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Test Method for Autoclave Expansion of Cement

水泥压蒸安定性试验方法

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Test Method for Autoclave Expansion of Cement

Warning: This test is dangerous. The autoclave shall be used safely during the test. Pay attention to the direction of the exhaust port to avoid steam burns.

1 Scope

This Document gives the method and principle of cement autoclave stability test; and stipulates the instruments, equipment, materials, test conditions, specimen molding and maintenance, specimen length measurement, specimen autoclave and result calculation and evaluation.

This Document is applicable to the determination of autoclave stability of hardened cement paste. Among them, the test method of 25 mm×25 mm×280 mm specimen is the reference method; and the test method of 25 mm×25 mm×146 mm specimen is the substitute method.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 1346 Standard test method for water requirement for normal consistency, setting time, and soundness of the portland cement

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 17671 Test method of cement mortar strength (ISO method)

JC/T 603 Test method for dry shrinkage of cement mortar

JC/T 681 Mixer for mixing mortars

JC/T 723 Cement mortar vibration compaction equipment

JC/T 729 Mixer for cement paste

3 Terms and Definitions

For the purposes of this Document, the following terms and definitions apply.

3.1 Autoclave

The specimen is treated for a certain period of time in a closed environment with saturated water vapor at 215.7°C and a corresponding pressure of 2.0 MPa.

4 Method Principle

Under saturated water vapor conditions, the temperature and pressure are increased to hydrate most of the periclase and free calcium oxide in the cement in a short period of time. The linear expansion rate of the specimen is used to determine the volume stability of the cement.

5 Instruments and Equipment

5.1 Cement mortar mixer

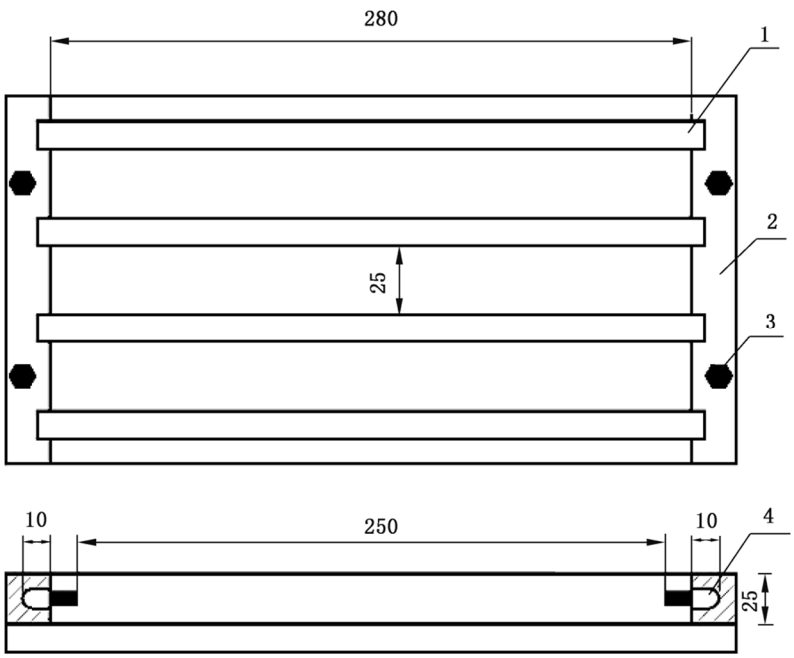
It shall meet the requirements of JC/T 681.

5.2 25 mm×25 mm×280 mm test mold

Triple or double, shall meet the requirements of the test mold in JC/T 603; and the size shall meet the requirements of Figure 1. The end plate and partition board of the test mold are made of 45# steel; the hardness shall be no less than HRC48; and the surface roughness $R\alpha$ shall be no greater than 1.6.

25 mm×25 mm×146 mm test mold can also be used. The test method of 25 mm×25 mm×146 mm specimen shall be carried out according to Appendix A.

Unit: mm



Key:

1 – partition board;

2 – end plate;

3 – tightening bolt;

4 – nail head.

Figure 1 – Schematic diagram of 25 mm×25 mm×280 mm test mold

5.3 Nail head

It shall meet the requirements of JC/T 603.

