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Indian Standard

**METHODS OF
PHYSICAL TESTS FOR HYDRAULIC CEMENT**

PART 3 DETERMINATION OF SOUNDNESS

(First Revision)

(Incorporating Amendment No. 1)

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Price Group 2

Indian Standard

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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 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 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

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

$\pm 2^{\circ}\text{C}$. The relative humidity of the laboratory shall be 65 ± 5 percent.

3.2 The moist closet or moist room shall be maintained at $27 \pm 2^{\circ}\text{C}$ 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*.

*Specification for apparatus used in Le-Chatelier test.

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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 $27 \pm 2^\circ\text{C}$ 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 $27 \pm 2^\circ\text{C}$ 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 $27 \pm 2^\circ\text{C}$ 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 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 $215.7 \pm 1.7^\circ\text{C}$. 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 $24 \pm \frac{1}{2}$ 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 1¼ 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 $27 \pm 2^\circ\text{C}$ in 15 min. The water surrounding the specimens shall then be maintained at $27 \pm 2^\circ\text{C}$ 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.

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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 $27 \pm 2^\circ\text{C}$ and relative humidity of 50 to 80 percent. Retest this cement as described in **6.2** and **6.3**.

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