



Designation: C1897 – 20

Standard Test Methods for Measuring the Reactivity of Supplementary Cementitious Materials by Isothermal Calorimetry and Bound Water Measurements¹

This standard is issued under the fixed designation C1897; 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 These two alternative test methods are used to assess the chemical reactivity of a supplementary cementitious material (SCM) as determined by measurements of cumulative heat release or bound water content of hydrated pastes composed of the SCM, calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide cured at 40 °C for 3 and 7 days.

1.1.1 These two test methods do not distinguish between hydraulic and pozzolanic reactivity.

1.2 The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.3 The text of the standard refers to notes and footnotes that provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of this standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. (Warning—Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.)*²

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ These test methods are under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.24 on Supplementary Cementitious Materials.

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² Section on Safety Precautions, Manual of Aggregate and Concrete Testing, Annual Book of ASTM Standards, Vol. 04.02.

2. Referenced Documents

2.1 *ASTM Standards*:³

C114 Test Methods for Chemical Analysis of Hydraulic Cement

C125 Terminology Relating to Concrete and Concrete Aggregates

C311/C311M Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete

C618 Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete

C989/C989M Specification for Slag Cement for Use in Concrete and Mortars

C1240 Specification for Silica Fume Used in Cementitious Mixtures

C1702 Test Method for Measurement of Heat of Hydration of Hydraulic Cementitious Materials Using Isothermal Conduction Calorimetry

C1709 Guide for Evaluation of Alternative Supplementary Cementitious Materials (ASCM) for Use in Concrete

C1738/C1738M Practice for High-Shear Mixing of Hydraulic Cement Pastes

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 *Definitions*:

3.1.1 For definitions of terms used in these test methods, refer to Terminology C125.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *chemically bound water, n*—water in hardened cement paste that has reacted and is part of the structure of hydrated reaction products.

3.2.1.1 *Discussion*—In these test methods, bound water is taken as the mass loss when a paste specimen dried previously at 40 °C is heated in a furnace to 350 °C. Some raw natural

³ 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.

pozzolans may contain bound water and lose mass over this temperature range. This mass loss needs to be determined and used to correct the bound water value for the paste.

4. Summary of Test Method

4.1 *Method A*—Isothermal calorimetry is used to determine the heat of hydration of hydrating pastes composed of the SCM, calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide. The heat of hydration value is used to determine the chemical reactivity of the SCM.

4.2 *Method B*—Chemically bound water of pastes composed of the SCM, calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide is determined as a measure of the chemical reactivity of the SCM.

5. Significance and Use

5.1 These test methods are used to assess the chemical (pozzolanic or hydraulic) reactivity of SCMs over a curing time of 7 days. The results of these test methods can be used to estimate the potential contribution of a SCM to the development of strength, or other properties such as lower permeability, when used with portland cement. However, the test results are not a substitute for direct measurement of the same properties of concrete made with that SCM.

5.2 The calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide are combined in proportions to provide a paste where the dissolved ions from these components simulate the pore solution in a portland cement system.

5.3 The pastes are cured at 40 °C to accelerate the rate of reaction of slowly reactive SCMs.

5.4 These test methods allow for the direct measurement of the hydraulic or pozzolanic reactivity of a potential SCM. These test methods are also suitable for screening purposes in the development and research of SCMs for use in portland cement-based systems. Furthermore, these test methods may be used in manufacturing control of portland cement-based products for assessing the hydraulic or pozzolanic reactivity of a SCM component.

5.5 These test methods are based on the work by Avet et al.⁴ and are a result of the work of RILEM Technical Committee 267 – Tests for Reactivity of Supplementary Cementitious Materials.⁵ The test methods are based on established correlations between strength development and evolution of heat and binding of water for SCMs covered by Specifications C618, C989/C989M, and C1240, and by Guide C1709. For other alternative SCMs, the validity of such correlations has not been established.

5.6 There is no requirement to use Method A and Method B for a given application. In many instances the choice is based

on the user's determination of available equipment. Method A can also provide an indication of rate of reactivity because measurements are taken continuously during the test period, while Method B provides the level of reactivity up to a single point in time.