5.4 Tamping rod

It shall meet the requirements of JC/T 603; the bottom surface of the square tamping rod shall be covered with a rubber layer.

5.5 Autoclave

5.5.1 The autoclave is a high-temperature and high-pressure steam container with a temperature and pressure measurement system, including a safety valve, an exhaust valve, an electric heater, a heat dissipation hole, an automatic pressure control device and a timing device. The safety valve is activated when the pressure in the autoclave reaches 2.2 MPa.

5.5.2 The autoclave shall be equipped with an exhaust valve to discharge air in the early heating stage and release the remaining steam pressure at the end of the later cooling stage.

5.5.3 The maximum range of the pressure gauge is 4.0 MPa, and the accuracy is no less than Level-1.6. When the working pressure is 2.0 MPa, the error of the pressure gauge shall not exceed ±0.06 MPa.

5.5.4 The autoclave shall be made of stainless steel, and the autoclave body and the autoclave cover shall be sealed with ground metal. No steam shall be discharged during the constant pressure process. The inner diameter of the autoclave is about 160 mm, and the volume of the autoclave cavity is about 8.5 L. Under the condition of maximum test load (with specimen and water in the autoclave), the electric heater can raise the steam pressure in the autoclave to 2.0 MPa within 65 min~95 min after closing the exhaust valve.

5.5.5 The automatic pressure control device shall be able to control the pressure in the autoclave within the range of 2.0 MPa±0.1 MPa (equivalent to a temperature of 215.7 ℃±2.6 ℃) and maintain it for more than 3 h.

5.5.6 After the autoclave stops heating, the heat dissipation holes can be opened manually or

automatically; and the pressure can be reduced from 2.0 MPa to below 0.1 MPa within 90 min. The use of the autoclave shall comply with the provisions of Appendix B.

5.6 Temperature measuring device

The measuring range shall be no less than 250 ℃; and the graduation value shall be no greater than 1 ℃.

5.7 Curing box

The temperature shall be maintained at 20 ℃ ±1 ℃; and the relative humidity shall be no less than 90%.

5.8 Comparator

It shall meet the requirements of JC/T 603. Automatic measuring equipment that meets the requirements may also be used. When in use, the comparator shall be consistent with the laboratory temperature. It is made of a specific alloy steel with a small thermal expansion coefficient, and both ends are processed into spherical surfaces of the same size as the measuring nail head. The middle hand grip shall be wrapped with rubber or other insulation materials and marked with vertical marks.

6 Materials

6.1 Cement sample

The cement sample shall pass through a 0.9 mm square hole sieve and be mixed evenly.

6.2 Mixing water

Drinking water shall be used, and Grade-3 water in accordance with GB/T 6682 shall be used for acceptance tests or in case of disputes.

6.3 Water for autoclaving

Use Grade-3 water in accordance with GB/T 6682.

7 Test Conditions

7.1 Molding laboratory

The temperature of the laboratory shall be maintained at 20 ℃ ± 2 ℃, and the relative humidity should be no less than 50%.

7.2 Autoclave laboratory

It shall be equipped with ventilation equipment and tap water source, and shall not be shared with other tests.

7.3 Length measurement environment

The length measurement environment of the specimen shall be consistent with that of the molding laboratory.

8 Molding and Curing of Specimens

8.1 Preparation of test mold

Before the test, apply a thin layer of engine oil to the inner cavity of the test mold; and install the nail head into the round holes at both ends of the mold cavity. The exposed part of the nail head shall not be stained with engine oil.

8.2 Preparation of cement paste

Weigh 800 g + 2 g of cement; and measure the mixing water according to the standard consistency water volume determined by GB/T 1346. Stir according to the procedure specified in GB/T 17671; lift the mixing pot appropriately; and stir the cement paste evenly after stopping stirring for 90 s.

