Designation: C 186 – 98

Standard Test Method for Heat of Hydration of Hydraulic Cement[1]

This standard is issued under the fixed designation C 186; 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 the determination of the heat of hydration of a hydraulic cement by measuring the heat of solution of the dry cement and the heat of solution of a separate portion of the cement that has been partially hydrated for 7 and for 28 days, the difference between these values being the heat of hydration for the respective hydrating period.

1.2 The results of this test method may be inaccurate if some of the components of the hydraulic cement are insoluble in the nitric acid/hydrofluoric acid solution.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 Values in SI units shall be obtained by measurement in SI units or by appropriate conversion, using the Rules for Conversion and Rounding given in Standard IEEE/ASTM SI 10, or measurements made in other units.

1.5 *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 or regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 109 Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)[2]

C 114 Test Methods for Chemical Analysis of Hydraulic Cement[2]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[3]

C 1005 Specification for Weights and Weighing Devices for Use in the Physical Testing of Hydraulic Cements[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System[4]

3. Significance and Use

3.1 The purpose of this test is to determine if the hydraulic cement under test meets the heat of hydration requirement of the applicable hydraulic cement specification.

3.2 This test may also be used for research purposes when it is desired to determine the heat of hydration of hydraulic cement at any age.

NOTE 1—When tests are performed for research purposes, useful additional information can be obtained by determining fineness, chemical and compound compositions.

3.3 Determination of the heat of hydration of hydraulic cements provides information that is helpful for calculating temperature rise in mass concrete.

4. Apparatus

4.1 *Calorimetric Apparatus*:

4.1.1 *Calorimeter*—The calorimeter, such as that illustrated in Fig. 1 shall consist of a 0.5-L (1-pt), wide-mouth vacuum jar, with cork stopper, or other suitable non-reactive stopper held in a suitably insulated container (see 4.1.2) to keep the vacuum jar in position and to protect the jar from undue temperature fluctuations. The vacuum jar shall be coated on the interior with a material resistant to hydrofluoric acid, such as a baked phenolic resin, a baked vinyl chloride acetate resin, or beeswax. The acid-resistant coating shall be intact and free of cracks at all times; it shall be examined frequently and renewed whenever necessary. As another means of protecting the vacuum jar, a plastic liner of suitable size may be used instead of coating the interior of the jar. The contents of the vacuum jar shall not change more than 0.001°C/min per degree difference from room temperature when filled with 425 g of the acid specified in 6.2, stoppered, and allowed to stand unstirred for 30 min. The temperature for this check shall approximate the starting temperatures to be used in making the determination.

4.1.2 *Insulated Container*—The container shall have an insulating layer of a material such as non-reactive foam, cotton, or fiber-glass, which shall be at least 25 mm (1 in.) in thickness and shall encase the sides and bottom of the vacuum jar, but shall be so arranged as to permit easy removal of the jar.

4.1.3 *Differential and Reference Thermometers*—The adjustable differential thermometer shall be of the Beckmann-type, graduated at least to 0.01°C, and shall have a range of approximately 6°C. The thermometer shall be so adjusted that

[1] This test method is under the jurisdiction of ASTM Committee C-1 on Cement and is the direct responsibility of Subcommittee C01.26 on Heat of Hydration.

