
**Cement — Test methods —
Determination of setting time and
soundness**

*Ciments — Méthodes d'essais — Détermination du temps de prise et
de la stabilité*

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This second edition cancels and replaces the first edition (ISO 9597:1989), the following clauses and subclauses of which have been technically revised, based on comments received by the secretariat.

- 4.1: Requirement for the laboratory relative humidity is reduced to a minimum of 50 % from a minimum of 65 %.
- 4.2.5: Drinking water is permitted for storing and boiling test specimens; drinking water is no longer permitted in the production of paste since such water varies in quality between places and even with time in the same laboratory.
- 5.1: The Vicat mould is permitted to have a cylindrical, in addition to truncated conical, shape.
- 5.2.1: The time permitted for scraping paste from the sides and bottom of the mixing bowl is increased from 15 s to 30 s.
- 5.2.3: The end-point for the determination of standard consistence is increased to (6 ± 2) mm from (6 ± 1) mm.
- Clause 6: The determination of the setting time is carried out whilst the specimen is immersed in water.
- 6.1.1: In the determination of the setting time, the use of automatic apparatus conforming to the requirements of the reference method is permitted.
- 6.1.3: In the determination of the setting time, the tolerance on storage temperature for specimens is reduced to $(20,0 \pm 1,0)$ °C from (20 ± 1) °C.
- 6.2.2: The end-point for the determination of the initial setting time is increased to (6 ± 3) mm from (4 ± 1) mm.
- 6.3.1: It is required to confirm the end-point for the determination of the final setting time by testing at two additional positions in the test specimen.
- 7.1.1: The material for the construction of the Le Chatelier apparatus to determine the soundness is extended to any non-corrodible spring metal.

- 7.1.3: The minimum relative humidity in which specimens for determination of soundness are stored is reduced to 90 % from 98 %.
- 7.2: The determination of soundness is carried out once.
- 7.4: Where a retest of the determination of soundness is required, the relative humidity at which the cement sample is stored is reduced to 50 % from 65 %.
- Annex A: An alternative method for testing the setting times of slow-setting cements is included.

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Cement — Test methods — Determination of setting time and soundness

1 Scope

This International Standard specifies the methods for determining standard consistence, setting times and soundness of cements.

The method applies to common cements and to other cements and materials, the standards for which call up this method. It might not apply to other cement types that have, for example, a very short initial setting time. The method is used for assessing whether the setting time and soundness of a cement is in conformity with its specification.

This International Standard describes the reference methods and allows the use of alternative procedures and equipment, as indicated, provided that they have been calibrated against the reference methods. In the event of a dispute, only the reference equipment and procedures described in this International Standard can be used, excluding any alternatives.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 679, *Cement — Test methods — Determination of strength*

3 Principles

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.

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 is determined by observing the volume expansion of cement paste of standard consistence as indicated by the relative movement of two needles.

4 Laboratory, equipment and materials

4.1 Laboratory

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

A laboratory temperature of $(25 \pm 2) ^\circ\text{C}$ or $(27 \pm 2) ^\circ\text{C}$ may be maintained in warm countries, provided the temperature is stated in the test report.

The temperature and relative humidity of the air in the laboratory and the temperature of the water in the storage containers shall be recorded at least once per day during working hours.

Cement, water and apparatus used to make and test specimens shall be at the laboratory temperature.

NOTE See Annex A for storage conditions applied when testing setting times by an alternative method.

Where temperature ranges are given, the target temperature at which the controls are set shall be the middle value of the range.

4.2 Equipment and materials

4.2.1 General requirements

The tolerances shown in Figures 1 and 2 are important for the correct operation of the equipment in the testing procedure. When regular control measurements show that the tolerances are not met, the equipment shall be rejected, adjusted or repaired. Records of control measurements shall be kept.

Acceptance measurements on new equipment shall cover mass, volume and dimensions to the extent that these are indicated in this International Standard, paying particular attention to those critical dimensions for which tolerances are specified.

