Important Tests on Cement (IS 4031)

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Indian Standard

METHOD OF PHYSICAL TESTS FOR HYDRAULIC CEMENT

PART 1 DETERMINATION OF FINENESS BY DRY SIEVING

(*Second Revision*)

1 SCOPE

1.1 This standard (Part 1) covers the procedure for determining the fineness of cement by dry sieving as represented by the mass of residue left on a standard 90 μm IS Sieve.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
460	Specification for test sieves:
(Part 1) : 1985	Wire cloth test sieves (*third revision*)
(Part 3) : 1985	Methods of examination of apertures of test sieves (*third revision*)
3535 : 1986	Methods of sampling hydraulic cements (*first revision*)
5165 : 1969	Interchangeable conical ground - glass joints

3 SAMPLING AND SELECTION OF TEST SPECIMENS

3.1 The samples of the cement shall be taken according to the requirements of IS 3535:1986 (*see* **2.1**) and the relevant standard specification for the type of cement being tested. The representative sample of the cement selected as above shall be thoroughly mixed before testing.

4 SIEVING METHOD

4.1 Principle

The fineness of cement is measured by sieving it on standard sieve. The proportion of cement of which the grain sizes are larger than the specified mesh size is thus determined.

A reference sample having a known proportion of material coarser than the specified mesh size is used for checking the specified sieve.

4.2 Apparatus

4.2.1 *Test Sieve*

It comprises 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 μm mesh sieve cloth of woven stainless steel, or other abrasion-resisting and non-corrodible metal wire.

The sieve cloth shall comply with the requirements of IS 460 (Part 1) : 1985 and IS 460 (Part 3) : 1985 and shall be free of visible irregularities in mesh size when inspected optically by the methods of IS 460 (Part 3) : 1985. A tray fitting beneath the sieve frame and a lid fitting above it shall be provided to avoid loss of material during sieving.

4.2.2 *Balance*

Capable of weighing up to 10 g to the nearest 10 mg.

4.2.3 *Brush*

A nylone or pure bristle brush, preferably with 25 to 40 mm bristle, for cleaning the sieve.

4.3 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.

4.4 Procedure

4.4.1 *Determination of the Cement Residue*

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.

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, R_1, 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.

Repeat the whole procedure using a fresh 10 g sample

to obtain R_2. Then calculate the residue of the cement R as the mean of R_1 and R_2 as a percentage, expressed to the nearest 0.1 percent.

When the results differ by more than 1 percent absolute, carry out a third sieving and calculate the mean of the three values.

The sieving process is carried out manually by a skilled and experienced operator.

NOTE - Alternatively a sieving machine may be used provided that it can be shown to give the same results as the manual operation.

4.4.2 *Checking the Sieve*

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.

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. Carry out the sieving procedure as in 4.4.1 including the repeat determination of residue to yield two values p_1 and P_2 expressed to the nearest 0.1 percent.

The two values of P_1 and P_2 for a satisfactory sieve should differ by not more than 0.3 percent. Their mean *P* characterizes the state of the sieve.

Given the known residue on the 90 μm mesh of the reference material, R_o, calculate R_o/P as the sieve factor, F, expressed to the nearest 0.01. The residue, R, determined as in 4.4.1 shall be corrected by multiplying by F, which may have a value of 1.00 ± 0.20.

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.

5 EXPRESSION OF RESULTS

Report the value of R, to the nearest 0.1percent, as the residue on the 90 µm 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.

ANNEX A
(*Foreword*)