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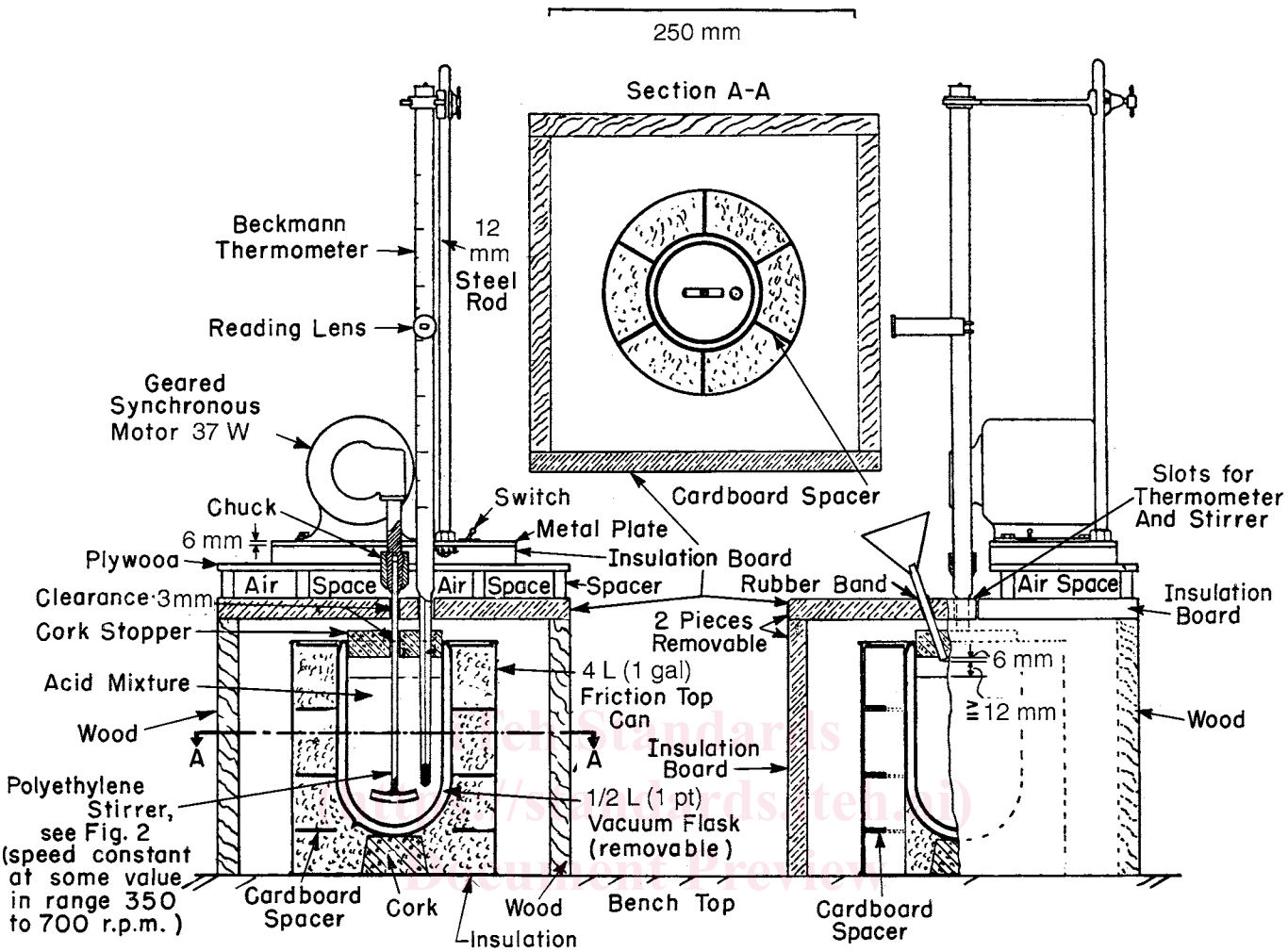
[2] *Annual Book of ASTM Standards*, Vol 04.01.

[3] *Annual Book of ASTM Standards*, Vol 04.02.

[4] *Annual Book of ASTM Standards*, Vol 14.02.



Metric Units	Inch-Pound Equivalents	Metric Units	Inch-Pound Equivalents
250 mm	10 in.	6 mm	1/4 in.
12 mm	1/2 in.	3 mm	1/8 in.
37 W	1/20 hp	0.47 litre	1 pt

FIG. 1 Calorimeter

the upper limit of the scale approximates room temperature. The portion of the thermometer that will rest inside the calorimeter shall be protected with a coating resistant to hydrofluoric acid (see 4.1.1). The thermometer shall be equipped with a suitable reading lens. The Beckmann thermometer zero must be determined by immersion in a liquid and comparison with the reference thermometer described. An accurate reference thermometer of the appropriate range and having 0.1°C divisions shall be placed in the proximity of the calorimetric apparatus and shall be used for room temperature readings and for establishing the Beckmann thermometer zero.

4.1.4 *Funnel*—The funnel through which the sample is introduced into the calorimeter shall be of glass or plastic and shall have a stem approximately 75 mm (3 in.) in length and with an inside diameter of not less than 6 mm (¼ in.).

4.1.5 *Stirring Assembly*—The stirrer shall be a three-bladed polyethylene propeller having the dimensions shown in Fig. 2, and shall extend as closely as possible to the bottom of the calorimeter. The motor shall be of the constant-speed type, at least 37 W (1/20 hp), and shall be equipped with a geared speed reducer so that one speed, in the range of 350 to 700 r/min, can be maintained constant.

NOTE 2—The stirrer shown in Fig. 2 may be readily made from a commercially available three-bladed polyethylene propeller having a propeller diameter of 34 mm (1⅜ in.), shaft diameter of 6 mm (¼ in.), and a shaft length of approximately 455 mm (18 in.). The function of the stirrer is two-fold: to maintain uniform temperature throughout the liquid and to supply sufficient agitation to keep the solid in suspension in the acid mixture. Since a stirrer capable of keeping the solid in suspension generates considerable heat in the calorimeter, it is important that the

