 10 to 15 percent). When sampling from a falling stream, the increments shall be taken in such a way as to represent the whole width and thickness of the stream. If the batch has been deposited in a heap of concrete, take the increments, wherever possible, distributed through the depth of the concrete as well as over the exposed surface. Increments shall not be taken from parts of the concrete that appear to be segregated. The increments shall be taken from at least four points. Deposit the increments into the container(s).

3.5.2 Obtaining a Spot Sample: Ensure that the apparatus is clean and dampen it with a moist, but not wet, cloth prior to use. Take the sample increment(s) by a scoop from the required part of a batch or mass of concrete. Deposit the increment(s) into the container(s).

3.5.3 Mixing, Transporting and Handling of Samples: The samples, obtained by either of the methods described above, shall be mixed thoroughly in non-absorbent container with shovel or by other suitable implement.

At all stages of sampling, transporting and handling, care shall be taken to protect the fresh concrete samples against contamination, increase or loss of moisture, excessive vibration, and against extreme variations of temperature.

The properties of fresh concrete change with time after mixing, depending upon climatic conditions, more so if the concrete contains admixture. This should be taken into account in deciding when test specimens are made and when tests are carried out.

It is recommended that the tests for slump, temperature, and air content should start within 5 min after obtaining the final portion of the composite sample. Complete these tests expeditiously.

3.5.4 Measuring the Temperature of the Sample: Whenever required, measure the temperature of the concrete in the container(s) at the time of sampling.

3.6 Sampling Record: The following information regarding the samples shall be included in the sample report:

(a) Clear identification of the sample (sample number),

(b) Type of sample (composite or spot),

(c) Date and time of sampling,

(d) Type and grade (if applicable) of cement and admixtures (if used),

(e) Identification of the works which the sample represents,

(f) Identification of the batch or truck mixer sampled,

(g) Ambient temperature,

(h) Temperature of the concrete sample (when

required),

(j) Any deviations from the standard method of sampling,

(k) A declaration by the person responsible for sampling that the sample was obtained in accordance with this Indian standard, except as noted in (j), and

(m) Name and signature of person responsible for sampling.

(A) By Slump Test

1. Introduction: It is the most common method for measuring the workability of freshly mixed concrete. The fresh concrete is compacted into a mould in the shape of a frustum of a cone. When the cone is withdrawn upwards, the distance that the concrete has slumped provides a measure of the consistency of the concrete.

It can be performed both in lab and at site. Uniformity of the concrete regarding workability and quality aspects can be assessed from batch to batch by observing the nature in which the concrete slumps.

The slump test is applicable to a range of consistency of concrete that corresponds to slumps between 10mm and 210mm. Outside this range, other method of determination of consistency should be considered.

2. Apparatus required

(i) Mould: It shall be made of metal, not readily attacked by cement paste and having at least 1.6mm thickness. The mould shall have a smooth internal surface and free from dents. It shall be in the form of a hollow frustum of a cone and shall have the following internal dimensions:

(a) Diameter of base: 200 ± 2 mm

(b) Diameter of top: 100 ± 2 mm

(c) Height: 300 ± 2 mm

The base and the top shall be open and parallel to

each other and at right angle to the axis of the cone. The mould shall be provided, on the upper portion, with two handles at two-thirds of the height, and at the bottom with fixing clamps or foot pieces to hold it steady. A typical mould without guide is shown in Fig. 5.1.1.

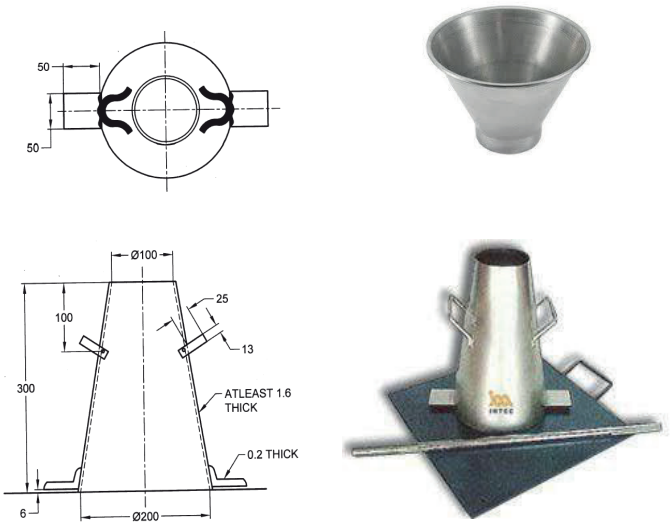


Fig. 5.1.1: Slump Test Mould and Funnel

The mould shall be visually inspected prior to each use to ensure that the interior is clean and free from concrete deposits.

The mould shall be checked annually to ensure that its dimensions and condition remain within specified tolerances.

(ii) Tamping rod: Made of steel, having a circular cross-section with a diameter of 16 ± 1mm, 600 ± 5mm in length and with rounded ends. The rod may be extended with a handle of plastic conduit, provided that the overall length does not exceed 1000mm. The tamping rod shall be checked annually to ensure that its dimensions and condition remain within the tolerances.

(iii) Funnel (optional): Made of non-absorbent material, not readily attacked by cement paste. The funnel shall consist of two coaxial conical frustums having a common diameter of 100 ± 2mm, the ends being of greater diameter, one frustum to act as a filling funnel and the other as a collar to enable the funnel to be located on the outer surface of the mould (Fig. 5.1.1). The funnel shall be checked annually to ensure that its dimensions and condition remain within tolerances.

(iv) Rule: Graduated from 0mm to 300mm, at intervals not exceeding 5mm, with zero point being at extreme end of the rule.

(v) Base plate/surface: It shall be rigid, flat, non-absorbent and smooth plate on which the mould is to be placed.

(vi) Shovel: It shall have a square blade.

(vii) Re-mixing tray: It shall be of good construction and fabricated from a non-absorbent material, not readily attacked by cement paste. It shall be of appropriate dimensions such that the concrete can be thoroughly remixed, using the square bladed shovel.

(viii) Scoop: It shall be of adequate size.

(ix) Timer: It shall allow time measurement accurate to 1s. The timer shall be calibrated at suitable frequency.

3. Procedure

(a) Dampen the mould and base plate with a moist cloth. Wipe any excessive water from the surfaces using an absorbent cloth. Place the mould on the surface of the rigid horizontal base plate free from extraneous vibration and shock.

(b) Immediately after obtaining the sample, fill the mould in three layers, each approximately one-third of the height of the mould when compacted. During filling, clamp or hold the mould firmly in place by

standing on the two foot pieces.

(c) When filling the mould, ensure that the concrete is distributed symmetrically around the mould.

(d) Tamp each layer with 25 strokes of the tamping rod. The strokes shall be distributed in an uniform manner over the cross-section area of the mould. For the bottom layer, this will necessitate inclining the rod slightly and positioning approximately half the strokes spirally towards centre.

(e) Tamp the second layer and top layer each throughout its depth, so that the strokes just penetrate into the underlying layer.

(f) In filling and tamping the top layer, heap the concrete above the mould before the tamping is started.

(g) If the tamping operation of the top layer results in subsidence of the concrete below the top edge of the mould, add more concrete to keep an excess above the top of the mould at all times. Also ensure that the addition of concrete to the top layer does not provide extra compaction of the concrete.

(h) After the top layer has been tamped, scrap off the surface of the concrete level with the top of the mould by means of a sawing and rolling motion of the tamping rod. Remove spilled concrete from the base plate/surface.

(j) Remove the mould in 5 ± 2s by a straight upward push with no lateral or torsional motion being imparted to the concrete.

(k) The entire operation from the start of the filling to the removal of the mould shall be carried out without interruption and shall be completed within 180 s.

(l) Immediately after removal of the mould, determine the slump, h, by measuring the difference between the height of the mould and the highest point of the slumped concrete (see Fig. 5.1.2).

(m) Measure slump to the nearest 5mm.

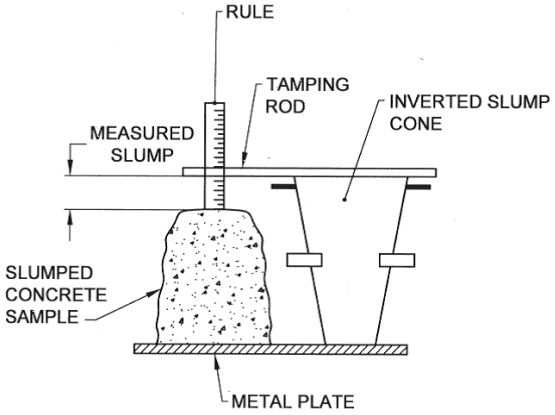


Fig. 5.1.2: Slump Measurement

4. Test Results: The test is valid only if it yields a true slump, this being a slump in which the concrete remains substantially intact and symmetrical as shown in 3A of Fig. 5.1.3. If a specimen shears as shown in 3B of Fig. 5.1.3 or collapses as shown in 3C of Fig. 4.1.3, take another sample and repeat the entire procedure as described above.

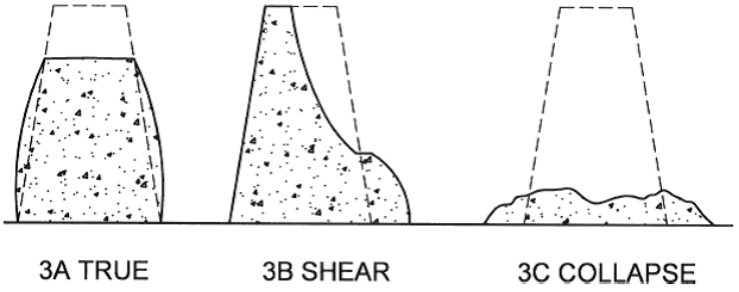


Fig. 5.1.3: Types of Slump

Record the true slump, to the nearest 5mm.

If two consecutive test show portion of concrete shearing of from the mass of the test specimen or collapsing, report the test as being invalid as the concrete lacks the

necessary plasticity and cohesiveness for the slump test to be suitable.

5. Test Report: The test report shall also include the following:

(a) Slump, if there is a true slump, measured to the nearest 5mm; or

(b) A notation that the test gave a sheared/collapsed slump.

(B) By Compacting Factor Test

1. Introduction: This test is generally carried out in laboratory but can be used in site also. It is particularly useful for concrete mixes of very low workability (or very dry concrete), with the nominal maximum size of the aggregate not exceeding 40mm, as they are insensitive to slump test. It is more precise and sensitive than the slump test.

This test works on the principle of determining the degree of compaction achieved by a standard amount of work done by allowing the concrete to fall through a standard height.

2. Apparatus: A diagram of the apparatus is shown in Fig. 5.1.4. It shall consist of the two conical hoppers (A and B) mounted above a cylindrical mould (C).

The essential dimensions of the hoppers and mould and distances between them shall be as shown in Table 5.1.

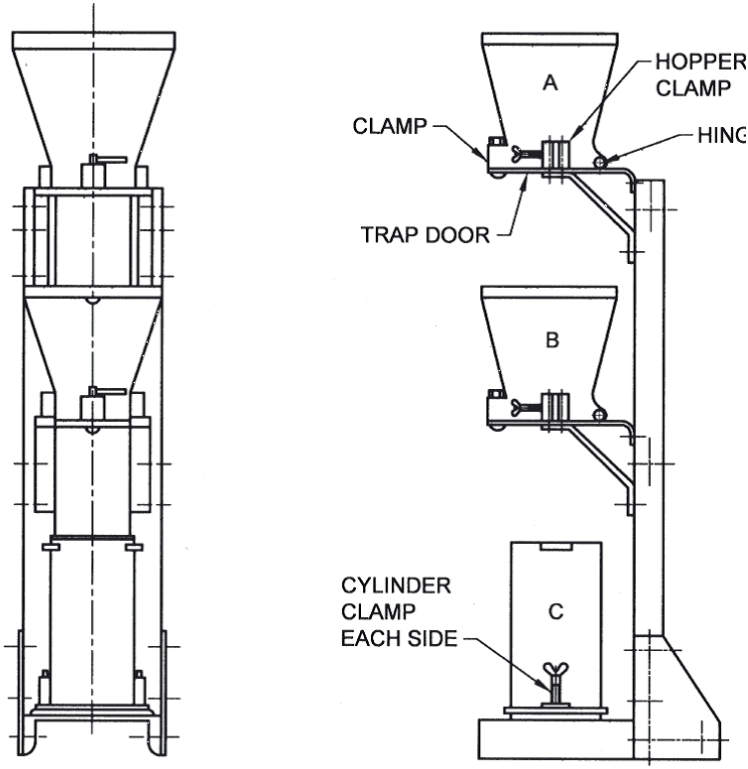


Fig. 5.1.4: compaction Factor Apparatus

Table 5.1: Dimensions of Compaction Factor Apparatus

Sl. No.	Details (see Fig. 4.1.5)	Dimension (mm)
(i)	Upper hopper, A:	
	(a) Top internal diameter	250
	(b) Bottom internal diameter	125
	(c) Internal height	280
(ii)	Lower hopper, B:	
	(a) Top internal diameter	230

	(b) Bottom internal diameter	125
	(c) Internal height	230
(iii)	Cylinder, C:	
	(a) Internal diameter	150
	(b) Internal height	300
(iv)	Distance between bottom of upper hopper and top of lower hopper	200
(v)	Distance between bottom of lower hopper and top of cylinder	200

The hopper and cylinder shall be of rigid construction, true to shape and smooth inside. They shall preferably be made of cast brass or bronze, but stout sheet brass or steel may also be considered satisfactory provided the inside surfaces of the joints are smooth and flush. The lower ends of the hoppers shall be closed with tightly fitting hinged trap-doors having quick release catches. Metal plate 3mm thick is suitable for the doors. The frame in which the hoppers and cylinder are mounted shall be of rigid construction and shall firmly locate them in the relative positions indicated in Table 4.1. The cylinder and hoppers shall be easily detachable from the frame.

The apparatus shall also include two ordinary trowels, one scoop of adequate size, a tamping rod made of steel, having a circular cross-section with a diameter of 16 ± 1mm, 600 ± 5mm in length and with rounded ends, and scales (or a balance) to weigh up to 50kg, with an accuracy to the nearest 10g.

3. Sampling: The sample shall be taken as per Para 3 above. In the case of concrete containing aggregate of maximum size more than 40mm, the concrete shall be wet sieved through 40mm screen to exclude aggregate particles bigger than 40mm.

4. Procedure: Dampen the inside of the hopper and cylinder with a moist cloth before its use. The sample of concrete to be tested shall be placed gently in the upper hopper, using the scoop. The hopper shall be filled level with its brim and the trap-door shall be opened so that the concrete falls into the lower hopper. Certain mixes have a tendency to stick in one or both of the hoppers. If this occurs, the concrete may be helped through by pushing the rod gently into the concrete from the top. During this process, the cylinder shall be covered by the trowels. Immediately after the concrete has come to rest, the cylinder shall be uncovered, the trap-door of the lower hopper opened, and the concrete allowed to fall into the cylinder. The excess of concrete remaining above the level of the top of the cylinder shall then be cut off by holding a trowel in each hand, with the plane of the blades horizontal, and moving them simultaneously one from each side across the top of the cylinder, at the same time keeping them pressed on the top edge of the cylinder. The outside of the cylinder shall then be wiped clean. The above operation shall be carried out at a place free from vibration or shock. The weight of the concrete in the cylinder shall then be determined to the nearest 50g. This weight shall be known as the weight of partially compacted concrete. The cylinder shall be refilled with concrete from the same sample in layers approximately 5cm deep, the layers being heavily rammed or preferably vibrated so as to obtain full compaction. The top surface of the fully compacted concrete shall be carefully struck off level with the top of the cylinder. The outside of the cylinder shall then be wiped clean. The weight of the concrete in the cylinder shall then be determined to the nearest 50g. This weight shall be known as the weight of fully compacted concrete.

Note: The test is sufficiently sensitive to enable differences in workability arising from the initial processes in the hydration of the cement to be measured. Each test, therefore, should be carried out at a constant time interval after the mixing is completed, if strictly comparable

results are to be obtained.

5. Calculation: The compacting factor is defined as the ratio of the weight of partially compacted concrete to the weight of fully compacted concrete. It shall normally be stated to the nearest second decimal place.

6. Test Report: The test report shall include the compacting factor value, expressed to the nearest two decimal places.

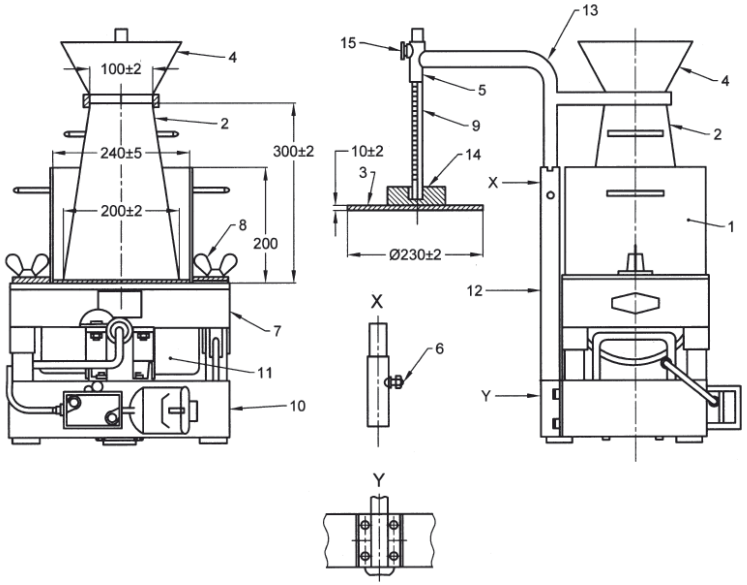
(C) By Vee-Bee Consistometer Test

1. Introduction: Vee-Bee test is a good laboratory test suitable for stiff concrete mixes having low and very low workability. The Vee-Bee consistometer test determines the time required for transforming, by vibration, a concrete specimen in the shape of a conical frustum into a cylinder. The fresh concrete is compacted into a slump mould. The mould (cone) is lifted of the concrete and a transparent disc is swung over the top of the concrete and carefully lowered until it comes in contact with the concrete. The slump of the concrete is recorded. The vibrating table is started and the time taken for the lower surface of the transparent disc to be fully in contact with cement paste of the concrete is measured.

If the Vee bee time is less than 5 s or more than 30 s, the use of the test method to determine consistency may be unsuitable and other methods should be considered for this purpose (modified Vee bee consistometer test can be used for testing of very stiff dry concretes such as roller compacted concrete).

2. Apparatus

2.1 Consistometer (Vee-Bee meter) consists of the following items as shown in Fig. 5.1.5:



(All dimensions are in mm)

Fig. 5.1.5: Vee Bee Consistometer

Key:

1 container	6 set-screw	11 vibrator unit
2 mould	7 vibrating table	12 holder
3 transparent disc	8 wing nuts	13 swivel arm
4 funnel	9 rod	14 weight
5 guide sleeve	10 base for vibrator	15 screw

(a) Container (see 1 in Fig. 5.1.5): It is cylindrical in shape, having an internal diameter of 240 ± 5mm and a height of 200 ± 2mm and made of a metal, not readily attacked by cement paste. The thickness of wall shall be 3mm and that of the base 7mm. The container shall be watertight and of sufficient rigidity to retain its shape under rough usage. It shall be fitted with handles and protected from corrosion. The container shall be

provided with suitable foot pieces to enable it to be securely clamped to the top of the vibrating table (see 7 in Fig. 5.1.5) by means of wing nuts (see 8 in Fig. 5.1.5).

(b) Mould (see 2 in Fig. 5.1.5), same as for the Slump Test, except that the fixing clamps or foot pieces are not required. The mould shall be visually checked prior to each use to ensure that it is clean and not damaged or dented.