In those cases when the material of the equipment can influence the results, the material is specified and shall be used.

The approximate dimensions shown in the figures are provided as guidance to equipment manufacturers or operators. Dimensions that include tolerances are obligatory.

4.2.2 Balance, capable of weighing to an accuracy of ± 1 g.

4.2.3 Graduated cylinder or burette, capable of dispensing to an accuracy of ± 1 ml.

4.2.4 Mixer, conforming to ISO 679.

NOTE A more homogeneous paste is produced at the lower limit of tolerance for the clearance between the blade and the bowl.

4.2.5 Water, distilled or deionized, shall be used for making the specimens.

Drinking water may be used for storing and boiling specimens.

4.2.6 Timer, capable of measuring to an accuracy of ± 1 s.

4.2.7 Ruler, capable of measuring to an accuracy of $\pm 0,5$ mm.

5 Standard consistence test

5.1 Apparatus

Use the manual Vicat apparatus as shown in Figure 1 a) and 1 b) with the plunger as shown in Figure 1 c). The plunger shall be of non-corrodible metal in the form of a right cylinder of at least 45 mm effective length and of $(10,00 \pm 0,05)$ mm diameter. The total mass of moving parts shall be (300 ± 1) 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, plastics or brass. It shall be of cylindrical or preferably truncated conical form $(40,0 \pm 0,2)$ mm deep and shall have an internal diameter of (75 ± 10) mm. It shall be adequately rigid and shall be provided with a base-plate larger than the

mould and at least 2,5 mm thick, constructed of impermeable material resistant to attack by cement paste, e.g. plane glass.

Moulds of other metal may be used, provided that they are of the specified depth and that their use has been calibrated against the specified mould.

It is recommended that a laboratory uses base-plates of equal thickness so that it is necessary only to adjust the scale of the Vicat apparatus once for several determinations.