8.3 Molding of the test specimen

Wearing rubber gloves, mix the prepared cement paste thoroughly and evenly; and then load it into the two cavities of the prepared test mold in two layers according to the following steps:

a) The first layer of paste is loaded to a height of about 3/5 height of the test mold. First, use a table knife to compact the paste at a 45° angle to the horizontal direction to evenly fill the mold cavity; then insert and scratch; insert several times on both sides of the nail head to compact the cement paste near the nail head. Starting from the inside of the nail head, between the tails of the two nail heads, compact the paste from one end to the other end in sequence until a dense test specimen is obtained;

b) Load the second layer of paste; fill the test mold evenly; use a table knife to insert and scratch; the insertion and scratch depth does not penetrate the compacted paste; and then compact the paste from one end to the other end in sequence;

c) Regularize the paste to the upper part of the mold cavity and compact it; lift one end of the test mold 30 mm~50 mm and let it fall freely on a hard plate to compact it 3 times; and lift the other end in the same way to compact it 3 times;

d) Use a table knife to scrape off the excess slurry and smooth it, number it.

8.4 Curing and demolding

Put the formed specimen together with the test mold in a moisture curing box for curing for 24 h±30 min and demolding; counting from the time when the cement is mixed with water. After measuring the initial length, place the specimen flat in 20 ℃ ±1 ℃ water for curing; and complete the autoclave test within 2 days.

9 Measurement of Specimen Length

9.1 Dial indicator calibration

Before measuring, use the calibration rod to calibrate the base length reading of the comparator dial indicator. After the measurement is completed, measure the calibration rod reading again. If the change exceeds ±0.01 mm, recalibrate the dial indicator.

9.2 Length measurement

Use a comparator to measure the length of each specimen. Keep the specimen in the comparator in the same position in each measurement. Rotate the specimen before reading. Read the result when the dial indicator pointer is stable. The result is retained to 0.001 mm.

10 Autoclave of Specimen

10.1 Sealed autoclave

10.1.1 Place the specimen on the specimen holder of the autoclave at room temperature.

10.1.2 Add about 850 mL of Grade-3 water in accordance with GB/T 6682 into the autoclave.

10.1.3 Use fine cotton yarn to clean the sealing opening of the autoclave body and the sealing groove of the autoclave cover. Put the autoclave cover vertically on the autoclave body through the bolts and make close contact.

10.1.4 Put 10 nuts with washers on the autoclave cover bolts; and then use a torque wrench to fix them one by one in the order of covering the diagonal lines of the bolts. As shown in Figure 2, first tighten 1 and 6, then 4 and 9, 2 and 7, 5 and 10, 3 and 8. Tighten all nuts repeatedly; and finally check whether the torque force reaches 100 N • m one by one in the order of 1, 2, 3, 4, 5, 6, 7, 8, 9, 10.

10.2 Start the temperature measuring device

Insert the temperature measuring device (5.6) into the temperature measuring hole on the autoclave cover.