(c) Disc (see 3 in Fig. 5.1.5): A transparent and horizontal disc attached to a rod (see 9 in Fig. 5.1.5) that slides vertically through a guide sleeve (see 5 in Fig. 5.1.5) mounted on swivel arm (see 13 in Fig. 5.1.5) and which can be fixed in a position by a screw (see 15 in Fig. 5.1.5). The swivel arm also supports a funnel (see 4 in Fig. 5.1.5), the bottom of which coincides with the top of the conical mould when the latter is positioned concentrically in the container. The swivel arm is located by a hold (see 12 in Fig. 5.1.5) and can be fixed in a position by set screw (see 15 in Fig. 5.1.5). When in the proper position, the axes of the rod and of the funnel shall be coincident with the axis of the container.

The transparent disc shall be 230 ± 2mm in diameter and 10 ± 2mm in thickness. A weight placed directly above the disc shall be provided such that the moving assembly consisting of the rod, the disc and the weight has a mass of 2 750 ± 50g. The rod shall be provided with a scale graduated to at least 5mm intervals to record the slump of the concrete.

(d) Vibrating table: It shall be 380 ± 3mm in length and 260 ± 3mm in width, supported on four rubber shock absorbers.

A vibrator unit (see 11 in Fig. 5.1.5), carried on a base resting on three rubber feet, shall be securely fixed beneath it. The vibrator shall

operate at a frequency of 55 ± 5.5 Hz and the vertical amplitude of vibration of the table with empty container on the top of it shall be approximately 0.5 ± 0.02mm.

The vibrating table shall be checked annually to ensure that the frequency and the vertical amplitude remain within tolerances. All the elements of the vibration table shall be checked annually to ensure that their dimensions remain within tolerances.

3.2 Tamping rod: It is a straight rod, made of steel or other suitable material, of circular cross-section, having a diameter of 16 ± 1mm, 600 ± 5mm in length, and with rounded ends.

3.3 Stopwatch or clock, capable of recording time to an accuracy of 1 s.

3.4 Remixing container: It is of rigid construction, made from non-absorbent material not readily attacked by cement paste.

3.5 Scoop, of adequate size.

3. Procedure

(a) Place the Vee-Bee meter (consistometer) on a rigid horizontal base free from extraneous vibration and shock. Make sure that the container is firmly fixed to the vibrating table by means of wing nuts. Dampen the inside of the mould with a moist cloth and place it in the container. Swing the funnel into position over the mould and lower the funnel on the mould. Tighten the screw so that the mould cannot rise from the bottom of the container. During the subsequent operations ensure that the mould does not rise or move until it is manually raised and ensure that concrete has not fallen into the container. From the sample of concrete, immediately fill the mould in three layers, each approximately one-third of the height of the mould when compacted. When adding the concrete, ensure that it is distributed

symmetrically around the mould.

(b) Tamp each layer with 25 strokes of the tamping rod. The strokes shall be distributed in a uniform manner over the cross-section area of the mould. For the bottom layer, this will necessitate inclining the rod slightly and positioning approximately half the strokes spirally towards centre. Tamp the second layer and top layer each throughout its depth, so that the strokes just penetrate into the underlying layer. In filling and tamping the top layer, heap the concrete above the mould before tamping is started. If necessary, add further concrete to maintain excess above the top of the mould throughout the tamping operation. After the top layer has been tamped, loosen the screw, raise and swing the funnel through 90° and tighten the screw. Scrape off the concrete level with the top of the mould with a sawing and rolling motion of the tamping rod. Lift the mould from the concrete

by raising it carefully in a vertical direction, using the handles. The operation of raising the mould shall be performed in 7 ± 2s by a steady upward lift with no lateral or torsional motion being imparted to the concrete. If the concrete shears, collapses or slumps to the extent that it touches the wall of the container, this information shall be recorded. Swing the transparent disc over the top of the concrete, loosen the screw and very carefully lower the disc until it just comes in contact with the concrete. When the disc just touches the highest point of the concrete without disturbing it, tighten the screw. When there is a true slump, the value of the slump shall be read from the scale and the value recorded. The screw shall be loosened to allow the disc to follow the concrete as it settles under the subsequent vibration. Simultaneously, start the vibration of the table and the timer. Observe through the transparent disc how the concrete is being re-moulded. As soon as the lower surface of the disc is fully in contact

with cement paste of the concrete, stop the timer and switch off the vibrating table. Record the time taken to the nearest second. Complete the procedure within a period of 5 min from the start of filling.

The consistency of the concrete mix changes with the time due to hydration of the cement and possibly, loss of moisture. Tests on different samples should, therefore, be carried out at a constant time interval after mixing, if strictly comparable results are to be obtained.

4. Test Result: Record the time read from the stopwatch to the nearest second. This is the Vee bee time which indicates the consistency of the concrete under test.

5. Test Report: The test report shall include the following:

(a) Time from completion of mixing of the concrete until the time of removal of the mould; and

(b) Vee bee time, in second.

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Chapter - 6

TESTS ON HARDENED CONCRETE

Following are the most commenly performed tests on hardened concrete :

(6.1) Compressive Strength of Concrete

(6.2) Flexural Strength of Concrete

(6.3) Splitting Tensile Strength of Concrete

(6.4) Permeability of Concrete

- (A) As per IS:3085
- (B) As per IRS:CBC

(6.5) Rebound Hammer Test

(6.6) Ultrasonic Pulse Velocity Test

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6.1 Compressive Strength of Concrete

1. Introduction: Concrete gains strength with time, after pouring and casting. It takes very long time to gain 100% strength and it often remains unknown. The rate of gain of compressive strength is higher during the first 28 days of casting, which gradually slows down. Therefore, compressive strength is checked after 28 days. The Characteristic Strength of Concrete (fck) is defined as the compressive strength of concrete cubes (cast as per relevant code of practice, cured for 28 days and then tested) below which not more than 5% of the test results are expected to fall.

2. Reference: IS-516 (Part-1/Sec-1) : 2021 "Hardened Concrete – Methods of Test. Part-1: Testing of Strength of Hardened Concrete. Section-1: Compressive, Flexural and Split Tensile Strength".

3. Test Specimen

3.1 The test specimen shall be a cube or a cylinder meeting the requirements of IS:1199 (Part 5) and IS:516 (Part 4) for concrete cores. The standard cube and cylinder specimen shall not be tested if they are badly honeycombed as this is an indication of poor specimen making. When such specimens are tested, the test report shall include the fact that the specimen was honeycombed.

3.2 Age at Test: Tests shall be made at recognized ages of the test specimens, the most usual being 7 and 28 days.

3.3 Number of Specimens: At least three specimens shall be tested at each selected age.

4. Apparatus required: The test shall be carried out using a compression testing machine conforming to IS:14858. The test machine shall be in calibration at the time of test. The calibration shall be carried out at least once per year.

5. Procedure:

5.1 Preparation and Positioning of Specimens: For specimens stored in water, excess moisture shall be wiped from the surface of the specimen. The dimensions of the specimens to the nearest 0.2 mm and their weight shall be noted before testing. The time between the extraction of the specimen from the curing tank, and the testing, shall be as short as possible or not more than 2 hour. During the time the specimen is outside the curing tank, it shall be protected from drying, may be by covering with wet cloth.

All testing machine bearing surfaces shall be wiped clean and any loose grit or other extraneous material removed from the surfaces of the specimen that will be in contact with the platens.

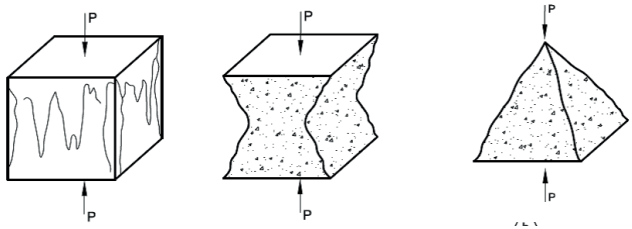
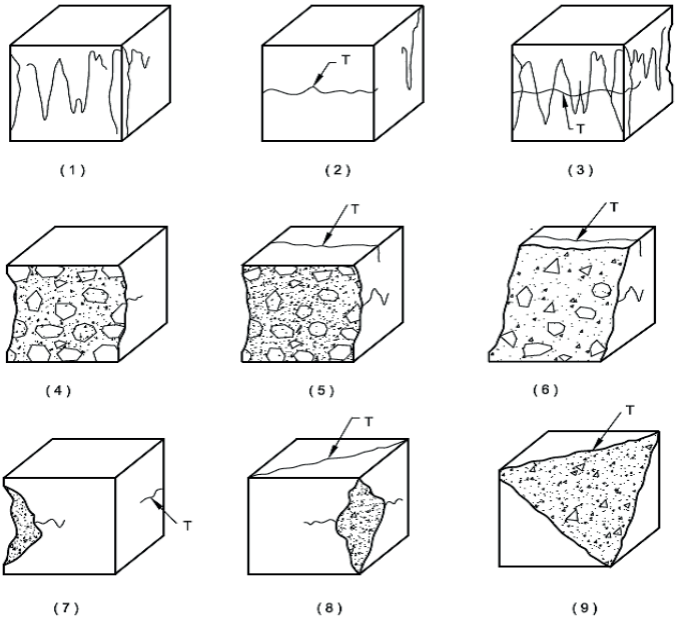


Fig. 6.1.1: Satisfactory Failure of Cube Specimen

Cube specimens shall be compressed perpendicularly to the direction of casting. The specimen shall be centered on the lower platen to an accuracy of 1 percent of the designated size of cubic, or diameter of cylindrical specimens.

5.2 Loading: The load shall be applied without shock and shall be increased continuously at a constant rate of 14 N/mm^2/min until no greater load can be sustained. The maximum load indicated shall be recorded.



Note T = Tensile Crack

Fig. 6.1.2: Unsatisfactory Failure of Cube Specimen

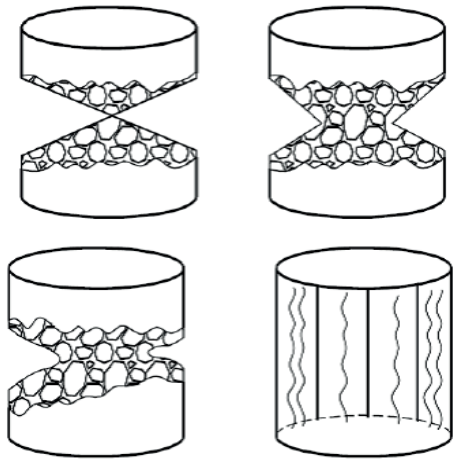


Fig. 6.1.3: Satisfactory Failure of Cylindrical

Specimen

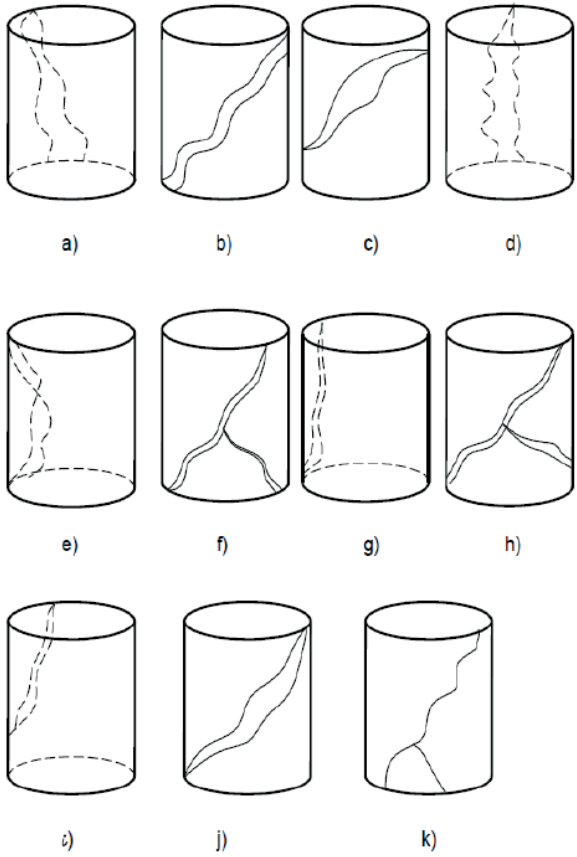


Fig. 6.1.4: Unsatisfactory Failure of Cylindrical Specimen

5.3 Assessment of Type of Failure: If the failure is satisfactory (see Fig. 6.1.1 or Fog. 6.1.3), this fact shall be recorded. If the failure pattern is unsatisfactory, this fact shall be recorded and the type of failure recorded using the pattern number in Fig. 6.1.2 or Fig. 6.1.4 closest to that observed.

6. Test Result: The compressive strength is given by the equation:

$$fc = F / Ac$$

where:

fc = compressive strength, in MPa;

F = maximum load, in N; and

Ac = cross-sectional area, in mm^2, of the specimen on which the compressive force acts.

Average of three values shall be taken as the representative of the batch provided the individual variation is not more than ± 15 percent of the average. Otherwise repeat test shall be made, however if there is no further sample, then the average of two closest values may be taken as the average result.

The actual dimensions of test specimens shall conform to IS:10086. If the actual dimensions are within the tolerance limits as mentioned in IS:10086, the strength may be calculated on the basis of designated size. If the actual dimensions are outside this tolerance, the strength calculation shall be based on the actual dimensions of the test specimen, however, perpendicularity of the surface of specimens should be maintained as per IS:10086.

The compressive strength shall be expressed to the nearest 0.5 MPa.

7. Test Report: The following information shall be included in the report:

(a) Details of the concrete like grade, mix details, etc, in case of cast specimens; and details of the structure, like structure type, origin member/ structure, in case of cored specimen;

(b) Type of specimen: cast (cube/cylinder) or drilled core;

(c) Size of the specimen, and capping details, if applicable;

(d) Identification mark;

(e) Age of specimen;

(f) Date of test;

(g) Curing conditions;

(h) Weight of specimen;

(j) Dimension of specimen;

(k) Maximum load;

(m) Details of the machine used for testing (manual/ automated, loading range, date of calibration, etc);

(n) Compressive strength of specimen (to the nearest 0.5 MPa); and

(p) Type of failure (satisfactory or unsatisfactory and, if unsatisfactory, the nearest type).

6.2 Flexural Strength of Concrete

1. Introduction: Flexural strength of Concrete, also known as Modulus of rupture, is an indirect measure of the tensile strength of unreinforced concrete. Apart from external loading, tensile stresses can also be caused by warping, corrosion of steel, drying shrinkage and temperature gradient. Designers of pavements use a theory based on flexural strength. Therefore, laboratory mix design based on flexural strength tests may be required, to obtain the needed design MR.

Unlike compression, tensile strength of a member cannot be found directly as no apparatus or specimen model has been developed to evenly distribute the tensile force to the member. However, the indirect measurement of the flexural strength like the One-point loading test and the Two-point loading test fetch satisfying results.

2. Reference: IS-516 (Part-1/Sec-1) : 2021 "Hardened Concrete – Methods of Test. Part-1: Testing of Strength of Hardened Concrete. Section-1: Compressive, Flexural and Split Tensile Strength".

3. Test Specimen: The test specimen shall be a prism conforming to IS 1199 (Part 5). Sawn specimens of nominal width of 100mm or 150mm with a square cross-section and overall length of 500mm and 700mm may also be used.

3.1 Age at Test: Tests shall be made at recognized ages of the test specimens, the most usual being 7 and 28 days. Ages of 90 days and 1 year are recommended if tests at greater ages are required.

3.2 Number of Specimens: At least three specimens shall be tested at each selected age.

4. Apparatus

4.1 Testing Machine: The permissible errors shall be not greater than ± 1 percent of the applied load. The bed of the testing machine shall be provided with

two steel rollers, 38mm in diameter, on which the specimen is to be supported, and these rollers shall be so mounted that the distance from centre to centre is 600mm for 150mm specimens and 400mm for 100mm specimens. The load shall be applied through two similar rollers mounted at the third points of the supporting span, that is, spaced at 200 or 133mm respectively centre-to-centre. The load shall be divided equally between the two loading rollers, and all rollers shall be mounted in such a manner that the load is applied axially and without subjecting the specimen to any torsional stresses or restraints. Each roller, except one of the lower ones shall be capable of rotating around its axis and of being inclined in a plane normal to the longitudinal axis of the test specimen.

4.2 Force Application: The device for applying loads shall consist of two upper rollers and two lower rollers (see Fig. 6.2.1).

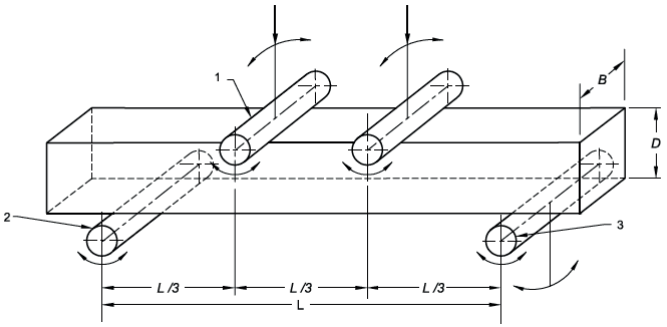


Fig. 6.2.1: Test Setup

Key:

1. Loading Roller (Capable of Rotation and being inclined)

2. Supporting Roller

3. Supporting Roller (Capable of Rotation and being inclined)

5. Procedure

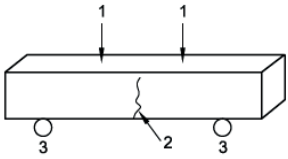
5.1 Preparation and Positioning of Specimens: The specimen shall be examined and any abnormalities shall be reported. For specimens stored in water, excess moisture shall be wiped from the surface of the specimen before placing in the testing machine. The time between the extraction of the specimen from the curing tank until the test shall be as short as possible or not more than 2 hour. During the time the specimen is outside the curing tank, it shall be protected from drying, like by covering with wet cloth. The test specimen shall be placed in the machine, correctly centred with the longitudinal axis of the specimen at right angles to the longitudinal axis of the upper and lower rollers. The reference direction of loading shall be perpendicular to the direction of casting of the specimen.

5.2 Loading: The bearing surfaces of the supporting and loading rollers shall be wiped clean, and any loose sand or other material removed from the surfaces of the specimen where they are to make contact with the rollers. The specimen shall then be placed in the machine in such a manner that the load shall be applied to the uppermost surface as cast in the mould.

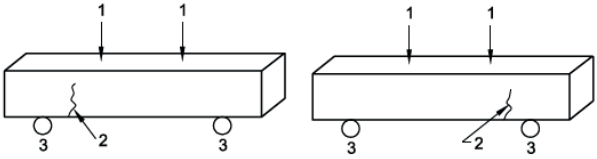
The load shall not be applied until all loading and supporting rollers are resting evenly against the test specimen. The load shall be applied without shock and shall be increased continuously at a constant rate until no greater load can be sustained. The load shall increase at a rate 0.7 N/mm^2/min (rate of loading being 4 kN/min for 150mm specimens and 1.8 kN/min for 100mm specimens).

The maximum load indicated shall be recorded.

5.3 Assessment of Type of Fracture: The fractured specimen shall be examined and the appearance of the concrete and type of fracture shall be recorded (see Fig. 6.2.1).



Failure Type A



Failure Type B

Fig. 6.2.1: Types of Failure Pattern

Key:

1. Loading Rollers
2. Line of Fracture
3. Supporting Rollers

6. Test Results: The flexural strength of the specimen shall be expressed as "Modulus of Rupture" Fb and shall be calculated to the nearest 0.05 MPa as follows in case of failure Type A (Fig. 6.2.1):

$$F_b = (P \times L) / B \times D^2$$

in which 'a' is greater than 200mm for 150mm specimen, or greater than 133mm for 100mm specimen, or in case of failure Type B (Fig. 6.2.1), "Modulus of Rupture" Fb shall be calculated to the nearest 0.05 MPa by the following formula:

$$F_b = (3P \times a) / B \times D^2$$

in which 'a' is less than 200mm but greater than 170mm for 150mm specimen or less than 133mm but greater than 110mm for 100 mm specimen.

