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Standard Test Method for Autoclave Expansion of Portland Cement¹

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1. Scope

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

1.2 The values stated in inch-pound units are to be regarded as 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:*

¹ This test method is under the jurisdiction of ASTM Committee C-1 C01 on Cement and is the direct responsibility of Subcommittee C01.31 on Volume Change. Current edition approved Oct. 10, 1998; 2000. Published February 1999; September 2000. Originally published as C 151 – 40 T. Last previous edition C 151 – 98a.

C 187 Test Method for Normal Consistency of Hydraulic Cement²

C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency²

C 490 Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete²

C 511 Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes²

C 1005 Specification for Weights and Weighing Devices for Use in the Physical Testing of Hydraulic Cements²

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 portland cement.³

4. Apparatus

4.1 *Weighing Devices and Weights*, for determining the mass of materials conforming to the requirements of Specification C 1005.

4.2 *Glass Graduates*, 200 or 250-mL capacity, and conforming to the requirements of Practice C 490.

4.3 *Molds*, 1 by 1-in. (25.4 by 25.4-mm) cross section, conforming to the requirements of Practice C 490.

4.4 *Flat Trowel*, having a straight-edged steel blade 4 to 6 in. (100 to 150 mm) in length.

4.5 *Autoclave*, consisting of a high-pressure steam vessel provided with a thermometer well. The autoclave shall be equipped with an automatic pressure control and a rupture disk with a bursting pressure of 350 psi (2.4 MPa) \pm 5 %. In locations where the use of a rupture disk is not permitted, the autoclave shall be equipped with a safety valve. In addition, the autoclave shall be equipped with a vent valve to allow the escape of air during the early part of the heating period and to release any steam pressure remaining at the end of the cooling period. The pressure gage shall have a nominal capacity of 600 psi (4.1 MPa), a dial with a nominal diameter of 4½ in. (114 mm) and shall be graduated from 0 to 600 psi (0 to 4.1 MPa) with scale divisions not exceeding 5 psi (0.03 MPa). The error in the gage shall not exceed \pm 3 psi (\pm 0.02 MPa) at the operating pressure of 295 psi (2 MPa). 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 gage pressure of 295 psi in 45 to 75 min from the time the heat is turned on. The automatic pressure control shall be capable of maintaining the gage pressure at 295 ± 10 psi (2 ± 0.07 MPa) for at least 3 h. A gage pressure of 295 ± 10 psi corresponds to a temperature of $420 \pm 3^\circ\text{F}$ ($216 \pm 2^\circ\text{C}$). The autoclave shall be designed to permit the gage pressure to drop from 295 psi to less than 10 psi in 1½ h after the heat supply has been shut off.

4.5.1 *Rupture Disk*—The rupture disk shall be made of a material having a tensile strength that is relatively insensitive to temperature in the range 68 to 420°F (20 to 216°C) and that is electrochemically compatible with the pipe leading to it and to its holder.⁴

4.6 *Length Comparator*—The comparator used for measuring length change of specimens shall conform to the requirements of Practice C 490.

5. Temperature and Humidity

5.1 *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 C 490.

5.2 *Moist Storage Facilities*—Maintain the temperature and humidity of the moist storage facilities to the requirements of Specification C 511.

6. Safety Precautions

6.1 The pressure gage shall have a capacity of 600 psi (4.1 MPa). A gage 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 gage, the pressure may be off scale, and with a larger capacity gage, the arc of movement may be too small to invite attention. The operator shall be sure the gage hand has not passed the maximum graduation on the scale.

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

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

6.4 Set the safety valve to relieve the pressure at about 6 to 10 % above the maximum of 305 psi (2.1 MPa) specified in this test method, that is, at about 330 psi (2.3 MPa). Unless the manufacturer has given specific instructions as to maintenance of the safety valve, test the valve twice each year.⁵ Test with a gage testing device, or by adjusting the automatic controls to allow the

² *Annual Book of ASTM Standards*, Vol 04.01.

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*, 1953, pp. 1-168.

⁴ A list of suppliers of autoclave rupture disks and gaskets is maintained at ASTM Headquarters.

⁵ A list of facilities for testing autoclave safety valves is maintained at ASTM Headquarters.

autoclave to reach a pressure of about 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 1—Unexpected combinations of conditions may occur. For example, in one case the automatic control had failed, the safety valve had become stuck, and the gage 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 gage was finally detected and the pressure, then of an unknown magnitude, was released before failure could occur in the apparatus.

6.5 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.

6.6 The operator shall be made aware that for many autoclave pressure gages the return of the gage 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.

7. Number of Test Specimens

7.1 Make at least one test specimen.

8. Preparation of Specimen Molds

8.1 Prepare the specimen molds in accordance with the requirements of Practice C 490 except that molds need not be sealed.

9. Preparation of Test Specimens

9.1 *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 C 187. Mix this batch in accordance with the procedure described in Practice C 305. 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.

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

9.3 *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.

10. Procedure

10.1 At 24 h \pm 30 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 68 to 82°F (20 to 28°C), 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.

10.2 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. (**Caution**—See the section on Safety Precautions). Close the valve and raise the temperature of the autoclave at a rate that will bring the gage pressure of the steam to 295 psi (2 MPa) in 45 to 75 min from the time the heat is turned on. Maintain the 295 \pm 10 psi (2 \pm 0.07 MPa) 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 10 psi at the end of 1½ h. At the end of the 1½-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 194°F (90°C). 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 74°F (23°C) in 15 min. Maintain the water surrounding the specimens at 74°F for an additional 15 min; then, surface-dry remove one specimen at a time from the water, blot the pins, but not the specimens, and obtain a length comparator reading for each specimen.

11. Calculation

11.1 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 gage 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.

12. Precision and Bias

12.1 *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.

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

13. Keywords

13.1 autoclave; autoclave expansion; expansion; portland 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.

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