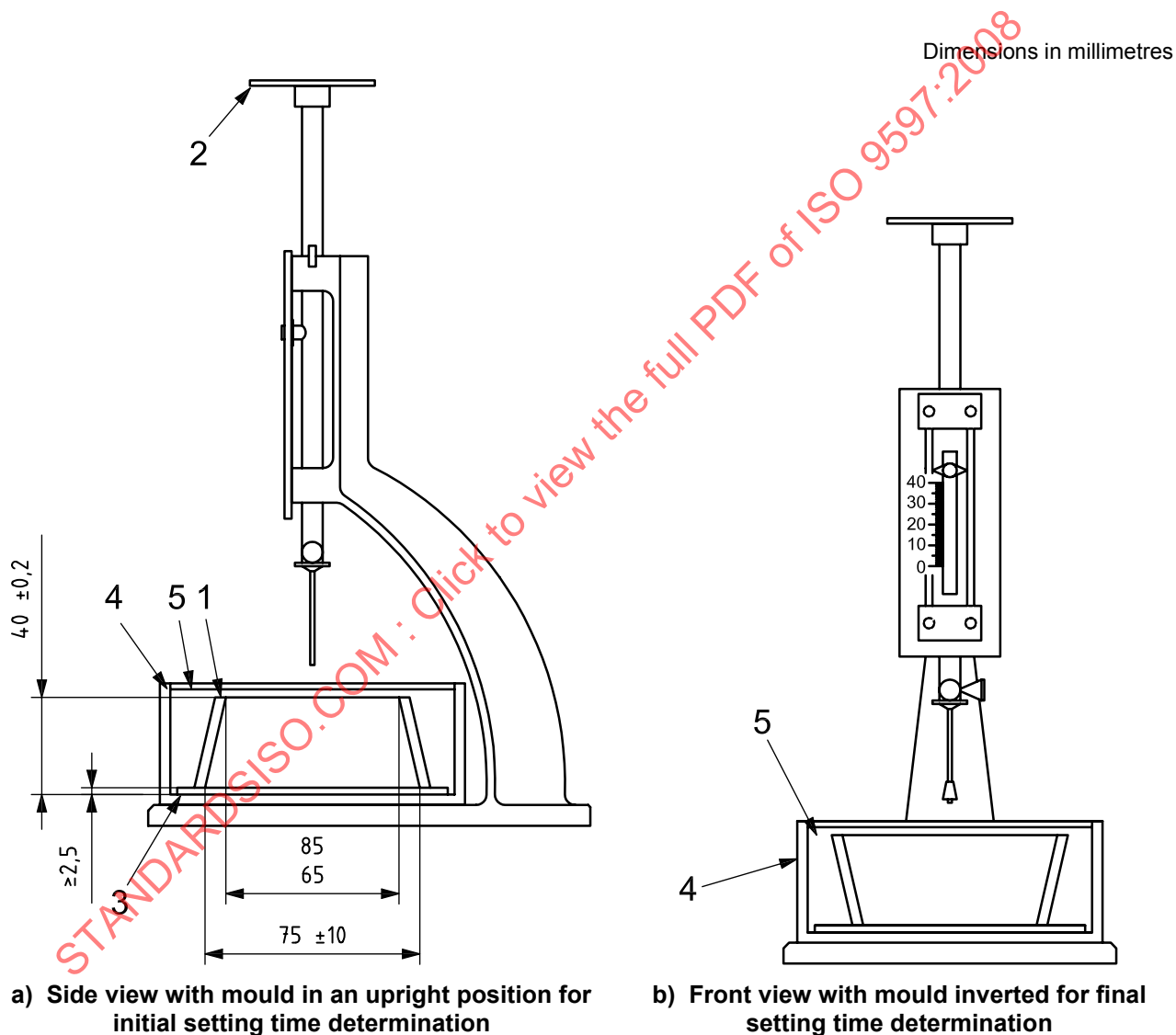
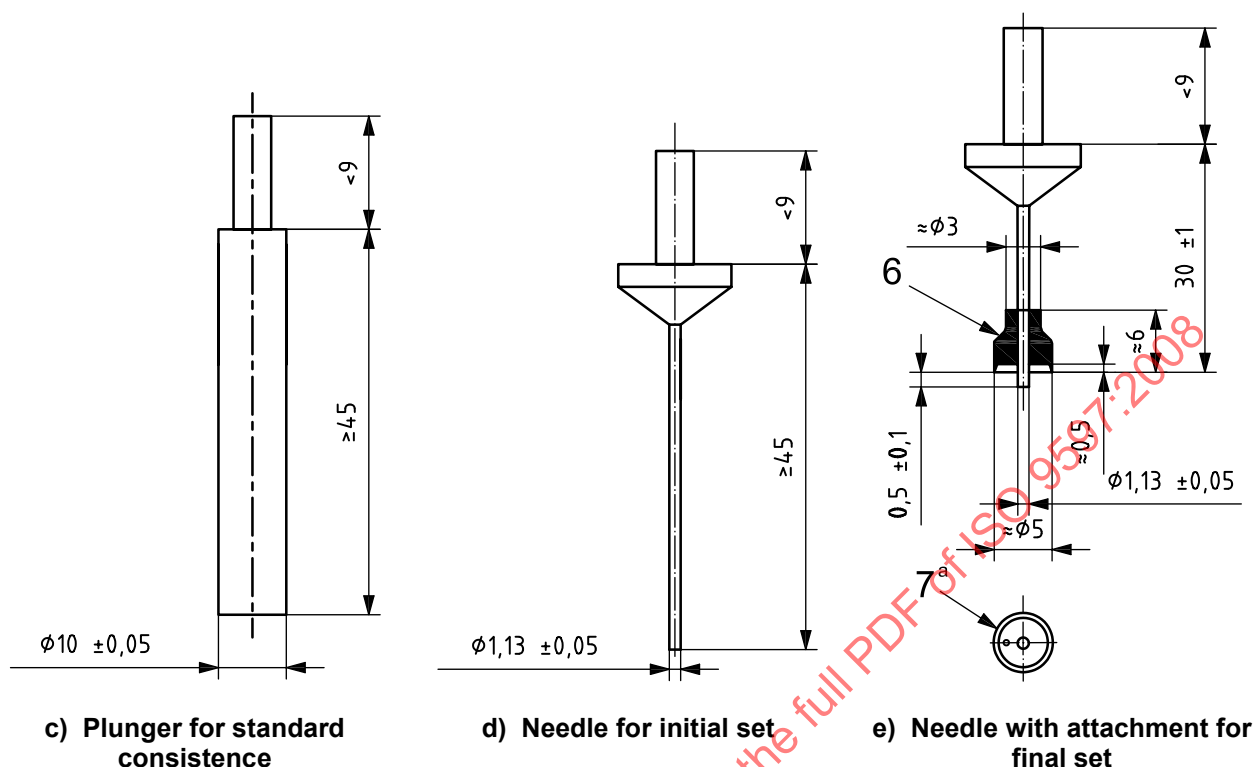