Where:

F_b= Flexural Strength, in MPa;

P= Maximum load, in N;

a= the distance between the line of fracture and the nearer support, measured on the centre line of the tensile side of the specimen, in mm;

B, D are the lateral dimensions (breadth and height) of the specimen, mm; and

L= Length of span on which the specimen is supported, expressed in mm.

If 'a' is less than 170mm for 150mm or less than 110mm for a 100mm specimen, the results of the test shall be discarded.

The flexural strength shall be expressed to the nearest 0.05 MPa.

7. Test Report: The following information shall be included in the report:

(a) Size of specimen;

(b) Identification mark;

(c) Age of specimen;

(d) Date of test;

(e) Details of concrete mix such as grade, cement content, curing condition, etc;

(f) Weight of specimen;

(g) Maximum load;

(h) Type of fracture and flexural strength of specimen (to the nearest 0.05 MPa);

(j) In case of failure Type B, value of 'a'.

6.3 Splitting Tensile Strength of Concrete

1. Introduction: One of the important properties of concrete is "tensile strength" as structural loads make concrete vulnerable to tensile cracking. Tensile strength of concrete is much lower than its compressive strength (that's why steel is used to carry the tension forces). To determine the tensile strength, indirect methods are applied due to the difficulty of the direct method. Splitting Tensile Strength test is one of these indirect methods to check tensile strength of the concrete.

2. Reference: IS-516 (Part-1/Sec-1) : 2021 "Hardened Concrete – Methods of Test. Part-1: Testing of Strength of Hardened Concrete. Section-1: Compressive, Flexural and Split Tensile Strength".

3. Specimens: The specimen shall be a cube or cylinder meeting the requirements of IS:1199(Part-5). Damaged or badly honeycombed specimens shall not be tested.

3.1 Age at Test: Tests shall be made at the recognized ages of the test specimens, the most usual being 7 and 28 days. Tests at any other age at which the tensile strength is desired may be made, if so required. The age at test shall be reported along with the results.

3.2 Number of Specimens: At least three specimens shall be tested for each age of tests.

4. Apparatus

4.1 Testing Machine: The test shall be carried out using a compression testing machine conforming to IS:14858. The test machine shall be in calibration at the time of test. The calibration shall be carried out at least once per year.

4.2 Jigs: Jig shown in Fig. 6.3.1 may be used for splitting cylindrical and cubic specimens. Curved steel loading pieces may be used in place of conventional plane platens when tests are carried out on cubical specimen. Alternatively, a jig shown in Fig. 6.3.2 may

be used for cubic specimen.

4.3 Two packing strips of tempered hardboard of nominal thickness 4mm conforming to IS:1658 having following dimensions of the test specimen shall be used for each specimen and shall be discarded after each such test:

(a) Width 15 ± 2 mm,

(b) Nominal thickness 4 ± 1 mm, and

(c) Length greater than the length of the line of content of the test specimen.

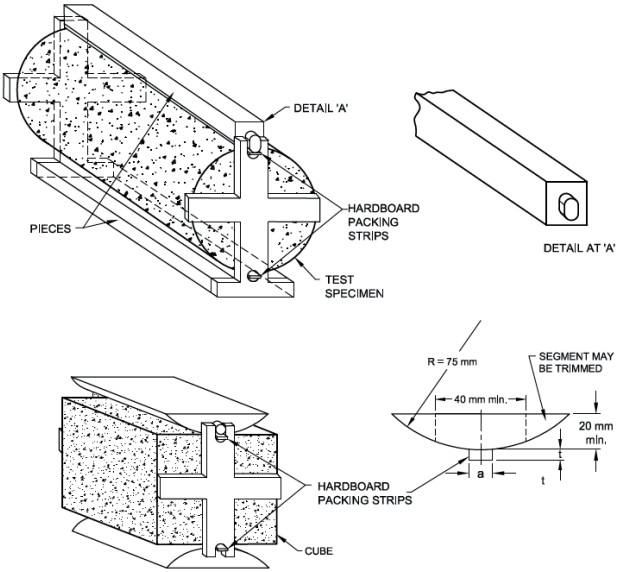


Fig. 6.3.1: Jig for Splitting Cylinder and Cube

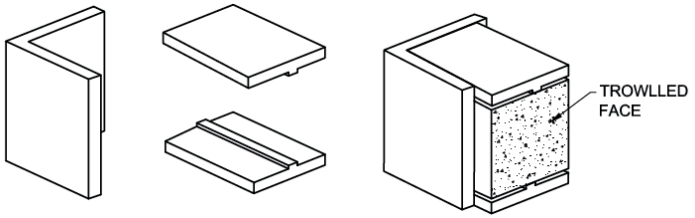


Fig. 6.3.2: Alternate Apparatus for Splitting Cubes

4.4 Steel Loading Strips: A steel loading plate having minimum hardness value, when tested in accordance with IS:1500 (Part 1) shall be used between the platen of the machine and the hardboard packing strip. The piece shall not be shorter than the specimen. For cylindrical specimens it shall be of rectangular cross-section and for cubic specimens, it shall be a section of a cylinder, with a radius of 75mm, so that the load is applied along a line on the surface of the specimen.

5. Procedure

5.1 Specimens when received dry shall be kept in water for 48 hour before testing. The specimens shall be tested immediately on removal from the water whilst they are still wet. Surface water and grit shall be wiped off the specimens and any projecting fins removed from the surfaces which are to be in contact with the packing strips.

5.2 Marking: Central lines shall be drawn on the two opposite faces of the cube using any suitable procedure and device that will ensure that they are in the same axial plane (Fig. 6.3.3).

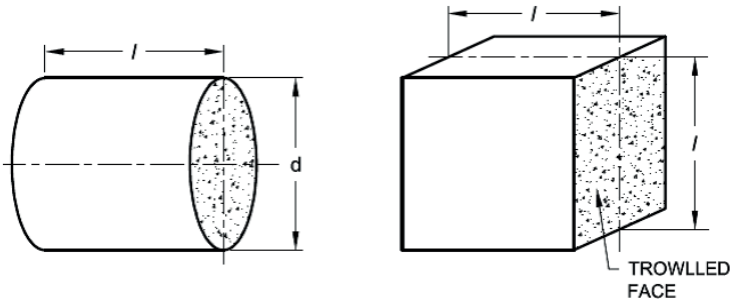


Fig. 6.3.3: Plane of Loading

5.3 Placing of the Specimen in the Testing Machine: The bearing surfaces of the testing machine and of the loading strips shall be wiped clean.

5.4 Positioning: The test specimen shall be placed in

the centering jig with packing strip and/or loading pieces carefully positioning along the top and bottom of the plane of loading of the specimen. The jig shall then be placed in the machine so that the specimen is located centrally. In the case of cubic specimens, the load shall be applied on the moulded faces in such a way that the fracture plane will cross the trowelled surface. For cylindrical specimen it shall be ensured that the upper platen is parallel with the lower platen.

5.5 Rate of Loading: The load shall be applied without shock and increased continuously at a nominal rate within the range 1.2 N/mm^2/min to 2.4 N/mm^2/min. Maintain the rate, once adjusted, until failure. The maximum load applied shall then be recorded. The appearance of concrete and any unusual features in the type of failure shall also be noted. The rate of increase of load may be calculated from the formula:

$$(1.2 \text{ to } 2.4) \times \pi/2 \times l \times d \quad \text{N/min}$$

6. Calculation: The measured splitting tensile strength Fc, of the specimen shall be calculated to the nearest 0.05 N/mm2 using the following formula:

(a) For cylinders: $Fc = 2P / \pi ld$

(b) For cubes: $Fc = P / 2l2$

Where:

P = Maximum load applied to specimen, in N;

l = Length of cylinder/side of the cube, in mm; and

d = cross sectional dimension of cylindrical specimen, in mm.

7. Examination of Specimen: The fractured specimen shall be examined and the appearance of the concrete and type of fracture, if unusual, shall be recorded. An example of unusual type of fracture is when the plane of fracture is not vertical.

8. Report: The following information shall be included in the report on each specimen:

(a) Date of test.

(b) Identification mark, shape and size of the specimen, in mm.

(c) Age of specimen.

(d) Splitting tensile strength to the nearest 0.05 N/ mm^2.

(e) Fracture pattern, in line with Fig. 6.3.3

(f) Weight of specimen.

6.4 Permeability of Concrete

1. Introduction: Concrete is a composite material comprising of Cement, Sand & coarse aggregate. Presence of voids in concrete makes it permeable, which in turn allows water flow into it. The permeability of concrete is the ability of concrete to resist the water flow into it when the external force is applied. Permeability is a measure of the amount of water, that can enter the concrete matrix. In reinforced concrete, ingress of water and air will cause steel erosion causing the concrete to expand, crack, and disintegrate. If the concrete becomes saturated with water because of permeability, it is more susceptible to frost action. It is critical in case of liquid retaining structures like water tanks and dams where water tightness is critical. This chapter describes two methods of testing permeability of concrete i.e. as per IS code which is to be used for concrete in other than Railway Bridges and as per IRS:CBC which is to be used for concrete in Railway Bridges.

(A) As per Bureau of Indian Standard (IS) Code

2. General: This chapter covers the method for determining the permeability of concrete specimens either cast in the laboratory or obtained by cutting out cores from existing structures.

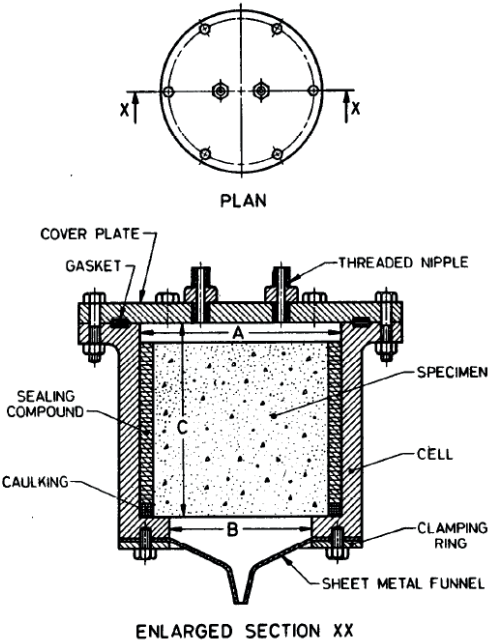
3. Reference: IS-3085:1965 (Reaffirmed-2021) "Method of Test for Permeability of Cement Mortar and Concrete".

4. Apparatus required

4.1 Permeability Cell: It consist of a metal cylinder with a ledge at the bottom for retaining the specimen, a flange at the top, a removable cover plate and a sheet metal funnel which can be securely bolted to the cell. Gunmetal, aluminium or other suitable corrosion-resistant metal shall be used for fabrication of the cell and cover plate. A rubber or neoprene O-ring or other suitable gasket, seated in matching grooves, shall be used between the cell and the cover plate to render the joint water-tight. A typical

permeability cell is shown in Fig. 6.4.1.

4.2 Water Reservoir: A suitable reservoir may consist of a length of metal pipe, 50 to 100 mm in diameter and about 500 mm long. The reservoir shall be fitted with a graduated side arm gauge-glass, and the necessary fittings and valves for admitting water and compressed air and for draining, bleeding and connection to the permeability cell, as shown in Fig. 6.4.2.



Specimen Dia. (mm)	Dimension of Cell (mm)		
	A	B	C
100	115	80	110
150	170	120	160
300	330	260	320

Fig. 6.4.1: Typical Permeability Cell

4.3 Pressure Lines: Heavy duty armoured rubber hose or suitable metal tubing or any other equally suitable hose or pipe shall be used for the various high pressure connections. All joints shall be properly made to render them leakproof.

5. Accessories

5.1 Compressed Air: Suitable arrangements shall be made for supplying compressed air at 5 kg/cm2 to 15 kg/cm^2 to the permeability cell assemblies. Compressed air (or nitrogen) cylinders or alternatively a compressor of adequate capacity may be used. Suitable regulating valves and pressure gauges shall be provided. Several cells at different operating pressures may be served by a common source as shown in Fig. 6.4.2.

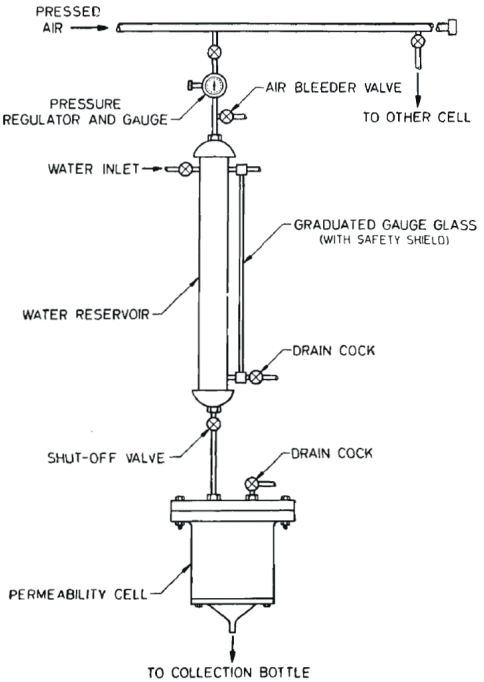


Fig. 6.4.2: Permeability Test Set-up

5.2 De-aired Water: An adequate supply of clean de-aired water shall be available. Water may be easily de-aired by boiling and cooling. It may be stored in closed containers, which should, as far as possible, be kept full. Unnecessary agitations and contact with air shall be avoided.

6. Test Specimens

6.1 Size of Specimens: The specimens shall be cylindrical in shape with height equal to the diameter. The standard size of specimen shall have diameter (and height) of 150mm. In the case of specimens containing aggregates whose nominal size does not exceed 20mm, the diameter (and the height) of the specimen may be reduced to 100mm. In the case of specimens containing aggregates whose nominal size exceeds 40mm, the diameter (and the height) of the specimen should not be less than about four times the nominal size of the aggregate.

6.2 Casting and Curing: The concrete mix shall be cast in split moulds of the required size, with a removable collar of about half the height set on the top. The material shall be compacted either by hand rodding or vibration, as proposed to be done during construction. The collar shall then be removed and the mould shall be struck off level with a straight-edge using a sawing motion without further trovelling or finishing, which might raise the fines to the surface. The specimen shall be cured for 28 days unless otherwise specified by the engineer-in-charge.

7. Procedure

7.1 Pressure Head: The standard test pressure head to be applied to the water in the reservoir should be 10 kg/cm2. This may, however, be reduced up to 5 kg/cm2 in the case of relatively more permeable specimens where steady state of flow is obtained in a reasonable time, and may be increased up to 15 kg/cm2 for relatively less permeable specimens and where sealing could be ensured to be fully effective.

7.2 Calibrating the Reservoir: Each reservoir shall be calibrated under the operating pressures of 5 kg/cm2 to 15 kg/cm2 as indicated below:

With the reservoir drain-cock and the shut-off valve between the reservoir and the cell closed, and with the air bleeder valve open, the reservoir shall be filled with water. The reservoir drain-cock shall then be opened to flush out any air and closed again. The reservoir shall be refilled to a point above the zero mark of the gauge-glass scale; the bleeder valve shall be closed and the desired air pressure applied. The drain-cock shall be carefully opened to bring the water to the zero mark and quickly closed. Water shall then be drawn off and caught in 250 ml increments in a graduated jar and the level in the gauge-glass read on the scale. The calibration constant for the reservoir shall be expressed in millilitres per division of the scale.

7.3 Preparing the Specimen: The specimen shall be thoroughly cleaned with a stiff wire brush to remove all laitance. The end faces shall then be sand-blasted or lightly chiselled.

7.4 Sealing the Specimen: The specimen shall be surface-dried and the dimensions measured to the nearest 0.5mm. It shall then be centred in the cell, with the lower end resting on the ledge. The annular space between the specimen and the cell shall be tightly caulked to a depth of about 10mm using a cotton or hemp cord soaked in a suitable molten sealing compound. The rest of the space shall be carefully filled with the molten sealing compound, level with the top of the specimen. Any drop in the level due to cooling shall be made up, using a heated rod to remelt the solidified compound before pouring fresh material over it. A mixture of bees-wax and rosin, applied smoking hot, forms an effective seal. Other suitable materials are stearin pitch, marine glue, and various asphaltic compounds.

7.5 Testing the Seal: It is essential that the seal is watertight. This may be checked very conveniently by bolting on the top cover plate, inverting the cell and applying an air pressure of 1 to 2 kg/cm2 from below. A little water poured on the exposed face of the specimen is used to detect any leaks through the seal, which would show up as bubbles along the ledge. In case of leaks the specimen shall be taken out and resealed.

7.6 Assembling the Apparatus: After a satisfactory seal has been obtained, the funnel shall be secured in position and the cell assembly connected to the water reservoir. With the air bleeder valve, the valve between the reservoir and the cell, and the drain-cock in the cell open, de-aired water shall be allowed to enter the reservoir. When water issues freely through the drain-cock, it shall be closed and the water reservoir filled. The reservoir water inlet and air bleeder valves shall then be closed.

7.7 Running the Test: With the system completely filled with water, the desired test pressure shall be applied to the water reservoir and the initial reading of the gauge-glass recorded. At the same time a clean collection bottle shall be weighed and placed in position to collect the water percolating through the specimen. The quantity of percolate and the gauge-glass readings shall be recorded at periodic intervals. In the beginning, the rate of water intake is larger than the rate of outflow. As the steady state of flow is approached, the two rates tend to become equal and the outflow reaches a maximum and stabilizes. With further passage of time, both the inflow and outflow generally register a gradual drop. Permeability test shall be continued for about 100 hours after the steady state of flow has been reached and the outflow shall be considered as average of all the outflows measured during this period of 100 hours.

NOTE - The steady state of flow is defined as the

stage at which the outflow and inflow of water become equal for the first time.

7.8 Test Temperature: The test shall preferably be carried out at a temperature of 27°±2°C. In case arrangements are not available for maintaining the above temperature, a record shall be maintained of the actual temperature. An approximate correction may be made on the basis that each 5°C increase of temperature above the standard temperature, results in 10 percent increase in the coefficient of permeability and vice versa.

7.9 Precautions: Following important precautions should be observed:

(a) The seal around the specimen shall be effective. Leakage through it can give rise to entirely misleading results.

(b) Air content of the water entering the specimen should not exceed about 0.2 percent. Excessive amounts of dissolved air can result in air locks in the specimen and apparent reduction in permeability. Periodical samples shall be drawn from the cell drain-cock and the dissolved air determined. The system shall be drained and replenished with fresh de-aired water as soon as the air content exceeds the above limit.

(c) The flow should be permitted to attain the steady state before the coefficient of permeability is calculated. Examination of the inflow and outflow rate data or suitable graphs of the same may be used to determine the establishment of the steady state.

(d) The observation of outflow from the specimen is liable to be influenced by evaporation of the percolate during collection. The collection bottle may be housed in a humid chamber, or alternatively, blank observations on a similar bottle containing water should be made and

the necessary correction for evaporation loss applied. The inflow measurement provides an additional check.

(e) It is very important that the specimen surface is carefully prepared by sand blasting or chiselling, as even a thin highly impervious skin can result in considerable underestimation of the permeability.

