



Standard Test Method for Autoclave Expansion of Hydraulic Cement¹

This standard is issued under the fixed designation C151/C151M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers determination of the autoclave expansion of hydraulic cement by means of a test on a neat cement specimen.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precaution statements, see the section on Safety Precautions.

2. Referenced Documents

2.1 ASTM Standards:²

- C187 Test Method for Amount of Water Required for Normal Consistency of Hydraulic Cement Paste
- C305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency
- C490 Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete
- C511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes
- C856 Practice for Petrographic Examination of Hardened Concrete
- C1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements

¹ This test method is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.31 on Volume Change.

Current edition approved Jan. 1, 2015. Published January 2015. Originally approved in 1940. Last previous edition approved in 2009 as C151/C151M – 09. DOI: 10.1520/C0151_C0151M-15.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

C1157 Performance Specification for Hydraulic Cement

3. Significance and Use

3.1 The autoclave expansion test provides an index of potential delayed expansion caused by the hydration of CaO or MgO, or both, when present in hydraulic cement.³

4. Interferences

4.1 Pozzolans containing fine-grained quartz have been reported to cause excessive expansion under autoclave test conditions due to alkali-silica reaction that will not occur under normal service conditions.⁴ When excessive expansion of paste containing pozzolans occurs, it is recommended that the specimens be examined (see Practice C856) for evidence of alkali-silica reaction (Note 1).

NOTE 1—If the excessive expansion has resulted from alkali-silica reaction under autoclave conditions, the pozzolan can be evaluated for alkali reactivity using the procedure described in Performance Specification C1157.

5. Apparatus

5.1 *Weighing Devices and Weights*, for determining the mass of materials conforming to the requirements of Specification C1005.

5.2 *Glass Graduates*, 200 or 250-mL capacity, and conforming to the requirements of Practice C490.

5.3 *Molds*, conforming to the requirements of Practice C490 for test specimens used in the determination of length change of cement paste.

5.4 *Flat Trowel*, conforming to the requirements of Test Method C187.

5.5 *Autoclave*, consisting of a high-pressure steam vessel provided with a thermometer well. The autoclave shall be equipped with automatic controls and either a rupture disk with a bursting pressure of 2.4 MPa [350 psi] \pm 5 % or a safety valve that actuates at this pressure. In addition, the autoclave shall be equipped with a vent valve to allow the escape of air

³ Gonnerman, H. F., Lerch, W. and Whiteside, T. M., "Investigations of the Hydration Expansion Characteristics of Portland Cements," *Portland Cement Association Research Department Bulletin 45*, pp. 1–168, 1953.

⁴ Wang, H., "Autoclave Soundness Test Mischaracterizes Cement-Fly Ash Blends by Introducing Alkali-Quartz Reaction," *Cement, Concrete, and Aggregates*, Vol 24, No. 2, pp. 68–72, 2002.

*A Summary of Changes section appears at the end of this standard

during the early part of the heating period and to release any steam pressure remaining at the end of the cooling period. The pressure gauge shall have a nominal capacity of 4.0 MPa [600 psi], a dial with a nominal diameter of 115 mm [4½ in.] and shall be graduated from 0 to 4.0 MPa [0 to 600 psi] with scale divisions not exceeding 0.03 MPa [5 psi]. The error in the gauge shall not exceed ± 0.02 MPa [± 3 psi] at the operating pressure of 2 MPa [295 psi]. The capacity of the heating unit shall be such that with maximum load (water plus specimens) the pressure of the saturated steam in the autoclave may be raised to a gauge pressure of 2 MPa [295 psi] in 45 to 75 min from the time the heat is turned on. The automatic control shall be capable of maintaining the gauge pressure at 2 ± 0.07 MPa [295 ± 10 psi] for at least 3 h. A gauge pressure of 2 ± 0.07 MPa [295 ± 10 psi] corresponds to a temperature of $216 \pm 2^\circ\text{C}$ [$420 \pm 3^\circ\text{F}$]. The autoclave shall be designed to permit the gauge pressure to drop from 2 MPa to less than 0.07 MPa [295 psi to less than 10 psi] in 1½ h after the heat supply has been shut off.

5.5.1 Rupture Disk—The rupture disk shall be made of a material having a tensile strength that is relatively insensitive to temperature in the range 20 to 216°C [68 to 420°F] and that is electrochemically compatible with the pipe leading to it and to its holder.⁵

5.6 Length Comparator—The comparator used for measuring length change of specimens shall conform to the requirements of Practice C490.

6. Temperature and Humidity

6.1 Molding Room—Maintain the temperature of the molding room, dry materials and mixing water, and the relative humidity of the molding room within the limits of Practice C490.

6.2 Moist Storage Facilities—Maintain the temperature and humidity of the moist storage facilities to the requirements of Specification C511.

7. Safety Precautions

7.1 The pressure gauge shall have a capacity of 4.0 MPa [600 psi]. A gauge with too small or too large a capacity may be a hazard, since for pressure above the specified maximum working pressure, with a smaller capacity gauge, the pressure may be off scale, and with a larger capacity gauge, the arc of movement may be too small to invite attention. The operator shall be sure the gauge hand has not passed the maximum graduation on the scale.

7.2 Test the pressure gauge for proper operation. Always use a thermometer together with the pressure gauge, so as to provide a means of detecting any failure of the pressure gauge to operate properly, and to indicate any unusual condition.

7.3 Maintain the automatic control in proper working order at all times.

7.4 Set the safety valve to relieve the pressure at about 6 to 10 % above the maximum of 2.1 MPa [305 psi] specified in

this test method, that is, at about 2.3 MPa [330 psi]. Unless the manufacturer has given specific instructions as to maintenance of the safety valve, test the valve twice each year.⁶ Test with a gauge testing device, or by adjusting the automatic controls to allow the autoclave to reach a pressure of about 2.3 MPa [330 psi], at which pressure the safety valve will either open or be adjusted to open. Direct the safety valve discharge away from the operator.

