



Standard Test Method for Heat of Hydration of Hydraulic Cement¹

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 Cement²

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials³

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

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁴

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System⁴

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. Current edition approved Jan. 10, 1998. Published July 1998. Originally published as C 186 – 44 T. Last previous edition C 186 – 97.

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.

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 bees-wax. 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 the upper limit of the scale approximates room temperature.

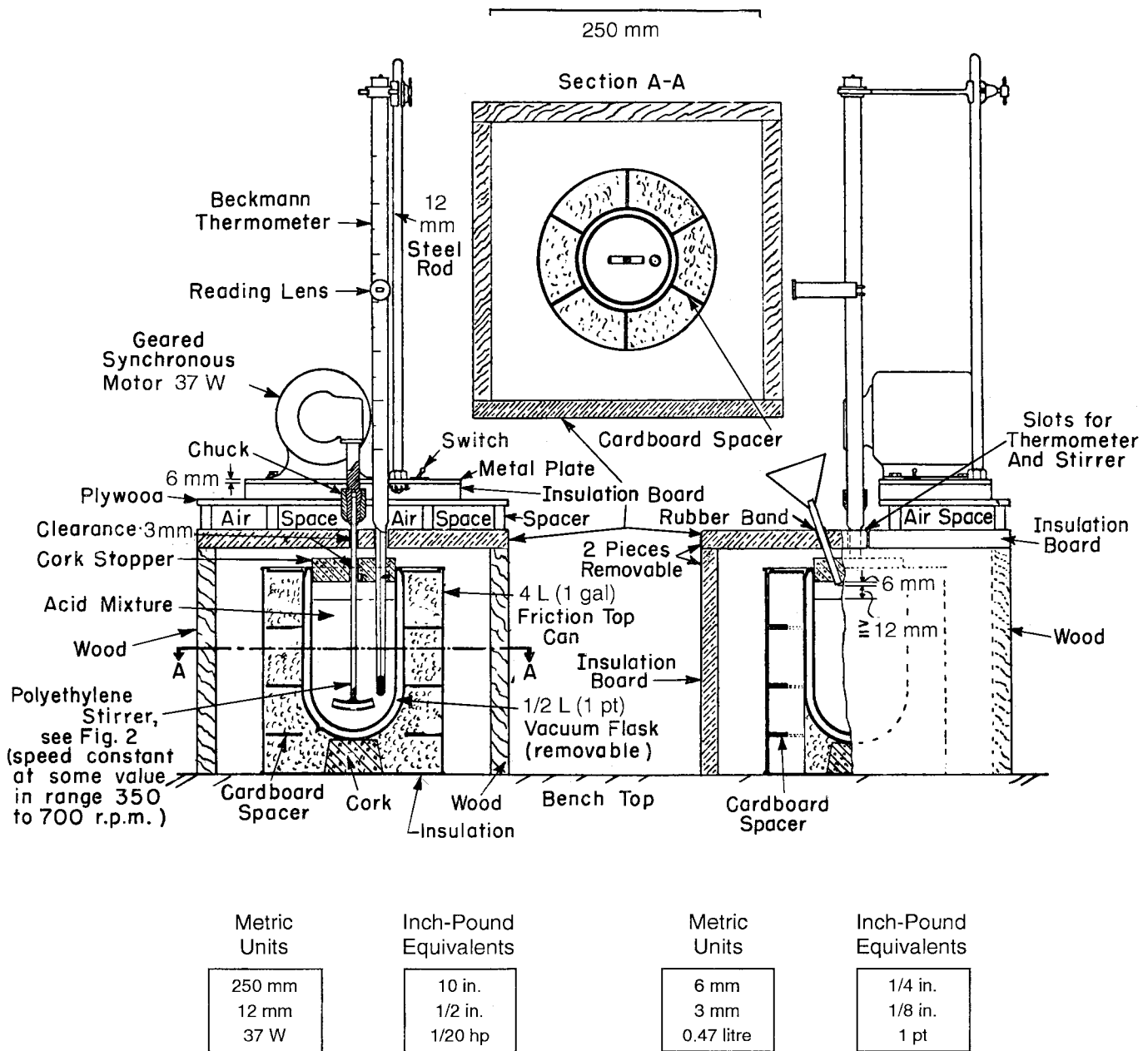


FIG. 1 Calorimeter

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 (1/4 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 (1 3/8 in.), shaft diameter of 6 mm (1/4 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 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.