Figure 1 (continued)

Dimensions in millimetres

**Key**

- | | | | |
|---|--------------------|---|---------------------------|
| 1 | mould | 5 | water |
| 2 | correcting weights | 6 | air vent ($\phi = 1,5$) |
| 3 | base-plate | 7 | air vent |
| 4 | container | | |

a View from below needle with attachment for determining final setting time.

NOTE Only one correcting weight is required for each Vicat apparatus if the mass of the plunger and the needles with and without the attachment is always the same, e.g. $(9,0 \pm 0,5)$ g.

Figure 1 — Typical manual Vicat apparatus for determination of standard consistence and setting time

5.2 Procedure

5.2.1 Mixing the cement paste

Weigh, to an accuracy of ± 1 g, by means of the balance (4.2.2), 500 g of cement and a quantity of water, e.g. 125 g. When water is measured by volume using the graduated cylinder or burette (4.2.3), it shall be dispensed to an accuracy of ± 1 ml. Mix each batch of paste mechanically using the mixer (4.2.4). The timing of the various mixing stages refers to the times at which mixer power is switched on/off and shall be maintained within ± 2 s. Proceed as follows.

- With the mixer in the operating condition, place the water and cement into the bowl, taking care to avoid loss of water or cement; complete the addition within 10 s.
- Immediately start the mixer at low speed whilst starting the timing of the mixing stages. In addition, record the time to the nearest minute as “zero time”.

NOTE “Zero time” is the point from which the initial (see 6.2) and final (see 6.3) setting times are calculated.

- c) Stop the mixer after 90 s for 30 s during which time detach, by means of a suitable rubber or plastics scraper, all the paste adhering to the wall and bottom part of the bowl and place it in the middle of the bowl.
- d) Restart the mixer and run at low speed for a further 90 s. The total mixer running time shall be 3 min.

Any other mixing method may be used, provided that it has been calibrated against the reference method.

5.2.2 Filling the mould

Transfer the paste immediately to the lightly oiled mould, which has previously been placed on the lightly oiled base-plate, and fill it to excess without undue compaction or vibration. Remove any voids in the paste by gently tapping the slightly overfilled mould against the ball of the hand. 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.

NOTE Some oils have been found to affect the setting time test; mineral-based oils have been found to be suitable.

WARNING — Wet cement paste is highly alkaline and can cause skin burns. Avoid direct contact with skin during manual operations by wearing protective gloves.