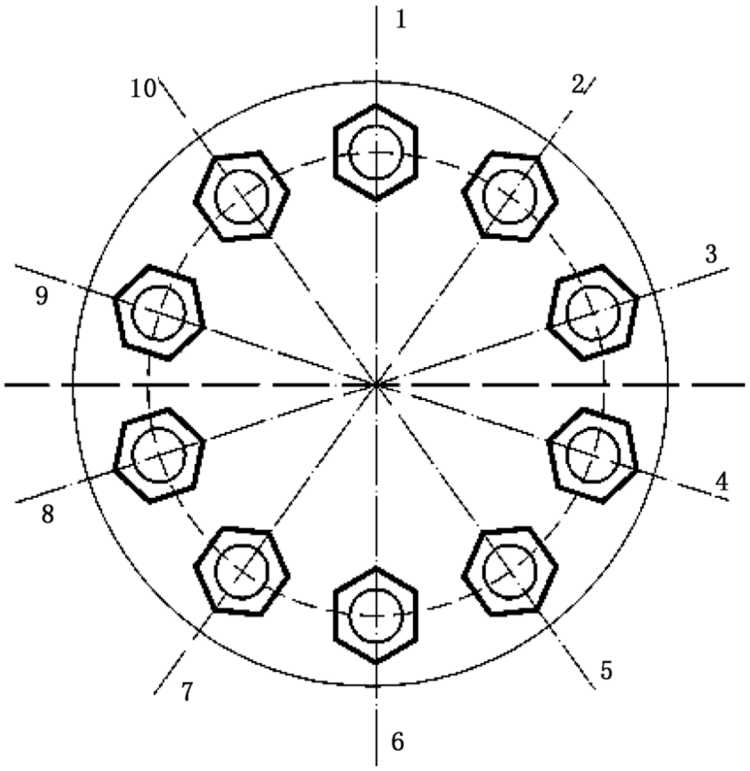


Figure 2 -- Schematic diagram of relative position of the autoclave cover bolts

10.3 Autoclave operation steps

10.3.1 Open the exhaust valve, electric heater and pressure control system; and the hot air in the autoclave cavity is discharged from the exhaust valve until continuous steam is released and then close the exhaust valve.

10.3.2 Record the temperature and pressure increase process of the autoclave. After 65 min ~ 95 min, the pressure in the autoclave reaches 2.0 MPa ± 0.1 MPa and is maintained at this pressure for 3 h.

10.3.3 Turn off the electric heater; open the heat dissipation hole; and cool to the pressure in the autoclave below 0.1 MPa within 90 min.

10.3.4 Open the exhaust valve to discharge the remaining steam in the autoclave.

10.3.5 During the autoclave experiment, observe the relationship between pressure and temperature at any time (the temperature is 215.7 ℃ ± 2.6 ℃ when the gauge pressure is 2.0 MP ± 0.1 MPa). The corresponding relationship between temperature and saturated water vapor pressure is shown in Table 1. If the temperature rises and the pressure drops, the power supply shall be cut off immediately and the experiment shall be stopped. For cement types with added steel slag, the pressure steaming time is 6 h.

Table 1 -- The corresponding relationship between saturated water vapor pressure and temperature

Pressure MPa	Temperature ℃	Pressure MPa	Temperature ℃
0.1	118	1.6	207
0.5	156	1.7	210
1.0	183	1.8	213
1.2	192	1.9	215
1.4	200	2.0	216

10.4 Steps to open the autoclave

10.4.1 The order of opening the nuts of the autoclave cover is still: first tighten 1 and 6, then tighten 4 and 9, 2 and 7, 5 and 10, 3 and 8.

10.4.2 Vertically extract the autoclave cover to separate the autoclave cover from the autoclave body, and take out the specimen support.

10.4.3 After the autoclave body temperature drops, use a ruler to measure the height of the remaining water and record it; and wipe the remaining water in the autoclave with a clean towel. Use two wooden strips of about 20 mm×20 mm×200 mm to pad between the autoclave body sealing port and the autoclave cover; and cover it with a dust cover.

10.5 Specimen cooling

After taking out the specimen, immediately place it in hot water above 90℃. The height of the water tank shall be higher than 3 times the height of the specimen. Then pour cold water into the hot water. Avoid pouring cold water directly onto the specimen surface. Reduce the water temperature to 20℃±2℃ within 15 min. Keep the water temperature constant for at least 1 h before measuring the specimen length.

10.6 Measure the length after autoclaving

Measure the length of the specimen after autoclaving (L_1) according to the steps in Clause 9. Record the phenomenon of specimen powdering, bending, cracking, bursting, and exceeding the measuring range of the comparator.

11 Calculation and Evaluation of Results

11.1 Calculation of autoclave expansion rate

The autoclave expansion rate of the specimen is calculated according to Formula (1), and the result is retained to 0.001%.

$$L_A = \frac{L_1 - L_0}{L} \times 100\% \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (1)$$

Where:

L_A - Expansion rate of the specimen after autoclaving;

L_1 - Length of the specimen after autoclaving, in mm;

L_0 - Initial length of the specimen, in mm;

L - Effective length of the specimen, in mm; generally, 250 mm.

11.2 Test results

The average value of the pressure expansion rate of two specimens in one group is taken as the test result; and the result is retained to 0.01%.

When the difference in the pressure expansion rate of the two specimens is no greater than 0.05%, the average value of the pressure expansion rate of the two specimens is taken as the test result. When the difference in the pressure expansion rate of the two specimens is greater than 0.05%, and the pressure expansion rate of the two specimens differs from the average value by no more than ±10%, the average value of the pressure expansion rate of the two specimens is taken as the test result. When the difference in the pressure expansion rate of the two specimens is greater than 0.05%, and the pressure expansion rate of the two specimens differs from the average value by more than ±10%, this test is invalid and shall be retested.

11.3 Qualification assessment

11.3.1 If the specimen is powdered, bent, cracked, burst, or exceeds the measurement range of the comparator, it is considered unqualified for pressure steam stability.

11.3.2 If the autoclave expansion rate of Portland cement is no more than 0.80%, it shall mean the autoclave stability is qualified; otherwise, the autoclave stability is unqualified.

11.3.3 If the autoclave expansion rate of ordinary Portland cement, slag Portland cement, pozzolanic Portland cement, fly ash Portland cement, composite Portland cement and other cement varieties with mixed materials is no more than 0.50%, it shall mean the autoclave stability is qualified; otherwise, the autoclave stability is unqualified.

Appendix A

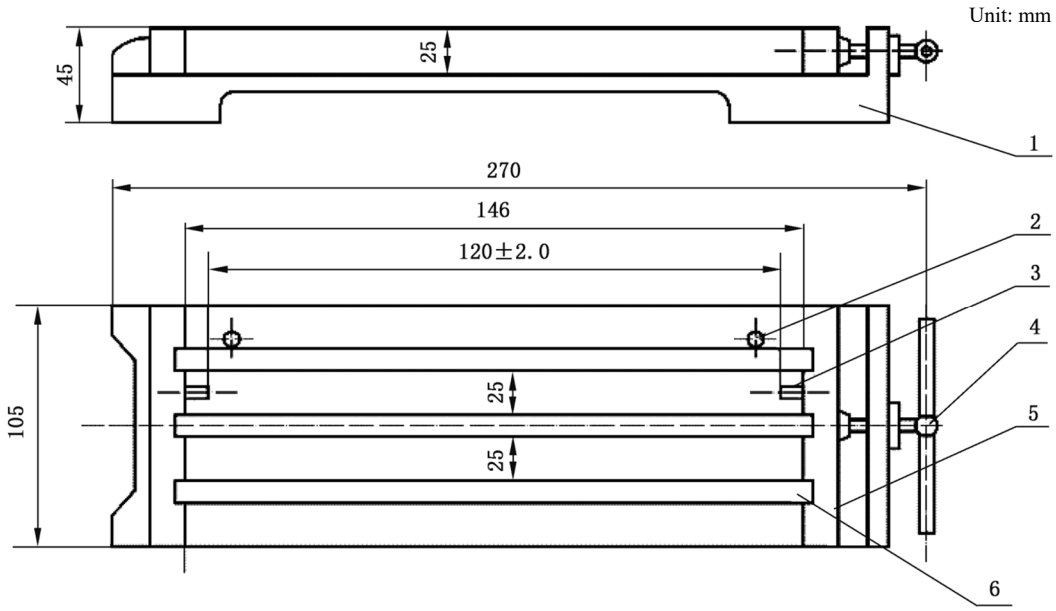
(Normative)

Test Method of 25mm×25mm×146mm specimen

A.1 Instruments and equipment

A.1.1 25 mm×25 mm×146 mm test mold

The test mold is a two-piece type; and its dimensions shall meet the requirements of Figure A.1. The end plate and partition board of the test mold are made of 45# steel, with a hardness no less than HRC48 and a surface roughness *R*α no greater than 1.6.



Key:

1 - base;

2 - positioning pin;

3 - nail head;

4 - fastening device;

5 - end plate;

6 – partition board.

Figure A.1 -- 25 mm×25 mm×146 mm pressure steaming two-piece test mold

A.1.2 Nail head

It is made of stainless steel or copper; the dimensions shall meet the requirements of Figure A.2. After the nail heads are fixed on the test mold, the distance between the inner sides of the nail heads is 120 mm±2 mm; and the depth of the nail heads penetrating into the test mold is 7 mm±1 mm.

Unit: mm

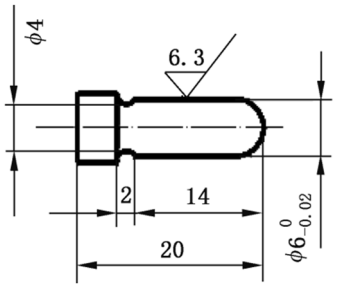


Figure A.2 – Nail head

A.1.3 Mold sleeve

The mold sleeve is made of steel and is used to block the material and fix the test mold during forming. The size shall meet the requirements of Figure A.3.

Unit: mm

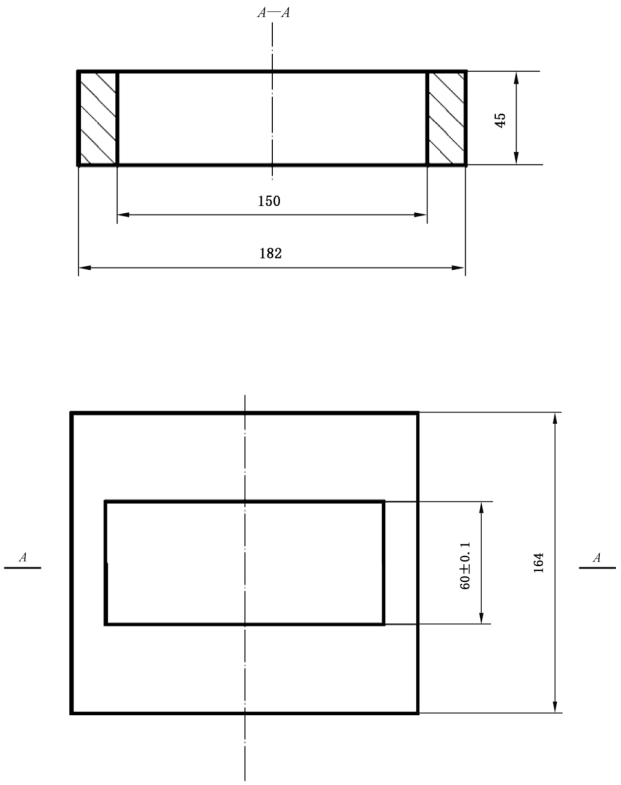


Figure A.3 – Mold sleeve

A.1.4 Cement slurry mixer

It shall meet the requirements of JC/T 729.

A.1.5 Mortar vibrating table

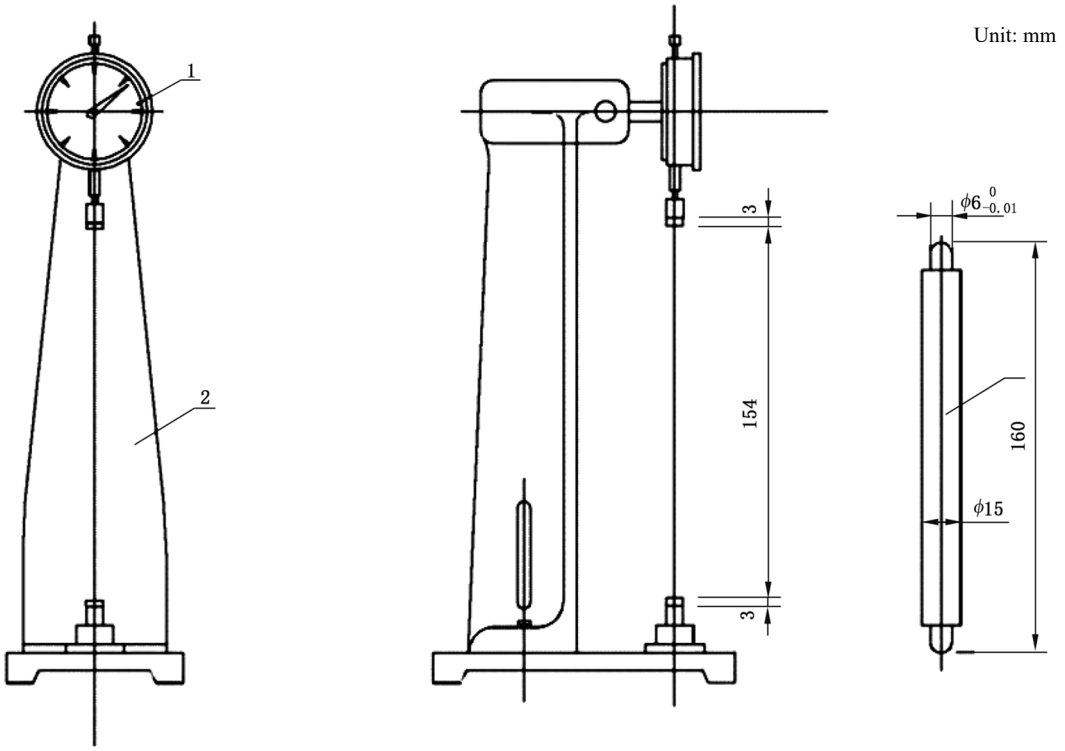
It shall meet the requirements of JC/T 723.