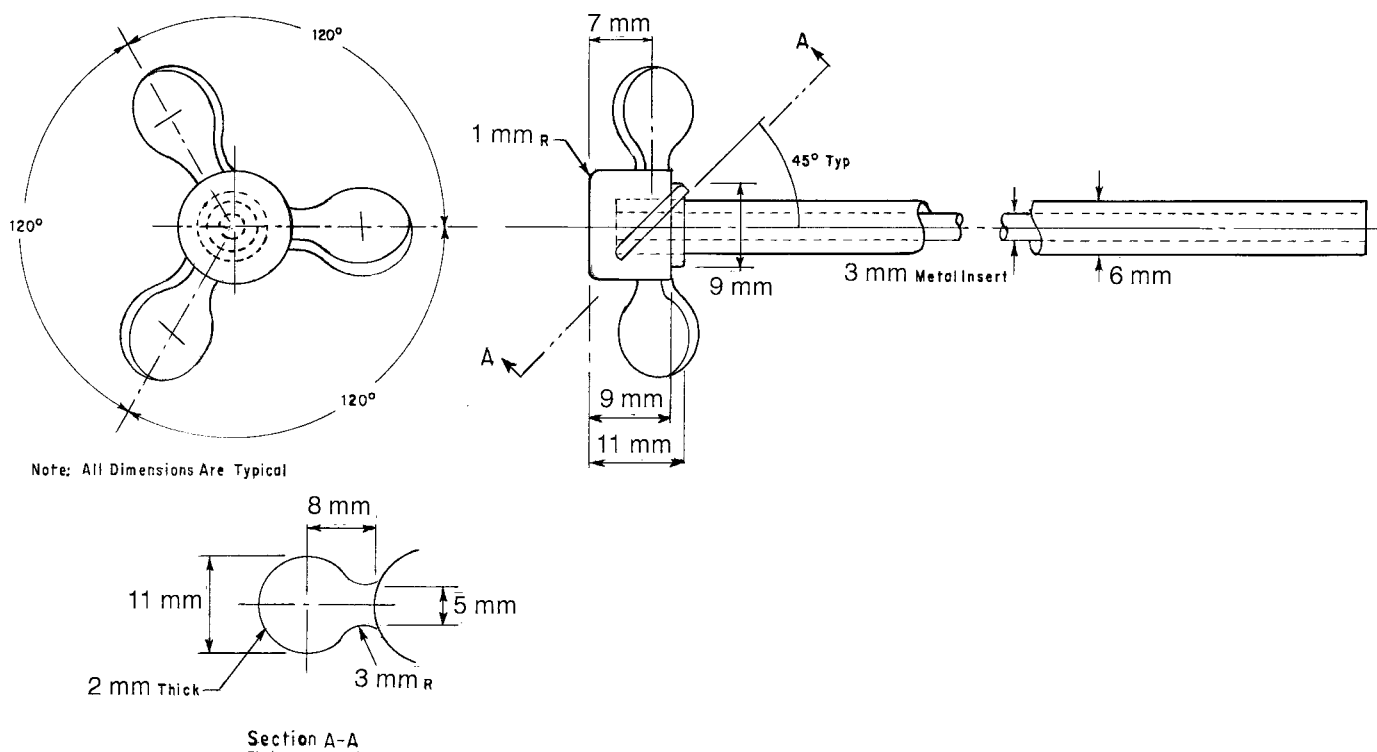


FIG. 2 Stirrer

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 $23.0 \pm 2.0^\circ\text{C}$ ($73.5 \pm 3.5^\circ\text{F}$).