5.2.3 Determination of standard consistence

Adjust the manual Vicat apparatus with the plunger [Figure 1 c)], attached in advance of the test, by lowering the plunger to rest on the base-plate being used and adjusting the pointer or the scale to read zero. 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 s and 2 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 $4 \text{ min} \pm 10 \text{ s}$ after zero time. Read the scale at least 5 s after penetration has ceased or 30 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 percent mass fraction 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 $(6 \pm 2) \text{ mm}$. 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 General

The apparatus described in 6.1.2 to 6.1.5 is that used in carrying out the reference method. An automatic setting-time apparatus conforming to the requirements of the reference method may be used.

Other automatic or manual setting-time apparatus, based on the same test principles, may be used, provided that they have been calibrated against the reference method.

NOTE Experience has shown that the reference method, in which the specimens are tested under water, is not suitable for some slow-setting cements. An alternative test method is set out in Annex A. The specifications for cements and other products state when it is necessary to use this alternative method.

6.1.2 Container, for immersing the filled moulds in water maintained at $(20,0 \pm 1,0) ^\circ\text{C}$ during storage.

A temperature of $(25 \pm 2) ^\circ\text{C}$ or $(27 \pm 2) ^\circ\text{C}$ may be maintained in warm countries, provided the temperature is stated in the test report.

Various storage containers may be used, providing that the surface of the specimen is immersed in water.

6.1.3 Temperature-controlled enclosure, consisting of a water bath or cabinet, thermostatically controlled at $(20,0 \pm 1,0) ^\circ\text{C}$, suitable for storing containers (6.1.2).

A temperature of $(25 \pm 2) ^\circ\text{C}$ or $(27 \pm 2) ^\circ\text{C}$ may be maintained in warm countries, provided the temperature is stated in the test report.

6.1.4 Vicat apparatus (manual or automatic) for initial set

Remove the plunger and replace it with the needle [Figure 1 d)], which shall be of steel and in the form of a right cylinder of effective length of at least 45 mm and diameter $(1,13 \pm 0,05) \text{ mm}$. The total mass of moving parts shall be $(300 \pm 1) \text{ g}$. Their movement shall be truly vertical and without appreciable friction, and their axis shall coincide with that of the needle.

Adjust the Vicat apparatus with the needle [Figure 1 d)], attached in advance of the test, by lowering the needle to rest on the base-plate being used, positioned inside the container, and adjusting the pointer or the scale to read zero. Raise the needle to the stand-by position.

6.1.5 Vicat apparatus (manual or automatic) for final set

Fit the needle with ring attachment of diameter approximately 5 mm [Figure 1 e)] to facilitate accurate observation of small penetrations. The total mass of moving parts shall be $(300 \pm 1) \text{ g}$. Their movement shall be truly vertical and without appreciable friction, and their axis shall coincide with that of the needle.

An automatic setting-time apparatus may be used, in which the needle [Figure 1 d)] is used for determination of final setting times, provided that they have been calibrated against the reference method.

6.2 Determination of the initial setting time

6.2.1 Procedure

Fill a Vicat mould (see 5.1) in accordance with 5.2.2 with paste of standard consistence mixed in accordance with 5.2.1.

Place the filled mould and base-plate in the container (6.1.2), add water so that the surface of the paste is submerged to a depth of at least 5 mm, and store in the temperature-controlled enclosure (6.1.3) at $(20,0 \pm 1,0) ^\circ\text{C}$, $(25 \pm 2) ^\circ\text{C}$ or $(27 \pm 2) ^\circ\text{C}$, as appropriate. After a suitable time, position the mould, base-plate and container under the needle of the Vicat apparatus. 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 parts 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 (see 5.2.1). Repeat the penetration on the same specimen at conveniently spaced positions, not less than 8 mm from the rim of the mould or 5 mm from each other and at least 10 mm from the last penetration position, at conveniently spaced intervals of time, e.g. at 10 min intervals. Between penetrations, keep the specimen in the container in the temperature-controlled enclosure (6.1.3). Clean the Vicat needle immediately after each penetration. Retain the specimen if a determination of the final setting time is also being made.