COMMITTEE COMPOSITION

Cement and Concrete Sectional Committee, CED 2

Chairman	*Representing*
DR H.C. VISVESVARYA	In personal capacity (*University of Roorkee, Roorkee 247 667*)
Members	
SHRI H. BHATTACHARYA	Orissa Cement Limited, New Delhi
SHRI G. R. BHARTIKAR	B.G. Shirke & Co , Pune
DR A. K. CHATTERJEE	The Associated Cement Companies Ltd, Bombay
SHRI S. H. SUBRAMANIAN (*Alternate*)	
CHIEF ENGINEER (DESIGN)	Central Public Works Department , New Delhi
SUPERINTENDING ENGINEER, (S&S) (*Alternate*)	
CHIEF ENGINEER, NAVAGAM DAM	Sardar Sarovar Narmada Nigam Ltd, Gandhinagar
SUPERINTENDING ENGINEER, QCC (*Alternate*)	
CHIEF ENGINEER , RESEARCH-CUM-DIRECTOR	Irrigation and Power Research Institute, Amritsar
RESEARCH OFFICER , CONCRETE TECHNOLOGY (*Alternate*)	
DIRECTOR	A.P. Engineering Research Laboratories, Hyderabad
JOINT DIRECTOR (*Alternate*)	
DIRECTOR (CMDD) (N&W)	Central Water Commission, New Delhi
DEPUTY DIRECTOR (CMDD) (NW&S) (*Alternate*)	
SHRI K. H. GANGWAL	Hyderabad Industries Ltd, Hyderabad
SHRI V. PATTABHI (*Alternate*)	
SHRI V. K. GHANEKAR	Structural Engineering Research Centre (CSIR), Ghaziabad

(*Continued on page* 3)

Indian Standard

METHODS OF PHYSICAL TESTS FOR HYDRAULIC CEMENT

PART 3 DETERMINATION OF SOUNDNESS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 3) (First Revision) was adopted by the Bureau of Indian Standards on 24 February 1988, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Standard methods of testing cement are essential adjunct to the cement specifications. This standard in different parts lays down the procedure for the tests to evaluate physical properties of different types of hydraulic cements. The procedure for conducting chemical tests of hydraulic cement is covered in IS : 4032-1985*.

0.3 Originally all the tests to evaluate the physical properties of hydraulic cements were covered in one standard but for facilitating the use of this standard and future revisions it has been decided to print the different tests as different parts of the standard and, accordingly this revised standard has been brought out in thirteen parts. This will also facilitate updating of individual tests. Further, since publication of the original standard in 1968, a number of standards covering the requirements of different equipment used for testing of cement, a brief description of which was also covered in the standard, had been published. In this revision, therefore, reference is given to different instrument specifications deleting the description of the instruments, as it has been recognized that reproducible and repeatable test results can be obtained only with standard testing equipment capable of giving desired level of accuracy. This part covers determination of soundness by Le-Chatelier method and autoclave test.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Method of chemical analysis of hydraulic cement (*first revision*).

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

1. SCOPE

1.1 This standard (Part 3) covers the procedures for determining the soundness of cement.

2. SAMPLING AND SELECTION OF TEST SPECIMEN

2.1 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 representative sample of the cement selected as above shall be thoroughly mixed before testing.

3. TEMPERATURE AND HUMIDITY

3.1 The temperature of the moulding room, dry materials and water shall be maintained at 27 ± 2°C. The relative humidity of the laboratory shall be 65 ± 5 percent.

3.2 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. GENERAL

4.1 Soundness of cement may be determined by two methods, namely Le-Chatelier method and autoclave method as described in **5** and **6**.

5. LE-CHATELIER METHOD

5.1 Apparatus

5.1.1 The apparatus for conducting the Le-Chatelier test shall conform to IS : 5514-1969*.

*Methods of sampling hydraulic cements *(first revision)*.

*Specification for apparatus used in Le-Chatelier test.

5.1.2 *Balance* — The balance shall conform to the following requirements:

On balance in use, the permissible variation at a load of 1 000 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 be not 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.

5.1.3 *Weights* — The permissible variations on weights in use in weighing the cement shall be as prescribed in Table 1.

TABLE 1 PERMISSIBLE VARIATIONS ON WEIGHTS

WEIGHT	PERMISSIBLE VARIATION ON WEIGHTS IN USE, PLUS OR MINUS
g	g
(1)	(2)
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

'5.1.4 *Water Bath* — Water bath with means of heating, capable of containing immersed Le-Chatelier moulds with specimens and of raising their temperature from 27 ± 2°C to boiling in 27 ± 3 minutes.'

5.2 Procedure

5.2.1 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 [*see* IS : 4031 (Part 4)-1988*]. 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°C and keep there for 24 hours.

5.2.2 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.

5.3 Calculation — Calculate the mean of two values to the nearest 0·5 mm to represent the expansion of cement.

5.4 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 **5.2**.

6. AUTOCLAVE METHOD

6.1 Apparatus

6.1.1 *Balance* — Same as **5.1.2**.

6.1.2 *Weights* — Same as **5.1.3**.