NOTE 2—Unexpected combinations of conditions may occur. For example, in one case the automatic control had failed, the safety valve had become stuck, and the gauge hand, which at first glance appeared to be at about zero, had 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.

7.5 Wear heavy leather work gloves to prevent burning of the hands when removing the top of the autoclave at the end of the test. Direct the vent valve away from the operator. When removing the autoclave lid, tilt it so that any steam escaping from beneath the lid will 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.

7.6 The operator shall be made aware that for many autoclave pressure gauges 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 dangerous pressure. Procedures in 11.2 shall be followed.

8. Number of Test Specimens

8.1 Make at least one test specimen.

9. Preparation of Specimen Molds

9.1 Prepare the specimen molds in accordance with the requirements of Practice C490 except that molds need not be sealed.

10. Preparation of Test Specimens

10.1 Mixing Cement Paste—Prepare the standard batch consisting of 650 g of cement and sufficient water to give a paste of normal consistency in accordance with the procedure described in Test Method C187. Mix this batch in accordance with the procedure described in Practice C305. Both a time of setting specimen and an autoclave bar may be made from the same batch. If the paste to be used for time of setting is the paste sample already used to determine normal consistency, then the autoclave bar may be prepared immediately from the remainder of the batch.

10.2 Molding Specimens—Immediately following preparation of the time of setting specimen or completion of mixing, mold the test specimen in two approximately equal layers, each layer being compacted with the thumbs or forefingers by pressing the paste into the corners, around the gauge studs, and along the surface of the mold until a homogeneous specimen is obtained. Compact the top layer, cut off the paste flush with the top of the mold with a thin-edged trowel, and smooth the

⁵ A list of suppliers of autoclave rupture disks and gaskets is maintained at ASTM Headquarters.

⁶ A list of facilities for testing autoclave safety valves is maintained at ASTM Headquarters.

surface with a few strokes of the flat trowel. During the operations of mixing and molding, protect the hands with rubber gloves.

10.3 *Storage of Test Specimens*—After filling the mold, place it in the moist closet or moist room. Store the specimens in the molds in the moist enclosure for at least 20 h; if removed from the molds before 24 h, they shall be kept in the moist closet or moist room until time of test.

11. Procedure

11.1 At $24\text{ h} \pm 30\text{ min}$ after molding, remove the specimens from the moist atmosphere, immediately obtain a length comparator reading for each specimen, and place in the autoclave at room temperature in a rack so that all sides of the specimen will be exposed to saturated steam. The autoclave shall contain enough water, at an initial temperature of $20\text{ to }28^\circ\text{C}$ [$68\text{ to }82^\circ\text{F}$], to maintain an atmosphere of saturated steam vapor during the entire test. Ordinarily 7 to 10 % of the volume of the autoclave should be occupied by the water.

11.2 To permit air to escape from the autoclave during the early portion of the heating period, leave the vent valve open until steam begins to escape. (**Warning**—See the section on Safety Precautions). Close the valve and raise the temperature of the autoclave at a rate that will bring the gauge pressure of the steam to 2 MPa [295 psi] in 45 to 75 min from the time the heat is turned on. Maintain the $2 \pm 0.07\text{ MPa}$ [$295 \pm 10\text{ psi}$] pressure for 3 h. At the end of the 3-h period, shut off the heat supply and cool the autoclave at such a rate that the pressure will be less than 0.07 MPa [10 psi] at the end of $1\frac{1}{2}\text{ h}$. At the end of the $1\frac{1}{2}\text{-h}$ period, slowly release any remaining pressure by partially opening the vent valve until atmospheric pressure is attained. Then open the autoclave and place the test specimen in water at a temperature above 90°C [194°F]. Cool the water surrounding the bars at a uniform rate by adding cold

water so that the temperature of the water will be lowered to 23°C [74°F] in 15 min. Maintain the water surrounding the specimens at 23°C [74°F] for an additional 15 min; then, remove one specimen at a time from the water, blot the pins, but not the specimen, and obtain a length comparator reading.

12. Calculation

12.1 Calculate the change in length of the test specimen by subtracting the length comparator reading before autoclaving from that after autoclaving, and report as percent of effective gauge length to the nearest 0.01 %. Report the percentage of increase in length as the autoclave expansion. Indicate a decrease in length by a minus sign prefixed to the percent value.

13. Precision and Bias

13.1 *Precision*—The single-operator (within-laboratory) standard deviation has been found to be 0.024 % throughout the range of 0.11 % to 0.94 % expansions. Therefore, results of two properly conducted tests by the same operator for expansions of similar batches should not differ from each other by more than 0.07 % expansion. The multi-laboratory (between laboratory) standard deviation has been found to be 0.030 % throughout the range of 0.11 % to 0.94 % expansions. Therefore, results of two properly conducted tests from two different laboratories for expansions of similar batches should not differ from each other by more than 0.09 % expansion.

13.2 *Bias*—Since there is no accepted reference material suitable for determining bias, bias has not been determined.

14. Keywords

14.1 autoclave; autoclave expansion; expansion; hydraulic cement; soundness

For additional useful information on details of cement test methods, reference may be made to the “Manual of Cement Testing,” which appears in the *Annual Book of ASTM Standards*, Vol 04.01.

SUMMARY OF CHANGES

Committee C01 has identified the location of selected changes to this standard since the last issue (C151/C151M – 09) that may impact the use of this standard. (Approved Jan. 1, 2015.)

(1) Revised subsections 5.5 and 7.6.

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