NOTE The time elapsed between "zero time" (see 5.2.1) and the time at which the distance between the needle and the base-plate is $(6 \pm 3) \text{ mm}$, measured to the nearest minute, is the initial setting time of the cement.

6.2.2 Report

Report the elapsed time, measured from zero to the time at which the distance between the needle and the base-plate is (6 ± 3) mm, as the initial setting time of the cement, to the nearest 5 min.

NOTE Accuracy can be improved by reducing the time interval between penetrations near the setting time.

6.3 Determination of final setting time

6.3.1 Procedure

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. Immerse the mould and base-plate in the container (6.1.2) and store in the temperature controlled enclosure (6.1.3) at $(20,0 \pm 1,0)$ °C, (25 ± 2) °C or (27 ± 2) °C, as appropriate. After a suitable time, position the mould, base-plate and container under the needle of the Vicat apparatus. 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 parts 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.

Repeat the penetrations on the same specimen at conveniently spaced positions, not less than 8 mm from the rim of the mould or 5 mm from each other and at least 10 mm from the last penetration position, at conveniently spaced intervals of time, e.g. at 30 min intervals. Between penetrations, keep the specimen in the container in the temperature controlled enclosure (6.1.3). Clean the Vicat needle immediately after each penetration.

Record the time at which the needle first penetrates only 0,5 mm into the specimen, together with the time from zero (see 5.2.1). 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 penetrations near the final setting time. The final setting time shall be confirmed by repeating the test in two other positions.

6.3.2 Report

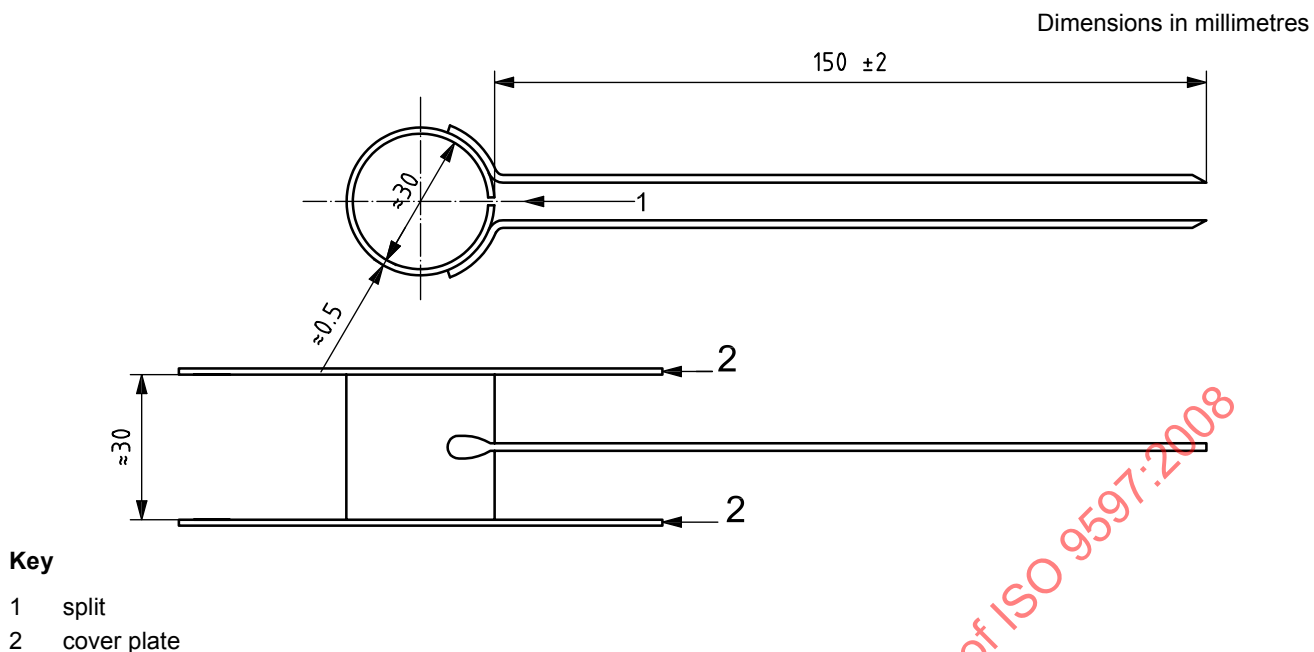
Report the elapsed time, measured from zero to that at which the needle first penetrates only 0,5 mm into the specimen, as the final setting time of the cement, to the nearest 15 min.