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 ($3\frac{5}{32}$ 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 room temperature in a desiccator, and weighed accurately for introduction into the calorimeter.

NOTE 3—The rate of solution of the ZnO varies with the preliminary treatment. The procedure described results in a product which dissolves at about the same rate as the dry cement.

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 Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

6. Determination of Heat Capacity of Apparatus

6.1 To determine the heat capacity of the system (that is, the number of joules or calories required to raise the temperature of the calorimeter and contents 1°C), measure the corrected temperature rise obtained by dissolving 7 g of ignited ZnO in the specified acid mixture (see 6.2-6.7).

6.2 Transfer approximately 400 g of the 2.00 N HNO₃, which has been cooled to the temperature indicated by the lower range of the Beckmann thermometer (ordinarily about 4 to 5°C below room temperature), into the vacuum jar, add 8.0 mL of HF (sp gr 1.15), weigh, and add sufficient additional 2.00 N HNO₃ to bring the total weight of the solution to 425.0 g. Then, assemble the calorimeter and start the stirring motor. Take care that the stirrer blades or shaft do not touch the thermometer, the sides or bottom of the jar, or the cork stopper. The lower end of the funnel stem shall extend approximately 6 mm (¼ in.) below the lower surface of the stopper and at least 12 mm (½ in.) above the level of the liquid. The upper end of the bulb of the Beckmann thermometer shall be at least 38 mm (1½ in.) below the surface of the liquid. Place it at the same depth in all determinations. After an initial stirring period of at least 20 min to allow the temperature of the system to become uniform, record the temperature of the room to the nearest 0.1°C, the temperature of the acid to the nearest 0.001°C, record the time, and then immediately introduce the prepared ZnO through the funnel at a uniform rate (see Note 4). Complete the introduction of the ZnO in not less than 1 or more than 2 min. Brush any ZnO clinging to the funnel stem into the acid mixture by means of a small “camel’s-hair” brush.

NOTE 4—The temperature of the sample shall be identical with that of the room when the sample is introduced into the calorimeter.

6.3 Read the temperature, to the nearest 0.001°C, at 20 min and again at 40 min after beginning the introduction of the sample. The first 20-min period is the uncorrected temperature rise, which covers the solution period. The second 20-min period is the rating period, and the temperature difference between the 20 and 40-min readings is the correction to be added to or subtracted from the uncorrected temperature rise, according to whether the calorimeter temperature rises or falls during the rating period.

6.4 Calculate the corrected temperature rise as follows:

$$\begin{aligned} R_o &= \theta_{20} - \theta_0 \\ R &= R_o - (\theta_{40} - \theta_{20}) \end{aligned} \quad (1)$$

where:

- R_o = observed temperature rise, °C,
- θ_{20} = calorimeter temperature at the end of the solution period,
- θ_0 = calorimeter temperature when sample was introduced,
- R = corrected temperature rise, °C, and
- θ_{40} = calorimeter temperature at the end of the rating period.

6.5 Calculate the heat capacity of the calorimeter and contents as follows (see Note 5):

$$C = \frac{W[1072 + 0.4(30 - t) + 0.5(T - t)]}{R} \quad (2)$$

where:

- C = heat capacity, kJ/°C,
- W = mass of ZnO, g,
- t = final temperature of the calorimeter, °C (θ_{20} plus temperature, °C, at which the Beckmann thermometer reading is zero),
- T = temperature of the ZnO (room temperature), °C, when introduced into the calorimeter, and
- R = corrected temperature rise, °C.

NOTE 5—The heat of solution of ZnO is 1072 kJ/kg (256.1 cal/g) at 30°C. This value increases 0.4 kJ/kg (0.1 cal/g) for each degree decrease in temperature below 30°C. The heat capacity of ZnO is 0.5 kJ/kg-K (0.12 cal/g·°C). The heat required to bring the ZnO to the final temperature of the calorimeter must be included in the effective heat of solution.