6. Apparatus

6.1 *Apparatus for Mixing Pastes:*

6.1.1 *A high-shear blender*, capable of maintaining a no-load speed of at least 1600 r/min.

6.2 *Apparatus for Method A (Cumulative Heat Release):*

6.2.1 *Isothermal calorimeter*, conforming to Test Method C1702 and calibrated for measurement at 40 °C.

6.2.2 *Analytical Balance*—The balance shall have a capacity of 160 g, a repeatability within 0.002 g, and a readability of 0.001 g or better.

6.2.3 *Calorimeter-specific specimen containers and lids* that can be sealed air-tight.

6.3 *Apparatus for Method B (Bound Water Content):*

6.3.1 *Ventilated oven*, capable of maintaining temperatures of 40 ± 2 °C.

6.3.2 *Furnace*, capable of maintaining temperature at 350 ± 10 °C.

6.3.3 *Porcelain crucibles*, complying with the crucibles for measuring loss on ignition in Test Methods C311/C311M.

6.3.4 *Glass petri dishes*, of at least 50 mm in diameter.

6.3.5 *Analytical Balance*—The balance shall have a capacity of 160 g, a repeatability within 0.002 g, and a readability of 0.001 g or better.

6.3.6 *Mortar and pestle, disk pulverizer, rotary mill, or crusher*, capable of decreasing the size of the paste particles to meet the size requirements given in 9.3.3.2.

6.3.7 *Desiccator*, with fresh desiccant, which can be silica gel or an alternative desiccant capable of maintaining a relative humidity below 10 % at room temperature.

6.3.8 *ASTM E11 Sieve*, either 2 mm (No. 10) or 2.38 mm (No. 8).

6.3.9 *High-density polyethylene (or polycarbonate) cylindrical specimen containers with air-tight caps*. The specimen container volume shall not exceed three times the volume of the 10 to 15 mL paste specimen.

7. Reagents and Materials

7.1 *Purity of Reagents*—Chemicals shall be reagent grade and, unless otherwise indicated, shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,⁶ if such specifications are available.

7.1.1 Calcium hydroxide, (Note 1);

7.1.2 Potassium hydroxide, which shall be dry (Note 2);

7.1.3 Potassium sulfate;

⁴ Avet, F., Snellings, R., Ben Haha, M., Alujas, A., and Scrivener, K., "Development of a new rapid, relevant and reliable (R3) test method to evaluate the pozzolanic reactivity of calcined kaolinitic clays," *Cement and Concrete Research*, Vol. 85, 2016, pp. 1–11.

⁵ Li, X., Snellings, R., Antoni, M. et al. (31 more authors), "Reactivity tests for supplementary cementitious materials: RILEM TC 267-TRM phase 1," *Materials and Structures*, Vol 51, No. 6, 2018, pp. 151

⁶ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

7.1.4 Calcium carbonate;

7.1.5 Reagent water conforming to the requirements in Test Methods C114.

NOTE 1—The calcium hydroxide should be protected from exposure to carbon dioxide. Material remaining in an open container after a test should not be used for subsequent tests.

NOTE 2—Potassium hydroxide should be stored dry and protected from exposure to moisture or humidity. Potassium hydroxide from open containers should be dried for at least 8 h at 105 °C prior to use.

7.2 Parafilm tape for sealing containers.

8. Proportioning and Mixing Test Mixtures

8.1 Proportioning:

8.1.1 The ratio of SCM to calcium hydroxide is 1 to 3 by mass.

8.1.2 The ratio of SCM to calcium carbonate is 2 to 1 by mass.

8.1.3 Prepare a potassium solution by dissolving 4.00 g of potassium hydroxide and 20.0 g of potassium sulfate in 1.00 L of reagent water conditioned at 23 ± 3 °C.

8.1.4 The ratio of potassium solution to solids (sum of the SCM, the calcium hydroxide, and the calcium carbonate) is 1.2 by mass.

NOTE 3—Table 1 gives an example of the mixture proportions for an SCM paste conforming to this standard. The mass ratio of the SCM in the fresh paste is 10 g/99 g = 0.101. The required total mass to be mixed will depend on the type and number of tests to be performed.

