LTRC Concrete Workshop
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# *Simple Thermal Measurements (Semi-Adiabatic Calorimetry) for Concrete QC and Troubleshooting*


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# QC Control of concrete set and early strength

- Greater variability today due to:
  - Greater cement replacement with SCM's
  - More aggressive admixture use
  - Frequent source changes / unfamiliar materials
  - Variability of individual materials
  - Greater seasonal / temp change sensitivity
- Effects of unpredictability include issues with:
  - Uniformity and quality of finish
  - Uncontrolled cracking & other aesthetic issues
  - Timing and effectiveness of joint sawing
  - Construction sequencing / schedules

*A lab concrete batch of the proposed mix design for a project is not an adequate prediction of field performance!!*

# How can thermal measurements help?

- Background:
  - Hydration of cementitious materials generates heat
  - Records of the mixture thermal changes over time can be used for various QC-related applications
  - Inexpensive equipment is available for very simple measuring, recording, and processing of this data

- “Semi-adiabatic calorimetry” (SAC):
  - *Indication of the heat evolved from a cementitious mixture hydrating with relatively little influence from ambient temperature changes, using a record of the mixture’s changing temperatures over time.*

# Applications of SAC

- QC – evaluation of proportions & materials
  - Evaluation of set time & early hydration influences:
    - New or changed mix designs
    - New (unfamiliar or changed) materials sources
    - Potential changes in admix dosage, SCM % or blend
    - Effects of changing field temperatures
    - Combinations of above variables
- Other QC areas - producer or contractor
  - Passive monitoring of set times & early hydration
  - Tracking variability of materials
- Troubleshooting of field problems
  - Unexplained performance changes

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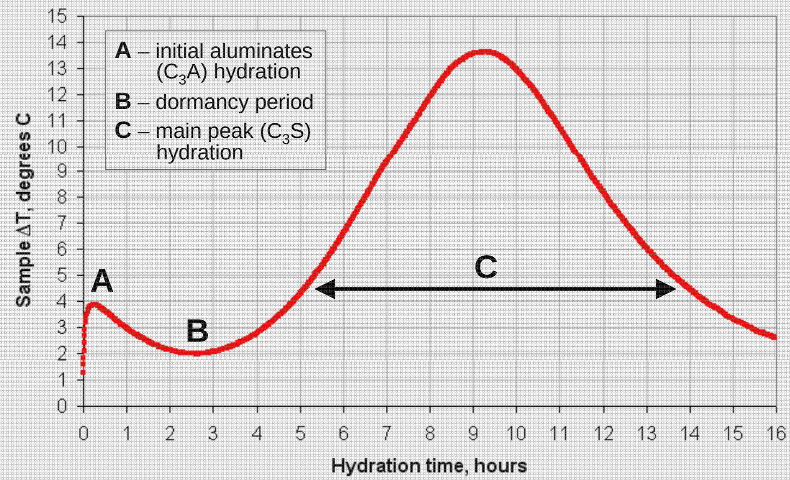
# Technical background


$3CaO \cdot Al_2O_3$ (tricalcium aluminate)
$3CaO \cdot SiO_2$ (tricalcium silicate)
$CaSO_4 \bullet 2H_2O = CaO \bullet SO_3 \bullet 2H_2O$ (calcium sulfate dihydrate)
$CaSO_4 \bullet \frac{1}{2}H_2O = CaO \bullet SO_3 \bullet \frac{1}{2}H_2O$ (calcium sulfate hemihydrate)
$CaSO_4 = CaO \bullet SO_3$ (anhydrous calcium sulfate)



# Cement hydration and heat

- Compounds in portland cement clinker:

  ***$C_3S$*** = $3CaO \cdot SiO_2$
  (Tricalcium silicate)

  ***$C_2S$*** = $2CaO \cdot SiO_2$
  (Dicalcium silicate)

  ***$C_3A$*** = $3CaO \cdot Al_2O_3$
  (Tricalcium aluminate)

  ***$C_4AF$*** = $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$
  (Tetracalcium aluminoferrite)

# Cement hydration and heat

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(Tetracalcium aluminoferrite)

← *Hydration of these compounds produces significant, measurable heat during the first 24 hours of hydration*