6.1.3 *Graduated Glass Cylinders* — Graduated glass cylinders of 150 ml capacity shall be used. The permissible variation on these cylinders shall be plus or minus one millilitre. The main graduation lines of the cylinders shall be in circles and shall be numbered. The least graduations shall extend at least one-seventh of the way around, and intermediate graduations shall extend at least one-fifth of the way around the cylinder. The graduation lines may be omitted for the lowest 5 ml.

6.1.4 *Moulds* — Moulds of 25 × 25 mm size and 282 mm internal length and other accessories conforming to IS : 10086-1982*.

6.1.5 *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

*Methods of physical tests for hydraulic cement: Part 4 Determination of consistency of standard cement paste (*first revision*).

*Specification for moulds for use in tests of cement and concrete.

to a gauge pressure of 2·1 MPa or to an absolute pressure of about 2·2 MPa, in 1 to 1½ 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°C. 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.

6.1.6 *Length Comparator* — Changes in length of the test specimen shall be measured by an apparatus conforming to IS : 9459-1980*.

6.2 Preparation of Test Specimens

6.2.1 *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.

6.2.2 *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.

6.2.3 *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.

6.2.4 *Storage of Test Specimen* — 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 h. If removed from the moulds before 24 h, they shall be kept in the moist closet or moist room until tested.

*Specification for apparatus for use in measurement of length change of hardened cement paste, mortar and concrete.

6.3 Procedure

6.3.1 At 24 ± ½ h 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.

6.3.2 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 (*see* **6.4**). 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 1½ h from the time the heat is turned on. The 2·1 ± 0·1 MPa pressure shall be maintained for 3 h. 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°C. 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°C in 15 min. The water surrounding the specimens shall then be maintained at 27 ± 2°C in 15 min when the specimens shall be surface-dried and their lengths measured again.

6.4 Safety Precautions

6.4.1 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.

6.4.2 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.

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

6.4.4 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.

NOTE — Unexpected combinations of conditions may really occur. For example, in one case the automatic control had failed, the safety valve had stuck, and the gauge hand, which at first glance appeared to be at about zero, had really passed the maximum graduation and had come to stop on the wrong side of the pin. This condition of the gauge was finally detected and the pressure, then of an unknown magnitude, was released before failure could occur in the apparatus.

6.4.5 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.

6.4.6 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.

6.4.7 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.

6.5 Calculations — 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.

6.6 Retests — 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 sample 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 **6.2** and **6.3**.

Indian Standard

METHODS OF PHYSICAL TESTS FOR HYDRAULIC CEMENT

PART 4 DETERMINATION OF CONSISTENCY OF STANDARD CEMENT PASTE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 4) (First Revision) was adopted by the Bureau of Indian Standards on 24 February 1988, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Standard methods of testing cement are essential adjunct to the cement specifications. This standard in different parts lays down the procedure for the tests to evaluate physical properties of different types of hydraulic cements. The procedure for conducting chemical tests of hydraulic cement is covered in IS : 4032-1985*.

0.3 Originally all the tests to evaluate the physical properties of hydraulic cement were covered in one standard but for facilitating the use of this standard and future revisions, it has been decided to print different tests as different parts of the standard and, accordingly this revised standard has been brought out in thirteen parts. This will also facilitate updating of individual tests. Further, since the publication of the original standard in 1968, a number of standards covering the requirements of different equipment used for testing of cement, a brief description of which was also covered in the standard, had been published. In this revision, therefore, reference is given to different instrument specifications deleting the description of the instruments as it has been recognized that reproducible and repeatable test results can be obtained only with standard testing equipment capable of giving desired level of accuracy. This part covers the procedure for determining the quantity of water required to produce a cement paste of standard consistency.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Method of chemical analysis of hydraulic cement (*first revision*).

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

1. SCOPE

1.1 This standard (Part 4) covers the procedure for determining the quantity of water required to produce a cement paste of standard consistency.

2. SAMPLING AND SELECTION OF TEST SPECIMEN

2.1 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 representative sample of the cement selected as above shall be thoroughly mixed before testing.

3. TEMPERATURE AND HUMIDITY

3.1 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.

4. APPARATUS

4.1 Vicat Apparatus —Vicat apparatus conforming to IS : 5513-1976*.

4.2 Balance — The balance shall conform to the following requirements.

4.2.1 On balance in use, the permissible variation at a load of 1 000 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.

*Methods of sampling hydraulic cements (*first revision*).

*Specification for Vicat apparatus (*first revision*).