6.6 If more than a trace of ZnO is found adhering to the tip of the funnel or to the stopper when the calorimeter is opened, reject the test.

6.7 Redetermine the heat capacity at the following times:

- 6.7.1 When the Beckmann thermometer is reset,
- 6.7.2 When a new coating is applied to thermometer, stirrer, or flask,
- 6.7.3 When a new thermometer, stirrer, or flask is put in service,
- 6.7.4 When a new batch of acid is used, and
- 6.7.5 At other times when, according to the judgment of the operator, the need is indicated.

7. Sampling and Test Specimens

7.1 *Preparation of Cement Paste*—Store the cement and the mixing water in a constant-temperature room at 23.0 ± 2.0 °C (73.5 ± 3.5°F) until the materials are at ambient temperature before preparation of the paste. Mix 150 g of cement and 60 mL of distilled water by means of a spatula, and then vigorously stir the mixture with a mechanical stirrer for 5 min. Place approximately equal representative portions of the paste in four or more plastic vials, filling the vials to within about 13 mm (½ in.) of the top. Immediately after filling the vials, close them with tight-fitting stoppers or caps. (If there is any doubt regarding the tightness of the seal, the sealed ends of the vials should be dipped in molten paraffin wax.) Store the vials in an upright position in a water bath at 23 ± 2.0°C until the time of test.

7.2 *Preparation of Partially Hydrated Sample for Heat of Solution Test*—At the specified age of test or age of interest, remove a vial of the partially hydrated sample from storage within the test time tolerances of Test Method C 109, and, during a 20-min initial stirring period of the calorimeter, break the plastic away from the sample and rapidly crush the entire sample with a mortar and pestle so that all the material will pass through a 850-µm (No. 20) sieve; then quickly place the sample in a well-stoppered weighing bottle. Take care, particularly with the 7-day partially hydrated sample, to expose the sample to the air as little as possible, and thus minimize the action of CO₂ or the loss of moisture from the sample.

8. Procedure

8.1 *Calorimetric Procedure, Dry Cement*—Determine the heat of solution of the dry cement sample according to the

procedure described for the heat capacity determination (Section 6), but use a 3-g sample (weighed to the nearest 0.001 g) of the dry cement instead of the prepared ZnO (see Note 4). (Exercise care in securing a uniform and representative sample.) Calculate and report the results on the ignited mass basis (8.3). Perform the heat of solution test on the dry cement just prior to the test on the corresponding 7-day partially hydrated sample.

8.2 Calorimetric Procedure, Partially Hydrated Sample—For the heat of solution of the partially hydrated sample follow the same procedure as for the dry cement described in 8.1, but use a 4.18 ± 0.05 -g calorimetric sample of the partially hydrated cement, weighed to the nearest 0.001 g (see Note 4). Calculate the results on the ignited basis.

8.3 Loss on Ignition:

8.3.1 Portland Cement—Immediately before and after the calorimetric sample is being weighed out, weigh a sample of similar amount into a platinum crucible for determination of loss on ignition, the value to be used being the average of the two determinations. Ignite the dry cement at $950 \pm 50^\circ\text{C}$ for at least 1½ h or to constant mass. Immediately place the crucible containing the sample in a desiccator and allow to cool to room temperature; then quickly weigh the crucible. When determining the loss on ignition of the hydrated cement, first dry the weighed sample in an oven at 100 to 110°C for 1 h; then place the sample in a muffle furnace at $950 \pm 50^\circ\text{C}$ overnight, or bring to constant mass. Reduce the mass of the cement sample that was introduced into the calorimeter to the ignited mass basis for use in the final calculations as follows:

$$W_i = (A/B)W \quad (3)$$

where:

- W_i = mass of calorimetric sample, on ignited basis, g,
- A = mass of ignited sample, g,
- B = mass of sample before ignition, g, and
- W = mass of calorimetric sample, g.