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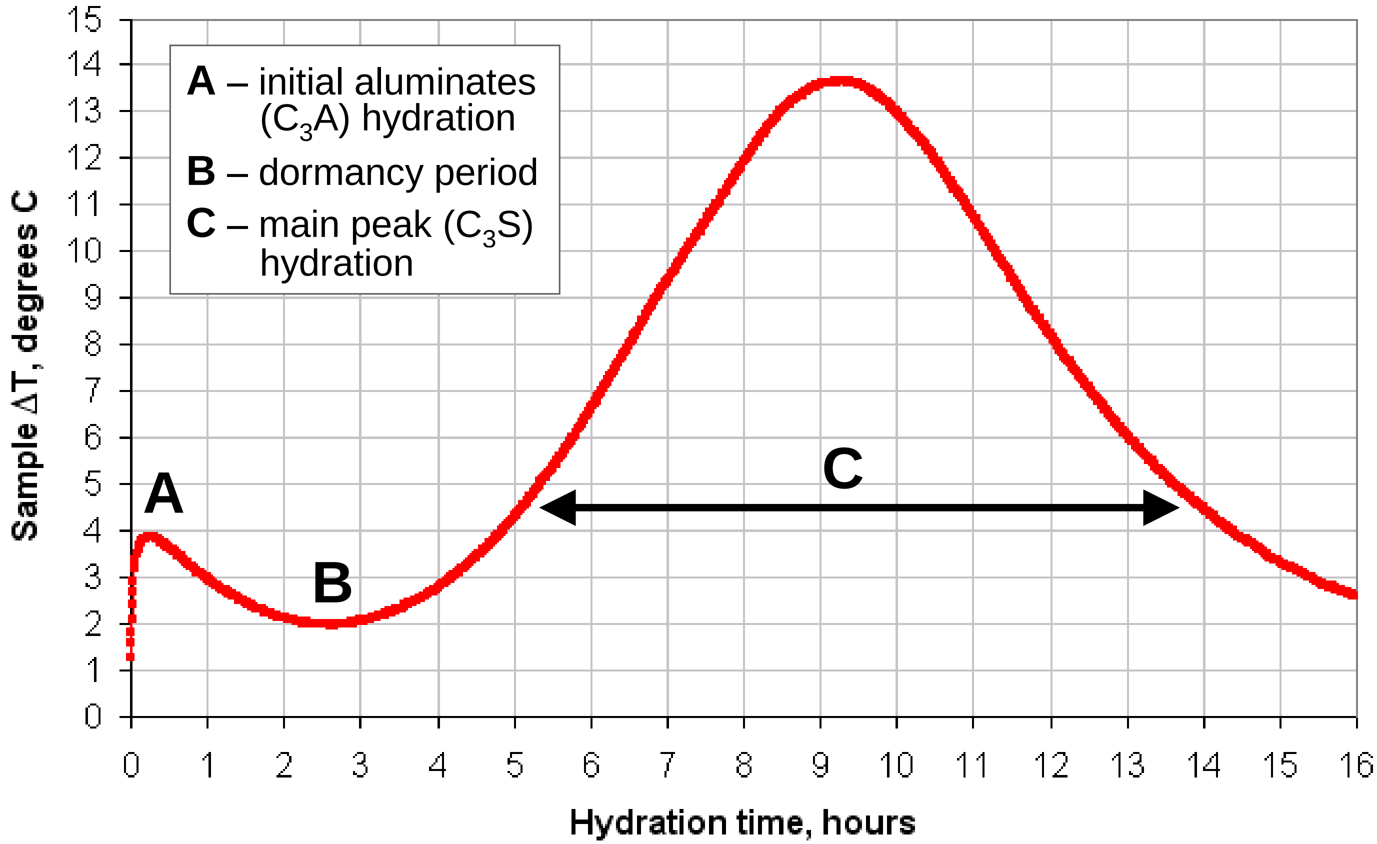
# Cement hydration and heat

*Thermal changes and their timing are influenced by cement chemistry, mix temps, interaction of admixtures and SCM's, mass effects, sulfate balance, etc.*




*Reactions involving $CaSO_4$ from gypsum added during cement grinding tame the initial, rapid aluminate hydration "A", resulting in the dormant period "B".*

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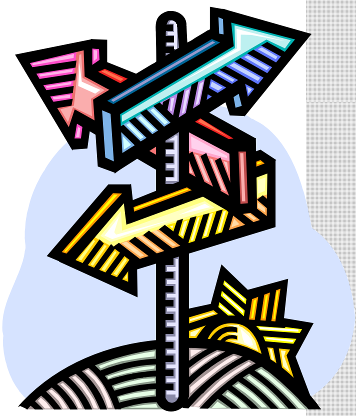
# Selection of equipment and methods