8. Calculation: The coefficient of permeability shall be calculated as follows:

$$K = Q / \{A * T * (H/L)\}$$

Where:

K = Coefficient of permeability in cm/sec;

Q = Quantity of water in millilitres percolating over the entire period of test after the steady state has been reached;

A = Area of the specimen face in cm2;

T = Time in seconds over which Q is measured; and

H/L= Ratio of the pressure head to thickness of specimen, both expressed in the same units.

9. Report: The following information shall be included in the report on each specimen:

(a) Identification mark of the specimen,

(b) Particulars of mix,

(c) Age at commencement of the test,

(d) Duration of test,

(e) Size of specimen,

(f) Test pressure,

(g) Test temperature,

(h) Coefficient of permeability at test temperature, and

(j) Corrected coefficient of permeability at standard temperature.

(B) <u>As per IRS: Concrete Bridge Code</u>

2. General: Permeability test is required in following cases:

(i) Mandatory for all RCC/PSC bridges under severe and extreme environment;

(ii) Under moderate environment, this test shall be mandatory for all major bridges and for other bridges this test is desirable to the extent possible; and

(iii) It is required for RCC/PSC structural element only.

3. Reference: Indian Railway Standard (IRS): Code of Practice for Plain, Reinforced & Prestressed Concrete for General, Bridge Construction (Concrete Bridge Code). Reprint-September 2014".

4. Test Specimen: Test specimen of 200mm dia and 120mm thick shall be used. After 24 hours of casting of specimen, central circular area of 100mm diameter shall be roughened with a wire brush on the side on which the water pressure is to be applied. The unroughened part of the side of the test specimen which is subjected to water pressure is to be sealed with two coats of cement water paste (W/C = 0.4).

5. Procedure:

5.1 After 28 days curing, test specimen is fitted in to a test apparatus where water pressure acts on the required face and remaining faces can be observed (Fig. 6.4.3).

5.2 At first, a pressure of 1 bar is applied for 48 hours, then 3 bar for 24 hours and 7 bar for 24 hours. After the test, the specimen is split in the middle by compression applied on two round steel bars lying on opposite sides, above and below. The side after test specimen exposed to the water pressure should face downwards.

5.3 The greatest water penetration depth, is taken as

the average value of the greatest penetration depths on three test specimens.

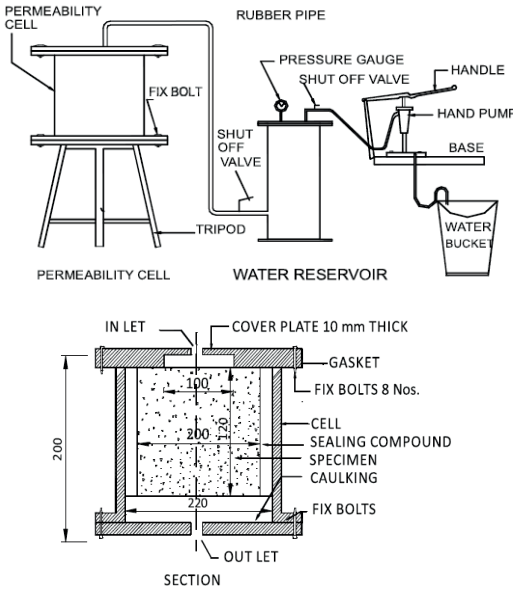


Fig. 6.4.3: Typical Permeability Test Set-up

6. Result: The average value of the greatest penetration depths of moisture shall not exceed 25mm.

6.5 Rebound Hammer Test

1. Introduction: Rebound Hammer test is a Non-destructive testing method of concrete which provide a convenient and rapid indication of the compressive strength of the concrete. The rebound hammer test for concrete is considerably factual when the concrete is at least 28 days old. It is a simple yet adequate tool that is useful to test concrete on site. It is easy to use and does not need special training.

2. Reference: IS-516 (Part-5/Sec-4) : 2020 "Hardened Concrete – Methods of Test. Part-5: Non-destructive Testing of Concrete. Section-4: Rebound Hammer Test".

3. Apparatus

3.1 Rebound Hammer: It consists of spring-loaded steel hammer (mass) that strikes the metal plunger in contact with the concrete surface when released (Fig. 6.5.1).

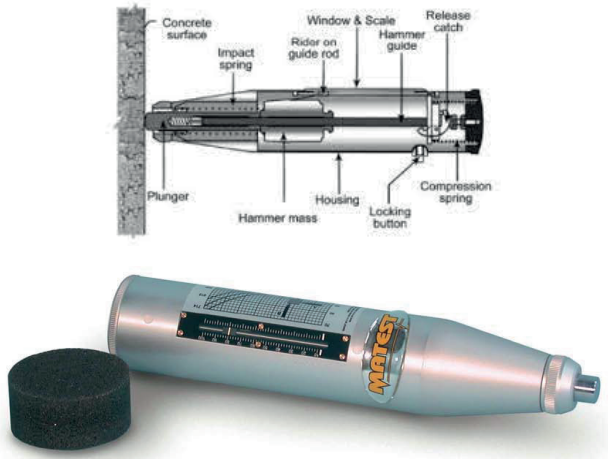


Fig. 6.5.1: Rebound Hammer

The impact energy required for rebound hammers for different applications is given in Table 6.1.

3.2 Abrasive Stone: It consists of medium-grain texture silicon carbide or equivalent material.

3.3 Testing Anvil: It consists of a steel cylinder with 150mm diameter and 150mm height (Fig. 6.5.2). The hardness Rockwell C (HRC) value of the impact area shall be 64 to 68. The supplier/manufacturer of the rebound hammer should indicate the range of readings on the anvil suitable for different types of rebound hammers. It is necessary that the rebound hammer is checked against the testing anvil before commencement of a test and after completion of test to ensure reliable results.

Table 6.1: Impact Energy for Different Applications

Sl. No.	Application	Approximate Impact Energy required (Nm)
1	For testing normal weight concrete	2.25
2	For light-weight concrete or small and impact sensitive part of concrete	0.75
3	For testing mass concrete, for example, in roads, air-field pavements and hydraulic structures	30.00



Fig. 6.5.2: Testing Anvil

4. Checking of Apparatus: To use this test method to estimate strength, it is necessary to establish a correlation

between rebound number and strength for a particular concrete and particular apparatus by any method given below:

4.1 Using Cube Compressive Strength: The satisfactory way of establishing a correlation between compressive strength of concrete and its rebound number is to measure both the properties simultaneously on concrete cubes. The correlation shall be derived on project specific concrete cubes for all bigger projects. The general correlation can be derived from concrete cubes used in smaller projects in a region with similar materials including cement type and the same shall be repeated every year. For bigger projects at least three cubes each for three different concrete grades shall be cast and tested for establishing the correlation. Cube specimens should be wet cured for 27 days and they should be removed from wet storage and kept in the laboratory atmosphere for about 24 hour before testing. The concrete cube specimens are held in a compression testing machine under a fixed load, measurements of rebound number taken using the particular hammer/ hammers for which conditions are to be established and then the compressive strength determined as per IS:516 (Part-1/Sec-1). The fixed load required is of the order of 7 N/mm^2 when the impact energy of the hammer is about 2.25 Nm. The load should be increased for calibrating rebound hammers of greater impact energy and decreased for calibrating rebound hammers of lesser impact energy. The test specimens should be as large a mass as possible in order to minimise the size effect on the test result of a full scale structure. 150mm cube specimens are preferred for calibrating rebound hammers of lower impact energy (2.25 Nm), whereas for rebound hammers of higher impact energy, for example 30 Nm, the test cubes should not be smaller than 300mm. Only the vertical faces of the cube as cast should be tested. At least nine readings should be taken on each of

the two vertical faces accessible in the compression testing machine when using the rebound hammers. The points of impact on the specimen must not be nearer an edge than 25mm and should be not less than 25mm from each other. The same points must not be impacted more than once.

4.2 Using Core Compressive Strength: To establish correlation between rebound number and strength for a particular concrete and particular apparatus, rebound numbers measured on the structure can be correlated with the few core strengths measured on the structure on corresponding members. At least two replicate cores shall be taken from at least six locations with different rebound numbers. The test conditions and surface conditions of the locations where strengths are to be estimated using developed correlation shall be similar to the locations used for development of correlation. For smaller projects the number of cores may be limited to six. The locations where these tests are conducted and cores are taken should have ultrasonic pulse value greater or equal to 3.50 km/s for grades ≤ M25, and 3.75 km/s for grades above M25, by direct method of probing, when tested as per IS:516 (Part 5/Sec 1).

NOTES :

1. Predetermined curve prepared for similar concrete in the same region may be used for approximate estimation of strength of concrete used in the structural members tested for cases where correlation cannot be developed either by cube compressive strength or in-situ core strengths.

2. Different instruments of the same type may give rebound numbers differing from 1 to 3 units. Therefore, tests should be made with the same instrument in order to compare results. If more than one instrument is to be used, perform comparative tests on a range of typical concrete surfaces so as to determine the magnitude of the differences to be

expected in the readings of different instruments.

For readings to be compared, the direction of impact must be the same or established correlation factors shall be applied to the readings. In the absence of data, manufacturer correlation for direction effect can be adopted.

5. Procedure

5.1 For testing, smooth, clean and dry surface is to be selected. If loosely adhering scale is present, this should be rubbed off with a grinding wheel or stone. Rough surfaces resulting from incomplete compaction, loss of grout, spalled or tooled surfaces do not give reliable results and should be avoided.

5.2 The point of impact should be at least 25mm away from any edge or shape discontinuity.

5.3 For taking a measurement, the rebound hammer should be held at right angles to the surface of the concrete member. The test can thus be conducted horizontally on vertical surfaces (preferably) or vertically upwards or downwards on horizontal surfaces. If the situation demands, the rebound hammer can be held at intermediate angles also, but in each case, the rebound number will be different for the same concrete.

NOTE: Digital angle gauges are available that can be attached to the body of the instrument to allow quick measurement of the angle with respect to horizontal. However, correlation taking into account the direction effect can also be developed between equivalent cube compressive strength of concrete cores (minimum 6 nos.) with rebound number in vertically upward or downward direction for the specific project.

5.4 Rebound hammer test shall be conducted around all the points of observation on all accessible faces of the structural element. Concrete surfaces shall be thoroughly cleaned before taking any measurement. Around each point of observation, six readings of

rebound indices are taken and average of these readings after deleting outliers as per IS/ISO:16269 (Part-4) becomes the rebound index for the point of observation.

6. Factors Influencing Test Results: The rebound numbers are influenced by a number of factors like:

6.1 Type of Cement: Concretes made with high alumina cement can give strengths 100 percent higher than that with ordinary Portland cement. Concretes made with super-sulphated cement can give 50 percent lower strength than that with ordinary Portland cement.

6.2 Type of Aggregate: Normal aggregates such as gravels and crushed rock aggregates give similar correlations, but concrete made with lightweight aggregates require special calibration.

6.3 Surface Condition and Moisture Content: This test is suitable only for close texture concrete. Open texture concrete typical of masonry blocks, honeycombed concrete or no-fines concrete are unsuitable for this test. All correlations assume full compaction, as the strength of partially compacted concrete bears no unique relationship to the rebound numbers. Trowelled and floated surfaces are harder than moulded surfaces, and tend to overestimate the strength of concrete.

A wet surface will give rise to underestimation of the strength of concrete calibrated under dry conditions. In structural concrete, this can be about 20 percent lower than in an equivalent dry concrete.

6.4 Curing and Age of Concrete: The relationship between hardness and strength varies as a function of time. Variations in initial rate of hardening, subsequent curing and conditions of exposure also influence the relationship. The effect of age can be ignored for concrete above 14 days old.

6.5 Carbonation of Concrete Surface: The influence

of carbonation of concrete surface on the rebound number is very significant. Carbonated concrete gives an overestimate of strength which in extreme cases can be up to 50 percent. The carbonation depth shall be checked in cases where the age of concrete is more than 6 months and same shall be reproduced in the test report.

6.6 Vertical Distance from the Bottom of Concrete: The influence of vertical distance from the bottom of concrete placement on the rebound number is very significant. Generally, a higher rebound number is observed near the bottom of concrete placement as during compaction, concentration of aggregates will be higher at the bottom.

6.7 Surface Conditions: The direct correlation between rebound numbers and strength of wet cured and wet tested cubes is not recommended. It is necessary to establish a correlation between the strength of wet tested cubes and the strength of dry tested cubes on which rebound readings are taken.

7. Interpretation of Results

7.1 The rebound hammer method provides a convenient and rapid indication of the compressive strength of concrete by establishing a suitable correlation between the rebound index and the compressive strength of concrete (Ref. Para 4). In general, the rebound number increases as the strength increases but it is also affected by several parameters as mentioned in Para 6. It is also pointed out that rebound indices are indicative of compressive strength of concrete to a limited depth from the surface. If the concrete in a particular member has internal micro-cracking, flaws or heterogeneity across the cross-section, rebound hammer indices will not indicate the same. As such, the estimation of strength of concrete by rebound hammer method cannot be held to be very accurate and probable accuracy of prediction of concrete strength in a structure can be

up to ± 25 percent depending upon correlation curve and methodology adopted for establishing correlation between rebound index and likely compressive strength. If the relationship between rebound index and compressive strength can be checked by tests on core samples obtained from the structure or standard specimens made with the same concrete materials and mix proportion, then the accuracy of results and confidence thereon are greatly increased.

7.2 Because of the various limitations in rebound hammer test, the combined use of ultrasonic pulse velocity (UPV) test [IS-516 (Part 5/Sec 1)] and rebound hammer test is a must for proper interpretation. If the quality of concrete assessed by ultrasonic pulse velocity method is 3.50 km/s for grades ≤ M25, and 3.75 km/s for above M25 or above, only then the in-situ compressive strength assessed from the rebound hammer test is valid. This shall be taken as indicative of strength of concrete in the entire cross-section of the concrete member represented by the both tests.

7.3 In cases the quality of concrete assessed by UPV is doubtful, no assessment of concrete strength shall be made from rebound hammer test.

8. Test Report: The report shall include the following:

(a) Date/period of testing;

(b) Identification of the concrete structure/element;

(c) Location of test area(s);

(d) Identification of the rebound hammer;

(e) Details of concrete and its condition;

(f) Date/time of performance of the test;

(g) Test result and hammer orientation for each test area.

6.6 Ultrasonic Pulse Velocity Test

1. Introduction: Ultrasonic Pulse Velocity test on concrete is a simple non-destructive test to assess the homogeneity and integrity of concrete. With this test the qualitative assessment of strength of concrete, its gradation in different locations of structural members, discontinuity in cross section like cracks, cover concrete delamination etc., depth of surface cracks and dynamic modulus of elasticity of concrete can be ascertained.

2. Reference: IS-516 (Part-5/Sec-1) : 2020 "Hardened Concrete – Methods of Test. Part-5: Non-destructive Testing of Concrete. Section-1: Ultrasonic Pulse Velocity Testing".

3. Apparatus required



Fig. 6.5.1: Test Set-up

3.1 Electronic Pulse Generator

3.2 Transducers: One Pair : Piezoelectric and magneto-strictive types of transducers may be used, the latter being more suitable for the lower part of the frequency range. Frequencies as low as 10 kHz

and as high as 200 kHz can sometimes be used. It is preferable to use high-frequency transducers (60 kHz to 200 kHz) for short path lengths (down to 50mm) and low frequency transducers (10 kHz to 40 kHz) for long path lengths (up to a maximum of 15m). Transducers with a frequency of 25 kHz to 100 kHz are found to be useful for most applications.

3.3 Standard Calibration Bar: Two standard calibration bars, as per details given in Para 4.1 shall be provided by the manufacturer of the apparatus for calibrating the apparatus.

3.4 Amplifier

3.5 Electronic Timing Device: It shall be capable of measuring the time interval elapsing between the onset of a pulse generated at the transmitting transducer and the onset of its arrival at the receiving transducer.

4. Performance Requirement of Apparatus

4.1 The apparatus shall be capable of measuring transit times to an accuracy of ±1 percent over a range of 20 microsecond to 10 millisecond. For this, it is necessary to check the overall performance by making measurements on two standard reference specimens in which the pulse transit times are known accurately. The two reference specimens (usually steel bars) shall have pulse transit times of about 25 microsecond to 100 microsecond respectively; these times being specified by the supplier of the equipment to an accuracy of ±0.2 microsecond. The shorter of the reference specimens shall be used to set the zero for the apparatus and the longer one shall be used to check the accuracy of transit time measurement of the apparatus. The measurement obtained shall not differ from the known value for the reference specimen by more than ±0.5 percent.

4.2 Along with the calibration of the equipment, it is also advisable to perform a zero time check on the

unit by applying a coupling agent to the transducers and pressing the faces together. Check the zero adjustment on regular intervals.

4.3 The electronic excitation pulse applied to the transmitting transducer shall have a rise time of not greater than one quarter of its natural period. This is to ensure a sharp pulse onset.

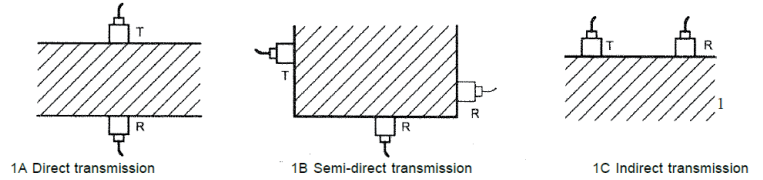
4.4 The interval between pulses shall be low enough to ensure that the onset of the received signal in small concrete test specimens is free from interference by the reverberations produced within the preceding working cycle.

4.5 The apparatus shall maintain its performance over the range of ambient temperature, humidity and power supply voltage as stated by the supplier.

4.6 When using long leads (above 20m) caution shall be taken during transit time measurement that the leads do not come into close contact with each other. In case the leads are close together, it may pickup unwanted signals from the transmitter lead resulting in incorrect and unstable readings.

5. Procedure

5.1 Surface Preparation: At the point of observation, the concrete surface shall be suitably prepared and any plaster or other coating shall be removed to expose the concrete surface. For this purpose, the use of carborundum stones or grinders may be adopted. However, care shall be taken to avoid any damage to concrete surface or concrete structure.



Key: T = Transmitter and R = Receiver

Fig. 6.6.1: Positioning of Transducers

5.2 Ultrasonic Measurements: Place the two transducers on opposite faces (direct transmission), or on adjacent faces (semi-direct transmission), or on the same face (indirect or surface transmission) (Fig. 6.6.1). Although the direction in which the maximum energy is propagated is at right angles to the face of the transmitting transducer, it is possible to detect pulses that have travelled through the concrete in some other direction. Direct transmission method of ultrasonic pulse velocity measurements is the most efficient method and shall be adopted, if possible. However, sometimes, it may be necessary to place the transducers on opposite faces but not directly opposite each other. Such arrangements shall be regarded as a semi-direct transmission. The third method for measurement of ultrasonic pulse velocity is the indirect transmission method. The indirect transmission arrangement is the least sensitive and shall be used when only one face of the concrete is accessible, or when the quality of the surface concrete relative to the overall quality is of interest.

5.3 Determination of Ultrasonic Pulse Velocity for Different Transducer Arrangements

5.3.1 Factors influencing pulse velocity measurements: There are various factors which influence pulse velocity measurements, such as:

(A) Surface Conditions and Moisture Content: For most concrete surfaces, the finish is usually sufficiently smooth to ensure good acoustical contact by the use of a coupling medium and by pressing the transducer against the concrete surface. When the concrete surface is rough and uneven, it is necessary to smoothen the surface to make the pulse velocity measurement possible.

In general, pulse velocity through concrete increases with increased moisture content of concrete. This influence is more for

low strength concrete than high strength concrete. The pulse velocity of saturated concrete may be up to 5 percent higher than that of similar dry concrete. In general, drying of concrete may result in somewhat lower pulse velocity.