8.3.2 Blended Hydraulic Cements—In addition to the procedures described in 8.3.1, determine the loss on ignition by the reference method given in Test Methods C 114 for portland blast-furnace slag cement and slag cement.

8.3.2.1 Determine the SO₃ content by the reference method given in Test Methods C 114 (see Note 6). Also determine the SO₃ content of a portion of the same cement that has not been ignited, using the same procedure.

8.3.2.2 Calculate the percentage of mass gain from sulfide sulfur as follows:

$$G = 0.8(S_1 - S_2) \quad (4)$$

where:

- G = percent mass gain in ignited sample,
 - S_1 = SO₃ determined on ignited sample, and
 - S_2 = SO₃ determined on unignited sample.
- 0.8 = molecular weight ratio of 4(0)/SO₃

NOTE 6—Some of the acid used for dissolving the sample may first be warmed in the platinum crucible to dissolve any adhering material.

8.3.2.3 Calculate the mass of the dry calorimetric sample on the ignited basis as follows:

$$W_i = \frac{\left(A - \frac{BG}{100}\right)W}{B} \quad (5)$$

where:

- W_i = mass of dry calorimetric sample, on ignited basis, g,
- A = mass of ignited dry sample, g,
- B = mass of dry sample before ignition, g,
- G = percentage mass gain from sulfide sulfur, and
- W = mass of dry calorimetric sample, g.

Calculate the mass of the partially hydrated calorimetric sample on the ignited basis as follows: (Note 7)

$$W_i = \frac{A W \left(1 - \frac{G}{100}\right)}{B} \quad (6)$$

where:

- W_i = mass of calorimetric sample, on ignited basis, g,
- A = mass of partially hydrated sample after ignition, g,
- B = mass of partially hydrated sample before ignition, g,
- G = percentage mass gain from sulfide sulfur, and
- W = mass of partially hydrated calorimetric sample, g.

NOTE 7—An assumption is made in the calculation that the same percentage of sulfide sulfur is present prior to ignition in the partially hydrated sample as was determined in the cement. Tests have confirmed that the assumption is reasonably correct and will not alter the precision of the test method.

9. Calculation

9.1 Heat of Solution of Dry Cement—Calculate the corrected temperature rise as described in 6.3 and 6.4. Also, correct the heat of solution value if the final calorimeter temperature of the heat of solution test is different from the temperature of the calorimetric sample when introduced. Thus, for the dry cement, which has a specific heat of approximately 0.8 kJ/kg·K (0.2 cal/g·°C), if the final calorimeter temperature exceeds the temperature of the cement sample at the time it was introduced, add a correction of 0.8 kJ/kg·K (0.2 cal/g·°C) difference in those temperatures when calculating the heat of solution. Calculate the heat of solution of the dry cement as follows:

$$H_1 = (RC/W_i) - 0.8(T - t_d) \quad (7)$$

where:

- H_1 = heat of solution of dry cement, kJ/kg,
- R = corrected temperature rise, °C,
- C = heat capacity, kJ/°C,
- W_i = mass of sample on ignited basis, g,
- T = room temperature, when sample is introduced, °C, and
- t_d = final calorimeter temperature at end of determination on dry cement, °C.

9.2 Heat of Solution of Partially Hydrated Sample—Calculate the heat of solution of the partially hydrated sample in the same way as for the dry cement (9.1), except make additional corrections, as follows:

9.2.1 Since an increase of 1°C in the temperature at which the heat of solution test occurs causes a decrease of approximately 1.3 kJ/kg (0.3 cal/g) in the heat of solution, if the temperature of the heat of solution test of the partially hydrated

sample exceeds the temperature of the dry cement determination, a correction of 1.3 kJ/kg·K (°C) difference in temperature shall be added to the heat of solution value obtained for the partially hydrated sample.