# Measuring & recording temperature changes

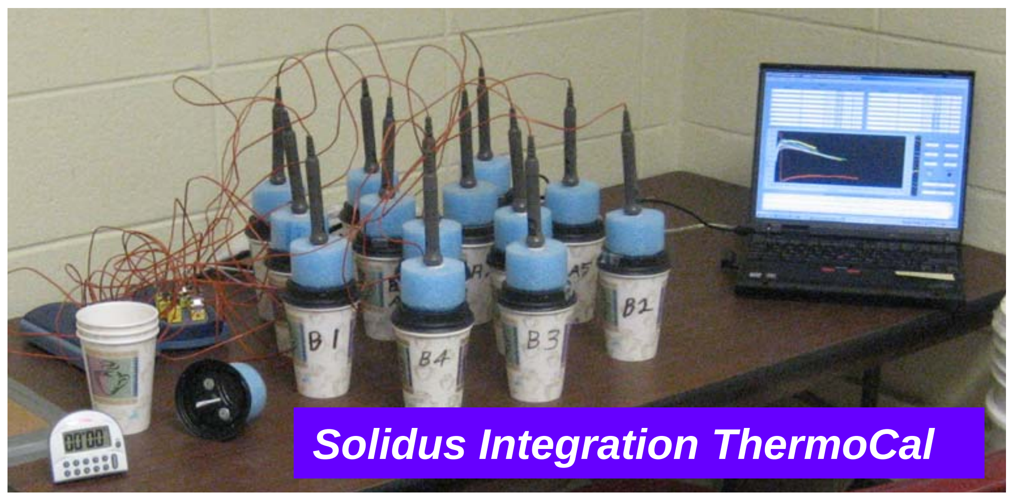


**Tipped Probe (Available in 1.5 meter and 3.0 meter cable length)**
A tipped 150mm length 5mm OD 316 stainless probe with no handle. Probe and Cable are FDA food rated.
Each probe comes with a 1.5 Meter or 3.0 Meter length cable terminated with an MCX plug for input into the LogTag TREX Recorder.


# Equipment designed or adapted for SAC

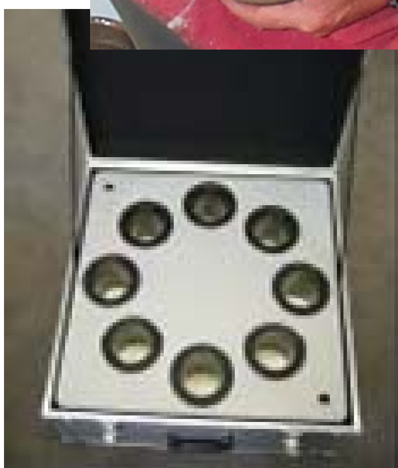
*Grace AdiaCal®*






*Solidus Integration ThermoCal*

# Method variables & considerations



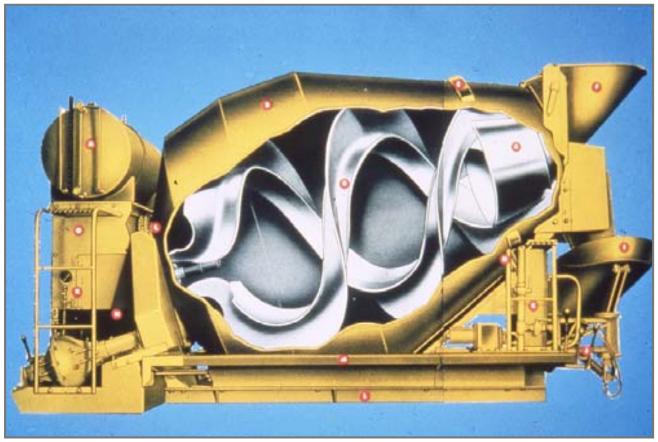
- **Concrete, mortar or paste?**
- **Mixing method / equipment**
- **Volume / mass of test sample?**
- **How much insulation?**
- **Initial mixture temperature**
- **Environment temperature**
- **Control of environment**
- **Sensor contact w/ sample**

# Test method variables & considerations

*General guidance:*

- Concrete or mortar vs. paste:
  - Concrete/mortar: more convenient on the job site, more representative set times
  - Paste: better for multi-variable lab studies
- Sample size (mass):
  - Larger samples for mortar / concrete
  - Smaller for paste (500g cementitious, ±)
- Insulation of container:
  - Insulation helps accentuate peaks, may modify shape of curves
  - Little to none for paste samples
  - More for mortar, most for concrete
  - Choice depends somewhat on objectives but mostly to produce reasonable curves