A.1.6 Autoclave

It shall meet the requirements of 5.5.

A.1.7 160 mm comparator

The range of the dial indicator is 0 mm ~10 mm, and the minimum graduation value is 0.01 mm. The length of the correction rod is 160 mm; and the structure is shown in Figure A.4.



Key:

1 – dial indicator;

2 – bracket;

3 – correction rod.

Figure A.4 – 160mm comparator

A.2 Forming and curing of test specimens

A.2.1 Preparation of test molds

Before the test, apply a thin layer of engine oil to the test mold and insert the nail heads (A.1.2) into the round holes at both ends of the mold groove. The exposed part of the nail heads shall not be contaminated with engine oil.

A.2.2 Preparation of cement paste

Weigh 500 g ± 1 g of cement and mix it with water of standard consistency according to the procedure specified in GB/T 1346.

A.2.3 Molding of test specimen

Clamp the test mold with installed nail heads and the mold sleeve on the working position of the mortar vibrating table (A.1.5); put the mixed cement paste of standard consistency into the test mold at one time and flatten it with a table knife; start the mortar vibrating table and use a knife to scratch back and forth along the length of the test mold to make the surface of the paste flat and fill the edges and corners of the test mold. The scratching operation shall be completed within 80 s after starting. After vibrating for 2 min, remove the mold sleeve and take off the test mold, and remove the excess paste.

A.2.4 Curing and demolding

Put the molded test specimen and the test mold horizontally in the curing box (5.7) for curing for 24 h ± 30 min and demolding. The counting starts from the time when the cement is mixed with water.

A.3 Measurement of specimen length

Use 160 mm comparator (A.1.7) to measure the length.

A.4 Autoclave of specimen

Perform according to Clause 10.

A.5 Calculation and evaluation of results

Perform according to Clause 11, but the effective length (L) of the specimen is 120 mm.

Appendix B

(Normative)

Safety Precautions

B.1 During the autoclave test, the temperature and saturated steam pressure shall be measured simultaneously. Because the temperature and saturated steam pressure have a certain relationship, using them simultaneously can timely detect the failure of the pressure gauge or the abnormal situation caused by the loss of water in the autoclave during the test.

B.2 The safety valve shall be adjusted to 10% higher than the working pressure of the autoclave test, which is about 2.2 MPa. The safety valve shall be inspected at least once a year. During the inspection, the pressure gauge can be used to inspect the equipment; or the pressure automatic controller can be adjusted to make the autoclave reach 2.2 MPa. At this time, the safety valve shall be opened immediately.

B.3 During the use of the autoclave, the following failures may occur at the same time: the automatic controller fails; the safety valve is not sensitive; the pressure pointer suddenly indicates zero, but it has actually exceeded the maximum scale and returned to zero from the opposite direction. The pressure gauge needle returns to the initial position of the test; and the autoclave may still maintain a certain pressure. If these situations are found, no matter how high the pressure in the autoclave is, the test shall be stopped immediately, the power supply shall be cut off, and safety measures shall be taken.

B.4 When the pressure steam test is completed and the air is released, the operator shall stand away from the release valve. When opening the autoclave cover, asbestos gloves shall be worn to avoid burns.

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