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

4.3 Standard Weights — The permissible variation on weights in use in weighing the cement shall be as prescribed in Table 1.

TABLE 1 PERMISSIBLE VARIATION ON WEIGHTS

WEIGHT	PERMISSIBLE VARIATION ON WEIGHTS IN USE, PLUS OR MINUS
g	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

4.4 Gauging Trowel — Gauging trowel conforming to IS 10086 : 1982†.

5. PROCEDURE

5.1 The standard consistency of a cement paste is defined as that consistency which will permit the Vicat plunger *G* shown in IS : 5513-1976* to penetrate to a point 5 to 7 mm from the bottom of the Vicat mould when the cement paste is tested as described in **5.2** to **5.4**.

5.2 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 minutes, 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 *E* 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.

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

5.3 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.

5.4 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 **5.1** is found.

6. CALCULATION

6.1 Express the amount of water as a percentage by mass of the dry cement to the first place of decimal.

*Specification for Vicat apparatus (*first revision*).

†Specification for moulds for use in tests of cement and concrete.

Indian Standard

METHODS OF PHYSICAL TESTS FOR HYDRAULIC CEMENT

PART 5 DETERMINATION OF INITIAL AND FINAL SETTING TIMES

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 5) (First Revision) was adopted by the Bureau of Indian Standards on 10 March 1988, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Standard methods of testing cement are essential adjunct to the cement specifications. This standard in different parts lays down the procedure for the tests to evaluate the physical properties of different types of hydraulic cements. The procedure for conducting chemical tests of hydraulic cement is covered in IS : 4032-1985*.

0.3 Originally all the tests to evaluate the physical properties of hydraulic cements were covered in one standard; but for facilitating the use of this standard and future revisions, it has been decided to print the different tests as different parts of the standard and, accordingly this revised standard has been brought out in thirteen parts. This will also facilitate updating of individual tests. Further, since publication of the original standard in 1968, a number of standards covering the requirements of different equipment used for testing of cement, a brief description of which was also covered in the standard, had been published. In this revision, therefore, reference is given to different instrument specifications deleting the description of the instruments, as it has been recognized that reproducible and repeatable test results can be obtained only with standard testing equipment capable of giving desired level of accuracy. This part covers determination of initial and final setting times of cement.

0.4 This edition 2.1 incorporates Amendment No. 1 (March 1993). Side bar indicates modification of the text as the result of incorporation of the amendment.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Method of chemical analysis of hydraulic cement (*first revision*).

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

1. SCOPE

1.1 This standard (Part 5) covers the procedure for determining the initial and final setting times of cement.

2. SAMPLING AND SELECTION OF TEST SPECIMEN

2.1 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 representative sample of the cement selected as above shall be thoroughly mixed before testing.

3. TEMPERATURE AND HUMIDITY

3.1 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.

3.2 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. APPARATUS

4.1 Vicat Apparatus — Vicat apparatus conforming to IS : 5513-1976*.

4.2 Balance — The balance shall conform to the following requirements:

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

*Methods of sampling hydraulic cements (*first revision*).

*Specification for Vicat apparatus (*first revision*).

4.3 Standard Weights — The permissible variations on weights in use in weighing the cement shall be as prescribed in Table 1.

TABLE 1 PERMISSIBLE VARIATION ON WEIGHTS
(*Clause* **4.3**)

WEIGHT	PERMISSIBLE VARIATION ON WEIGHTS IN USE,
g	g
(1)	(2)
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

4.4 Gauging Trowel — Gauging trowel conforming to IS : 10086-1982*.

5. PROCEDURE

5.1 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 *E* 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.

5.1.1 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.

5.2 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 (*C*); 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.

5.3 Determination of Final Setting Time — Replace the needle (*C*) 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.

6. REPORTING OF RESULTS

6.1 The results of initial and final setting time shall be reported to the nearest five minutes.

*Specification for moulds for use in tests of cement and concrete.

†Methods of physical tests for hydraulic cement: Part 4 Determination of consistency of standard cement paste (*first revision*).

Indian Standard

METHODS OF PHYSICAL TESTS FOR HYDRAULIC CEMENT

PART 6 DETERMINATION OF COMPRESSIVE STRENGTH OF HYDRAULIC CEMENT OTHER THAN MASONRY CEMENT

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 6) (First Revision) was adopted by the Bureau of Indian Standards on 10 March 1988, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Standard methods of testing cement are essential adjunct to the cement specifications. This standard in different parts lays down the procedure for the tests to evaluate the physical properties of different types of hydraulic cements. The procedure for conducting chemical tests of hydraulic cement is covered in IS : 4032-1985*.