7 Soundness test

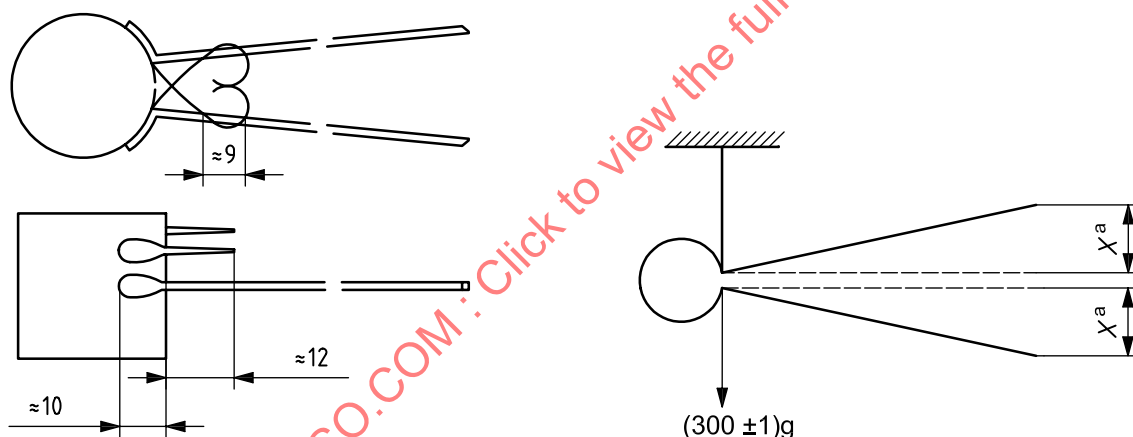
7.1 Apparatus

7.1.1 Le Chatelier apparatus. The mould shall be of non-corrodible spring metal, e.g. brass, with indicator needles and shall have the dimensions shown in Figure 2 a). The resilience of the mould shall be such that the action of a mass of (300 ± 1) g applied as shown in Figure 2 c) shall increase the distance between the indicator ends of the needles by not less than 15,0 mm without permanent deformation.

A pair of base and cover plates of impermeable material, non-corrodible by cement paste, e.g. plane glass, 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 the plate to satisfy this requirement.



a) Apparatus for determining the soundness of cements



NOTE Provision of the two loops shown soldered to the upper half of the mould, on each side of the central split, facilitates demoulding of the hardened paste specimen after test.

^a Increase in separation, X , of indicator ends ($2X \geq 15,0$).

b) Apparatus for determining standard consistence and setting time — Optional arrangement of loops for demoulding

c) Apparatus for determining standard consistence and setting time — Resilience test arrangement

Figure 2 — Typical Le Chatelier apparatus

7.1.2 Water bath, with means of heating, capable of containing immersed Le Chatelier specimens and of raising the temperature of the water from $(20 \pm 2)^\circ\text{C}$ to boiling in (30 ± 5) min.

An initial temperature of $(25 \pm 2)^\circ\text{C}$ or $(27 \pm 2)^\circ\text{C}$ may be maintained in warm countries, provided the temperature is stated in the test report.

7.1.3 Room or humidity cabinet, of adequate size, maintained at $(20 \pm 1)^\circ\text{C}$ and not less than 90 % relative humidity.