9. Test Procedures

9.1 Paste Mixing:

9.1.1 Weigh the designated amounts of the dry SCM, calcium hydroxide, and calcium carbonate on weighing papers, then combine and mix until a homogeneous color is achieved. Mass measurements are to be accurate to ±0.01 g. Store the dry mixture in an air-tight container in a storage environment at 40 ± 2 °C until the temperature of the dry mixture is stabilized at 40 ± 2 °C.

NOTE 4—Mixing the dry mixture by hand with a spatula, passing through sieves multiple times, or use of a gyratory type mixer until a homogeneous color is attained, are methods that have been found to be satisfactory. Gentle mixing is often required to avoiding creating dust.

9.1.2 Place and seal the potassium solution in an air-tight container in a storage environment at 40 ± 2 °C until the solution is at 40 ± 2 °C.

9.1.3 Mix the dry mixture and the potassium solution at 1600 ± 50 r/min for 2 min using the high-shear blender so that a homogeneous paste is achieved. Alternatively, use the procedure for mixing pastes in Practice C1738/C1738M, where the dry mixture of solids is considered cementitious materials or cement and the potassium solution is considered to be mixing water. Record the time at start of mixing and use that as time zero.

TABLE 1 Example of Proportioning of Paste Mixture

	SCM	Ca(OH) ₂	CaCO ₃	Potassium Solution
Mass (grams)	10.00	30.00	5.00	54.00

9.2 Method A, Cumulative Heat Determination by Isothermal Calorimetry:

9.2.1 Preparation of the Apparatus:

9.2.1.1 The calorimeter shall be set at 40 ± 0.5 °C for at least 16 h before test.

9.2.1.2 Prior to mixing the pastes (9.1), place the calorimeter specimen containers, lids, and pipettes in a 40 ± 2 °C storage environment until they reach 40 ± 2 °C.

NOTE 5—If the isothermal calorimeter has multiple test chambers, and a sufficient volume of paste is prepared, more than one test specimen can be tested simultaneously. However, inserting new test specimens into the calorimeter while other test specimens are in test may create significant additional noise depending on the ambient temperature, the reactivity of the materials tested, and the specific design of the calorimeter. It is not recommended to insert new test specimens or otherwise open the calorimeter beyond 1 h after the mix time of the first test specimens inserted into the calorimeter.

9.2.1.3 Insert sealed, air-tight containers filled with 9.40 ± 0.05 g of deionized water into the reference channels of the calorimeter (Note 6).

NOTE 6—Because the total specimen mass to be placed in the calorimeter specimen chamber is instrument specific, to ensure that the thermal signal is within the measurement range of the instrument, the manufacturer’s recommendations for reference and test specimen masses should be followed.

9.2.2 Paste Specimen Preparation:

9.2.2.1 Determine the mass of the empty calorimeter specimen container to the nearest 0.01 g. Cast 15.0 g of the freshly mixed paste in the specimen container (Note 6). Record the mass (m_p) of the paste to the nearest 0.01 g.

9.2.2.2 When placing the paste into the calorimeter specimen container, avoid spilling paste on the opening and outer sides of the container.

9.2.3 Measurement of Heat Release:

9.2.3.1 Place each sealed, air-tight calorimeter specimen into the calorimeter measurement chambers within 10 min of the start of mixing and record the heat release at 40 ± 0.5 °C for 168 h (7 days) after mixing.

9.2.4 Calculations:

9.2.4.1 Calculate the cumulative heat release (H) from 75 min until 3 days (72 h ± 10 min) and 7 days (168 h ± 10 min) after start of mixing (time zero) by integration of the recorded heat release (see Note 5). Calculate the cumulative heat release in units of J/(g of SCM) using Eq 1:

$$H_{SCM} = \frac{H}{(m_p \times 0.101)} \quad (1)$$

where:

H = cumulative heat release (H) from 75 min until 3 days (72 h ± 10 min) and 7 days (168 h ± 10 min) after start of mixing,

m_p = the mass of the paste, and

0.101 = mass fraction of the SCM in the paste specimen

NOTE 7—Calculation of cumulative heat release is started at 75 min after mixing to allow enough time for the paste and container to equilibrate at 40 ± 0.5 °C inside the calorimeter.

9.3 Method B, Bound Water Content Determination:

9.3.1 For each paste specimen cast, perform the following: