Mix Performance Evaluation using the ASTM C1753 Thermal Testing Protocol

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Standard Practice for Evaluating Early Hydration of Hydraulic Cementitious Mixtures Using Thermal Measurements

1. Scope

1.1 This practice describes the apparatus and procedure for evaluating relative differences in early hydration of hydraulic cementitious mixtures such as paste, mortar, or concrete, including those containing chemical admixtures, various supplementary cementitious materials (SCMs), and other finely divided materials, by measuring the temperature history of a specimen.

1.2 Calorimetry is the measurement of heat lost or gained during a chemical reaction such as cement hydration; calorimetric measurements as a function of time can be used to describe and evaluate hydration and related early-age property development. Calorimetry may be performed under isothermal conditions (as described in Practice C1679) or under adiabatic or semi-adiabatic conditions. This practice cannot be described as calorimetry because no attempt is made to measure or compute the heat evolved from test specimens due to hydration, but it can in many cases be used for similar evaluations. Variables that should be considered in the application of this practice are discussed in the Appendix.

1.3 Units—The values stated in either SI units or inch-pound units shall be regarded separately as standard. The

2. Referenced Documents

2.1 ASTM Standards:

C39/C39M Test Method for Compressive Strength of Cylindrical Concrete Specimens
C125 Terminology Relating to Concrete and Concrete Aggregates
C172/C172M Practice for Sampling Freshly Mixed Concrete
C192/C192M Practice for Making and Curing Concrete Test Specimens in the Laboratory
C219 Terminology Relating to Hydraulic Cement
C305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency
C403/C403M Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance
C494/C494M Specification for Chemical Admixtures for Concrete
C1679 Practice for Measuring Hydration Kinetics of Hy-
About the ASTM C1753 Standard Practice

- Published fall, 2015: *Standard Practice for Evaluating Early Hydration of Hydraulic Cementitious Mixtures Using Thermal Measurements*
- Sometimes referred to as “semi-adiabatic calorimetry”
  - True calorimetry (isothermal or adiabatic) measures and quantifies heat release from hydration (more sophisticated)
  - Thermal testing produces similar *indications* of evolved heat via records of temperature changes in hydrating samples
- Most applications evaluate *relative* behavior of similar, compared mixtures
- Samples can be concrete, mortar, paste, or slurry
- Equipment needed (simple and inexpensive) can be manufactured or devised (homemade)
About the ASTM C1753 Standard Practice

One of very few ASTM standards with extensive guidance on use & applications

The Appendix is 12 of the 18 pages, with examples and recommendations for many applications.
Some equipment variations, manufactured and adapted
Holcim device: HolcimHeat™

HolcimHeat™ stands for: HolcimHeat-evolution-analysis-tool

HolcimHeat™ – application-based testing:

- Investigation of admixture incompatibility issues at cement and RMX plants
- Detection of cement reactivity issues (sulfate balance, reactivity, heat evolution, etc.)
- Study of workability and setting issues related to delayed or accelerated hydration reactions
- Enhanced technical services
Hydration and thermal profile key features

- Initial C₃A hydration
- Dormant period from interaction of CaSO₄ with C₃A
- “Main Peak” - C₃S hydration
Hydration and thermal profile time of set indications

Approximate timing of initial set of concrete

Main peak rise “M”

0.5 x M

0.2 x M

“50% fraction” indicator, a relative setting reference
Comparisons for concrete – time of set by thermal methods vs. C403

- Fractions of 21% and 42% have found to be good default values for using thermal testing to estimate C403 times (initial and final set)

Comparisons for concrete – time of set by thermal methods vs. C403

- New standard under development in ASTM C09.23
- Will be an application of C1573 for setting of concrete as an alternative to C403

Standard Test Method for Using Temperature as a Relative Indication of Time of Set of Cementitious Mixtures

1. Scope

1.1. This method is a quick method to get a relative comparison of time of set of cementitious materials under similar curing conditions by temperature measurements. This relative difference in time to achieve a fraction of the temperature rise is an indicator of relative time of setting. This method is not meant to match or replace the C403 method or any other penetration resistance method. It can be applied to compare a limited number of scenarios where the microstructural aspects remain relatively constant, namely when comparing different binder combinations and admixture dosage rates.