# Test method variables & considerations

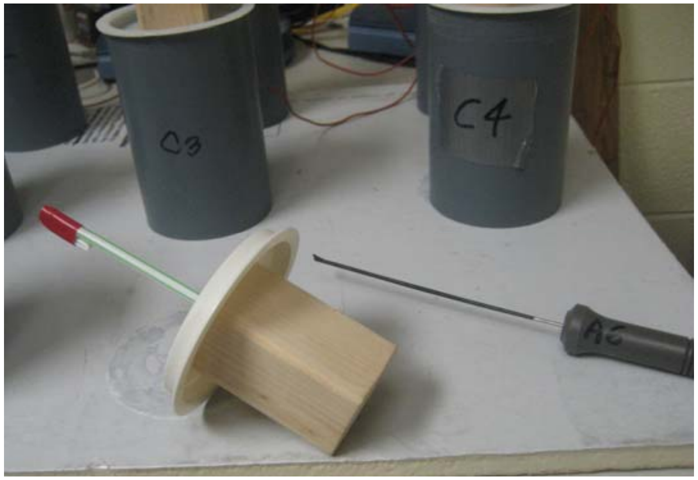


*Mixing equipment and procedures:*

- Mortar
  - Wet-sieved concrete
  - C 305 (4 min.)
  - Others
- Paste
  - C 305 (2¼ min.)
  - Hand-held kitchen mixer
  - 1 minute or less may be adequate mixing time
- Mixing shear differences may influence results

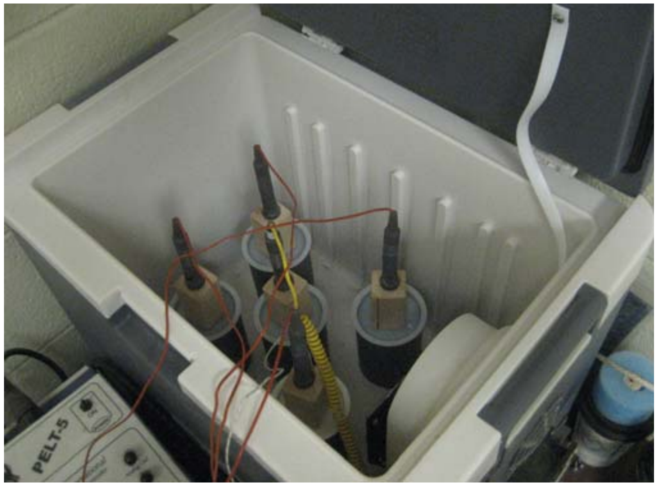
# Lab mix series recommendations

- All mixes in a series done the same way under the same conditions
- Pre-weigh all cementitious combinations
- Use a mix timer (60 sec. works for paste)
- Assure the same thermal sensor position for all samples
- Begin data collection ASAP



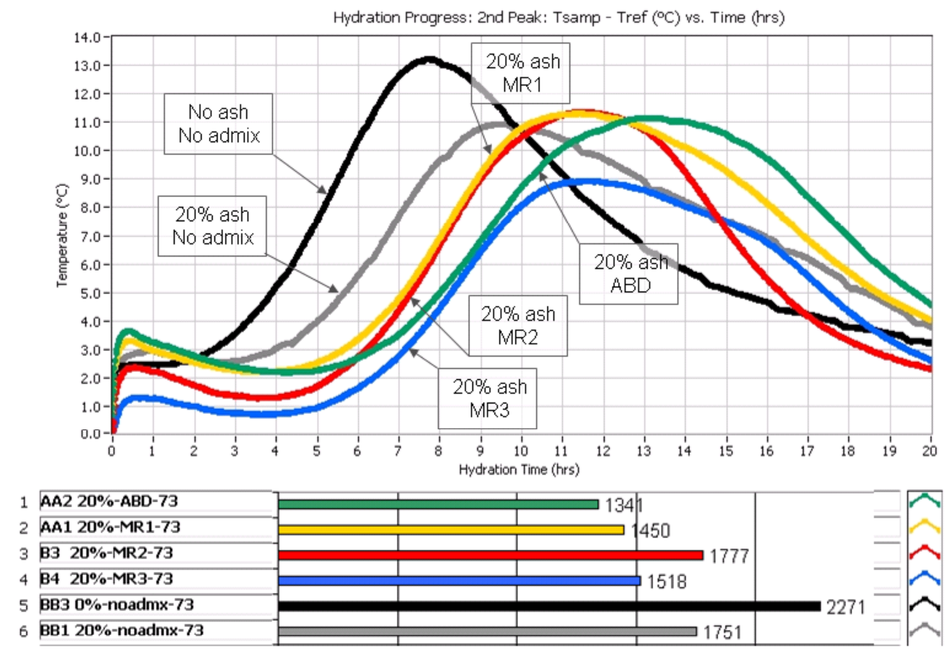
# Lab mix series recommendations

- Control curing environment temp to match initial mix temps as per test plan
- Water-bath curing tanks work well
- Avoid any air movement around samples, HVAC vents, areas near equipment cooling fans, etc.
- Document environment temp with a dedicated test channel & inert sample



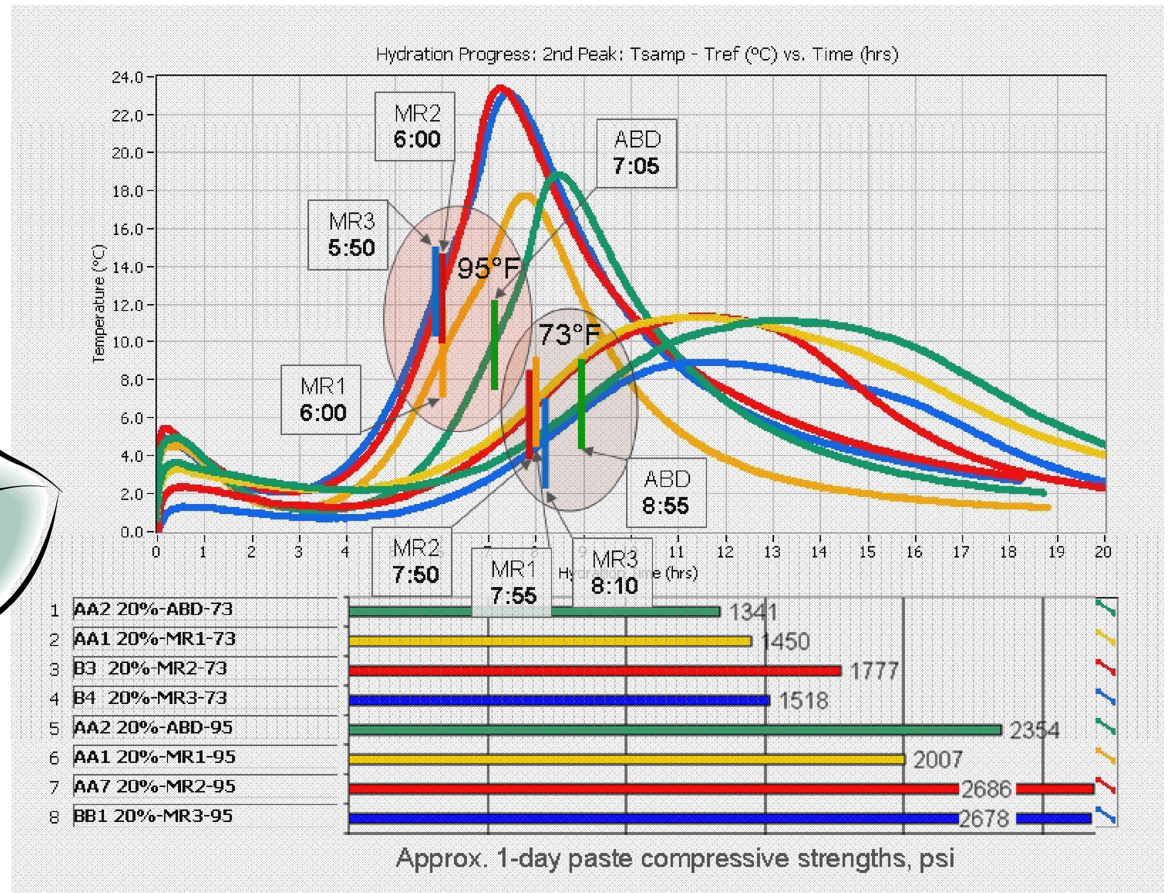

# Specimen strength data – also recommended

- Hardened test samples of paste or mortar can be prepared and compression tested after thermal data collection (usually 1-day)
- Parallel mortar cubes – another option
- Bar charts of strengths presented with thermal profiles assist in data interpretation & facilitate early strength comparisons




Approx. 1-day paste compressive strengths, psi

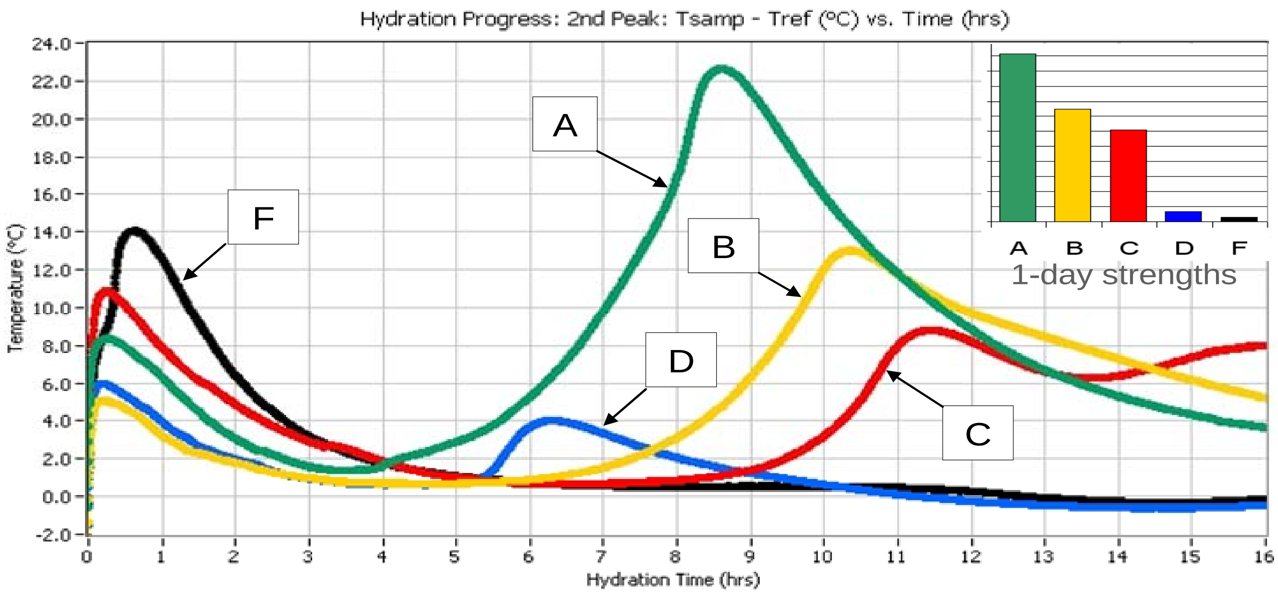
# Applications & example studies



# Normal vs. abnormal hydration behavior

*Sulfates-related abnormalities can easily be spotted using SAC:*

- Normal behavior = traditional thermal profiles with distinct peaks
- Abnormal behavior = misshapen profiles or non-traditional behavior, indicating a mixture sulfate imbalance, or “incompatibility of materials”




- **A**: Normal, robust peak and strength development
- **B**: Normal behavior but attenuated peak with significant retardation
- **C**: Marginal and retarded silicate hydration with recurring aluminate activity
- **D**: No useful silicate hydration or strength gain, may contain recurring aluminate activity
- **F**: Flash set from uncontrolled initial aluminate hydration, no silicate activity or strength

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# Concrete setting time

*Ongoing work by ASTM subcommittee C09.23 for an alternative to C 304:*

**Designation: X XXXX-XX** **Ballot Item**

**Standard Test Method for**
**Determination of Setting Time of Concrete by the Temperature Method**[1]

This standard is issued under the fixed designation X XXXX; 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.

- Development of a "Thermal" set time method:
  - Insulated samples of fresh concrete or mortar
  - Spreadsheet routine for determining "thermal" set times from SAC data

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# ASTM C 09.23 "round robin" testing – Atlanta 11/19/08

- 12 teams from suppliers, testing labs, universities
- 3 concrete mixtures
- C 403 in triplicate, each mixture, each team
- Thermal set determinations: specified SAC devices built by teams, Adiacal, others

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# ASTM C 09.23 “round robin” testing – Atlanta 11/19/08

| | Mix 1 | Mix 2 | Mix 3 |
|---|---|---|---|
| Cementitious content | 611 lb/yd³ (363 kg/m³) | 611 lb/yd³ (363 kg/m³) | 590 lb/yd³ (350 kg/m³) |
| Cement type | Type III | Type I | Type I |
| Fly ash replacement | none | none | 20% Class F |
| Type A/F admixture | 2 fl oz/100 lb (130 ml/100 kg) | ---- | ---- |
| Type B/D admixture | ---- | 2 fl oz/100 lb (130 ml/100 kg) | 0.75 fl oz/100 lb (50 ml/100 kg) |
| w/cm | 0.477 | 0.491 | 0.508 |
| Slump | 5.5 in. (140 mm) | 5 in. (125 mm) | 7 in. (180 mm) |
| Air content | 1.6% | 1.6% | 0.8% |
| Initial temp. | 49° F (9° C) | 56° F (13° C) | 59° F (15° C) |

*Round robin concrete mixtures*

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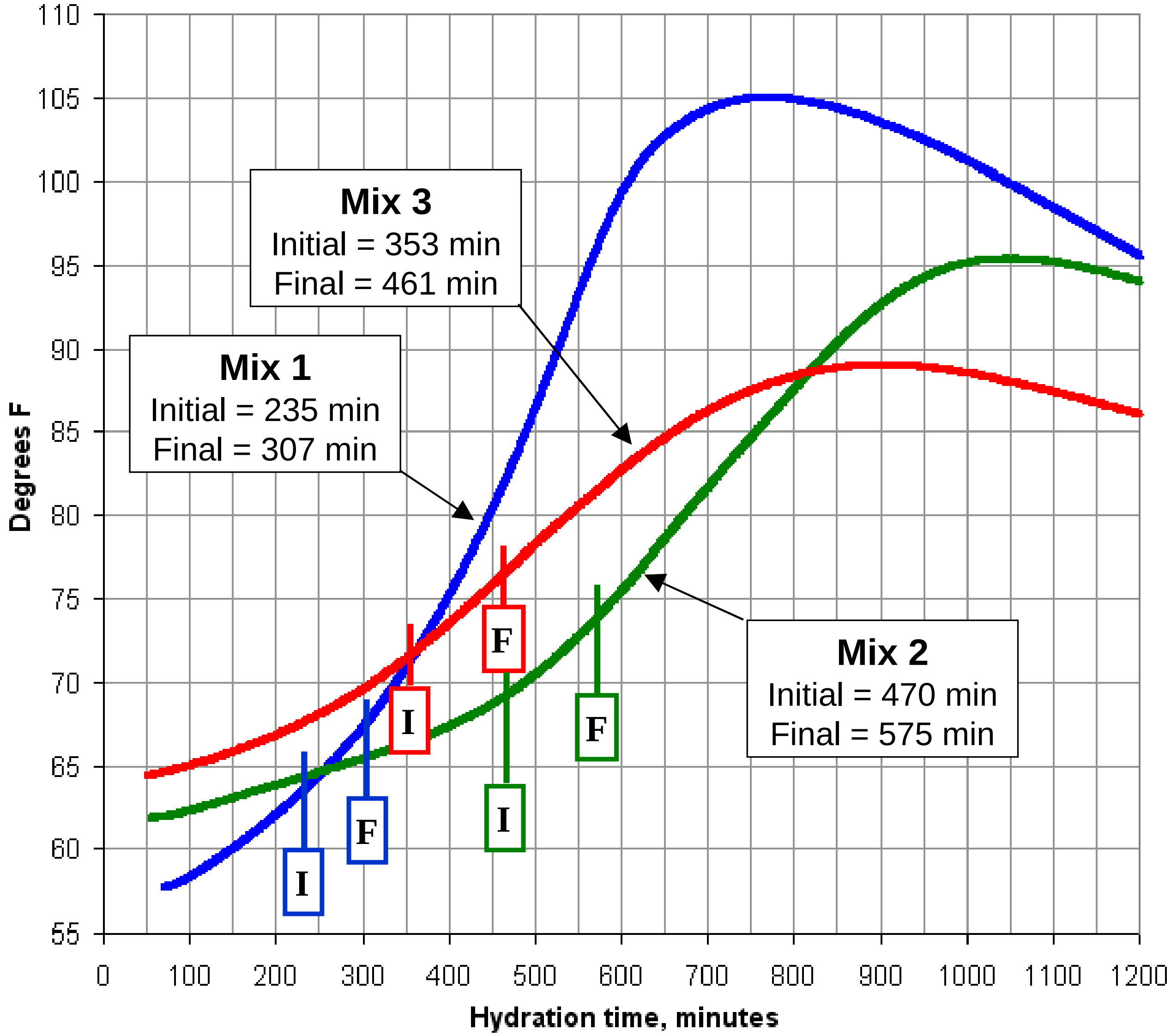
# ASTM C 09.23 “round robin” testing – Atlanta 11/19/08

