Mix Performance Evaluation using the ASTM C1753 Thermal Testing Protocol

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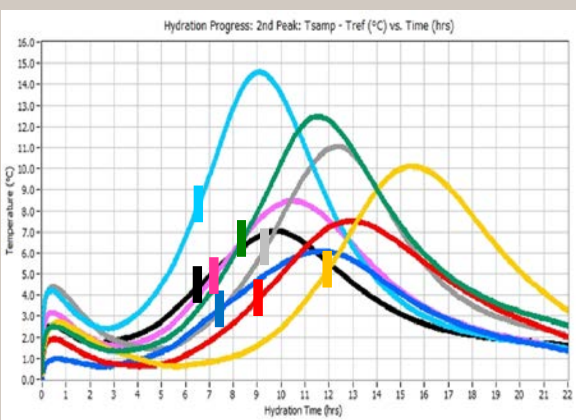
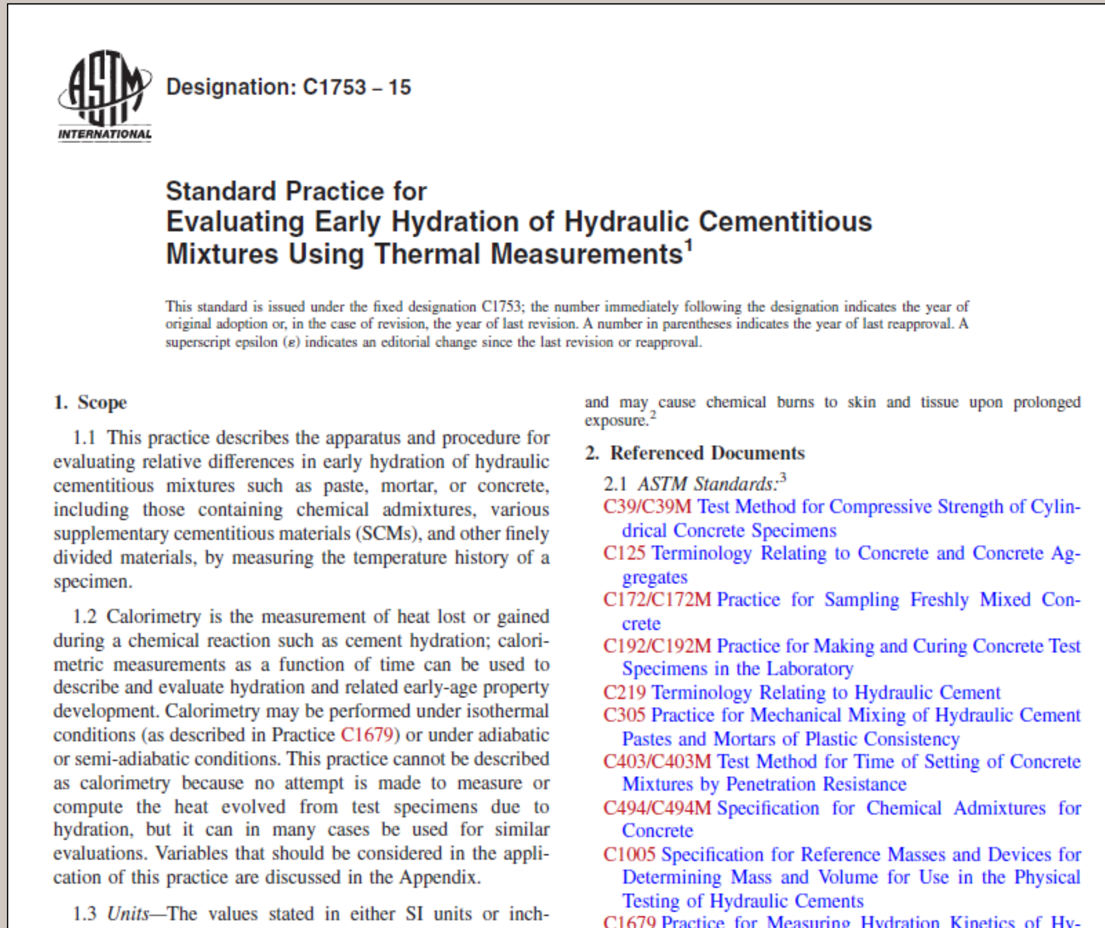
LafargeHolcim




Designation: C1753 – 15

INTERNATIONAL

Standard Practice for Evaluating Early Hydration of Hydraulic Cementitious Mixtures Using Thermal Measurements[1]

This standard is issued under the fixed designation C1753; 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 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

and may cause chemical burns to skin and tissue upon prolonged exposure.[2]

2. Referenced Documents

2.1 *ASTM Standards:*[3]
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
C1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements
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

TABLE X1.2 Mixture Proportions for Temperature Effects Example Mixtures (Fig. X1.8)

NOTE 1—1 mL/100 kg = 0.0154 oz/100 lb; 1 mL = 0.0338 oz.

Mixture description	cement, g	fly ash, g	w/cm	water, g	admix dosage rate	dose, mL	sand, g
No ash or admix	500		0.45	225			1360
25% ash, no admix	375	125	0.45	225			1360
25% ash, Type A dose	375	125	0.40	200	Type A, 260 mL/100 kg	1.30	1360
25% ash, Type D dose	375	125	0.40	200	Type D, 390 mL/100 kg	1.95	1360

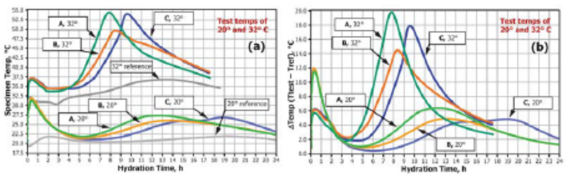
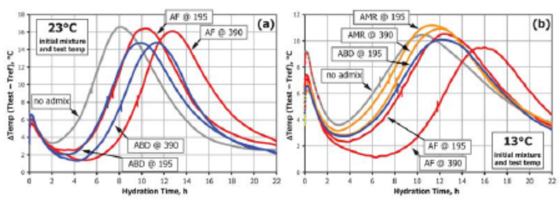


FIG. X1.9 Effects of Different Test Temperatures for Paste Specimens Mixed at 32 °C [90 °F] (a) Plotted without Subtraction of Reference Temperatures; (b) Same Data Plotted with Subtraction of Reference Temperatures



NOTE 1—See Table X1.3 for descriptions of admixtures; indicated dosage rates are mL/100 kg of cement.

FIG. X1.10 Effects of Admixtures and Dosages on Paste (a) at 23 °C [73 °F]; (b) at 13 °C [55 °F]

for the 13 °C [55 °C] test temperature and cooling of materials before mixing; the 23 °C [73 °C] test temperature condition was ambient room temperature. Descriptions and dosages of the admixture products are summarized in Table X1.3. Note that with this cement, the AF admixture appeared to result in more retardation at the higher dosage than even the ABD product, and also contributed more retarding influence at the lower temperature, even though it is marketed as a "set neutral" product. The AMR product included among the 13 °C [55 °C] mixtures shown in Fig. X1.10(b) showed little associated retardation or temperature sensitivity for the same dosages, which were on the lower end of the recommended range for each product.

X1.5.4.2 *Evaluation of Fly Ash Replacement Level*—Thermal measurement testing can be useful in evaluating SCM effects in a proposed mixture for a given set of materials and expected field temperatures, to aid in proportioning decisions. Fig. X1.11 shows thermal profiles from a series of laboratory paste mixtures (*w/cm* = 0.40) made and tested at the highest expected project field temperature of 34°C [93 °F], using a

TABLE X1.3 Admixtures and Dosages as Used in Example Test Series (Fig. X1.10)

NOTE 1—1 mL/100 kg = 0.0154 oz/100 lb.

Product ID in graphs	Manufacturer's product information			Test dosages used, mL/100 kg cement	
	water reduction categories	setting influences	recommended dosage range, mL/100 kg cement	low dose	high dose
AMR	Type A & mid-range	set neutral	195 to 980	195	390
AF	Type A, F, & mid-range	set neutral	195 to 780	195	390
ABD	Type A, B, & D	some retardation	195 to 460	195	390

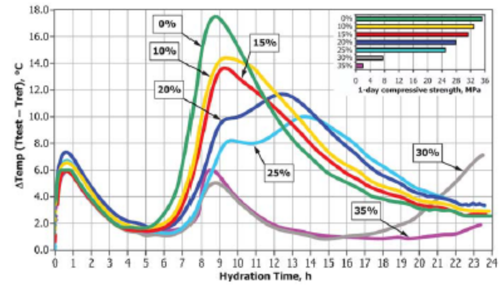


FIG. X1.11 Effects of Fly Ash Replacement Level in Paste Mixtures Made with a Type A/D Admixture at 34°C [93 °F] Initial Mixture and Test Temperature (Note: 1 MPa = 145 psi)

single sample of the Type II cement to be used and the highest expected dosage (390 mL/100 kg [6 oz/100 lb], based on total cementitious materials content) of a Type A/D admixture. Fly ash replacement level was incrementally increased from 0% to 35% using a single sample of Class C fly ash from the source planned for use in the project. Paste specimens were each about 350 g, tested in non-insulated 51 × 102 mm [2 × 4 in.] specimen containers. The fly ash replacement levels used for each mixture are indicated on the thermal profiles, and corresponding 1-day compressive strengths of the same specimens are shown in the inset bar chart. Note that sulfate imbalance begins to be indicated as fly ash replacement level is increased above 15%. Considering the possible effects of variability of other mixture components not included as test variables, it might be prudent to limit the project fly ash replacement level to around 15% with this set of materials and other proportions, if concrete temperatures in the field are to be this high.