1.2. Units - The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
Kinetics of both *normal* and *abnormal* hydration are studied for evaluation of materials & proportions, etc.

**Normal hydration** –
Series of otherwise identical mixtures comparing admixtures and dosages for retardation effects, robustness of hydration

**Abnormal hydration** –
Series of mixtures with varying admix dosage or fly ash replacement driving varying stages of incompatibility (sulfate depletion) effects, with corresponding 1-day strengths
• Paste batching with two technicians:
  ▶ A batch every 4 to 6 minutes
  ▶ 48 or more batches in a morning
Examples / Applications
### Mix plan for a paste testing series

- **3 cements**
  - 1 C150 Type II
  - 2 C595 Type II
- **3 SCMs**
  - C618 Class C
  - C618 Class F
  - C989 slag cement
- **No SCM controls**
- **4 replacement rates**
- **42 mixtures total**
- **3 specimens each mix**
  - Strengths: 1, 7, 28 days
  - Thermal set
Screening of WR admixtures for a 30% Class C ash mix

5 different WR admixtures (2 Type A/D, 2 Type A/F, 1 MR) compared in paste mixtures, dosages selected for approx. 6% water reduction

“A/F 1” causes the least retardation, with good early strength influence.
Initial NCA dosage rate for a HVFA mixture

HVFA paste mixtures with incremental NCA dosages compared against target performance ranges established for traditional mixtures

Examples

50% F ash
w/cm = 0.32
910 mL/100kg A/F

1.30 L/100 kg NCA

0.65 L/100 kg NCA

No accelerator
Examples

Effects of incremental fly ash replacement, C vs. F ash

Class C ash, 0% to 35%
- 0%
- 10%
- 15%
- 20%
- 25%
- 30%
- 35%

Class F ash, 0% to 40%
- 0%
- 15%
- 20%
- 25%
- 30%
- 35%
- 40%

1-day strength

Paste strength, MPa

Hydration time, hours
Examples

Verification of sulfate balance issue via sulfate addition

Otherwise identical paste mixtures with incremental addition of calcium sulfate

Insert graph showing hydration time vs. temperature change with different SO₃ additions.
Comparison of 7 cements in an aggressive mix design

Examples

Otherwise identical paste mixtures comparing 7 cements, with 25% Class C fly ash, upper-limit dose of Type A/D WR, and 35°C (95°F) mix and cure temps
Temperature influences on incompatibility potential

4 identical paste mixtures compared at different initial mixture and curing temps (70°F (21°C) vs. 93°F (34°C))

A) 100% OPC, no WR, w’c = 0.45
B) 25% C ash, no WR w/cm = 0.45
C) 25% C ash, 4 oz/cwt WR, w/cm = 0.40
D) 25% C ash, 6 oz/cwt WR, w/cm = 0.40

Higher temps alone drive incompatible behavior in the mixtures with WRA
Comparison of effects of different gypsum sources on cements made from two different clinkers

Cement plant QC testing – effects of different gypsum sources, 32 degrees C mixtures with 25% Class C ash + 390 ml/100kg admix (upper) or 585 ml/100kg (lower)

Examples
Optimum SO$_3$ via ASTM C1702 on mill-ground cement samples cement with varying SO$_3$ contents

Examples
Published papers using (or about) thermal testing


- Cost, Tim, “Thermal Measurements of Hydrating Concrete Mixtures – A Useful Quality Control Tool for Concrete Producers,” NRMCA Publication 2PE004, National Ready Mixed Concrete Association, 900 Spring Street, Silver Spring, MD, August 2009.


- Cost, Tim, “Preliminary Optimization of Concrete Paving Mixtures for Sustainability and Performance,” 10th International Conference on Concrete Pavements, Quebec City, Quebec, July 8-12, 2012, 11 pp.


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Questions?

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