0.3 Originally all the tests to evaluate the physical properties of hydraulic cements were covered in one standard but for facilitating the use of this standard and future revisions, it has been decided to print the different tests as different parts of the standard and, accordingly this revised standard has been brought out in thirteen parts. This will also facilitate updating of individual tests. Further, since publication of the original standard in 1968, a number of standards covering the requirements of different equipment used for testing of cement, a brief description of which was also covered in the standard, had been published. In this revision, therefore, reference is given to different instrument specifications deleting the description of the instruments, as it has been recognized that reproducible and repeatable test results can be obtained only with standard testing equipment capable of giving desired level of accuracy. This part covers the method for determining the compressive strength of hydraulic cement other than masonry cement which is covered in Part 7 of this standard. The criteria for accepting compressive strength values has been incorporated and the use of single graded sand has been deleted in this revision.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2 - 1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Method of chemical analysis of hydraulic cement (*first revision*).

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

1. SCOPE

1.1 This standard (Part 6) covers the procedure for determining the strength of cement as represented by compressive strength tests on mortar cubes compacted by means of standard vibration machine.

2. SAMPLING AND SELECTION OF TEST SPECIMEN

2.1 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 representative sample of the cement selected as above shall be thoroughly mixed before testing.

*Methods of sampling hydraulic cement (*first revision*).

3. TEMPERATURE AND HUMIDITY

3.1 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~~ not less than 65%

3.2 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. GENERAL

4.1 Standard Sand — The standard sand to be used in the test shall conform to IS : 650-1966*.

5. APPARATUS

5.1 Vibration Machine — Vibration machine

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

conforming to IS : 10080-1982*.

5.2 Poking Rod — Poking rod conforming to IS : 10080-1982*.

5.3 Cube Mould — The mould shall be of 70·6 mm size conforming to IS : 10080-1982*.

5.4 Gauging Trowel — Gauging trowel shall have a steel blade 100 to 150 mm in length with straight edges weighing 210 ± 10 g. *

5.5 Balance — The balance shall conform to the following requirements :

On balance in use, the permissible variation at a load of 1 000 g shall be ± 1·0 g. The permissible variation on new balance shall be one-half of this value. The sensibility reciprocal shall be not 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 at a non-automatic indicating scale a definite amount of any load.

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

5.6 Standard Weights — The permissible variation on weights in use in weighing the cement shall be as prescribed in Table 1.

TABLE 1 PERMISSIBLE VARIATIONS ON WEIGHTS

WEIGHT	PERMISSIBLE VARIATION ON WEIGHTS IN USE
(1)	(2)
(g)	(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

5.7 Graduated Glass Cylinders — Graduated glass cylinders of ~~150 to 200~~ 100 to 250 ml capacity. The permissible variation on these cylinders shall be ± 1 ml. The main graduation lines of the cylinders shall be in circles and shall be numbered. The least graduations shall extend at least one-seventh of the way around, and intermediate graduations shall extend at least one-fifth of the way around the cylinder. The graduation lines may be omitted for the lowest 5 ml.

6. PREPARATION OF TEST SPECIMENS

6.1 Mix Proportions and Mixing

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

6.1.2 The material for each cube shall be mixed separately and the quantity of cement, standard sand and water shall be as follows :

Cement	200 g
Standard Sand	600 g
Water	$\left(\frac{P}{4} + 3{\cdot}0\right)$ percent of combined mass of cement and sand, where ~~whether~~ P is the percentage of water required to produce a paste of standard consistency determined as described in IS : 4031 (Part 4) - 1988*

6.1.3 Place on a nonporous plate, a mixture of cement and standard sand. Mix it dry with a trowel for one minute and then with water until the mixture is of uniform colour. The quantity of water to be used shall be as specified in **6.1.2**. The time of mixing shall in any event be not less than 3 min and should the time taken to obtain a uniform colour exceed 4 min, the mixture shall be rejected and the operation repeated with a fresh quantity of cement, sand and water.

6.2 Moulding Specimens

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

6.2.2 Place the assembled mould on the table of the vibration machine and hold it firmly in position by means of a suitable clamp. Attach a hopper of suitable size and shape securely at the top of the mould to facilitate filling and this hopper shall not be removed until the completion of the vibration period.

6.2.3 Immediately after mixing the mortar in accordance with **6.1**, place the mortar in the cube mould and prod with the rod specified in **5.2** The mortar shall be prodded 20 times in about 8 s to ensure elimination of entrained air and honeycombing. Place the remaining quantity of mortar in the hopper of the cube mould and prod again as specified for the first layer and then compact the mortar by vibration.

6.2.4 The period of vibration shall be two minutes at the specified speed of 12 000 ± 400 vibration per minute.

*Specification for vibration machine for casting standard cement mortar cubes.

Specification for moulds for use in tests of cement & concrete

*Methods of physical tests for hydraulic cement: Part 4 Determination of consistency of standard cement paste (*first revision*).

*Amendment-1: Gauging Trowel conforming to IS 10086-1982

6.2.5 At the end of vibration, remove the mould together with the base plate from the machine and finish the top surface of the cube in the mould by smoothing the surface with the blade of a trowel.

6.3 Curing Specimens — Keep the filled moulds in moist closet or moist room for 24* hours after completion of vibration. At the end of that period, remove them from the moulds and immediately submerge in clean fresh water and keep there until taken out just prior to breaking. The water in which the cubes are submerged shall be renewed every 7 days and shall be maintained at a temperature of 27 ± 2°C. After they have been taken out and until they are broken, the cubes shall not be allowed to become dry.

7. TESTING

7.1 Test three cubes for compressive strength for each period of curing mentioned under the relevant specifications for different hydraulic cements, the periods being reckoned from the completion of vibration.

7.1.1 The cubes shall be tested on their sides without any packing between the cube and the steel plattens of the testing machine. One of the plattens shall be carried on a base and shall be self-adjusting, and the load shall be steadily and uniformly applied, starting from zero at a rate of 35 N/mm²/min.

8. CALCULATION

8.1 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 the 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.

* Amendment 2: 24 (+/-) 1

Indian Standard

METHODS OF PHYSICAL TESTS FOR HYDRAULIC CEMENT

PART 11 DETERMINATION OF DENSITY

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 11) (First Revision) was adopted by the Bureau of Indian Standards on 22 April 1988, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Standard methods of testing cement are essential adjunct to the cement specifications. This standard in different parts lays down the procedure for the tests to evaluate the physical properties of different types of hydraulic cements. The procedure for conducting chemical tests of hydraulic cement is covered in IS : 4032-1985*.

0.3 Originally all the tests to evaluate the physical properties of hydraulic cements were covered in one standard ; but for facilitating the use of this standard and future revisions, it has been decided to print the different tests as different parts of the standard and accordingly, this revised standard has been brought out in thirteen parts. This will also facilitate updating of individual tests. Further, since publication of the original standard in 1968, a number of standards covering the requirements of different equipment used for testing of cement, a brief description of which was also covered in the standard, had been published. In this revision, therefore, reference is given to different instrument specifications deleting the description of the instruments, as it has been recognized that reproducible and repeatable test results can be obtained only with standard testing equipment capable of giving desired level of accuracy. This part (Part 11) covers determination of density of hydraulic cement.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Method of chemical analysis of hydraulic cement (*first revision*).

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

1. SCOPE

1.1 This standard (Part 11) covers the procedure for determining the density of hydraulic cement.

2. SAMPLING AND SELECTION OF TEST SPECIMENS

2.1 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 representative sample of the cement selected as above shall be thoroughly mixed before testing.

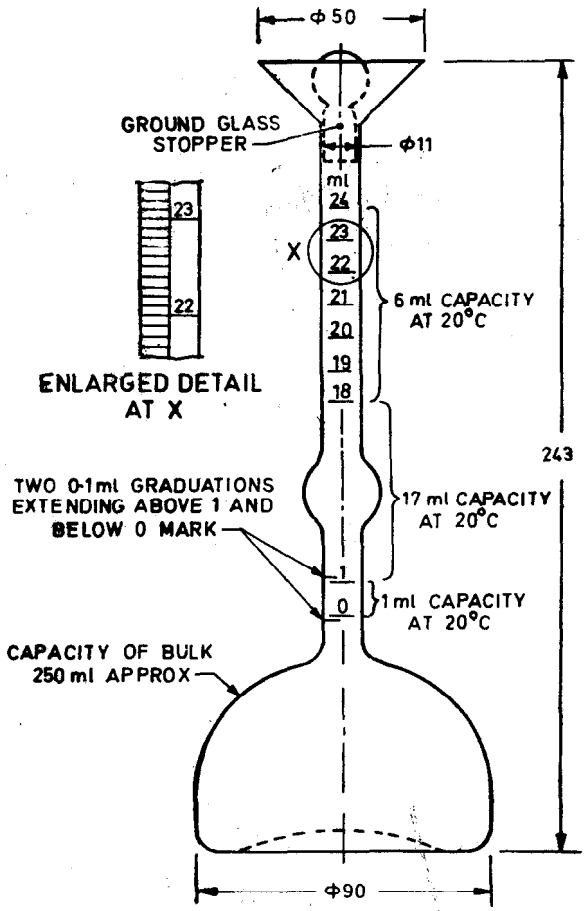
*Methods of sampling hydraulic cements.

3. TEMPERATURE

3.1 The temperature of the laboratory shall be maintained at 27 ± 2°C.

4. APPARATUS

4.1 Le Chatelier Flask — Standard LeChatelier flask conforming to the dimensions shown in Fig. 1.



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

All dimensions in millimetres.

FIG. 1 LE CHATELIER FLASK FOR DENSITY TEST

4.2 Analytical Balance — Analytical balance capable of reproducing results within 0·000 2 g with an accuracy of ± 0·000 2 g.

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

4.3 Standard Weights

4.4 Constant Temperature Water Bath — The constant temperature water bath shall be capable of maintaining temperature within ± 0·2°C.

5. MATERIAL

5.1 Kerosine free of water, or naphtha having a specific gravity not less than 0·731 3 shall be used in the density determination.

6. PROCEDURE

6.1 Density of cement shall be determined on the material as received, unless otherwise specified. If the density determination on a loss-free sample is required, the sample shall first be ignited according to the test for loss on ignition.

6.2 The flask shall be filled (*see* Note 1) with either of the liquids specified in **5.1** to a point on the stem between the zero and the 1-ml mark. The inside of the flask above the level of the liquid shall be dried, if necessary, after pouring. The first reading shall be recorded after the flask has been immersed in the water bath (*see* Note 2) in accordance with **6.3**. A weighed quantity of cement (about 64 g for Portland cement) shall then be introduced in small amounts at the same temperature as the liquid (*see* Note 1). Care shall be taken to avoid splashing and to see that the cement does not adhere to the inside of the flask above the liquid. A vibrating apparatus may be used to accelerate the introduction of the cement into the flask and to prevent the cement from sticking to the neck. After all the cement has been introduced, the stopper shall be placed in the flask and the flask rolled in an inclined position (*see* Note 1), or gently whirled in a horizontal circle, so as to free the cement from air until no further air bubbles rise to the surface of the liquid. If a proper amount of cement has been added, the level of the liquid will be in its final position at some point of the upper series of graduations. The final reading shall be taken after the flask has been immersed in the water bath in accordance with **6.3**.

NOTE 1 — It is advisable to use a rubber pad on the table top when filling or rolling the flask.

NOTE 2 — Before the cement has been added to the flask, a loose-fitting lead-ring weight around the stem of the flask will be helpful in holding the flask in an upright position in the water bath or the flask may be held in the water bath by a burette clamp.

NOTE 3 — For cleaning of Le Chatelier flasks, acid cleaning will not be effective in removing any deposition of silicic acid gel. They may be satisfactorily cleaned by using warm sodium carbonate solution.

6.3 The flask shall be immersed in a constant-temperature water bath, maintained at about room temperature, for a sufficient interval before making either of the readings so as to avoid variations greater than 0·2°C in the temperature of the liquid in the flask. All readings shall be checked until they are constant to ensure that the contents of the flask have reached the temperature of the water bath.

7. CALCULATION

7.1 The difference between the first and the final readings represents the volume of liquid displaced by the mass of cement used in the test. The density shall be calculated as follows to the second place of decimal :

$$\text{Density} = \frac{\text{Mass of cement in g}}{\text{Displaced volume in cm}^3}$$

7.2 Two tests shall be carried out and the average shall be reported.

8. RETEST

8.1 If the difference between the two values differs by more than 0·03, the test shall be repeated.

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