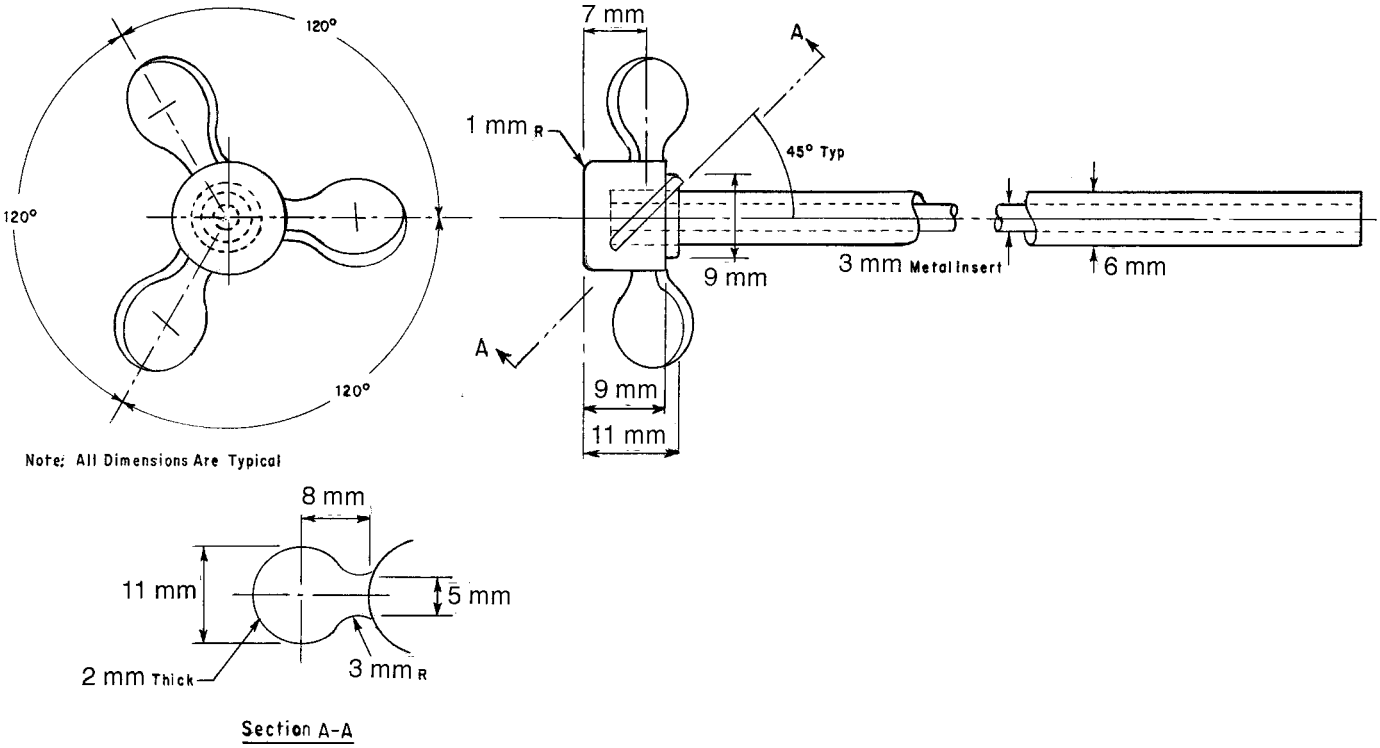


FIG. 2 Stirrer

stirrer speed, and hence the rate of heat generation, be maintained constant. Because such constancy is difficult to achieve with other types of motors, a synchronous motor with a geared speed reducer is recommended.

4.2 *Mixer*—A moderate-speed mechanical mixer, such as a milk-shake type stirrer, capable of intimately mixing the cement and water to a uniform paste.

4.3 *Storage*—Storage space with temperature controlled at 23.0 ± 2.0°C (73.5 ± 3.5°F).

4.4 *Mortar*, approximately 200 mm (8 in.) in diameter, and pestle for grinding the partially hydrated samples.

4.5 *Plastic Vials*, approximately 80 by 25-mm (3⅝₃₂ by 1-in.), shell-type, with tight-fitting stoppers or caps.

4.6 *Drying Oven*, maintained at 100 to 110°C.

4.7 *Weighing Bottles*, approximately 40 mm high and 25 mm wide, with matching stoppers.

4.8 *Stop Watch or Clock Timer*.

4.9 *Sieves*, 150-µm (No. 100) and 850-µm (No. 20), conforming to Specification E 11.

4.10 *Crucibles*, platinum, 30-mL capacity, with covers, for loss on ignition determination.

4.11 *Muffle Furnace*, or suitable burners capable of maintaining a temperature of 900 to 950°C.

4.12 *Analytical Balance and Analytical Weights*, conforming to the requirements prescribed in Test Methods C 114 for weighing out calorimetric samples and for loss on ignition weighings.

4.13 *Weights and Weighing Devices*, conforming to the requirements of Specification C 1005. The weighing device shall be evaluated at a total load of 1000 g.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Hydrofluoric Acid (sp gr 1.15)*—Concentrated hydrofluoric acid (HF).

5.3 *Nitric Acid (2.00 N)*—The 2.00 *N* HNO_3, for use in the calorimeter, shall be prepared and standardized in large quantities. Optionally, the dilute HNO_3 may be made up with 127 mL of concentrated HNO_3(sp gr 1.42) per litre of solution, provided that heat capacity determinations are made with each batch of diluted HNO_3 so prepared.

5.4 *Wax*—Paraffin wax, or other suitable wax, for sealing vials.

5.5 *Zinc Oxide (ZnO)*—The ZnO shall be heated at 900 to 950°C for 1 h, then cooled in a desiccator, ground to pass a 150-µm (No. 100) sieve, and stored. Immediately prior to a heat capacity determination, 7 g of the ZnO so prepared shall be heated for not more than 5 min at 900 to 950°C, cooled to

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.