X1.5.4.3 *Evaluation of Sulfate Imbalance Effects of Admixture Dosage*—The effects of possible sulfate imbalance resulting from higher admixture dosages can also be evaluated using thermal measurement testing. Fig. X1.12 shows thermal profiles of laboratory paste mixtures made and tested at 32 °C [90 °F] using a single sample of Type II cement, 25% replacement with a single sample of Class C fly ash, and dosages of a Type A/D admixture ranging from 0 to 520 mL/100 kg [0 to 8 oz/100 lb], based on total cementitious materials content. Paste specimens were each about 350 g [0.8 lb], tested in 51 × 102 mm [2 × 4 in.] non-insulated specimen containers. Admixture dosage is indicated for each thermal profile, and corresponding 1-day compressive strengths of the same specimens are shown in the inset bar chart. Note that effects of dosage increases up to 260 mL/100 kg [4 oz/100 lb] appear to result only in expected retardation, but sulfate imbalance indication is evident at the

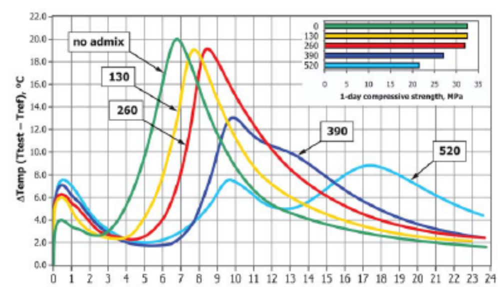
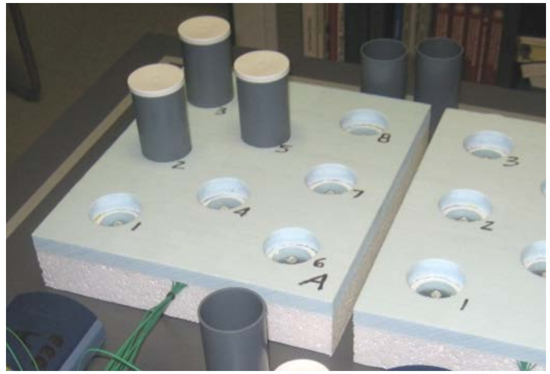
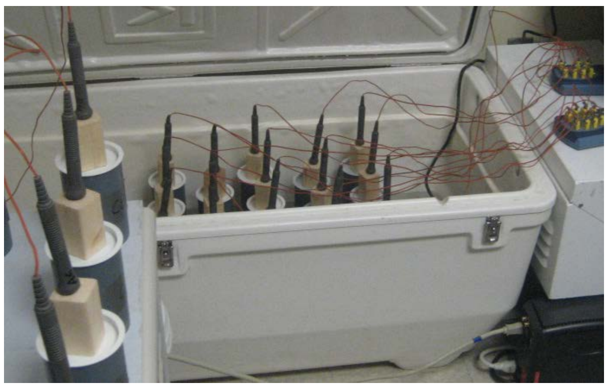
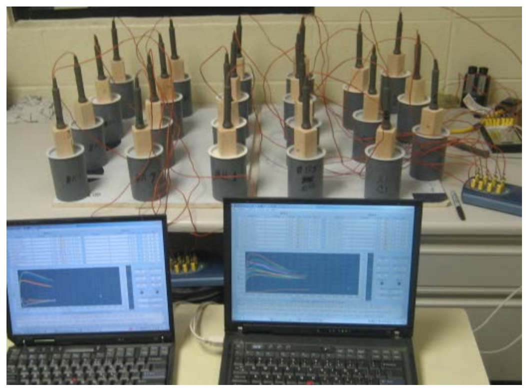


FIG. X1.12 Effects of Admixture Dosage (as Indicated, in mL/100 kg of Total Cementitious Materials) for 32°C Paste Mixtures Made with 25% Class C Fly Ash Replacement of Cement (Note: 1 mL/100 kg = 0.0154 oz/100 lb., 1 MPa = 145 psi)

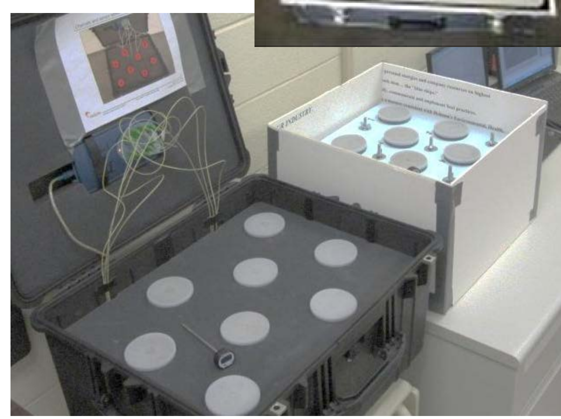
The Appendix is 12 of the 18 pages, with examples and recommendations for many applications.

Some equipment variations, manufactured and adapted





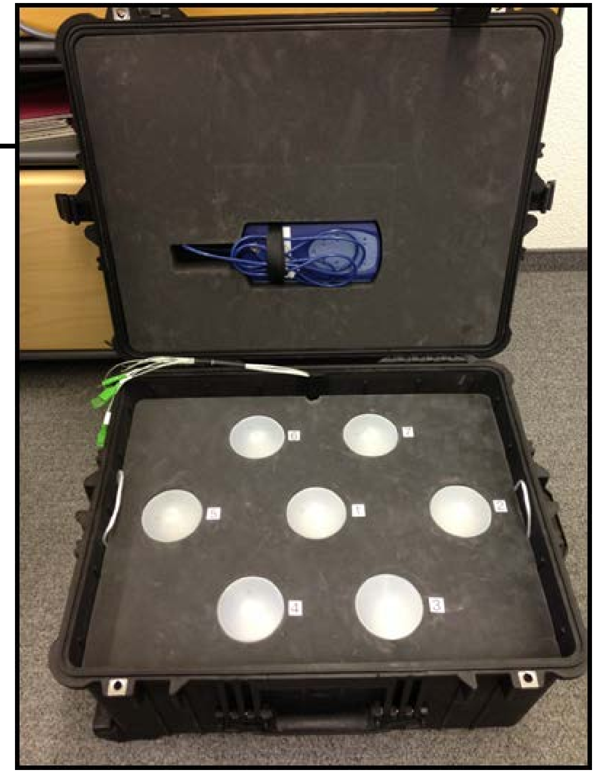
Holcim device: HolcimHeat™

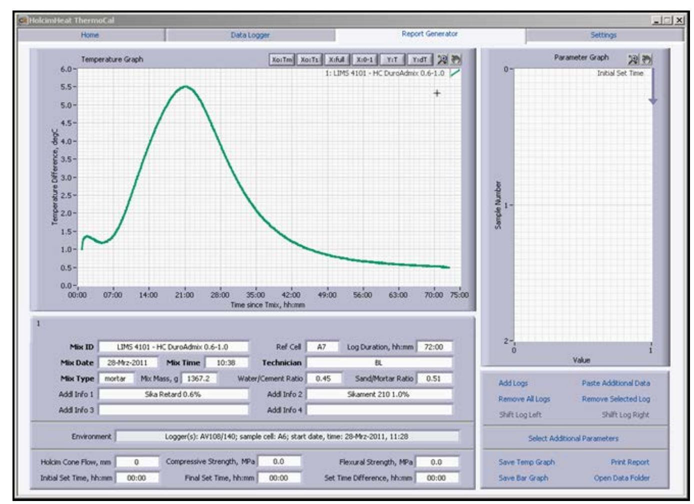
HolcimHeat™ stands for:

HolcimHeat-**e**volution-**a**nalysis-**t**ool

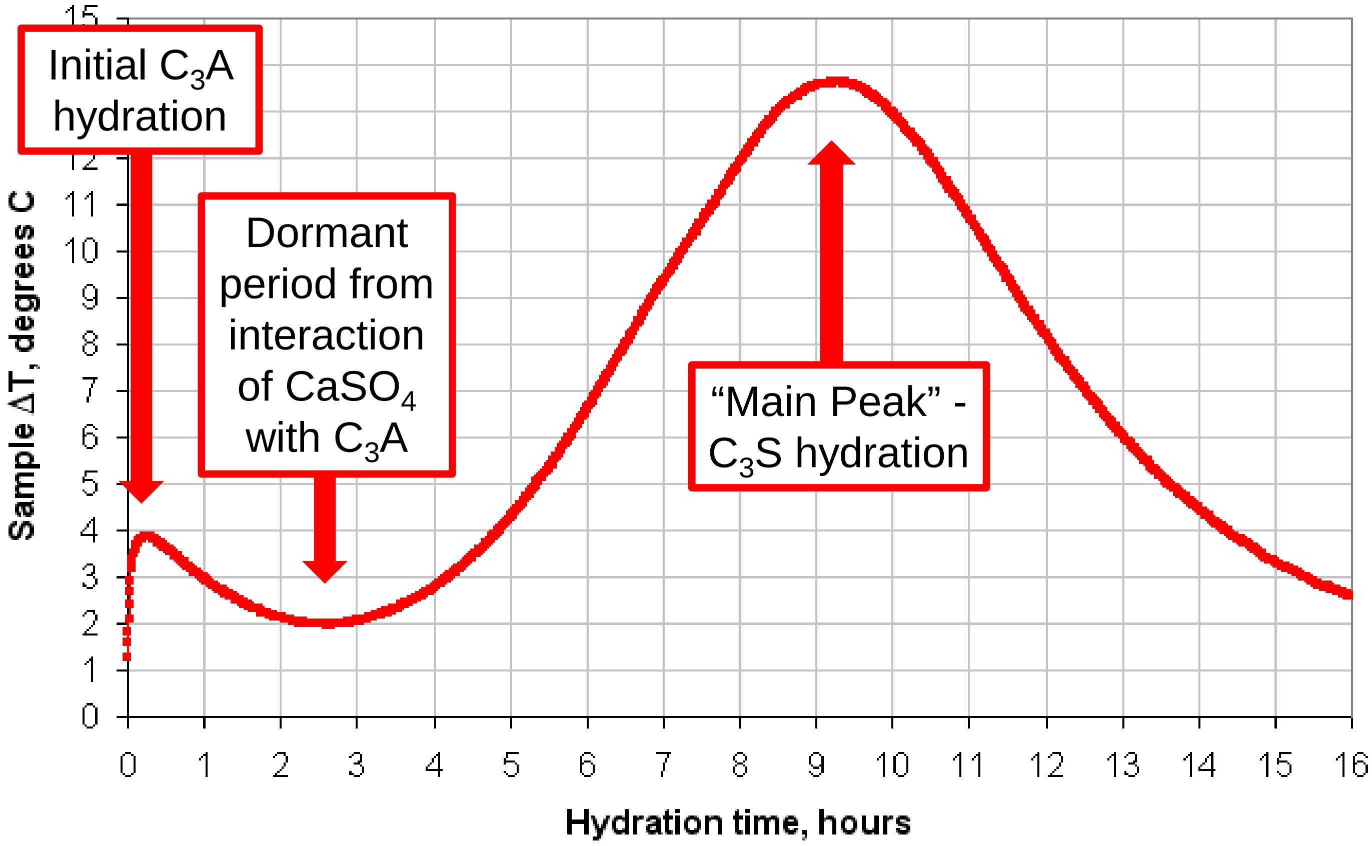
HolcimHeat™ – application-based testing:

- 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

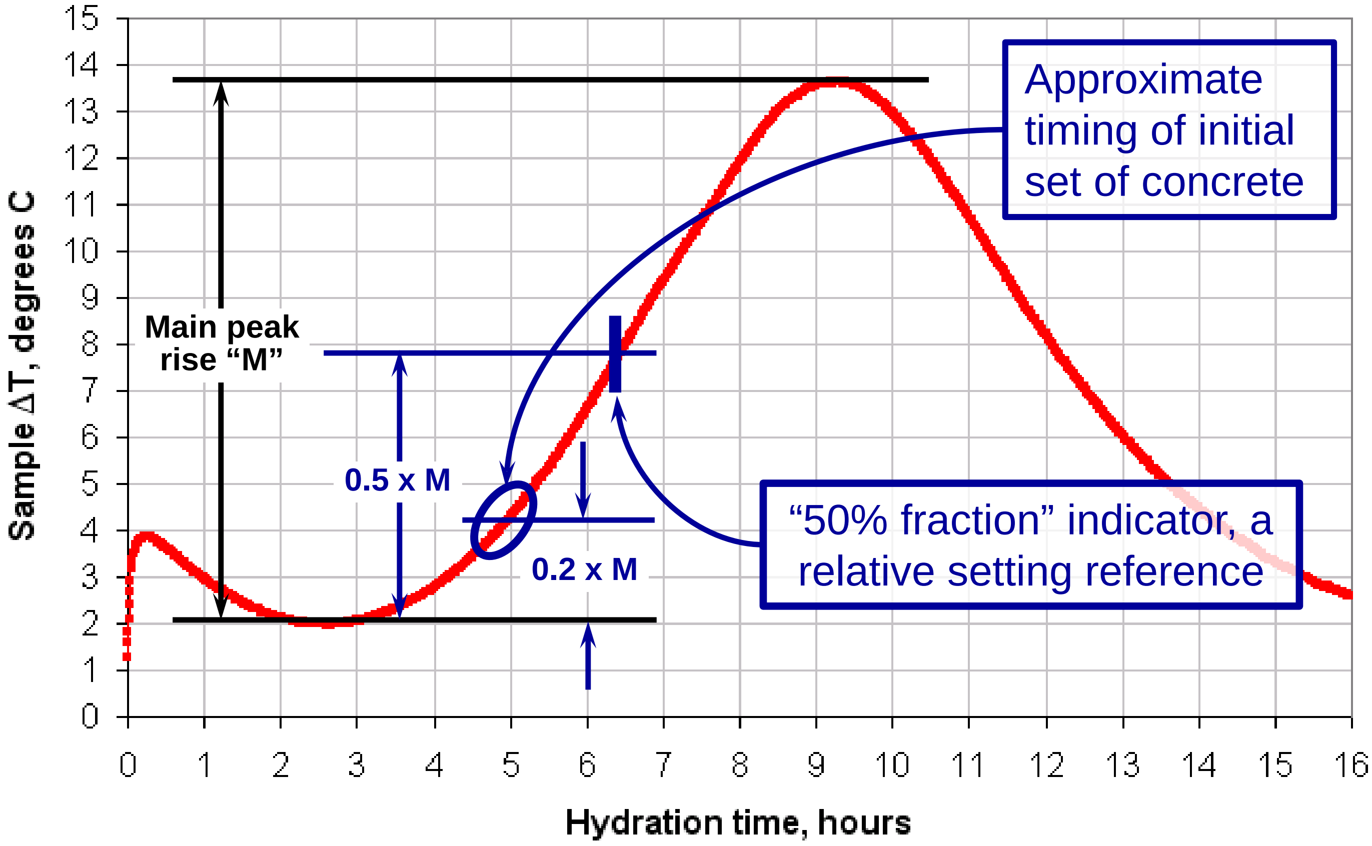




Hydration and thermal profile key features

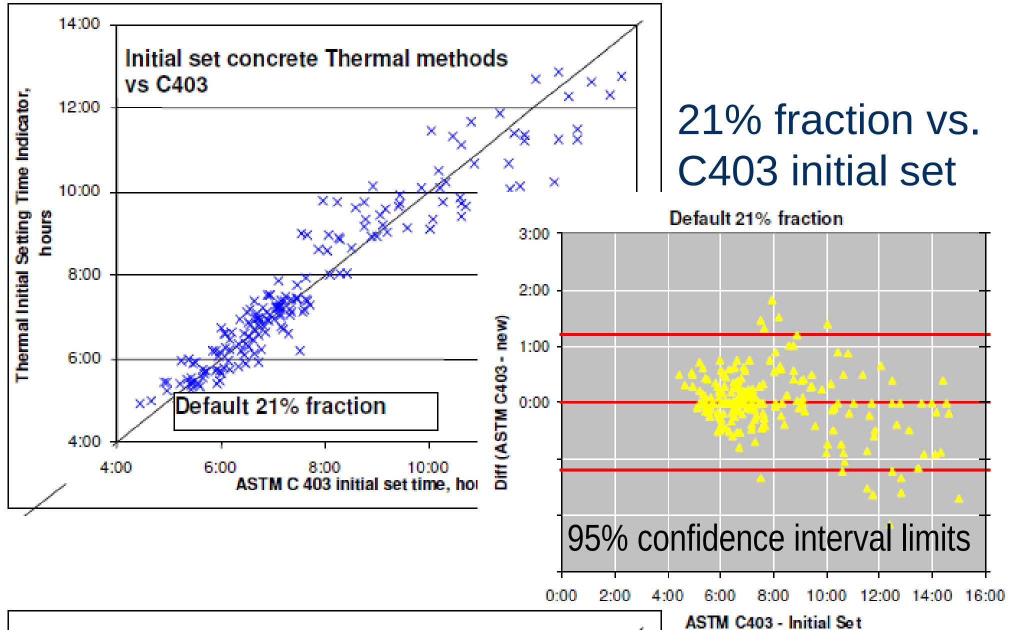


Hydration and thermal profile time of set indications

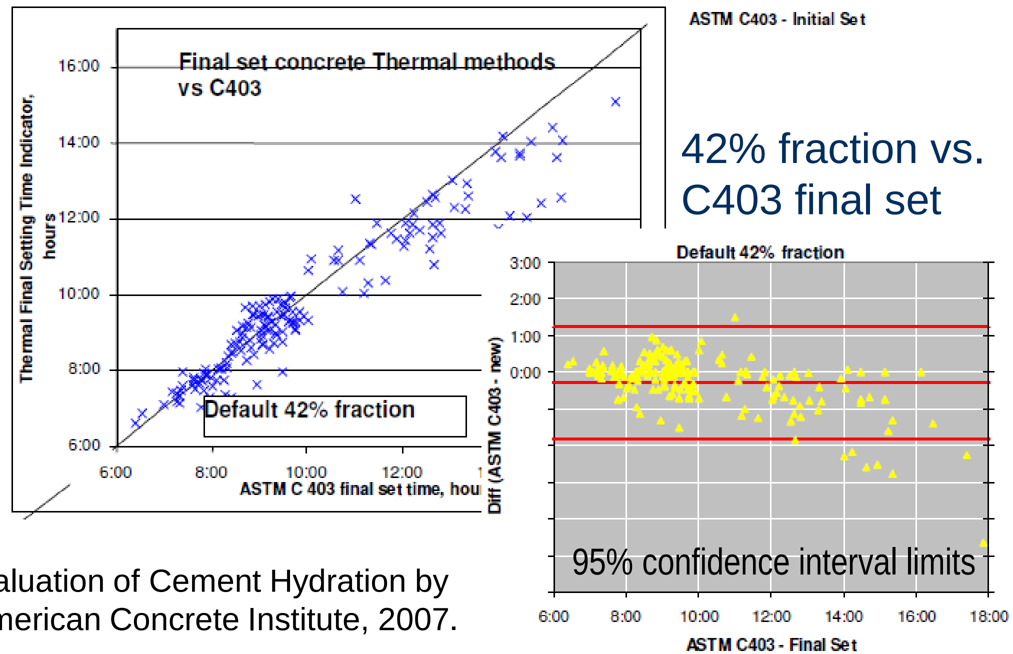


Comparisons for concrete – time of set by thermal methods vs. C403

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



21% fraction vs. C403 initial set



42% fraction vs. C403 final set

From: Sandberg and Liberman, “Monitoring and Evaluation of Cement Hydration by Semi-Adiabatic Field Calorimetry,” ACI SP-241-2, American Concrete Institute, 2007.

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

X XXXX

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

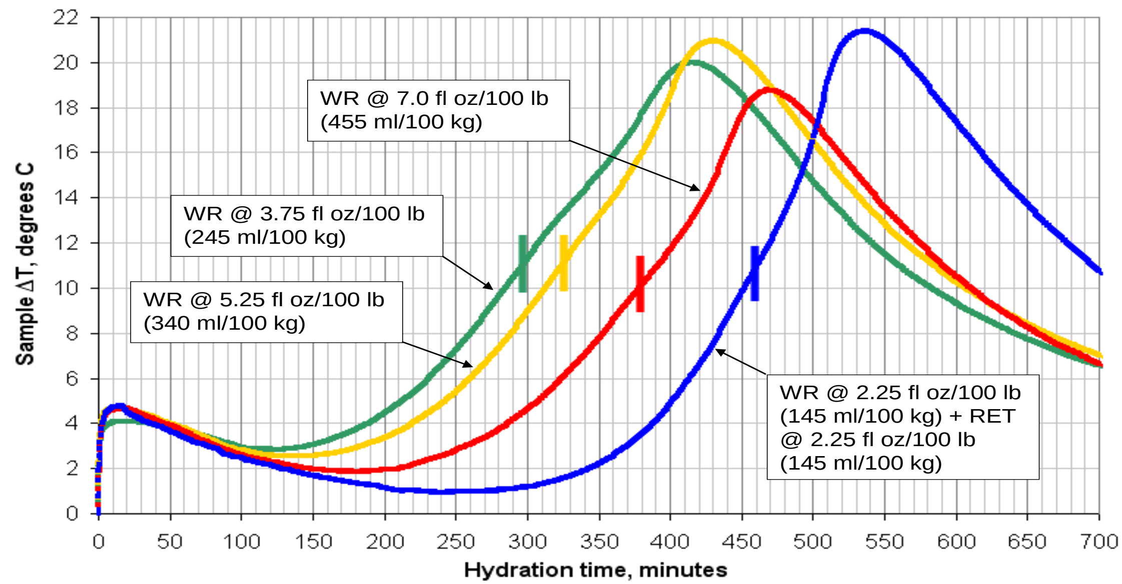
1. Scope

1.1. This method is 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.

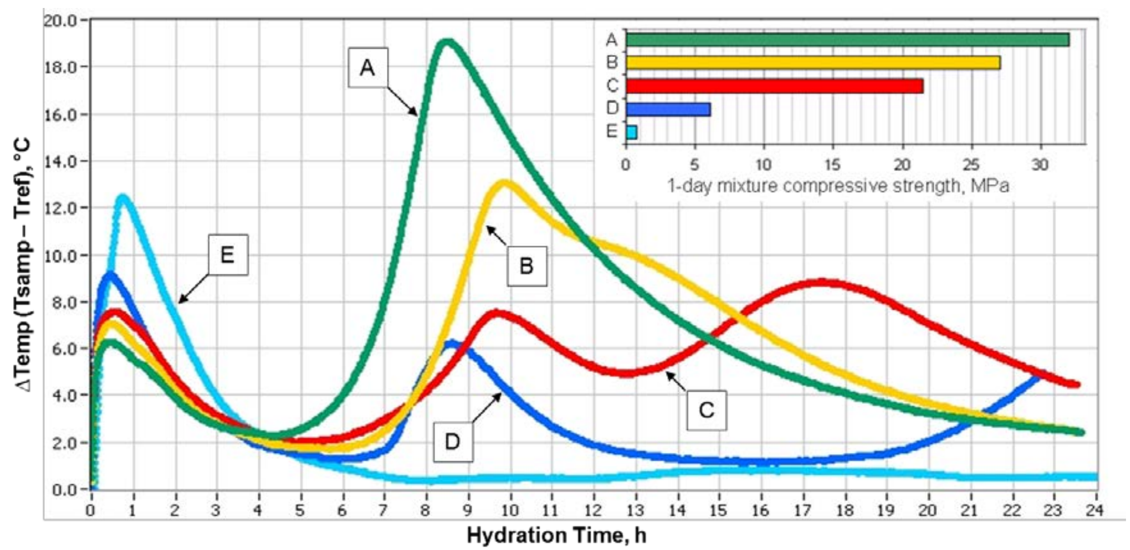
1.2. *Units* - The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

Kinetics of both *normal* and *abnormal* hydration are studied for evaluation of materials & proportions, etc.

<u>Normal hydration</u> –
Series of otherwise identical mixtures comparing admixtures and dosages for retardation effects, robustness of hydration

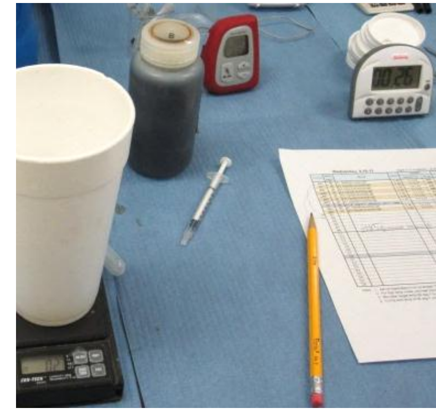


<u>Abnormal hydration</u> –
Series of mixtures with varying admix dosage or fly ash replacement driving varying stages of incompatibility (sulfate depletion) effects, with corresponding 1-day strengths



Lab paste mixture batches – equipment and procedures






- Paste batching with two technicians:
  - A batch every 4 to 6 minutes
  - 48 or more batches in a morning

Examples / Applications

Examples

Mix plan for a paste testing series

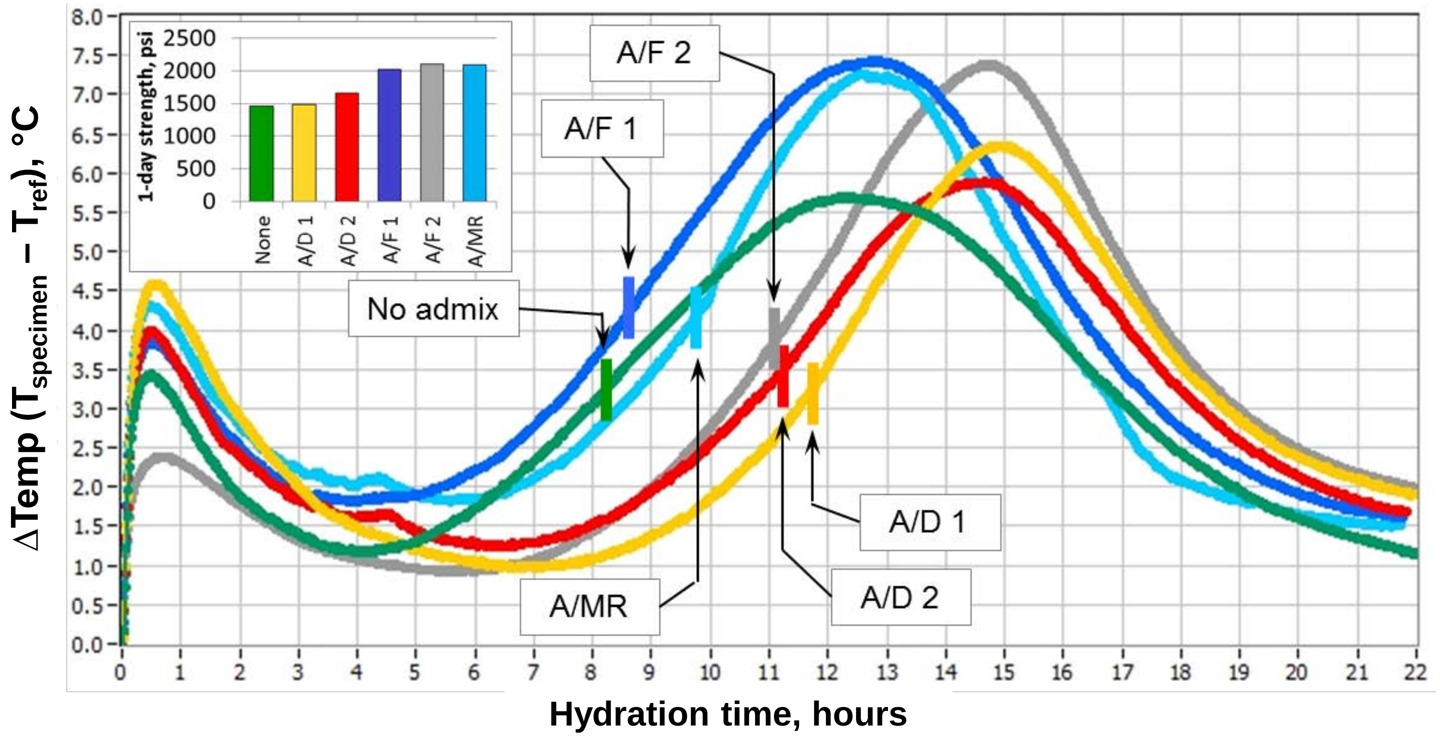
- 3 cements
  - 1 C150 Type II
  - 2 C595 Type IL
- 3 SCMs
  - C618 Class C
  - C618 Class F
  - C989 slag cement
  - No SCM controls
  - 4 replacement rates
- 42 mixtures total
- 3 specimens each mix
  - Strengths: 1, 7, 28 days
  - Thermal set

Paste thermal profile & strength testing, lab ambient, 2" x 4" cylinders **10/9/2013**

2x4 Channel	Mix ID	total cem(g)	w/cm	cement		SCM			water	Glen. 7500		data start time
				source / ID	wt (g)	material	%	wt (g)	(g)	oz/cwt	ml	
A1	A1 OPC-NoAdm-NoSCM	720	0.51	TH I/II	720				367.2			
A2	A2 OPC-7500-NoSCM	850	0.43	TH I/II	850				372.7	5	2.76	
A3	A3 OPC-20%Cash	850	0.43	TH I/II	680	C ash	20%	170	372.7	5	2.76	
A4	A4 OPC-30%Cash	850	0.43	TH I/II	595	C ash	30%	255	372.7	5	2.76	
A5	A5 OPC-40%Cash	850	0.43	TH I/II	510	C ash	40%	340	372.7	5	2.76	
A6	A6 OPC-50%Cash	850	0.43	TH I/II	425	C ash	50%	425	372.7	5	2.76	
A7	A7 OPC-20%Fash	850	0.43	TH I/II	680	F ash	20%	170	372.7	5	2.76	
A8	A8 73 degrees F reference - sand + water											
B1	B1 OPC-30%Fash	850	0.43	TH I/II	595	F ash	30%	255	372.7	5	2.76	
B2	B2 OPC-40%Fash	850	0.43	TH I/II	510	F ash	40%	340	372.7	5	2.76	
B3	B3 OPC-50%Fash	850	0.43	TH I/II	425	F ash	50%	425	372.7	5	2.76	
B4	B4 OPC-40%slag	850	0.43	TH I/II	510	GranCem	40%	340	372.7	5	2.76	
B5	B5 OPC-50%slag	850	0.43	TH I/II	425	GranCem	50%	425	372.7	5	2.76	
B6	B6 bad channel											xxx
B7	B7 OPC-60%slag	850	0.43	TH I/II	340	GranCem	60%	510	372.7	5	2.76	
B8	B8 OPC-70%slag	850	0.43	TH I/II	255	GranCem	70%	595	372.7	5	2.76	
C1	C1 PLC1-NoAdm-NoSCM	720	0.51	TH GU nrm	720				367.2			
C2	C2 PLC1-7500-NoSCM	850	0.43	TH GU nrm	850				372.7	5	2.76	
C3	C3 PLC1-20%Cash	850	0.43	TH GU nrm	680	C ash	20%	170	372.7	5	2.76	
C4	C4 bad channel											xxx
C5	C5 PLC1-30%Cash	850	0.43	TH GU nrm	595	C ash	30%	255	372.7	5	2.76	
C6	C6 PLC1-40%Cash	850	0.43	TH GU nrm	510	C ash	40%	340	372.7	5	2.76	
C7	C7 PLC1-50%Cash	850	0.43	TH GU nrm	425	C ash	50%	425	372.7	5	2.76	
C8	C8 73 degrees F reference - sand + water											
D1	D1 PLC1-20%Fash	850	0.43	TH GU nrm	680	F ash	20%	170	372.7	5	2.76	
D2	D2 PLC1-30%Fash	850	0.43	TH GU nrm	595	F ash	30%	255	372.7	5	2.76	
D3	D3 PLC1-40%Fash	850	0.43	TH GU nrm	510	F ash	40%	340	372.7	5	2.76	
D4	D4 PLC1-50%Fash	850	0.43	TH GU nrm	425	F ash	50%	425	372.7	5	2.76	
D5	D5 PLC-40%slag	850	0.43	TH GU nrm	510	GranCem	40%	340	372.7	5	2.76	
D6	D6 PLC-50%slag	850	0.43	TH GU nrm	425	GranCem	50%	425	372.7	5	2.76	
D7	D7 PLC-60%slag	850	0.43	TH GU nrm	340	GranCem	60%	510	372.7	5	2.76	
D8	D8 PLC-70%slag	850	0.43	TH GU nrm	255	GranCem	70%	595	372.7	5	2.76	
E1	E1 PLC2-NoAdm-NoSCM	720	0.51	TH GU fine	720				367.2			
E2	E2 PLC2-7500-NoSCM	850	0.43	TH GU fine	850				372.7	5	2.76	
E3	E3 PLC2-20%Cash	850	0.43	TH GU fine	680	C ash	20%	170	372.7	5	2.76	
E4	E4 PLC2-30%Cash	850	0.43	TH GU fine	595	C ash	30%	255	372.7	5	2.76	
E5	E5 PLC2-40%Cash	850	0.43	TH GU fine	510	C ash	40%	340	372.7	5	2.76	
E6	E6 PLC2-50%Cash	850	0.43	TH GU fine	425	C ash	50%	425	372.7	5	2.76	
E7	E7 PLC2-20%Fash	850	0.43	TH GU fine	680	F ash	20%	170	372.7	5	2.76	
E8	E8 73 degrees F reference - sand + water											
F1	F1 PLC2-30%Fash	850	0.43	TH GU fine	595	F ash	30%	255	372.7	5	2.76	
F2	F2 PLC2-40%Fash	850	0.43	TH GU fine	510	F ash	40%	340	372.7	5	2.76	
F3	F3 PLC2-50%Fash	850	0.43	TH GU fine	425	F ash	50%	425	372.7	5	2.76	
F4	F4 PLC2-40%slag	850	0.43	TH GU fine	510	GranCem	40%	340	372.7	5	2.76	
F5	F5 PLC2-50%slag	850	0.43	TH GU fine	425	GranCem	50%	425	372.7	5	2.76	
F6	F6 PLC2-60%slag	850	0.43	TH GU fine	340	GranCem	60%	510	372.7	5	2.76	
F7	F7 PLC2-70%slag	850	0.43	TH GU fine	255	GranCem	70%	595	372.7	5	2.76	
F8												

Screening of WR admixtures for a 30% Class C ash mix

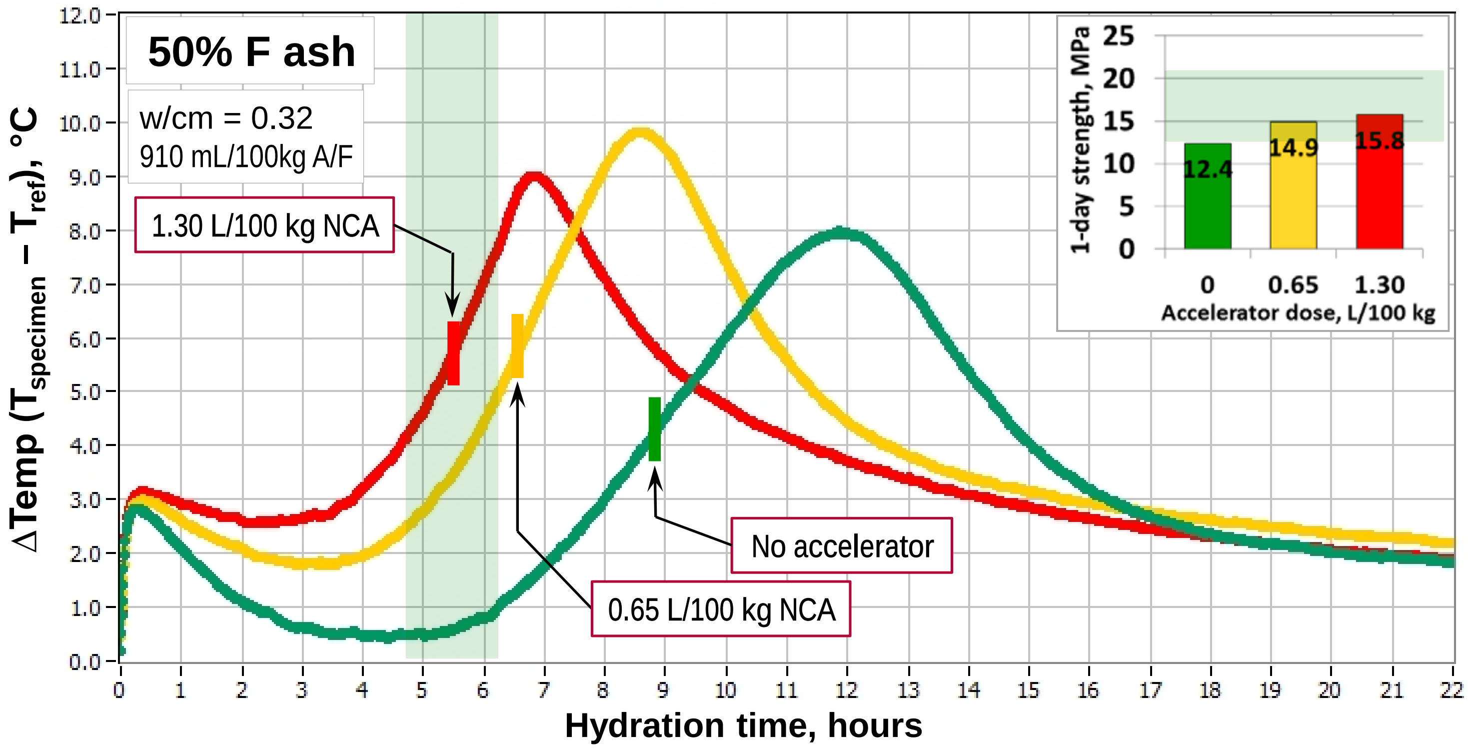
5 different WR admixtures (2 Type A/D, 2 Type A/F, 1 MR) compared in paste mixtures, dosages selected for approx. 6% water reduction



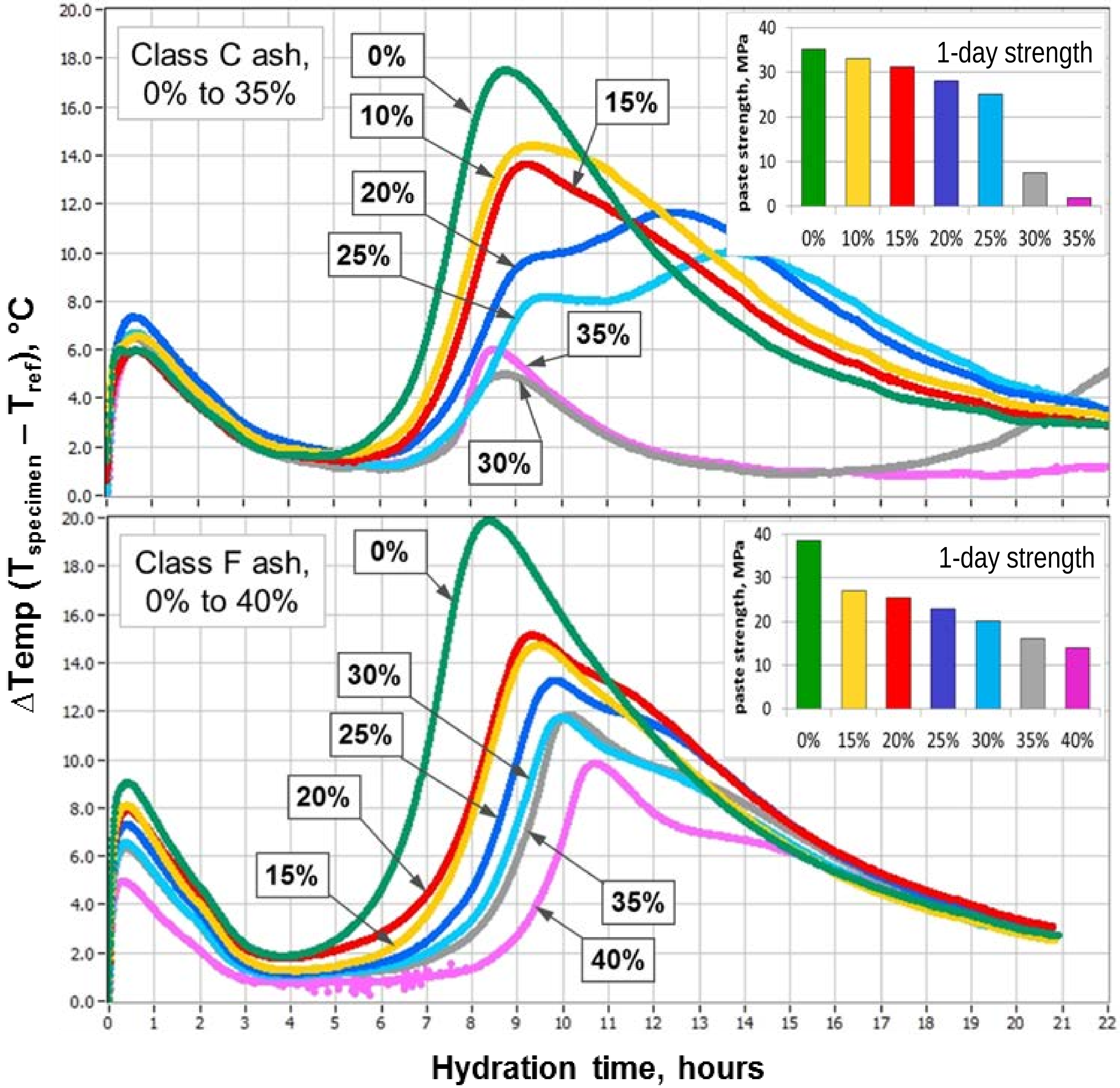
"A/F 1" causes the least retardation, with good early strength influence.

Initial NCA dosage rate for a HVFA mixture

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

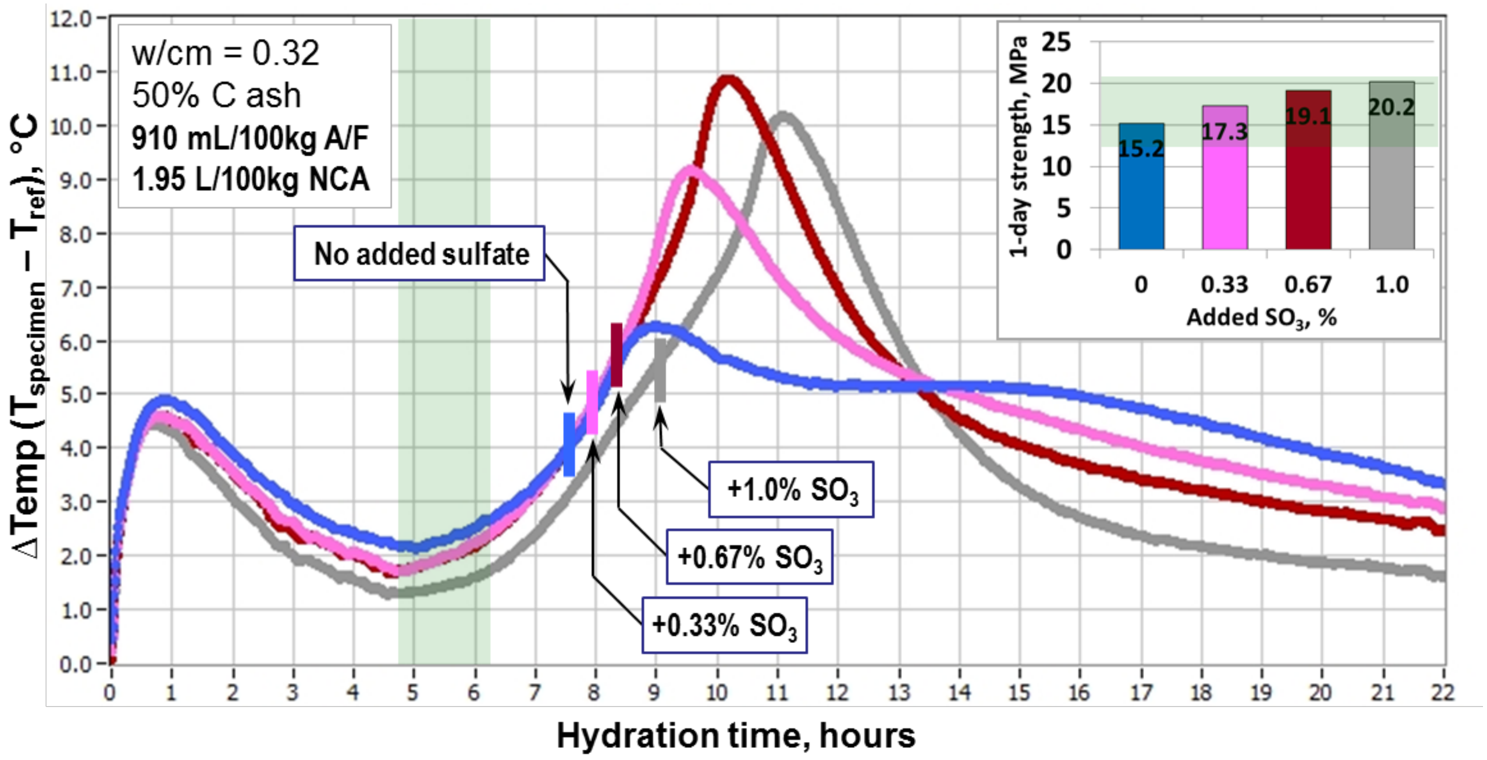


Effects of incremental fly ash replacement, C vs. F ash

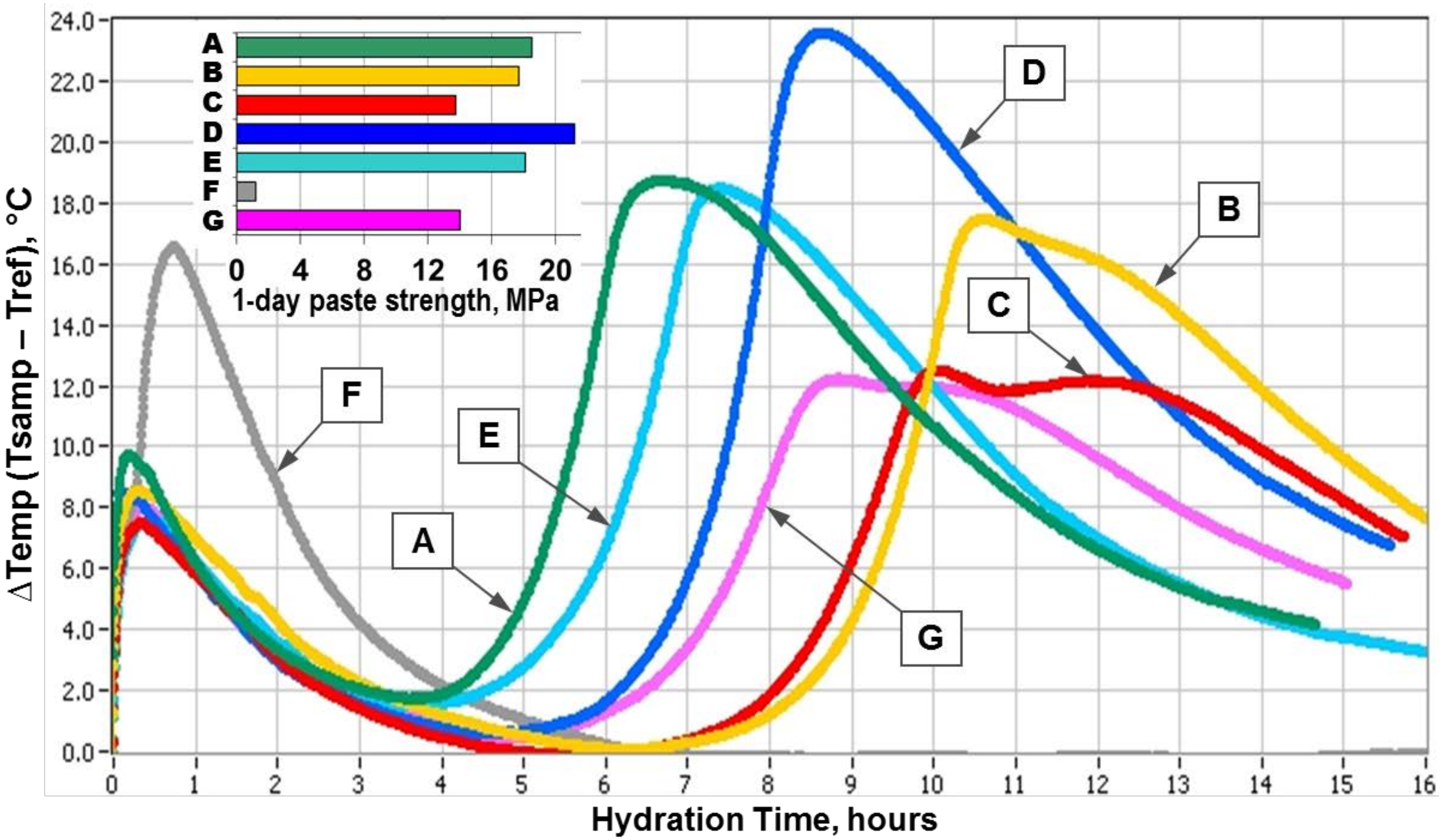


Verification of sulfate balance issue via sulfate addition

Otherwise identical paste mixtures with incremental addition of calcium sulfate

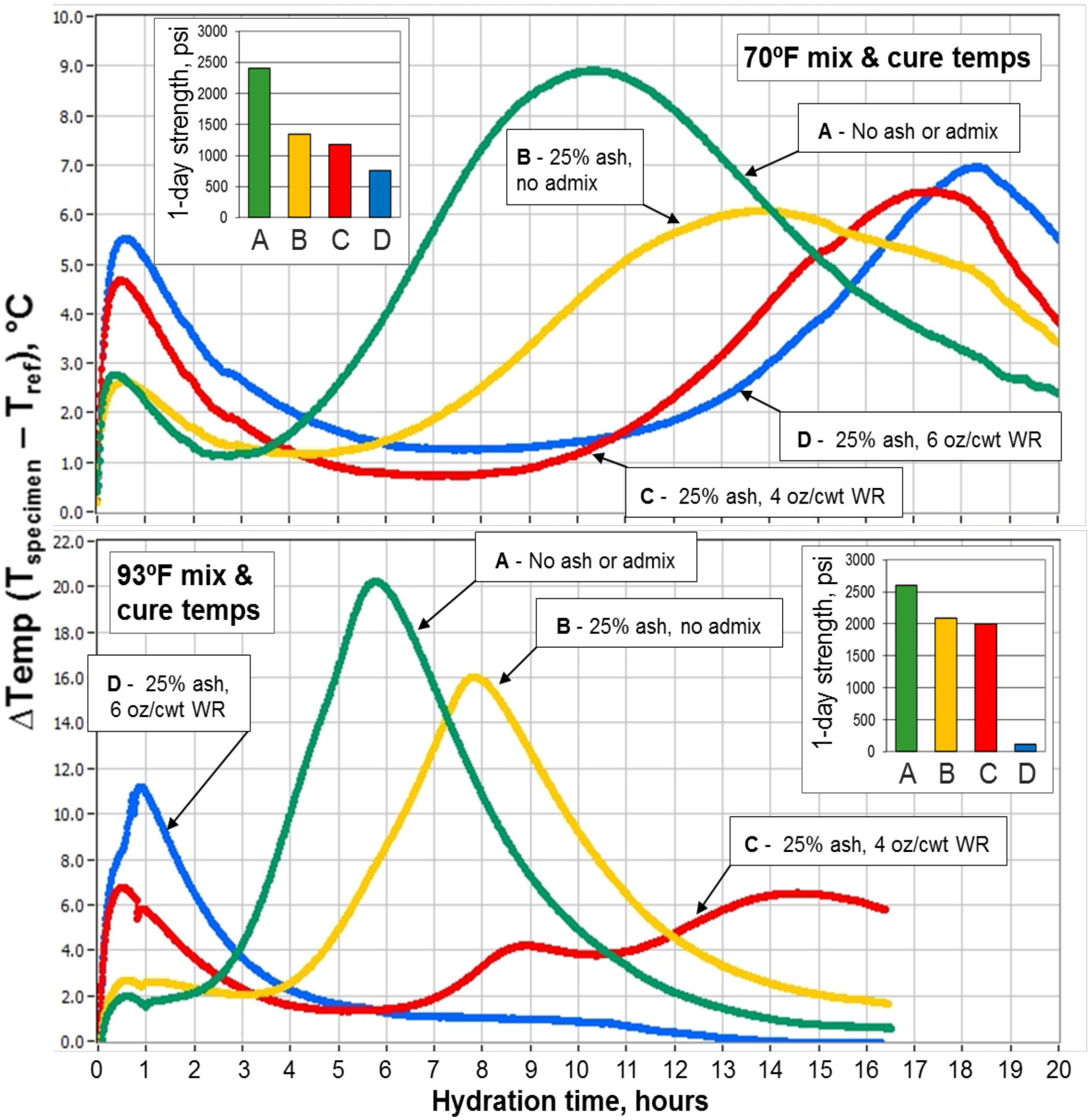


Comparison of 7 cements in an aggressive mix design



Otherwise identical paste mixtures comparing 7 cements, with 25% Class C fly ash, upper-limit dose of Type A/D WR, and 35ºC (95ºF) mix and cure temps

Temperature influences on incompatibility potential

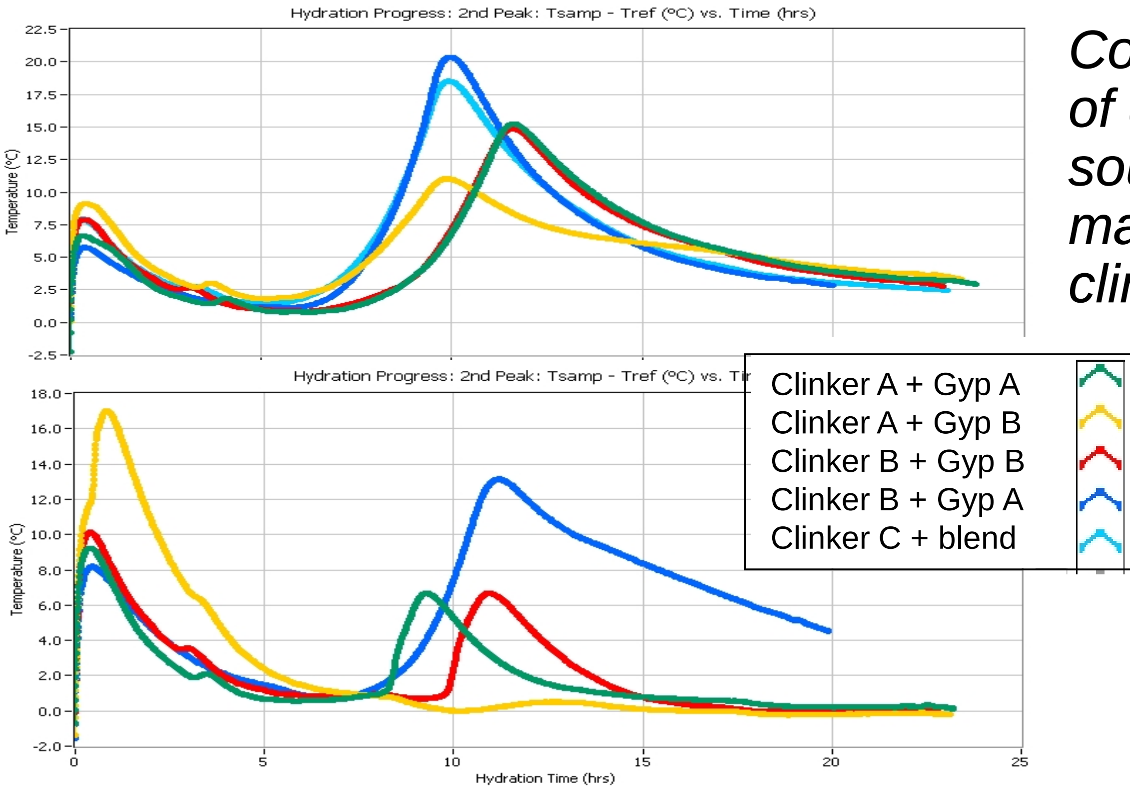


4 identical paste mixtures compared at different initial mixture and curing temps (70°F (21°C) vs. 93°F (34°C)

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

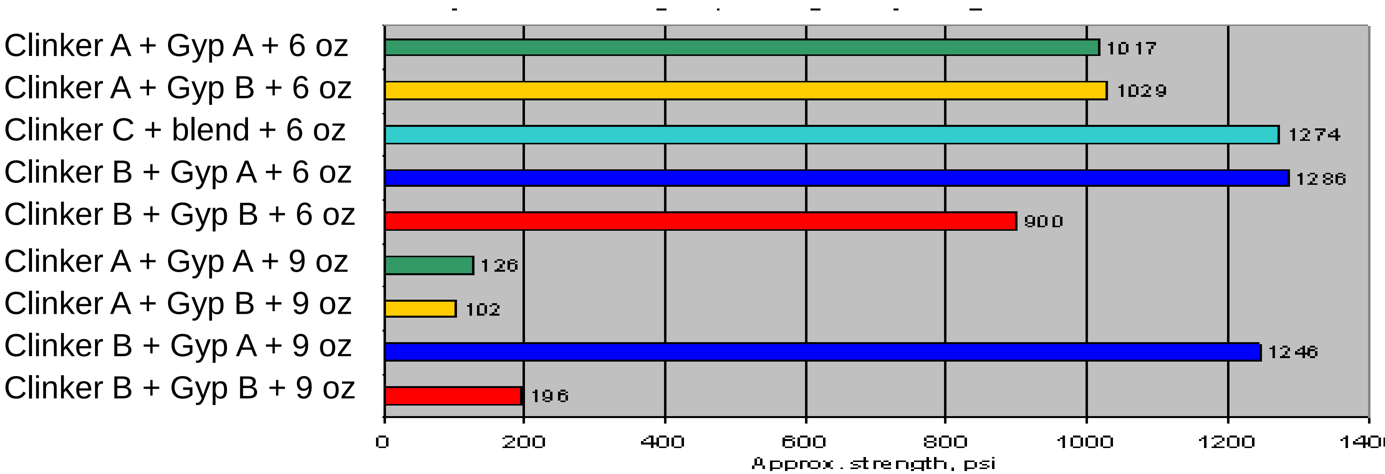
Higher temps alone drive incompatible behavior in the mixtures with WRA

Examples

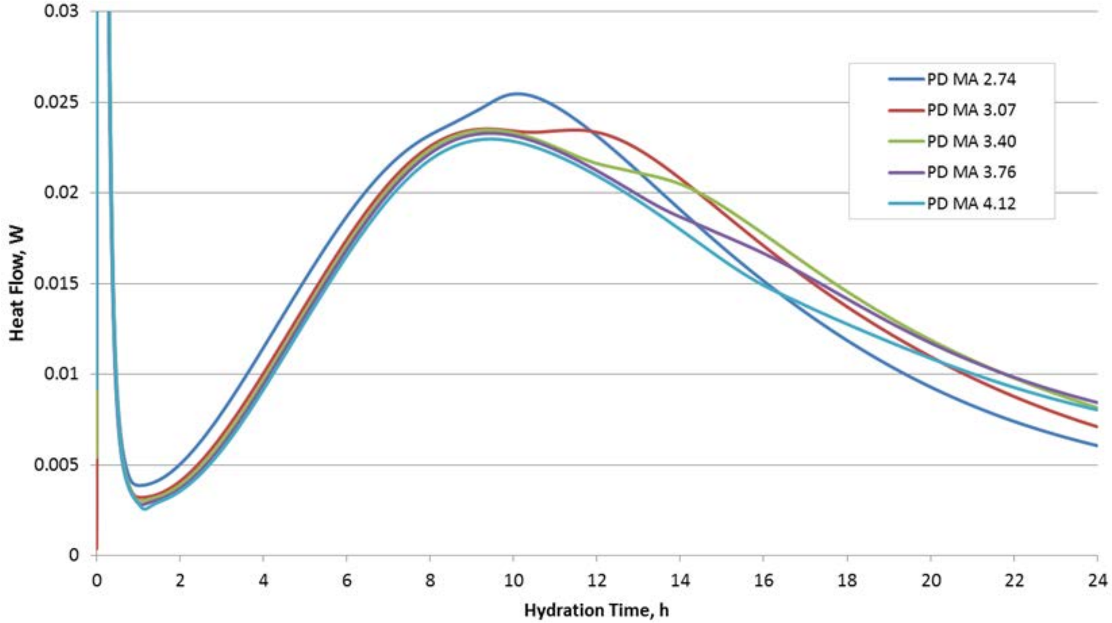


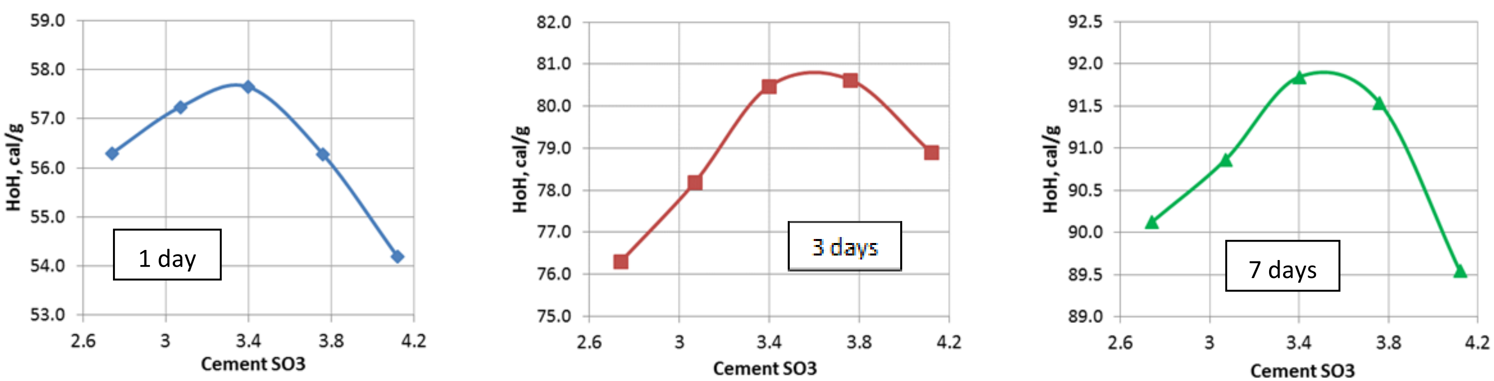
Comparison of effects of different gypsum sources on cements made from two different clinkers

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)



Optimum SO_3 via ASTM C1702 on mill-ground cement samples cement with varying SO_3 contents





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Questions?

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