(B) Path Length, Shape and Size of the Concrete Member: As concrete is inherently heterogeneous, it is essential that path lengths be sufficiently long so as to avoid any error introduced due to its heterogeneity. In field work, this does not pose any difficulty as the pulse velocity measurements are carried out on thick structural concrete members. However, in the laboratory where generally small specimens are used, the path length can affect the pulse velocity readings. The velocity of short pulses of vibrations is independent of the size and shape of the specimen in which they travel, unless its least lateral dimension is less than a certain minimum value. Below this value, the pulse velocity may be reduced appreciably. The extent of this reduction depends mainly on the ratio of the wave length of the pulse vibrations to the least lateral dimension of the specimen but is insignificant, if the ratio is less than unity. Table 6.2 gives the relationship between the pulse velocity in the concrete, the transducer frequency and the minimum permissible lateral dimension of the specimen.

If the minimum lateral dimension is less than the wavelength or if the indirect transmission arrangement is used, the mode of propagation changes and, therefore, the measured velocity will be different. This is particularly important in cases where concrete elements of significantly different sizes are being compared.

Table 6.2: Minimum Specimen Dimension

Sl. No.	Transducer Frequency (kHz)	Pulse Velocity in Concrete		
		3.50 km/s	4.00 km/s	4.50 km/s
		Minimum Permissible Lateral Specimen Dimension (mm)		
1	24	146	167	188
2	54	65	74	83
3	82	43	49	55
4	150	23	27	30

(C) Temperature of Concrete: Variations of the concrete temperature between 5°C and 30°C do not significantly affect the pulse velocity measurements in concrete. At temperatures between 30 to 60°C there can be reduction in pulse velocity up to 5 percent. Below freezing temperature, the free water freezes within concrete, resulting in an increase in pulse velocity up to 7.5 percent.

(D) Stress: When concrete is subjected to a stress which is abnormally high for the quality of the concrete, the pulse velocity may be reduced due to the development of micro-cracks. This influence is likely to be the greatest when the pulse path is normal to the predominant direction of the planes of such micro-cracks. This occurs when the pulse path is perpendicular to the direction of a uniaxial compressive stress in a member. This influence is generally insignificant unless the stress is greater than about 60 percent of the ultimate strength of the concrete.

(E) Reinforcing Bars: The pulse velocity measured in reinforced concrete in the vicinity of reinforcing bars is usually higher than in plain concrete of the same composition. This is because the pulse velocity in steel is 1.2 to 1.9 times the velocity in plain concrete and, under certain conditions, the first pulse to arrive at the receiving transducer travels partly in concrete and partly in steel. The apparent increase in pulse velocity depends upon the proximity of the measurements to the reinforcing bar, the diameter and number of the bars and their orientation with respect to the path of propagation.

(F) Contact Between Transducer and Concrete: Poor contact will affect the reading. It is essential to use grease or other couplants to improve the contact between the transducer and the concrete.

(G) Cracks and Voids: When an ultrasonic pulse travelling through concrete meets a concrete-air interface, there is negligible transmission of energy across this interface. Thus, any air-filled crack or void lying immediately between two transducers will obstruct the direct ultrasonic beam when the projected length of the void is greater than the width of the transducers and the wavelength of sound used. When this happens, the first pulse to arrive at the receiving transducer will have been diffracted around the periphery of the defect and the transit time will be longer than in similar concrete with no defect. It is possible to make use of this effect for locating flaws, voids or other defects greater than about 100mm in diameter or depth. Relatively small defects have little or no effect on transmission times, but equally are probably

of minor engineering importance. Plotting contours of equal velocity often gives significant information regarding the quality of a concrete unit. In cracked members, where the broken faces of the members are held tightly together in close contact by compression forces, the pulse energy may pass unimpeded across the crack. If the crack is filled with liquid which transmits the ultrasonic energy (like in marine structures), the crack is undetectable using digital reading equipment. Measurements of attenuation may give valuable information in these cases.

A grid shall be drawn on the concrete member with its points of intersection spaced to correspond to the size of void that might significantly affect its performance. A large survey of measurements at the grid points enables a large cavity to be investigated by measuring the transit times of pulses passing between the transducers when they are placed so that the cavity lies in the direct path between them.

The size of such cavities may be estimated by assuming that the pulses pass along the shortest path between the transducers and around the cavity. Such estimates are valid only when the concrete around the cavity is uniformly dense and the pulse velocity can be measured in that concrete.

5.3.2 Procedure

(A) The ultrasonic pulse is produced by the transducer which is held in contact with one surface of the concrete member under test. After traversing a known path length L in the concrete, the pulse of vibrations is converted into an electrical signal by

the second transducer held in contact with the other surface of the concrete member and an electronic timing circuit enables the transit time (T) of the pulse to be measured. The pulse velocity (V) is given by:

$$V = L / T$$

Once the ultrasonic pulse impinges on the surface of the material, the maximum energy is propagated at right angles to the face of the transmitting transducer and best results are, therefore, obtained when the receiving transducer is placed on the opposite face of the concrete member (direct transmission or cross probing).

However, in many situations two opposite faces of the structural member may not be accessible for measurements. In such cases, the receiving transducer is also placed on the same face of the concrete member (surface probing or indirect transmission). Surface probing is not as efficient as cross probing, because the signal produced at the receiving transducer has amplitude of only 2 to 3 percent of that produced by cross probing and the test results are greatly influenced by the surface layers of concrete which may have different properties from that of concrete inside the structural member. The indirect velocity is invariably lower than the direct velocity on the same concrete element. This difference may vary from 5 to 20 percent depending largely on the quality of the concrete under test. For good quality concrete, a difference of about 0.5 km/s may generally be encountered. For the procedure and for calculating the exact value of ultrasonic pulse velocity by surface probing

(see Annex A).

(B) To ensure that the ultrasonic pulses generated at the transmitting transducer pass into the concrete and are then detected by the receiving transducer, it is essential that there be adequate acoustical coupling between the concrete and the face of each transducer. Typical couplants are petroleum jelly, grease, liquid soap and kaolin glycerol paste. If there is very rough concrete surface, it is required to smoothen and level an area of the surface where the transducer is to be placed. If it is necessary to work on concrete surfaces formed by other means, for example trowelling, it is desirable to measure pulse velocity over a longer path length than would normally be used. A minimum path length of 150mm is recommended for the direct transmission method involving one unmoulded surface and a minimum of 400mm for the surface probing method along an unmoulded surface.

(C) Since size of aggregates influences the pulse velocity measurement, it is recommended that for direct transmission method, the minimum path length shall be 100mm for concrete in which the nominal maximum size of aggregate is 20mm or less and 150mm for concrete in which the nominal maximum size of aggregate is between 20 and 40mm.

(D) In view of the inherent variability in the test results, sufficient number of readings are taken by dividing the entire structure in suitable grid markings of 300 × 300 mm or even smaller. Each junction point of the grid becomes a point of observation. Larger grid spacing up to maximum 500 × 500mm may be adopted for general overall assessment

of larger structures having uniform cross-section showing no signs of distress. The number of individual test points or grid spacing depends upon the size of the structure, the accuracy required and the variability of the concrete. Transducers are held on corresponding points of observation on opposite faces of a structural element to measure the ultrasonic pulse velocity by direct transmission, that is, cross probing. If one of the faces is not accessible, ultrasonic pulse velocity is measured on one face of the structural member by surface probing.

(E) Surface probing in general gives lower pulse velocity than in case of cross probing and depending on number of parameters, the difference could be of the order of about 0.5 km/s. In view of this, it is recommended that, in surface probing method the pulse velocity may be increased by 0.5 km/s, for values > 3.0 km/s.

6. Interpretation of Results

6.1 The ultrasonic pulse velocity of concrete is mainly related to its density and modulus of elasticity. This in turn, depends upon the materials and mix proportions used in making concrete as well as the method of placing, compaction and curing of concrete. For example, if the concrete is not compacted as thoroughly as possible, or if there is segregation of concrete during placing or there are internal cracks or flaws, the pulse velocity will be lower, although the same materials and mix proportions are used.

6.2 The quality of concrete in terms of uniformity, incidence or absence of internal flaws, cracks and segregation, etc (indicative of the level of workmanship employed) can be assessed using the guidelines given in Table 6.3. This table is only for concrete quality grading and shall not be used for

estimating the concrete grades from ultrasonic pulse velocity values.

Table 6.3: Velocity Criterion for Concrete Grading

Sl. No.	Average Value of Pulse Velocity by Cross Probing (km/s)	Concrete Quality Grading
1	Above 4.40	Excellent
2	3.75 to 4.40	Good
3	3.00 to 3.75	Doubtful (*)
4	Below 3.00	Poor
(*) In case of "Doubtful" quality, it may be necessary to carry out further tests.		

6.3 Since actual values of the pulse velocity obtained, depend on a number of parameters, any criterion for assessing the quality of concrete on the basis of pulse velocity as given in Table 6.3 can be held as satisfactory only to a general extent. However, when the comparison is made amongst different parts of a structure, which have been built at the same time with supposedly similar materials, construction practices and supervision, the assessment of quality becomes more meaningful and reliable. Whenever the UPV values are lesser by more than 10 percent of average value of the member/part of structure, the location shall be considered as having internal flaws or segregation caused by poor workmanship or there could be micro cracks.

6.4 The assessment of compressive strength of concrete from ultrasonic pulse velocity values is not adequate because the statistical confidence of the correlation between ultrasonic pulse velocity and the compressive strength of concrete is not very high. The reason is that a large number of parameters are involved, which influence the pulse velocity and compressive strength of concrete to different

extents. However, if actual concrete materials and mix proportions adopted in a particular structure are available, then estimate of concrete strength can be made by establishing suitable correlation between the pulse velocity and the compressive strength of concrete specimens made with such materials and mix proportions, under environmental conditions similar to that in the structure. The estimated strength may vary from the actual strength by ± 20 percent. The correlation so obtained may not be applicable for concrete of another grade or made with different types of materials. If correlation graph is not available but the velocities on concrete cubes are available, the average pulse velocities in members of structures are not expected to deviate by more than 10 to 15 percent of the pulse velocity values obtained on concrete cubes (dry surface).

7. Estimating depth of a surface crack

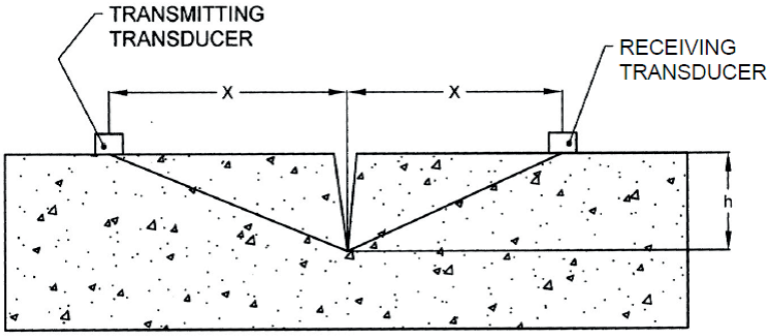


Fig. 6.6.2: Measurement of Depth of Crack

Longitudinal pulse velocity = ɑ

Distance travelled in uncracked concrete = 2X (Fig. 6.6.2)

Distance travelled in cracked concrete = $2 * (X^2 + h^2)0.5$

Tc^2 (in cracked concrete) = $(4X^2 + 4h^2) / ɑ^2$

Ts^2 (in uncracked concrete) = $4X^2 / ɑ^2$

Therefore, $(Tc^2 / Ts^2) = (X^2 + h^2)/X^2$

Or

$$h = X * [(Tc^2 / Ts^2) - 1]^{0.5}$$

where:

Tc = Travel time around the crack,

Ts = Travel time along the surface of the same type of concrete without any crack, and

h = Depth of crack (see Fig. 6.6.2)

8. Determining dynamic Young's Modulus of Elasticity (E): The dynamic Young's modulus of elasticity (E) of the concrete may be determined from the pulse velocity and the dynamic Poisson's ratio (μ), using the following relationship:

$$E = [\rho (1 + \mu) (1 - 2\mu) V2] / (1 - \mu)$$

Where:

E = Dynamic Young's Modulus of elasticity, in MPa;

ρ = Density, in kg/m3; and

V = Pulse velocity, in m/s.

The above relationship may be expressed as:

$$E = \rho f(\mu) V2$$

Where:

$$f(\mu) = [(1 + \mu) (1 - 2\mu)] / (1 - \mu)$$

The value of the dynamic Poisson's ratio varies from 0.20 to 0.35, with 0.24 as average. However, it is desirable to have an independent measure of it for the particular type of concrete under test. The dynamic Poisson's ratio may be obtained from measurements on concrete test-beams of the pulse velocity (V) along with length (l) of the beam and the fundamental resonant frequency (n) of the beam in longitudinal mode of vibration. From these measurements, the factor f(μ) is calculated by the relation:

$$f(\mu) = (2nl)^2 / V^2$$

Where:

n = Fundamental resonant frequency in cycles per second; and

l = length of specimen, in m.

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Chapter - 7

TESTS ON BITUMEN

As per the current instructions of Railway Board, the Items and Specifications of CPWD's DSOR and the corresponding Technical Specifications of CPWD are to be used for works related to roads (the works in which Bitumen is used as a constituent material). In CPWD's Specifications, Vol. 2, 2019, Sub Head: 16.0 for "Road Works" contains the "List of Mandatory Tests" for various constituent of Road Work (Page 751) and Sl. No. 3 of this list is about "Bitumen" wherein it is stipulated that tests should be done as Per IS:73.

Clause 6.2 of the IS:73-2013 (Reaffirmed in 2018) specifies that the Bitumen shall conform to the requirements prescribed in Table-1 therein. The requirements listed in Table-1 prescribes following tests:

(7.1) Determination of Penetration

(7.2) Determination of Absolute Viscosity

(7.3) Determination of Kinematic Viscosity

(7.4) Determination of Flash Point

(7.5) Determination of Solubility in Trichloroethylene

(7.6) Determination of Softening Point

(7.7) Determination of Viscosity Ratio

(7.8) Determination of Ductility

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7.1 Determination of Penetration

1. Introduction: Penetration value test on bitumen is a measure of hardness or consistency of bituminous material. It is carried to determine (i) Consistency of bituminous material, and (ii) Suitability of bitumen for use under different climatic conditions & various types of construction.

This chapter covers the method for the determination of penetration of semi-solid and solid bituminous materials.

2. Reference: IS-1203:2022 "Methods for Testing Tar and Bituminous Materials — Determination of Penetration".

3. Apparatus



Fig. 7.1.1: Test setup

3.1 Container: A metal or glass cylindrical, flat bottom container of essentially the following dimensions shall be used:

For penetration below 225:

Diameter, mm	55
Internal depth, mm	35

For penetration between 225 and 350:

Diameter, mm	70
Internal depth, mm	45

3.2 Needle: A straight, highly polished, cylindrical, stainless steel (SS 316), rod, with conical and parallel portions co-axial, having the shape, dimensions and tolerances given in Fig. 7.1.2. The needle is provided with a shank approximately 3mm in diameter into which it is immovably fixed. The taper shall be symmetrical and the point shall be 'blunted' by grinding to a truncated cone.

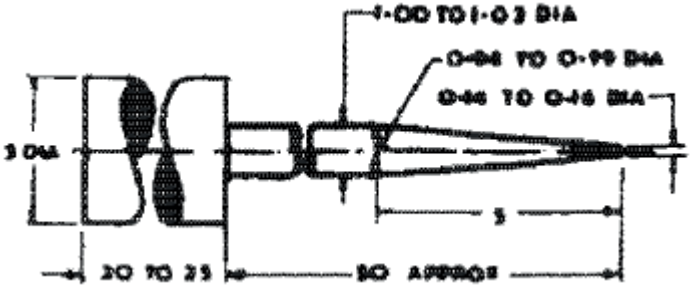


Fig. 7.1.2: Needle for Penetration test

3.3 Water Bath: A water bath preferably with a thermostat maintained at 25.0 ± 0.1 containing not less than 10 litres of water. The sample being immersed to a depth of not less than 100 mm from the top and supported on a perforated shelf not less than 50 mm from the bottom of the bath.

3.4 Transfer Dish: A small dish or tray provided with some means which ensure a firm bearing and prevent the rocking of the container and of such capacity as will ensure complete immersion of the container during the test.

3.5 Penetration Apparatus: Any apparatus that will allow the needle to penetrate without appreciable friction, and that is accurately calibrated to yield results in tenths of millimetre shall be adopted.

3.6 Thermometer: It shall conform to the following requirements:

Characteristic	Requirement
Range	0 to 440 C
Least Count	0.10 C

3.7 Time Device: For hand-operated penetrometers, any convenient timing device, such as electric timer, stop watch, or any other spring actuated device may be used provided it is graduated 0.1 s or less and is accurate to within ± 0.1 s for a 60 s interval. An audible seconds counter adjusted to provide 1 beat each 0.5 s may also be used. The time for a 1 1-count interval shall be 5 ± 0.1 s. Any automatic timing device attached to a penetrometer shall be accurately calibrated to provide the desired test interval within ± 0.1 s.

4. Procedure

4.1 Preparation of Test Sample

4.1.1 Soften the material to a pouring consistency at a temperature not more than 60°C for tars and pitches and not more than 90°C for bitumen above the respective approximate softening point and stir it thoroughly until it is homogeneous and is free from air bubbles and water. Pour the melt into the container to a depth at least 10mm in excess of the expected penetration. Protect the sample from dust and allow it to cool in atmosphere at a temperature between 15 to 30°C for 1 to 2 hour for 45mm deep container and 1 hour when the container of 35mm depth is used. Then place it along with the transfer dish in the water bath at 25.0 ± 0.1°C and allow it to remain for 1 to 2 hour and 1 hour for 45mm and 35mm deep container respectively.

4.2 Testing

4.2.1 Unless otherwise specified, testing shall be carried out at 25.0 ± 0.1°C.

4.2.2 Fill the transfer dish with water from the

water bath to a depth sufficient to cover the container completely; place the sample in it and put it upon the stand of the penetration apparatus. Adjust the needle to make contact with the surface of the sample.

4.2.2.1 This may be accomplished by placing the needle point in contact with its image reflected by the surface of the material from a suitably placed source of light.

4.2.2.2 Unless otherwise specified, load the needle holder with the weight required to make a total moving weight (that is, the sum of the weights of the needle, carrier and superimposed weights) of 100 ± 0.25 g.

4.2.3 Note the reading of the dial or bring the pointer to zero. Release the needle and adjust the points, if necessary to measure the distance penetrated. Make at least three determinations at points on the surface of the sample not less than 10mm apart and not less than 10mm from the side of the dish. After each test, return the sample and transfer dish to the water bath, and wash the needle clean with toluene and dry. In the case of material of penetration greater than 225, three determinations on each of two identical test specimens using a separate needle for each determination shall be made, leaving the needle in the sample on completion of each determination to avoid disturbance of the specimen.

4.2.4 For determining the penetration ratio, testing shall also be carried out a 4.0° ± 0.1° C.

NOTE: For test at 4° C, the total weight on the penetration needle shall be 200 ± 0.25 g and the time of penetration shall be 60 s.

5. Report

5.1 Express the depth of penetration of the needle in tenths of millimetre.

5.2 The value of penetration reported shall be the mean of not less than three determinations whose values do not differ by more than the amount given below:

Penetration	Maximum Difference
0 to 49	2
50 to 149	4
150 to 249	6
250 and above	8

5.3 Determine the penetration ratio as under:

$$\text{Penetration Ratio} = \frac{\text{Pen. At } 4^0\text{C, 200 g, 60 s}}{\text{Pen. At } 25^0\text{C, 200 g, 60 s}} \times 100$$

6. Precision

67.1 The duplicate results should not differ by more than the following:

Penetration	Repeatability	Reproducibility
Below 50	1 Unit	4 Units
Above 50	3% of their mean	8% of their mean

7. Precautions

7.1 If the sample contains extraneous matter, it should be sieved through IS Sieve 30 (see IS 460).

7.2 To avoid overheating at the bottom of the container, use of an air oven or sand bath is recommended.

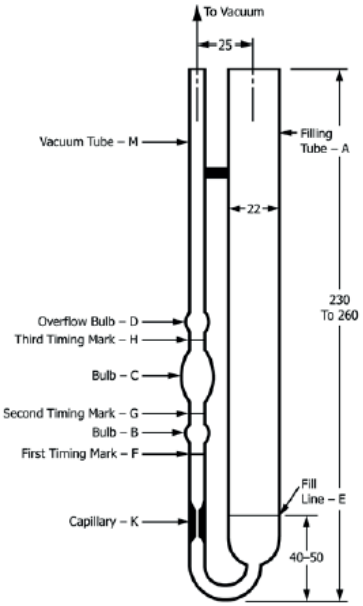
7.3 While the needle is penetrating into the sample, if there is any movement of the container, that determination shall be discarded.

7.2 Determination of Absolute Viscosity

1. Introduction: Viscosity shows how easily bitumen flows. The higher the viscosity of the bitumen, the harder it is to flow. Consequently, it behaves more like semi-solid matter. This chapter covers the method for the determination of absolute viscosity of bitumen by vacuum capillary viscometers at any specified temperature. It is applicable to materials having a viscosity range of 42 to 200000 Poises.

2. Reference: IS-1206 (Part-2):2022 "Methods for Testing Tar and Bituminous Material – Determination of Viscosity: Part-2: Absolute Viscosity".

3. Terminology: For the purpose of this standard, the following definitions and those given in IS 334 shall apply.



All dimensions are in mm

Fig. 7.2.1: Cannon-Manning Vacuum Capillary Viscometer

3.1 Absolute or Dynamic Viscosity of a Newtonian Liquid: The ratio between the applied shear stress and rate of shear is called the coefficient of viscosity. This coefficient is thus a measure of the resistance to flow of the liquid. It is commonly called the viscosity of the liquid. The SI unit of viscosity is 1 Pa. s (1 $N.s/m^2$) and is called a Pascal-second. The CGS unit of viscosity is 1 g/cm.s (1 $dyne.s/cm^2$) and is called a poise (P). 1 Pa.s is equivalent to 10 P.

3.2 Newtonian Liquid: A liquid in which the shear stress is directly proportional to the rate of shear. The constant ratio of shear stress to the rate of shear is called the coefficient of viscosity of the liquid. If this ratio is not constant then the liquid is non-Newtonian.

4. Apparatus

4.1 Viscometers: Capillary type made of borosilicate glass, annealed suitable for this test are given in Para 4.1.1 to 4.1.3.

4.1.1 Cannon-Manning Vacuums Viscometer: (Fig. 7.2.1) The size numbers/approximate bulb factors K and viscosity ranges for the series of Cannon-Manning Vacuum Capillary Viscometer are as follows:

Viscometer Size No.	Approximate Calibration Factor 30cm Hg Vacuum (Poises per s)		Viscosity Range (Poises)
	Bulb B	Bulb C	
10	2.0	0.6	36 to 800
11	6.0	2.0	120 to 2400
12	20.0	6.0	360 to 8000
13	60.0	20.0	1200 to 24000
14	200.0	60.0	3600 to 80000

For all viscometer sizes the volume of measuring bulb C is approximately three times that of bulb B. The viscosity ranges correspond to a filling time of 60 and 400 s for both measuring bulbs.

Sizes 10 through 14 are best suited to viscosity measurements of bituminous binders at 60 °C.

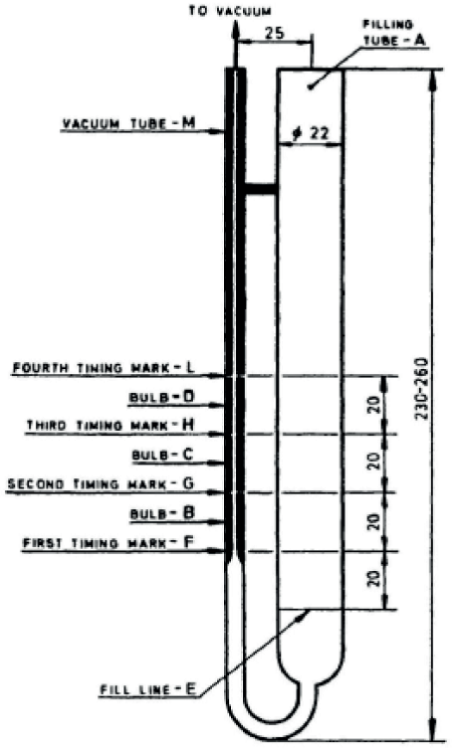
NOTE: The calibration factors have to be determined either by calibration through viscosity standards or through calibration by competent agency.

4.1.2Asphalt Institute Vacuum Viscometer: (Fig. 7.2.2) The size numbers. Approximate radii. approximate bulb factors K and viscosity ranges for the series of Asphalt Institute Vacuum Capillary Viscometer are as follows:

Viscometer Size No.	Capillary Radius K (cm)	Approximate Calibration Factor 30cm Hg Vacuum (Poises per s)			Viscosity Range (Poises)
		Bulb B	Bulb C	Bulb D	
25	0.0125	2	1	0.7	42 to 800
50	0.025	8	4	3	180 to 3200
100	0.050	32	16	10	600 to 12800
200	0.100	128	64	40	2400 to 52000
400	0.200	500	250	160	9600 to 200000

This viscometer has measuring bulbs B, C and D located on the viscometer arm M which is a precision bore glass capillary. The measuring bulbs are 2cm long capillary segments separated by timing marks F, G, H and L.

Sizes 50 through 200 are best suited to viscosity measurements of bituminous binders at 60°C.

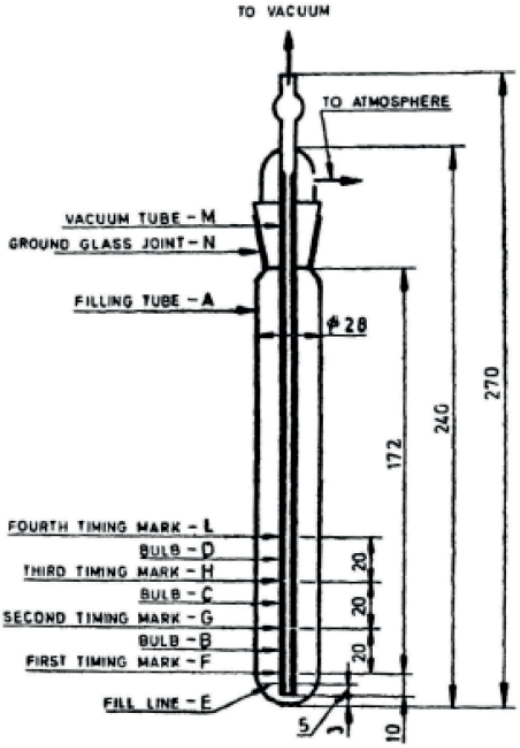


All dimensions are in mm

Fig. 7.2.2: Asphalt Institute Vacuum Capillary Viscometer

Note: The calibration factors have to be determined either by calibration through viscosity standards or through calibration by competent agency.

4.1.3 Modified Koppers Vacuum Viscometer: (Fig. 7.2.3) The size numbers approximate radii. approximate bulb factors K and viscosity ranges for the series of modified Koppers vacuum capillary viscometer are as follows:



All dimensions are in mm

Fig. 7.2.3: Modified Koppers Vacuum Capillary Viscometer

Viscometer Size No.	Capillary Radius K (cm)	Approximate Calibration Factor 30cm Hg Vacuum (Poises per s)			Viscosity Range (Poises)
		Bulb B	Bulb C	Bulb D	
25	0.0125	2	1	0.7	42 to 800
50	0.025	8	4	3	180 to 3200
100	0.050	32	16	10	600 to 12800

200	0.100	128	64	40	2400 to 52000
400	0.200	500	250	160	9600 to 200000

Sizes 50 through 200 are best suited to viscosity measurements of bituminous binders at 60 °C.

NOTE: The calibration factors have to be determined either by calibration through viscosity standards or through calibration by competent agency.

This viscometer consists of a separate filling tube A, and a precision bore glass capillary vacuum tube M. These two parts are joined by borosilicate ground glass joint N, having a 24/40 standard taper. The measuring bulbs B, C, and D on the glass capillary are 2 cm capillary segments separated by timing marks F, G, H and L.

A viscometer holder can be made by drilling a 28mm hole through the center of a No. 11 rubber stopper and setting the stopper between the hole and the edge. When placed in a 5cm diameter hole in the bath cover, it holds the viscometer in place.

4.2 Thermometer: The thermometer shall be calibrated from a competent agency with least count of 0.1°C.

4.3 Bath: A suitable bath for immersion of the viscometer so that the liquid reservoir or top of the capillary, whichever is uppermost is at least 20mm below the upper bath level, and with a provision for the visibility of the viscometer and the thermometer. Firm support for the viscometer shall be provided. The efficiency of the stirring and the balance between heat losses and heat input must be such that the temperature of the bath medium does not vary by more than ± 0.1°C.

NOTE: All tubing is of glass with 6.35mm OD

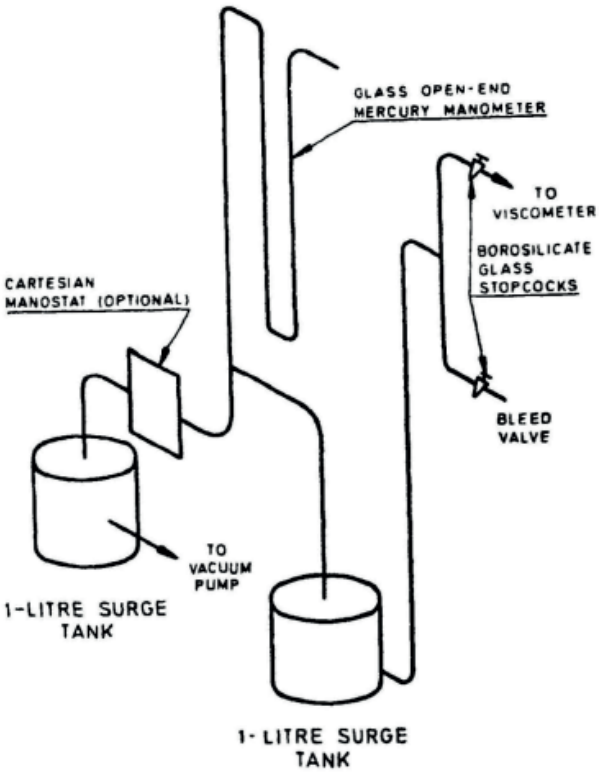


Fig. 7.2.4: Suggested Vacuum System

4.4 Vacuum System: A vacuum system capable of maintaining a vacuum to within ± 0.05cm of the desired level up to and including 30cm of mercury. One such system is shown in Fig. 7.2.4. The glass tubing of 6.35mm diameter and all glass joints should be completely airtight and no loss of vacuum should be permitted till the experiment is on. A vacuum or aspirator pump is suitable for the vacuum source.

NOTE: The vacuum measuring system for this test method must be standardized at least once a year.

4.5 Timing Device: A Stop watch or other timing device graduated in divisions of 0.2 s or less, and

accurate to within 0.1 percent when tested over a 60 min period.

5. Calibration of Viscometer

5.1 Reference Material: Viscosity standard (certified viscosity reference standard) may be used for calibration purposes.

5.2 Calibration: Charge a clean-dry viscometer by pouring the reference material to within ± 2 mm of fill line E (Fig. 7.2.1, 7.2.2 and 7.2.3). Place the charged viscometer in the viscometer bath maintained within ± 0.1°C at the calibration temperature. Establish a 30 ± 0.05cm vacuum in the vacuum system and connect it to the viscometer with valve closed in the line leading to the viscometer. After the viscometer has been in the bath for 30 ± 5 min, start the flow of liquid in the viscometer by opening the stop cock in the line leading to the vacuum system. Measure to within 0.5 s the time required for the leading edge of the meniscus to pass between timing marks F and G. Also measure to within 0.5 s the time required for the leading edge of the meniscus to pass between timing marks G and H. Calculate the calibration factor K for each bulb as follows:

$$K = V / t$$

Where:

K Viscometer bulb calibration factor poises/s at 30.0 cm Hg;

V Absolute viscosity of reference material at calibration temperature in poises; and

t Flow time, in seconds.

Repeat the calibration procedure using the same viscosity standard or another reference material. Record the average calibration constant K.

5.2.1 The duplicate determination of calibration constant K for each bulb shall be within 2 percent of the mean value. The value of viscometer

constants shall be expressed to the nearest 0.1 percent.

6. Procedure

6.1 Preparation of the Sample: Heat the sample to a temperature not more than 60°C for the tars and pitches and not more than 90°C for bitumen above their respective approximate softening point temperature respectively until it has become sufficiently fluid to pour. Transfer about 20 ml into a suitable container and maintain it to a temperature of 135 ± 5.5°C stirring occasionally to prevent local overheating and allow the entrapped air to escape.

6.1.1 Charge the viscometer by pouring the prepared sample to within ± 2mm of fill line E. Place the charged viscometer in an oven or bath maintained at 135 ± 5.5°C for a period of 10 ± 2 min to allow large air bubbles to escape.

6.2 Testing: Maintain the bath at the test temperature within ± 0.1°C. Place the charged viscometer vertically in the water bath with the help of a holder so that the uppermost timing mark is at least 2cm below the surface of the bath liquid. Establish a vacuum of 30 ± 0.05cm of mercury in the vacuum system and connect it to the viscometer with the valve closed. After the viscometer has remained in the bath for 30 ± 5 min open the valve and allow the asphalt to flow into the viscometer. Measure to within ± 0.5 s the time required for the leading edge of the meniscus to pass between successive pairs of timing marks. Upon completion of the test, remove the viscometer from the bath and place it in an inverted position in an oven maintained at 135 ± 5°C until asphalt is drained off thoroughly from the viscometer. Clean the viscometer thoroughly by rinsing several times with an appropriate solvent completely. Dry the tube by passing a slow stream of filtered dry air through the capillary for 2 min. Periodically clean the instrument with chromic acid to remove organic deposits. Rinse

thoroughly with distilled water and acetone and dry with clean air.

7. Calculation

7.1 Calculate and report the absolute viscosity to by the following equation:

Viscosity (in Poises) = K x t

Where:

K = selected calibration factor, in poise per second; and

t = flow time, in seconds.

NOTE: Measure the time required for the leading edge of the meniscus to pass between successive pairs of timing marks. Report the first flow time which exceeds 60s between a pair of timing marks, noting the identification of the pair of timing marks.

7.2 Always report the test temperature and vacuum with the viscosity test results. For example, viscosity at 60°C, 30 cm Hg vacuum in poises.

8. Precision

8.1 The duplicate test results should not differ by more than the following:

(a) Repeatability: The duplicate test results by the same operator using the same viscometer should not differ by more than 7 percent of their mean.

(b) Reproducibility: Results obtained by two laboratories should not differ by more than 10 percent of their mean.

Please also note that this precision is only for meant for measurement made at 60°C and hence should not be used for any other temperature measurements.

7.3 Determination of Kinematic Viscosity

1. Introduction: Viscosity shows how easily bitumen flows. The higher the viscosity of the bitumen, the harder it is to flow. Consequently, it behaves more like semi-solid matter. This chapter covers the method for the determination of kinematic viscosity of paving grade and cut-back bitumen and distillation residues of cut-backs. It is applicable to the materials having a viscosity range of 30-100 000 cSt.

2. Reference: IS-1206 (Part-3) : 2021 "Method or Testing Tar and Bituminous Materials – Determination of Viscosity. Part-3: Kinematic Viscosity".

3. Terminology: For the purpose of this standard the following definitions and those given in IS 334 shall apply.

3.1 Viscosity of a Newtonian Liquid: The ratio between the applied shear stress and rate of shear is called the coefficient of viscosity. This coefficient is thus a measure of the resistance to flow of the liquid. It is commonly called the viscosity of the liquid. The SI unit of viscosity is 1 Pa.s (1 $N.s/m^2$) and is called a Pascal-second. The CGS unit of viscosity is 1 g/cm.s (1 $dyne.s/cm^2$) and is called a poise (P). 1 Pa.s is equivalent to 10 P.

3.2 Density: It is mass per unit volume, the CGS unit of density is g/cm^3 and the SI unit of density is kg/m^3.

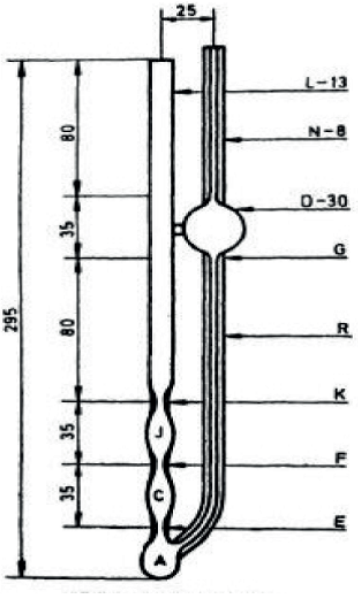
3.3 Kinematic Viscosity of a Newtonian Liquid: The ratio of the viscosity to the density of a liquid. It is a measure of the resistance to flow of a liquid under gravity.

3.4 Newtonian Liquid: A liquid in which the shear stress is directly proportional to the rate of shear. The constant ratio of shear stress to the rate of shear is called the coefficient of viscosity of the liquid. If this ratio is not constant then the liquid is non-Newtonian.

4. Apparatus

4.1 Viscometers : The capillary type of viscometer of borosilicate glass annealed suitable for the test are as given in Para 4.1.1 and 4.1.2.

4.1.1 Cannon-Fenske Viscometer for Opaque Liquids: Detailed drawings of the reverse-flow Cannon-Fenske viscometer is given in Fig. 7.3.1. The size, dimensions, approximate constant. kinematic viscosity range, capillary diameter and bulb volumes shall be as given in Table 7.1.

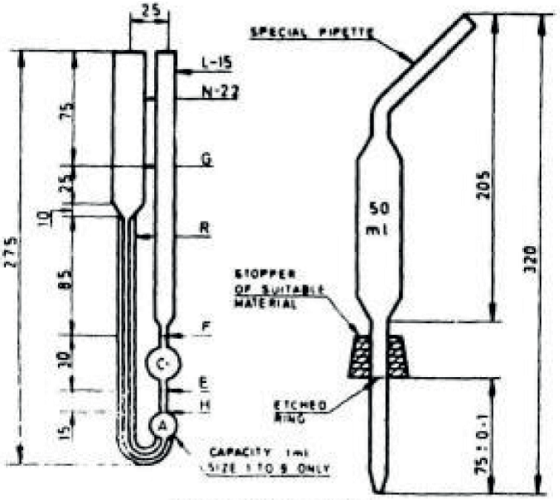


All dimensions are in mm

Fig. 7.3.1: Cannon-Fenske Viscometer

Table 7.1: Dimensions of Cannon-Fenske Viscometer

Size No.	Approx. Constant	Kinematic Viscometer Range	Inside Dia. of Tube	Inside Dia. of Tube	Volume Bulbs A	Volume Bulbs
	cSt/s	cSt	R mm (±2%)	N & G Tube E, F & K mm (±5%)	C & J ml (±5%)	D ml (±5%)
150	0.035	2.1 to 35	0.78	3.2	2.1	11
200	0.1	6 to 100	1.02	3.2	2.1	11
300	0.25	15 to 250	1.26	3.4	2.1	11
350	0.5	30 to 500	1.48	3.4	2.1	11
400	1.2	72 to 1200	1.88	3.4	2.1	11
450	2.5	150 to 2500	2.20	3.7	2.1	11
500	8	480 to 8000	3.1	4.0	2.1	11
600	20	1200 to 20000	4.00	4.7	2.1	13



All dimensions are in mm

Fig. 7.3.2: BS/IP/RF U-Tube Reverse Flow Viscometer

Table 7.2: Dimensions of BS/IP/RF-U Viscometer

Size No.	Approx. Constant	Kinematic Viscometer Range	Inside Dia. of Tube	Length of Tube	Inside Dia. at E, F, & G	Bulb C ml
	cSt/s	cSt	R mm (±2%)	R mm	mm	(±5%)
4	0.1	6-100	1.26	185	3.0-3.3	4.0
5	0.3	18-300	1.64	185	3.0-3.3	4.0
6	1.0	60-1000	2.24	185	3.0-3.3	4.0
7	3.0	180-3000	2.93	185	3.3-3.6	4.0
8	10	600-10000	4.00	185	4.4-4.8	4.0
9	30	1800-30000	5.5	185	6.0-6.7	4.0
10	100	6000-100000	7.00	210	7.7	4.0
11	300	18000-300000	10.00	210	10	4.0

4.1.2 BS U Tube Modified Reverse Flow Viscometer: The viscometer shall be made of clear borosilicate or other heat resistant glass free from visible defects. All glass tubing used in the construction of a single viscometer shall be of the same composition and the finished instrument shall be thoroughly annealed. The design and dimensions of the viscometer are given in Fig. 7.3.2 and Table 7.2.

4.2 Bath: A suitable bath for immersion of the viscometer so that the liquid reservoir or top of the capillary whichever it; uppermost is at least 20mm below the upper bath level. Provision shall be there for visibility of the viscometer and the thermometer. The efficiency of the stirring and the balance between heat losses and heat input shall be such that the temperature of the bath medium is maintained at ± 0.1°C over the entire length of the viscometer.

4.3 Thermometers: The thermometer shall be with least count reading of 0.1°C and shall be calibrated from competent agency.

4.4 Timing Device: Any timing device, such as stop-watch or stop clock capable of being read up to 0.5 s.

5. Calibration of Viscometer

5.1 Reference Material: Viscosity standard (Certified Viscosity reference standard) may be used for calibration purposes.

5.2 Calibration: Charge the clean dry viscometer by pouring the reference material. Place the charged viscometer in the viscometer bath maintained at calibration temperature within ± 0.1°C. Allow the charged viscometer to remain in the bath for 30 m to reach the test temperature. Measure to within 0.1 s the time required for the leading edge of the meniscus to pass from the first timing mark to the second. Calculate the viscometer constant C, as follows:

$$C = V / t$$

Where:

V = viscosity in centistokes for the standard liquid, and

t = efflux time, in seconds.

5.2.1 The duplicate determination of calibration constant K for each bulb shall be within 2 percent of the mean value. The value of Viscometer constants shall be expressed to the nearest 0.1 percent.

5.3 If the viscometer is used at a location other than the calibrating laboratory the constant C should be corrected for the difference in the acceleration of gravity "g" at the two locations as follows:

$$C2 = (g2 / g1) \times C1$$

Where:

C2 = Calibration constant in the testing laboratory,

C1= Calibration constant in the calibration

laboratory,

g2 = Acceleration of gravity at the testing laboratory, and

g1 = Acceleration of gravity at the calibration laboratory.

6. Preparation of Sample

6.1 Procedure for Cut-Back Bitumen and Oil Distillates: Open the sample container and mix the sample thoroughly by stirring for 30 s taking care to avoid entrapped air. For too viscous samples, heat the sealed container in a bath or oven maintained at about 60°C. Pour immediately 20 ml into a clean dry container having a capacity of about 30 ml and seal the container immediately.

6.2 Procedure for Bitumen-Heat: the sample to a temperature not more than 60°C for tars and pitches and not more than 90°C for bitumen above the corresponding approximate softening point temperature respectively until it attains pouring consistency. Stir it thoroughly and transfer approximately 20 ml in a 30 ml container. Local over-heating and entrapped air should be avoided.

7. Procedure

7.1 Procedure for Cannon-Fenske Viscometer

7.1.1 To charge the Carmon-Fenske viscometer invert the viscometer and apply suction to the tube L, immersing tube N in the liquid sample. Draw liquid through tube N filling bulbs D to fill mark G, wipe excess sample off tube N and invert the viscometer to its normal position. Align the viscometer vertically in the bath. Visual observation is sufficient. However, it can be done more accurately and quickly by suspending a plumb bulb in the tube L. Allow the viscometer to remain in the constant temperature bath for a sufficient time to ensure that the sample reaches

temperature equilibrium. Allow the viscometer to remain in the constant temperature bath a sufficient time to ensure that the sample reaches temperature equilibrium (10 min minimum and 30 min maximum). When the test temperature is reached, remove the stopper in the tubes N and L respectively and allow the sample to flow by gravity. Measure to the nearest 0.1 s the time required for the leading edge of the meniscus to pass from timing mark E to timing mark F. If this efflux time is less than 60 s, select a viscometer of smaller capillary diameter and repeat the operation.

7.1.2 Upon completion of the test, clean the viscometer thoroughly by several rinsing with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. Dry the tube by passing a slow stream of filtered dried air through the capillary for 2 min or until last trace of solvent is removed.

7.2 Procedure for BS U - Tube Modified Reverse Flow Viscometer

7.2.1 Mount the BS U-tube viscometer in the constant temperature bath keeping tube L vertical. Pour sample through tube N to a point just above filling mark G, allow the sample to flow freely through capillary R, taking care that the liquid column remains unbroken until the lower mark H and then arrest its flow by closing the timing tube with a cork or rubber stopper in tube L. Add more liquid, if necessary to bring the upper meniscus slightly above mark G. After allowing the sample to attain bath temperature and any air bubble to rise to the surface (usually about 20-30 min is required), gently loosen the stopper allowing the sample to flow until it is approximately at the lower filling mark H and press back the stopper to arrest flow. Remove

the excess sample above filling mark G by inserting the special pipette until its cork rests on top of the tube N and apply gentle suction until air is drawn through. The upper meniscus shall coincide with mark G. Allow the viscometer to remain in the constant temperature bath for a sufficient time to ensure that the sample reaches temperature equilibrium. It takes about 20 min at 38°C, 25 min at 100°C and 30 min at 135°C. Remove the stopper in the tube N and L respectively and allow the sample to flow by gravity. Measure to the nearest 0.1 s the time required for the leading edge of the meniscus to pass from timing mark E to timing mark F. If this efflux time is less than 60 s select a viscometer of smaller capillary diameter and repeat the operation.

7.2.2 Upon completion of the test, remove the viscometer from the bath and place it in an inverted position in an oven maintained at 135 ± 5°C until asphalt is drained off thoroughly from the viscometer. Clean the viscometer thoroughly by rinsing several times with an appropriate solvent completely. Dry the tube by passing a slow stream of filtered dry air through the capillary for 2 minutes. Periodically clean the instrument with chromic acid to remove organic deposits. Rinse thoroughly with distilled water and acetone and dry with clean air.

8. Calculation: Calculate the kinematic viscosity up to three significant figures with the help of following equation:

Kinematic viscosity cSt = C x t

Where:

C = Calibration constant of the viscometer, in centistokes per second, and

t = Efflux time, in seconds.

9. Report: Report always the test temperature along with the results as follows:

Kinematic Viscosity at 135 °C = X

where X = C.t

C = Calibration factor

t = Time

1 mm^2s^{-1} = 1cSt

10. Precision: The duplicate test results should not differ by more than the values given in Table 7.3.

10.1 Repeatability: The closeness of agreement between independent results obtained with the same method on identical test material, under the same conditions (same operator, same apparatus, same laboratory and after short intervals of time).

10.2 Reproducibility: The closeness of agreement between independent results obtained with the same method on identical test material but under different conditions (different operators, different apparatus, different laboratories and/or after different intervals of time).

Table 7.3: Precision of Test Results

Sl. No.	Material	Repeatability	Reproducibility
		% of Mean	% of Mean
i	Bitumen at 135°C	1.8	8.8
ii	Cut back Bitumen at 60°C		
	(a) Below 3000 cSt	1.5	3
	(b) 3000 to 6000 cSt	2.0	9.9
	(c) Above 6000 cSt	8.9	10.0

7.4 Determination of Flash Point

1. Introduction: The "flash point" of a material is the lowest temperature at which the application of test flame causes the vapours from the material momentarily catch fire in the form of a flash under specified conditions of test. In practical view the "fire point" is the lowest temperature at which the application of test flame causes the material to ignite and burn at least for 5 seconds under specified conditions of test. At high temperatures, bituminous materials emit hydrocarbon vapours which are susceptible to catch fire. Therefore, the heating temperature of bituminous material should be restricted to avoid hazardous conditions. Flash point and fire point tests are used to determine the temperature to which bituminous material can safely be heated.

This chapter describes a procedure for determination of flash and fire points of petroleum products using the Cleveland open cup apparatus. It is applicable to petroleum products having open cup flash points between 79°C and 400°C.

2. Reference: IS-1448 [P:69]:2019 "Methods of Test Petroleum and its Products. P:69- Determination of Flash and Fire Points – Cleveland Open Cup Method".

3. Terms and definitions: For the purpose of this standard, following terms and definitions apply:

3.1 Flash point: Lowest temperature of the test portion, corrected to a standard atmospheric pressure of 101.3 kPa, at which application of a test flame causes the vapour of the test portion to ignite under the specified conditions of test.

3.2 Fire point: Lowest temperature of the test portion, corrected to a barometric pressure of 101.3 kPa, at which application of a test flame causes the vapour of the test portion to ignite and sustain burning for a minimum of 5 sec under the specified conditions of test.

4. Principle: The test cup is filled to a specified level with the test portion. The temperature of the test portion may be increased rapidly (50 C/min to 170 C/min) at first and then at a slow constant rate (50 C/min to 6°C/min) as the flash point is approached. At specified temperature intervals, a small test flame is passed across the test cup.

The lowest temperature at which application of the test flame causes the vapour above the surface of the liquid to ignite is taken as the flash point at ambient barometric pressure. To determine the fire point, the test is continued until the application of the test flame causes the vapour above the test portion to ignite and burn for at least 5 sec. The flash point and fire point obtained at ambient barometric pressure are corrected to standard atmospheric pressure using a formula.

5. Chemicals and materials

5.1 Cleaning solvent, for removal of traces of sample from the test cup and cover. The choice of solvent depends upon the previous material tested and the tenacity of the residue. Low volatility aromatic (benzene-free) solvents may be used to remove traces of oil, and mixed solvents can be efficacious for the removal of gum type deposits.

5.2 Verification liquids, certified reference material (CRM) or secondary working standards (SWS).

5.3 Steel wool, any grade capable of removing carbon deposits without damage to the test cup.

6. Apparatus

6.1 Cleveland open cup apparatus: (Fig. 7.4.1) as per the specifications given in the Anne. A of the IS 1448 [P:69]:2019. If automated testers are used, the user shall ensure that all the manufacturer's instructions for adjusting and operating the instrument are followed.



Fig. 7.4.1: Cleveland Open Cup Apparatus

6.2 Shield, to cover at least three sides of the test cup. The apparatus may include built-in draught shield.

6.3 Temperature measuring device, which shall meet the requirements for accuracy and have the response as specified in Annex. B of the IS 1448 [P:69]:2019.

6.4 Barometer, reading absolute pressure accurate to 0.5 kPa with a resolution of 0.1 kPa. Barometers pre-corrected to give sea level readings, such as those used at weather stations and airports, shall not be used.

7. Preparation of apparatus

7.1 Location of apparatus: Place the apparatus on a level and steady surface in a draught-free room. Shield the top of the manual apparatus from strong light by any suitable means to permit detection of the flashpoint. When draughts cannot be avoided, it is recommended good practice to surround the apparatus with a shield.

7.2 Cleaning the test cup: Wash the test cup with an appropriate solvent to remove any traces of gum or residue remaining from a previous test. Dry the test cup using a stream of clean air to ensure complete removal of the solvent used. If any deposits of carbon are present, remove them by rubbing with steel wool.

7.3 Preparing the test cup: Before use, cool the test cup to at least 56°C below the expected flash point.

7.4 Assembly of apparatus: Support the liquid in glass thermometer in a vertical position with the bottom of the bulb (6.4 ± 0.5) mm from the bottom of the test cup, and located at a point halfway between the Centre and side of the test cup on a diameter perpendicular to the arc (or line) of the sweep of the test flame, and on the side opposite to the test flame applicator. It is not necessary to restrict electronic temperature measuring devices to be mounted vertically, provided their performance is in accordance with the requirements in the test method. The vertical position of the temperature measuring device may be set by lowering until it contacts the bottom of the test cup, and then raise it by (6.4 ± 0.5) mm.

7.5 Verification of apparatus

7.5.1 Verify the correct functioning of the apparatus at least once a year by testing a certified reference material (CRM). The result obtained shall be equal to or less than $R/\sqrt{2}$ from the certified value of the CRM, where R is the reproducibility of the method.

It 1s recommended practice during verification of an automated apparatus to visually observe the detected flash point for correct operation.

It is recommended that more frequent verification checks are made using secondary working standards (SWSs) or other verification materials with a proven value.

7.5.2 The numerical values obtained during the verification check shall not be used to provide a bias statement, nor shall they be used to make any correction to the flash points subsequently determined.

7.5.3 It is good practice to select a CRM or SWS that has a certified value similar to the flash point of products being tested.

8. Sampling

8.1 Place samples in tightly sealed containers, appropriate to the material being sampled, and for safety purposes, ensure that the sample container is only filled 85% to 95% of its capacity.

8.2 Store the samples in conditions to minimize vapour loss and pressure build up. Avoid storing the samples at temperatures in excess of 30° C.

8.3 Subsampling: Subsample at a temperature at least 56°C below the expected flash point. If a part of the original sample is to be stored prior to testing, ensure that the container is filled to more than 50 % of its capacity.

NOTE: The results of flash point determinations can be affected if the sample volume falls below 50% of the container capacity.

8.4 Samples containing undissolved water: Flash point results can be affected by the presence of water; if a sample contains undissolved water, decant a water-free aliquot prior to mixing.

NOTE: Flash and fire point results can be affected by the presence of water, and splashing can occur.

8.5 Samples that are liquid at ambient temperature: Mix samples by gently shaking by hand prior to the removal of the test portion, taking care to minimize the loss of volatile components, and proceed in accordance with Para 9.

8.6 Samples that are semisolid or solid at ambient temperature: Heat the sample in its container in a heating bath or oven at a temperature not exceeding 56°C below the expected flashpoint. Ensure that high pressures do not develop in the container. Avoid overheating the sample as this could lead to the loss of volatile components. After gentle agitation, proceed in accordance with Para 9.

9. Procedure for determining flash point

9.1 Samples that can form a skin during testing may be tested by removing the skin formed as described in Para 9.6 to 9.8.1, An alternative procedure is given in Annex. D of IS 1448 [P:69]: 2019.

9.2 Record the absolute barometric pressure μsing a barometer in the vicinity of the apparatus at the time of test.

NOTE: It is not considered necessary to correct the barometric pressure reading to 0°C, although some Barometers are designed to make this correction automatically.

9.3 Fill the test cup at ambient or elevated temperature (see Para 9.6) so that the top of the meniscus is level with the filling mark. Position the test cup on the centre of the heating plate. If too much sample has been added to the test cup, remove the excess using a pipette or other suitable device; however, if there is any sample on the outside of the apparatus, empty, clean and refill It. Destroy or remove any air bubbles or foam on the surface of the sample while maintaining the correct level of test portion in the test cup. If foam persists in the final stages of the test, discard the result.

9.4 Light the test flame and adjust it to a diameter between 3.2mm and 4.8mm. As a safety practice, it is strongly advised, before heating the test cup and test portion, to pass the test flame across the test portion in the test cup to check for the presence

of unexpected volatile material. Thereafter, it is recommended to test for a flash every 10°C until the test portion is within 56°C of the expected flashpoint.

9.5 Apply heat initially so that the rate of temperature rise of the test portion is 5°C/min to 17°C/min. When the test portion temperature is approximately 56 °C below the expected flash point, decrease the heat so that the rate of temperature rise for the last 28°C before the expected flash point is 5°C/min to 6°C/min.

9.6 Avoid disturbing the vapours in the test cup by careless movements or breathing near the test cup.

9.7 Apply the test flame when the temperature of the test portion is 28°C below the expected flash point. The test flame is applied in one direction with a smooth continuous motion, taking (1 ± 0.1) sec, to pass across the centre of the test cup, at right angles to the diameter which passes through the thermometer, either in a straight line or along the circumference of a circle having a radius of at least 150mm. The Centre of the test flame shall move in a horizontal plane not more than 2mm above the plane of the upper edge of the test cup. For the next test flame application, pass the flame in the opposite direction.

NOTE: Some automated apparatus pass the test flame in one single direction.

9.8 If a flash is detected on this application of the test flame or during a preliminary application (see Para 9.4), discontinue the test and repeat the test using a fresh test portion with an expected flash point of at least 280C below the previous test value.

9.8.1 If a skin forms over the test portion, carefully move it aside with a spatula or comb and continue the determination.

9.8.2 If a flash has not been detected, continue applying the test flame each time thereafter at a

temperature reading that is a multiple of 20C.

NOTE: Higher flash points have been detected when skins formed on the surfaces of test portions have not been removed.

9.9 When testing a sample whose expected flash point temperature is not known, bring the test portion in the lest cup to a temperature no greater than 50°C, or if the sample required heating to be transferred into the test cup, bring the test portion In the test cup to that temperature. Apply the test flame, in the manner described in Para 9.7, beginning at least 50C above the starting temperature. Continue heating the test specimen at 5°C/min to 6°C/min and testing the test specimen every 2°C as described in Para 9.7 until the flash point is obtained.

This value may be used as the expected flashpoint when a fresh test portion is tested in the standard mode of operation. Flash point results determined in an unknown expected flash point mode should be considered approximate.

9.10 Record as the detected flash point, the temperature of the test portion, read on the thermometer, when application of the test flame causes the vapours of the test portion to ignite at any point on the surface of the test portion and a large flame propagate over the surface of the test portion, under the specified conditions of test. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame.

10. Procedure for determining fire point: To determine the fire point, after carrying out the procedure specified in Para 9, continue heating so that the test portion temperature increases at a rate of 5° C/min to 6° C/min. Continue the application of the test flame at 2° C intervals until the vapour of the test portion ignites and continues to burn for at least 5 sec. Record the temperature at this point as the detected fire point of the sample.

If the fire persists for more than 5 sec, extinguish it with a cover made of metal or other fire-resistant material fitted with a handle.

11. Calculation

11.1 If the barometric pressure reading is measured in a unit other than kPa, convert it to kPa using one of the following formulae:

Reading in hPa x 0.1 = kPa

Reading in mbar x 0.1 = kPa

Reading in mmHg x 0.1333 = kPa

11.2 Calculate the corrected flash point or fire point, tc, using the following formula:

$$tc = td + 0.25 (101.3 - p)$$

where

td is the detected flash point or fire point at ambient barometric pressure, in °C;

p is the absolute barometric pressure, in kPa;

0.25 is a constant with dimensions °C/kPa;

101.3 is used as the standard atmospheric pressure in kPa.

12. Expression of results: Record the following:

(a) The flash point, corrected to standard atmospheric pressure, rounded to the nearest 10 C, and, if required,

(b) The fire point, corrected to standard atmospheric pressure, rounded to the nearest 1° C.

13. Precision

13.1 Repeatability (r): The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method,

exceed the following values in only one case in 20.

Flash point, R = 8^0C

Fire point, R = 14^0C

13.2 Reproducibility (R): The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in 20.

Flash point, R = 18^0C

Fire point, R = 14^0C

14. Test report: The test report shall contain at least the following information:

(a) A reference to the testing standard;

(b) The type and complete identification of the product tested;

(c) The result of the test (see Para 12);

(d) Whether the procedure in Annex. D was used or not;

(e) Any deviation, by agreement or otherwise, from the procedure specified;

(f) The date of the test.

7.5 Determination of Solubility in Trichloroethylene

1. Introduction: The pure bitumen is completely soluble in some solvents like carbon di-sulphide or trichloroethylene. Hence, the solubility test is carried out to know the amount of pure bitumen in a sample of bitumen. This chapter covers the solubility in carbon disulphide or trichloroethylene for asphaltic bitumen and native asphalts.

2. Reference: IS-1216:1978 "Methods for Testing Tar and Bituminous Materials. Determination of Solubility in Carbon Di-sulphide or Trichloroethylene".

(A) Method A (For Asphaltic Bitumen)

3. Apparatus

3.1 Crucible: Gooch Crucible (Fig. 7.5.1)

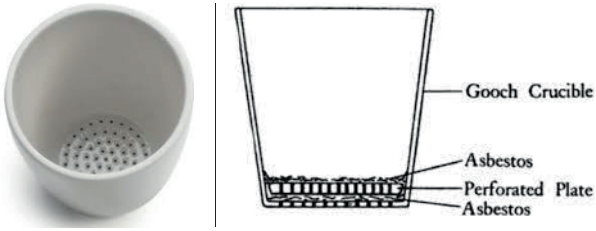


Fig. 7.5.1: Gooch Crucible

3.2 Conical Glass Flask - of 200 ml capacity.

4. Solvent

4.1 Carbon disulphide: Redistilled grade, conforming to IS:717.

4.2 Trichloroethylene: Conforming to IS:245.

5. Preparation of the Material: If the material contains water, heat it to a temperature not exceeding 130°C until the water has been removed, stirring constantly, when possible, during heating. Perform this operation as expeditiously as possible.

6. Preparation of Gooch Crucible: Insert the filter tube in the stopper of the filtering flask, set the Gooch crucible in the filter tube, and connect the flask to the suction pump. Fill the crucible with some of the suspension of asbestos in the water, allow it to settle partly in the crucible and apply a light suction to draw off the water, leaving a firm mat of asbestos in crucible. Add more suspended asbestos and repeat the process until a mat weighing 0.5 ± 0.1 g is built up after drying. Wash the asbestos mat thoroughly with water and dry in the oven at a temperature of 150°C. Coll the crucible in the desiccator, weigh and replace it in the dry filter tube supported in the clean, dry filtering flask.

6.1 In the determination, the asbestos apparently absorbs a small amount of soluble bitumen (usually 1 to 5 mg/g of asbestos) which is not removed by a subsequent washing with solvent. The weight of asbestos used, therefore, shall be kept within the specified limits to ensure reproducible results.

7. Procedure: Weigh about 2 g of the dry material correct to the nearest 0.001 g into 1 200 ml conical flask and add 100 ml of carbon disulphide or trichloroethylene. Stir the contents of the flask, and then allow it to stand for a period of one hour. Filter the contents of the flask through the Gooch crucible prepared as described under Para 6 which has been weighed to the nearest 0.001 g. Moisten the asbestos pad with carbon disulphide before commencing filtration, and filter at a rate of not more than two drops per second at first. The filtrate shall be quite clear. Transfer the insoluble matter remaining in the flask to the crucible by washing out the flask with a stream of carbon disulphide or trichloroethylene from a wash bottle. Wash the material retained in the crucible with successive small amounts of carbon disulphide or trichloroethylene until a filtrate is obtained which is not discoloured. Allow the crucible to dry in air for 30 minutes, after which place it in an oven at 100 to 110^{0}C for one hour. Allow the crucible to cool in a desiccator and then weigh.

8. Calculation and Report: Calculate the matter soluble in carbon disulphide or trichloroethylene as follows:

Matter soluble in carbon disulphide or trichloroethylene (in %) =

$$[(W_1 - W_2) / W_1] \times 100$$

where:

W_1 = Weight in g of dry sample taken for the test, and

W_2 = Weight in g of insoluble material retained in the Gooch crucible.

Report the result to the nearest 0.05 percent as the matter soluble in carbon disulphide or trichloroethylene of the dry material.

9. Precision: Test results shall not differ from the mean by more than the following:

Matter soluble in Carbon Disulphide or Trichloroethylene	Repeatability	Reproducibility
Below 98 percent	0.5	1.0
98 to 100 percent	1.0	0.2

(B) Method B (For Native Asphalts)

3. Apparatus

3.1 Glass Tap Funnel: Approximately 100 mm in diameter, the stem fitted with a tap and the top ground flat.

3.2 Glass Plate: To cover the funnel, about 120 mm in diameter, with a hole of about 16mm diameter in centre.

3.3 Glass Funnel: Smaller than that described under Para 3.1, with its stem passing through a cork placed in the hole in the glass plate, the lower end of the

stem reaching half-way down the tap funnel.

3.4 Filter Papers: Two, Whatman No. 5 or equivalent filter papers, 185mm in diameter, in a suitable oven, cooled in a desiccator, counterpoised and folded together as a cone.

4. Solvent and Material

4.1 Carbon Disulphide: Redistilled, conforming to IS:717.

4.2 Cement: Suitable for sealing the glass plate to the funnel. It may be prepared with 10 g of gelatin, 80 ml of water and 20 g of glycerine.

5. Preparation of Sample: If the sample contains water, heat a 100 g portion at a temperature not exceeding 130°C, the material being constantly stirred, when possible, until the rate of loss in weight of the sample does not exceed 0.1 g during a 15-minute period of heating.

5.1 If the loss in weight on drying exceeds 0.1 percent, determine the water content of another sample of the the material in accordance with IS:1211.

6. Procedure: Dry the two filter papers in an oven at 100 to 110^{0}C, cool in a desiccator, counterpoise, fold together and place in the funnel. Weigh about 2 g of the dry material, to the nearest 0.001 g in the filter papers and secure the cover to the funnel by means of the cement. Now add carbon disulphide or trichloroethylene through the small funnel until the filter paper is about half filled, and then allow it to stand for 30 minutes. Draw the solution off the tap. Place a watch glass on the small funnel to minimize evaporation. Close the tap and introduce a further quantity of carbon disulphide or trichloroethylene through the small funnel and draw the solution off after 30 minutes. Repeat this procedure until the solvent drawn off is no longer discoloured. When filtration is completed, remove the cover and allow the filter papers to dry in air for 30 minutes before placing them in a ventilated air oven at 100 to 110°C for one hour. Next place the filter papers in a desiccator and when cool, the inner filter paper and

contents, using the outer filter paper as a counterpoise.

7. Calculation: Calculate the solubility of the dry material as follows:

Matter soluble in carbon disulphide or trichloroethylene (in %) =

$$[(W_1 - W_2) / W_1] \times 100 \times [100 / (100 + W_3)]$$

where:

W_1 = Mass in g of dry sample taken for the test;

W_2 = Mass in g of recovered insoluble matter; and

W_3 = Water content of sample, if determined.

8. Report: Report the result to the nearest 0.05 percent as the matter soluble in carbon disulphide or trichloroethylene.

9. Precision: Test result shall not differ from the mean by more than the following:

Repeatability	0.5
Reproducibility	0.5

7.6 Determination of Softening Point

1. Introduction: Softening point is defined as the temperature at which bitumen softens beyond some specified softness. This test gives an idea of the temperature at which the bitumen attain certain viscosity. Softening point should be higher than the hottest day temperature, otherwise bitumen may sufficiently soften and result in bleeding and development of ruts. Bitumen with higher softening point may be preferred in warmer places. This chapter covers the determination of the softening point of bitumen using the ring-and-ball apparatus immersed in distilled water (up to 80° C) or glycerine (above 80° C)

2. Reference: IS-1205:2022 "Methods for Testing Tar and Bituminous Materials. Determination of Softening Point – Ring and Ball Apparatus".

3. Apparatus

3.1 Ring and Ball Apparatus: A convenient form of apparatus is illustrated in Fig. 7.6.1.



Fig. 7.6.1: Ring and Ball Apparatus

3.1.1 Steel Balls: Two; each 9.5mm in diameter and weighing 3.50 ± 0.05 g.

3.1.2 Brass Rings: Two; the rings shall be tapered and shall conform to the following dimensions:

Depth	6.4 ± 0.1 mm
Inside diameter at bottom	15.9 ± 0.1 mm
Inside diameter at top	17.5 ± 0.1 mm
Outside diameter	20.6 ± 0.1 mm

For convenience in mounting the rings in the support, the outside diameter of the ring at the bottom may be smaller, but shall be not less than 19.0 mm.

3.1.3 Ball Guide: A convenient form of ball centering guide.

3.1.4 Support: Any means of supporting the rings may be used provided the following conditions are observed:

(a) The rings shall be supported in a horizontal position with the upper surface of the rings 50mm below the surface of the bath liquid.

(b) There shall be a distance of exactly 25mm between the bottom of the rings and the top surface of the bottom plate of the support, if any, or the bottom of the bath.

(c) The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the rings, and within 10 mm of the rings, but not touching them.

3.1.5 Thermometer: The dimensions, tolerances and graduations of the thermometer shall be as follows and it shall be calibrated and with LC 0.1° C:

	Low Temperature	**High Temperature**
Range	-2^0 to 80^0C	30^0 to 200^0C
Graduation at each	0.2^0C	0.5^0C
Longer lines at each	1^0C	1^0C

Figured at each	2^0C	5^0C
Immersion, mm	Total	Total
Overall length	378 to 384 mm	378 to 384 mm
Length of graduated portion	243 to 279 mm	243 to 279 mm
Distance from bottom of bulb to 0 °C	75 to 90 mm	75 to 90 mm
Scale error, Max	± 0.2	± 0.3

3.1.6 Bath: A heat resistance glass vessel not less than 85mm in diameter and 120mm in depth. The bath liquid shall be freshly boiled with distilled water when testing materials having softening points up to 80^0C, and pure glycerin for materials having softening points above 80° C.

3.1.7 Stirrer: Manual or mechanical, which operates smoothly to ensure uniform heat distribution at all times throughout the bath. The stirrers shall be so placed that the moulds are not disturbed when the stirrer is in operation.

4. Procedure

4.1 Preparation of Test Sample: Heat the material to a temperature between 75° C and 100° C above its softening point, stir until it is completely fluid and free from air bubbles and water, and filter, if necessary, through IS Sieve 30 (see IS:460). Place the rings, previously heated to a temperature approximating to that of the molten material, on a metal plate which has been coated with a mixture of equal parts of glycerin and dextrin, and fill with sufficient melt to give an excess above the level of the ring when cooled. After cooling for 30 min in air, level the material in the ring by removing the excess with a warmed, sharp knife.

4.2 Materials of Softening Point up to 80° C: Assemble the apparatus with the rings, thermometer

and ball guides-in position, and fill the bath to a height of 50mm above the upper surface of the rings with freshly boiled distilled water at a temperature of 5° C. Maintain the bath at a temperature of 5° C for 15 min after which place a ball, previously cooled to a temperature of 5° C, by means of forceps in each ball guide. Apply heat to the bath and stir the liquid so that the temperature rises at a uniform rate of 5 ± 0.5° C per minute until the material softens and allows the ball to pass through the ring. The rate of temperature rise shall not be averaged over the period of the test, and any test in which the rate of temperature rise does not fall within the specified limits after the first three minutes shall be rejected. Make the determination in duplicate.

4.3 Materials of Softening Point Above 80° C: The procedure for materials of softening point above 80° C is similar to that described under Para 4.2 with the difference that glycerine is used in place of water in the bath and the starting temperature of the test is 30 + 1° C. Make the determination in duplicate.

5. Report

5.1 Record for each ring and ball, the temperature shown by the thermometer at the instant the sample surrounding the ball touches the bottom plate of the support, if any, or the bottom of the bath.

5.2 Report to the nearest 0.5° C the mean of the temperature recorded in duplicate determinations, without correction for the emergent stem of the thermometer, as the softening point.

NOTES:

1. For a given bitumen specimen, the softening point determined in a water bath will be lower than that determined in a glycerine bath.

2. To convert softening points slightly above 80°C determined in water to those determined in glycerine, the correction for bitumen is + 4.2° C. For referee

purposes, repeat the test in a glycerine bath.

3. Under any circumstances, if the mean of the two temperatures determined in water is 85.0° C or higher, repeat the test in a glycerin bath.

6. Precision

6.1 Test results shall not differ from the mean by more than the following:

Softening Point ^{0}C	Repeatability ^{0}C	Reproducibility ^{0}C
40 to 60	1.0	5.5
61 to 80	1.5	5.5
81 to 100	2.0	5.5
101 to 120	2.5	5.5
121 to 140	3.0	5.5

7. Precautions

7.1 Only freshly boiled distilled water shall, be used in the test, as otherwise air bubbles may form on the specimen and affect the accuracy of the results.

7.2 The prescribed rate of heating shall be rigidly adhered to for ensuring accuracy of results.

7.3 A sheet of filter paper or thin amalgamated sheet, placed on the bottom of the glass vessel and conveniently weighed would prevent the material from sticking to the glass vessel, and considerable time and trouble in cleaning would thereby be saved.

7.7 Determination of Ductility

1. Introduction: Ductility of bitumen is its property to elongate under traffic load without getting cracked in road construction works. Temperature changes cause bitumen to expand and contract. If the bitumen is not ductile enough, cracking will occur. Therefore, bitumen must have sufficient ductility to be resistant to temperature changes and it must not tear in heavy traffic and must adhere well to the aggregates. This chapter covers the method of determination of ductility of distillation residue of cutback bitumen, blown type bitumen and other bituminous products.

2. Reference: IS-1208:1978 (Reaafirmed-2010) "Methods for Testing Tar and Bituminous Material – Determination of Ductility".

3. Apparatus

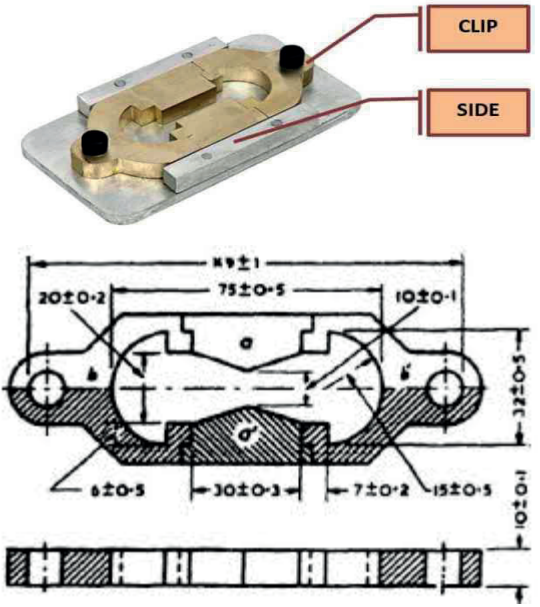


Fig. 7.7.1: Mould for Ductility Test

3.1 Mould: Made of brass with the shape, dimensions and tolerances as shown in Fig. 7.7.1. The ends b and b' are known as clips, and the parts a and a' as sides of the mould. The dimensions of the mould shall be such that when properly assembled, it will form a briquette specimen having the following dimensions:

Total length	75.0 ± 0.5 mm
Distance between clips	30.0 ± 0.3 mm
Width at mouth of clip	20.0 ± 0.2 mm
Width at minimum cross-section	10.0 ± 0.1 mm
(half way between clips)	
Thickness throughout	10.0 ± 0.1 mm

3.2 Water Bath: A bath preferably with a thermostat maintained within ± 0.1^0C of the specified test temperature, containing not less than 10 litres of water, the specimen being immersed to a depth of not less than 100mm and supported on a perforated shelf not less than 50mm from the bottom of the bath.

3.3 Testing Machine: For pulling the briquette of bituminous material apart, any apparatus may be used which is so constructed that the specimen will be continuously immersed in water as specified under Para 4.3 while the two clips are pulled apart horizontally with minimum vibrations at a uniform speed as specified and with suitable arrangement for stirring the water for attaining uniformity in temperature.

3.4 Thermometer: Conforming to the following requirements:

Characteristic	Requirement
Range	0 to 44^0C
Graduations	0.2^0C
Immersion	65mm

Overall length	340 ± 10 mm
Stem diameter	5.5 to 8.0 mm
Bulb length	10 to 16 mm
Bulb diameter	Not larger than stem diameter
Length of graduated portion	150 to 190 mm
Longer lines at each	1°C and 5°C
Figured at each	5°C
Scale	± 0.2^0C

4. Procedure

4.1 Unless otherwise specified, the test shall be conducted at a temperature of 27.0 ± 0.5° C and at a rate of pull of 50.0 ± 2.5 mm/min.

4.1.1 When a low temperature ductility test is desired, the test shall be made at a temperature of 4.0 ± 0.5° C and at a rate of pull 10.0 ± 0.5 mm/min.

4.2 Completely melt the bituminous material to be tested to a temperature of 75 to 100° C above the approximate softening point until it becomes thoroughly fluid. Assemble the mould on a brass plate and in order to prevent the material under test from sticking, thoroughly coat the surface of the plate and interior surfaces of the sides of the mould (a and a' in Fig. 7.7.1) with a mixture of equal parts of glycerine and dextrin. In filling, pour the material in a thin stream back and forth from end to end of the mould until it is more than level full. Leave it to cool at the room temperature for 30 to 40 min, and then place in a water bath maintained at the specified temperature for 30 min after which cut off the excess bitumen by means of a hot, straight edge putty knife or spatula so that the mould shall be just level full.

4.3 Testing: Place the brass plate and mould with briquette specimen, in the water bath and at the

specified temperature for about 85 to 95 minutes. Then remove the briquette from the plate, detach the side pieces, and test the briquette immediately.

4.3.1 Attach the rings at each end of the to the clips to the pins or hooks in the resting machine and pull the two clips apart horizontally at a uniform speed as specified until the briquette ruptures. Measure the distance in centimetres through which the clips have been pulled to produce rupture. While the test is being made, make sure that the water in the tank of the testing machine covers the specimen both above and below it by at least 25mm and is maintained continuously within ±0.5° C of the specified temperature.

5. Report

5.1 A normal test is one in which the material between the two clips pulls out to a point or to a thread and rupture occurs where the cross-sectional area is a minimum. Report the average of three normal tests as the ductility of the sample, provided the three determinations be within ± 5 percent of their mean value.

5.1.1 If the value of three determinations do not lie within ± 5 percent of their mean but the two higher values are within ± 5 percent of their mean then record the mean of the two higher values as test result.

5.2 If the bituminous material comes in contact with the surface of the water or the bottom of the bath, the test shall not be considered normal. Adjust the specific gravity of the water in the bath by the addition of either methyl alcohol or sodium chloride so that the bituminous material does not either come to the surface of the water, or touch the bottom of the bath at any time during the test.

5.3 If a normal test is not obtainable on three

successive tests. Report the ductility as being unobtainable under the conditions of test.

6. Precision

6.1 Test results shall not differ by more than the following:

Repeatability	10 percent of the mean
Reproducibility	10 percent of the mean

7. Precautions

7.1 The plate upon which the mould is placed shall be perfectly flat and level so that the bottom surface of the mould touches it throughout.

7.2 In filling the mould, care shall be taken not to disarrange the parts and thus distort the briquette and to see that no air pocket shall be within the moulded sample.

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