9.2.2 Also, correct the heat of solution value if the final calorimeter temperature of the solution test is different from the temperature of the calorimetric sample when introduced. Thus, for the partially hydrated sample, which has a specific heat of approximately 1.7 kJ/kg (0.4 cal/g) of ignited cement, if the final calorimeter temperature exceeds the temperature of the sample at the time it was introduced, add a correction of 1.7 kJ/kg·K (°C) difference in those temperatures when calculating the heat of solution.

9.2.3 Calculate the heat of solution of the partially hydrated sample as follows:

$$H_2 = (RC/W_i) - 1.7(T - t_h) - 1.3(t_d - t_h) \quad (8)$$

where:

H_2 = heat of solution of partially hydrated sample, kJ/kg,

R , C , W_i , and T = the same definition as in 9.1 except that they relate to the partially hydrated sample,

t_d = the same numerical value as in 9.1, and

t_h = final calorimeter temperature at end of determination on partially hydrated sample, °C.

9.3 *Heat of Hydration*—A final calorimeter temperature of 25°C shall be considered as the basis to which the heat of hydration shall be referred, and the effects of variation in that temperature should be kept in mind when considering test results. An increase in the final temperature raises the heat of hydration approximately 0.4 kJ/kg·K (0.1 cal/g·°C) of ignited cement. For example, if the final temperature is 27°C, 0.8 kJ/kg (0.2 cal/g) should be subtracted from the observed heat of hydration in order to refer the results to 25°C. In borderline cases, proper correction should be made for the effects of final calorimeter temperature. Calculate the heat of hydration of the cement to the nearest kilojoule, as follows:

$$H = H_1 - H_2 - 0.4(t_h - 25.0) \quad (9)$$

where:

H = heat of hydration of ignited cement, kJ/kg,

H_1 = heat of solution of dry cement (9.1),

H_2 = heat of solution of partially hydrated sample (9.2),
and

t_h = the same numerical value as in 9.2.3

NOTE 8—To convert cal/g to kJ/kg multiply by 4.184 in accordance with Standard IEEE/ASTM SI 10.

10. Retests

10.1 In case of failure to meet the 28-day requirement for heat of hydration, a reserve sample of paste may be tested at a later age and a correction of 2.1 kJ/kg (0.5 cal/g) per day of excess age added to bring the retested heat of solution to a 28-day basis. The period over which this correction may be made shall be limited to 4 days. In case of failure to meet the 7-day requirement, a complete retest including mixing of the paste should be made.

11. Report

11.1 Report the following information:

11.1.1 Sample identification, which may include the source and type of hydraulic cement and sampling date, and

11.1.2 The heat of hydration results at each of the test ages required by the applicable specification.

12. Precision and Bias

12.1 *Precision*:

12.1.1 *Single-Operator Precision*—The single-operator standard deviations have been found to be 12.2 kJ/kg(1s) (2.91 cal/g)(1s) and 14.8 kJ/kg(1s) (3.54 cal/g)(1s) for the determinations of heat of solution and heat of hydration, respectively. Therefore, results of two properly conducted tests by the same operator on samples of the same cement should not differ from each other by more than 34 kJ/kg (8 cal/g) in the determination of heat of solution or 42 kJ/kg (10 cal/g) in the determination of heat of hydration.⁶

12.1.2 *Multilaboratory Precision*—The multilaboratory standard deviations have been found to be 18.5 kJ/kg(1s) (4.42 cal/g)(1s) and 16.9 kJ/kg(1s) (4.03 cal/g)(1s) for the determinations of heat of solution and heat of hydration respectively. Therefore, results of two properly conducted tests from two different laboratories on samples of the same cement should not differ from each other by more than 52 kJ/kg (13 cal/g) in the determination of heat of solution or 48 kJ/kg (11 cal/g) in the determination of heat of hydration.⁶

12.2 *Bias*—Since there is no accepted reference material, no statement on bias is being made.

13. Keywords

13.1 blended cement; heat of hydration; heat of solution; hydraulic cements; portland cement

⁶ These numbers represent, respectively the (1s) and (d2s) limits as described in Practice C 670.

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