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## **Cements — Test methods — Determination of setting time and soundness**

**iTeh STANDARD PREVIEW**  
*Ciments — Méthodes d'essai — Détermination du temps de prise et de la stabilité*  
**(standards.iteh.ai)**

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9597 was prepared by Technical Committee ISO/TC 74, *Cement and lime*.

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# Cements — Test methods — Determination of setting time and soundness

## iTeh STANDARD PREVIEW (standards.iteh.ai)

### 1 Scope and field of application

This International Standard specifies the methods for determining setting time and soundness of cements.

It describes the reference procedure; it allows the use of alternative procedures as indicated in the notes provided that they do not affect the results significantly. In the event of a dispute, only the reference procedure described in this International Standard may be used, excluding any alternatives.

### 2 Reference

ISO 679, *Methods of testing cements — Determination of strength*.

### 3 Principle

The setting time is determined by observing the penetration of a needle into cement paste of standard consistence until it reaches a specified value.

The soundness, by the Le Chatelier method, is determined by observing the volume expansion of cement paste of standard consistence as indicated by the relative movement of two needles.

Cement paste of standard consistence has a specified resistance to penetration by a standard plunger. The water required for such a paste is determined by trial penetrations of pastes with different water contents.

### 4 Laboratory and equipment

#### 4.1 Laboratory

The laboratory in which specimens are prepared and tested shall be maintained at a temperature of  $20\text{ °C} \pm 2\text{ °C}$  and a relative humidity of not less than 50 %.

The laboratory temperature may be maintained in warm countries at  $25\text{ °C} \pm 2\text{ °C}$  or  $27\text{ °C} \pm 2\text{ °C}$ , provided the temperature is specified in the test report.

#### 4.2 Apparatus

**4.2.1 Balance**, capable of weighing to the nearest 1 g.

**4.2.2 Graduated cylinder or burette**, capable of measuring to the nearest 1 % of the volume measured.

**4.2.3 Mixer**, complying with the specifications of ISO 679.

## 4.3 Materials

**4.3.1** Distilled or deionized water shall be used for making, storing and boiling specimens.

NOTE — Other water may be used, provided that it can be shown to give the same test results.

**4.3.2** Cement, water and apparatus used to make and test specimens shall be at a temperature of  $20\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ . The temperature may be maintained at  $25\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  or  $27\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  in warm countries, provided the temperature is specified in the test report.

## 5 Standard consistence test

### 5.1 Apparatus

Use the Vicat apparatus as shown in figure 1 a) and 1 b) with the plunger shown in figure 1 c). The plunger shall be of non-corrodible metal in the form of a right cylinder of  $50\text{ mm} \pm 1\text{ mm}$  effective length and of  $10\text{ mm} \pm 0,05\text{ mm}$  diameter. The total mass of moving parts shall be  $300\text{ g} \pm 1\text{ g}$ . Their movement shall be truly vertical and without appreciable friction, and their axis shall coincide with that of the plunger.

The Vicat mould [see figure 1 a)] to contain the paste under test shall be of hard rubber. It shall be of truncated conical form  $40\text{ mm} \pm 0,2\text{ mm}$  deep and shall have internal diameters at top and bottom of  $70\text{ mm} \pm 5\text{ mm}$  and  $80\text{ mm} \pm 5\text{ mm}$  respectively. It shall be adequately rigid and shall be provided with a plane glass base-plate larger than the mould and at least  $2,5\text{ mm}$  thick.

NOTE — Moulds of metal or plastics, or of cylindrical form, may be used provided that they are of the specified depth and that they can be shown to give the same test results as the specified hard rubber mould of truncated conical form.

### 5.2 Procedure

#### 5.2.1 Mixing cement paste

Weigh, to the nearest  $1\text{ g}$ ,  $500\text{ g}$  of cement. Weigh a quantity of water, e.g.  $125\text{ g}$ , in the mixer bowl (4.2.3) or measure the water from the graduated cylinder or burette (4.2.2) into the mixer bowl.

Add the cement carefully to the water in order to avoid loss of water or cement. The time of addition shall be not less than  $5\text{ s}$  nor more than  $10\text{ s}$ . Note the time of completion of the addition as zero time from which later measurements of time shall be made. Start the mixer immediately and run at low speed for  $90\text{ s}$ .

Stop the machine after  $90\text{ s}$  for  $15\text{ s}$  during which remove any paste adhering to the bowl outside the mixing zone with a suitable scraper and return it to the mix. Restart the machine and run at low speed for a further  $90\text{ s}$ . The total mixer running time shall be  $3\text{ min}$ .

NOTE — Any other mixing method, whether by machine or hand, may be used provided that it can be shown to give the same test results as the specified method.

#### 5.2.2 Filling mould

Transfer the paste immediately to the mould, which has previously been placed on a lightly greased plane glass base-plate, and fill it to excess without undue compaction or vibration. Remove the excess by a gentle sawing motion with a straight-edged implement in such a way as to leave the paste filling the mould and having a smooth upper surface.

#### 5.2.3 Penetration test

Calibrate the Vicat apparatus with the plunger [see figure 1 c)], attached in advance of the test, by lowering the plunger to rest on the base-plate to be used and adjusting the pointer to read zero on the scale. Raise the plunger to the stand-by position.

Immediately after levelling the paste, transfer the mould and base-plate to the Vicat apparatus and position it centrally under the plunger. Lower the plunger gently until it is in contact with the paste. Pause in that position for between  $1\text{ s}$  and  $2\text{ s}$  in order to avoid initial velocity or forced acceleration of the moving parts. Then release the moving parts quickly and allow the plunger to penetrate vertically into the centre of the paste. The release of the plunger shall occur  $4\text{ min}$  after zero time. Read the scale when penetration has ceased, or  $30\text{ s}$  after the release of the plunger, whichever is the earlier.

Record the scale reading, which indicates the distance between the bottom face of the plunger and the base-plate, together with the water content of the paste expressed as a percentage by mass of the cement. Clean the plunger immediately after each penetration.

Repeat the test with pastes containing different water contents until one is found to produce a distance between plunger and base-plate of  $6\text{ mm} \pm 1\text{ mm}$ . Record the water content of that paste to the nearest  $0,5\%$  as the water for standard consistence.

## 6 Setting time test

### 6.1 Apparatus

**6.1.1** Use a room or a humidity cabinet of adequate size and maintained at  $20\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$  and not less than  $90\%$  relative humidity.

NOTE — Provided that it can be shown to give the same test results, a water-bath capable of maintaining filled moulds above water at  $20\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$  may be used.

The temperature may be maintained at  $25\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$  or  $27\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$  in warm countries, provided it is specified in the test report.

**6.1.2** Use a Vicat apparatus for the determination of initial and final set. Remove the plunger and replace it by the needle [see figure 1 d)] which shall be of steel and in the form of a right cylinder of effective length  $50\text{ mm} \pm 1\text{ mm}$  and diameter

1,13 mm  $\pm$  0,05 mm. The total mass of moving parts shall be 300 g  $\pm$  1 g. Their movements shall be truly vertical and without appreciable friction, and their axis shall coincide with that of the needle.

## 6.2 Determination of initial setting time

Calibrate the Vicat apparatus with the needle attached in advance of the test, by lowering the needle to rest on the base-plate to be used and adjusting the pointer to read zero on the scale. Raise the needle to the stand-by position.

Fill a Vicat mould with paste of standard consistence and level it, in accordance with 5.2.1 and 5.2.2.

Place the filled mould and base-plate in the room or humidity cabinet (see 6.1.1) and, after a suitable time, transfer to the Vicat apparatus and position under the needle. Lower the needle gently until it is in contact with the paste. Pause in that position for between 1 s and 2 s in order to avoid initial velocity or forced acceleration of the moving parts. Then release the moving part quickly and allow the needle to penetrate vertically into the paste. Read the scale when penetration has ceased, or 30 s after the release of the needle, whichever is the earlier.

Record the scale reading, which indicates the distance between the end of the needle and the base-plate, together with the time from zero. Repeat the penetration test on the same specimen at conveniently spaced positions, not less than 10 mm from the rim of the mould or from each other, at conveniently spaced intervals of time, e.g. at 10 min intervals. Between penetration tests keep the specimen in a room or humidity cabinet. Clean the Vicat needle immediately after each penetration. Record the time measured from zero at which the distance between the needle and the base-plate is 4 mm  $\pm$  1 mm as the initial setting time of the cement to the nearest 5 min. The required accuracy may be ensured by reducing the time interval between penetration tests near the end-point and observing that successive results do not fluctuate excessively.

## 6.3 Determination of final setting time

Invert the filled mould used in 6.2 on its base-plate so that the tests for final set are made on the face of the specimen originally in contact with the base-plate. Fit the needle with a ring attachment [see figure 1 e)] to facilitate accurate observation of small penetrations. Use the procedure described in 6.2. The intervals of time between penetration tests may be increased to, for example, 30 min.

Record, to the nearest 15 min, the time measured from zero at which the needle first penetrates only 0,5 mm into the specimen as the final setting time of the cement. This time is that at which the ring attachment first fails to mark the specimen and may be accurately established by reducing the time interval between tests near the end-point and observing that successive test results do not fluctuate excessively.

NOTE — Automatic setting time machines are commercially available and may be used provided that they can be shown to give the same test results as the specified apparatus and procedure. When using automatic machines, there is no need to turn the mould over.

## 7 Soundness test

### 7.1 Apparatus

7.1.1 Use Le Chatelier apparatus (see figure 2), where the mould is of spring brass with indicator needles. The resilience of the mould shall be such that the action of a mass of 300 g applied as shown in figure 2 c) shall increase the distance between the indicator ends of the needles by 17,5 mm  $\pm$  2,5 mm without permanent deformation.

A pair of plane glass base and cover plates shall be provided for each mould. Each plate shall be larger than the mould. The cover plate shall weigh at least 75 g; an additional small mass may be placed on a thin plate to satisfy this requirement.

7.1.2 Use a water-bath with means of heating, capable of containing immersed Le Chatelier specimens and of raising the temperature of the water from 20 °C  $\pm$  2 °C (25 °C  $\pm$  2 °C or 27 °C  $\pm$  2 °C in warm countries) to boiling in 30 min  $\pm$  5 min.

7.1.3 Use a humidity cabinet, of adequate size and maintained at 20 °C  $\pm$  1 °C (25 °C  $\pm$  1 °C or 27 °C  $\pm$  1 °C in warm countries) and not less than 98 % relative humidity.

### 7.2 Procedure

Carry out the test simultaneously on two specimens from the same batch of cement paste.

Prepare a cement paste of standard consistence. Place a lightly oiled Le Chatelier mould (7.1.1) on the lightly oiled base-plate and fill it immediately without undue compaction or vibration using only the hands and a straight-edged implement, if desired, to level the top surface. During filling, prevent the split in the mould from accidentally opening e.g. by gentle pressure with the fingers or by tying or by use of a suitable rubber band.

Cover the mould with the lightly oiled cover plate, add the additional mass, if necessary, and then immediately place the complete apparatus in the humidity cabinet. Maintain it for 24 h  $\pm$  0,5 h at 20 °C  $\pm$  1 °C (25 °C  $\pm$  1 °C or 27 °C  $\pm$  1 °C in warm countries) and not less than 98 % relative humidity.

NOTE — Provided that it can be shown to give the same test results, the mould may be placed, between its glass plates, with the additional mass, if necessary, in a water-bath and maintained under water for 24 h  $\pm$  0,5 h at 20 °C  $\pm$  1 °C (25 °C  $\pm$  1 °C or 27 °C  $\pm$  1 °C in warm countries).

At the end of the 24 h  $\pm$  0,5 h period, measure the distance *A* between the indicator points to the nearest 0,5 mm. Bring the water gradually to boiling, with the moulds kept submerged, during 30 min  $\pm$  5 min and maintain the water-bath at boiling temperature for 3 h  $\pm$  5 min.

NOTE — Where it can be shown that the expansion after a shorter boiling period is the same as that after 3 h, such a shorter period may be used.

At the end of the boiling period, the distance *B* between the indicator points may be measured to the nearest 0,5 mm.

Allow the mould to cool to  $20\text{ °C} \pm 2\text{ °C}$  ( $25\text{ °C} \pm 2\text{ °C}$  or  $27\text{ °C} \pm 2\text{ °C}$  in warm countries). Measure the distance  $C$  between the indicator points to the nearest 0,5 mm.

For each specimen, record the measurements  $A$  and  $C$  and calculate the difference  $C - A$ . Calculate the mean of the two values of  $C - A$  to the nearest 0,5 mm.

### 7.3 Interpretation

The main purpose of the soundness test is to assess the possible risk of late expansion due to hydration of uncombined calcium oxide and/or magnesium oxide. For this purpose, report the mean difference  $C - A$ .

NOTE — Where it can be shown that the conditions of the test lead to no significant difference between measurements  $B$  and  $C$ , the difference  $B - A$  may be reported since this reduces the time needed for the test.

### 7.4 Retest

Should the fresh cement fail to comply with the specified soundness requirement it may be retested after storage. For this purpose, spread out the cement in a layer of 7 cm thickness and store it for 7 days in an atmosphere maintained at  $20\text{ °C} \pm 2\text{ °C}$  ( $25\text{ °C} \pm 2\text{ °C}$  or  $27\text{ °C} \pm 2\text{ °C}$  in warm countries) and not less than 50 % relative humidity. Then retest the cement according to the test procedure given in 7.2.

### 7.5 Cold soundness

It may also be desirable to determine the cold soundness.

In this case, prepare the specimens and put into the water at  $20\text{ °C} \pm 2\text{ °C}$  ( $25\text{ °C} \pm 2\text{ °C}$  or  $27\text{ °C} \pm 2\text{ °C}$  in warm countries) according to 7.2, and immediately measure the distance  $A$  between the indicator points to the nearest 0,5 mm. After 7 days  $\pm 12$  h, measure their distance  $D$  with the same precision.

Shorter time spans can be chosen to determine, if need be, the moment when the phenomenon occurs.

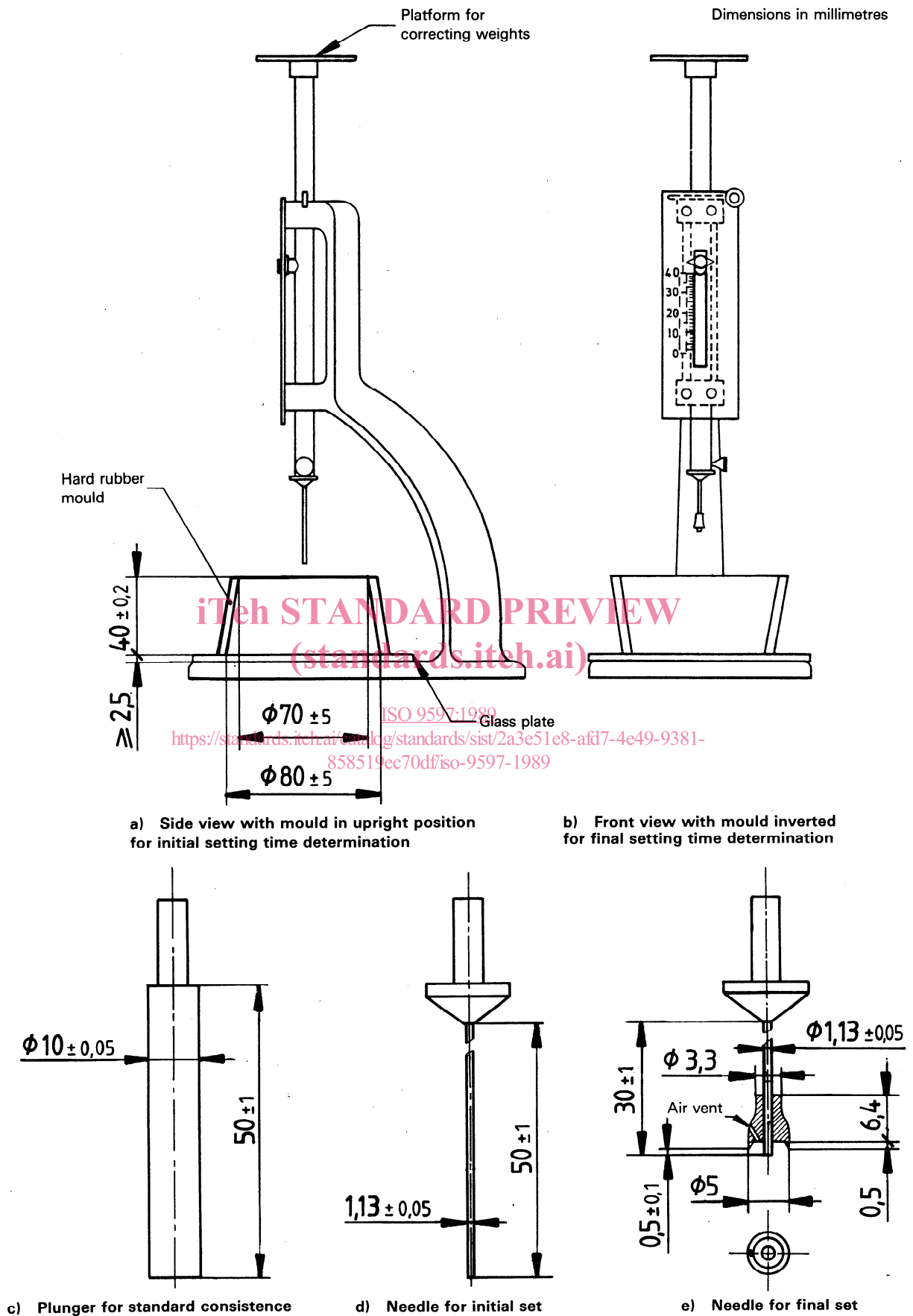
Cold soundness is given by the difference  $D - A$ .

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NOTE — The specified dimensions shall be met. If the plunger, needle and needle with attachment are all adjusted to have the same mass, e.g.  $9 \text{ g} \pm 0,5 \text{ g}$ , one correcting weight is sufficient for each apparatus.

Figure 1 — Vicat apparatus for determining standard consistence and setting time of cement