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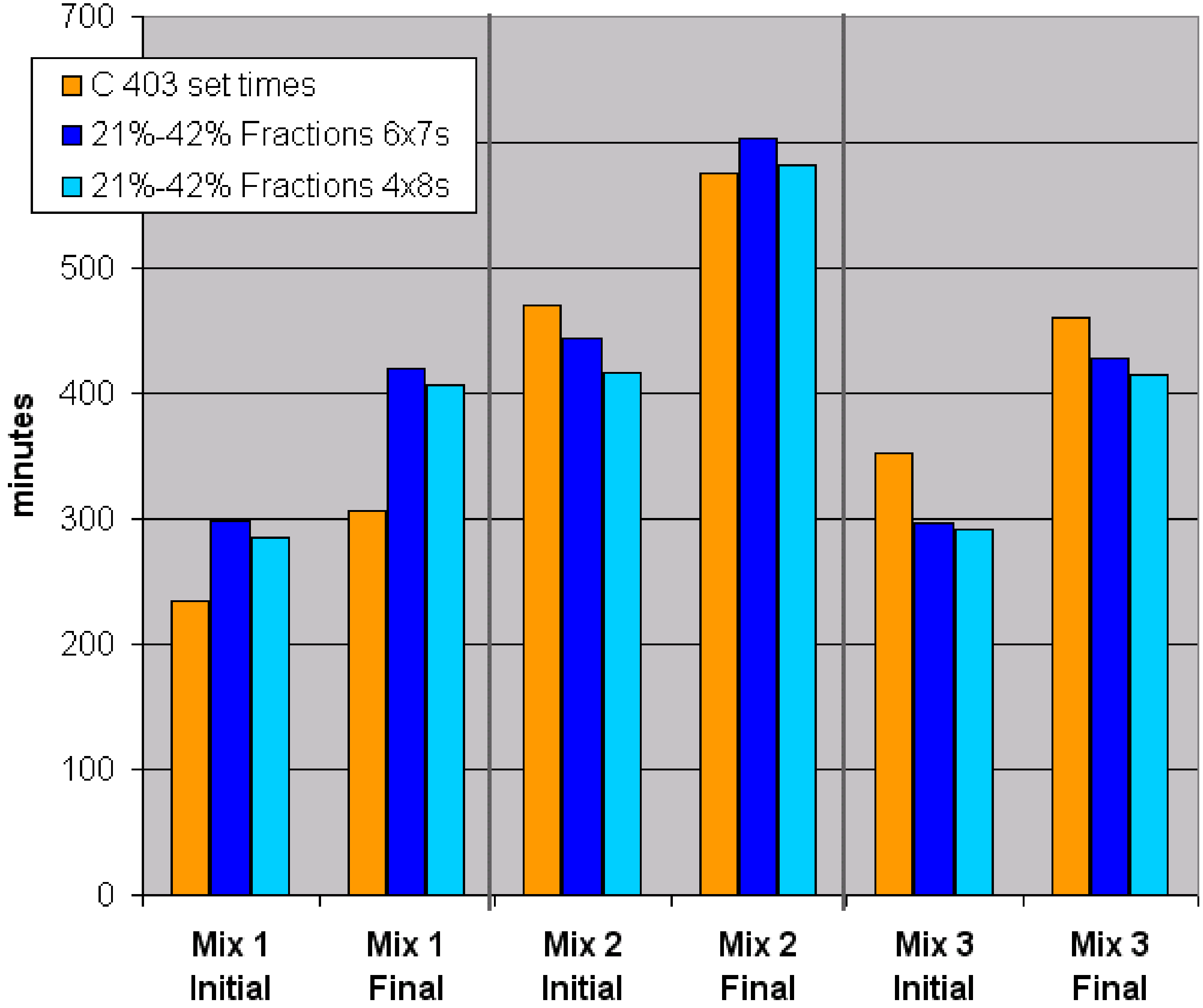
# ASTM C 09.23 “round robin” testing – Atlanta 11/19/08

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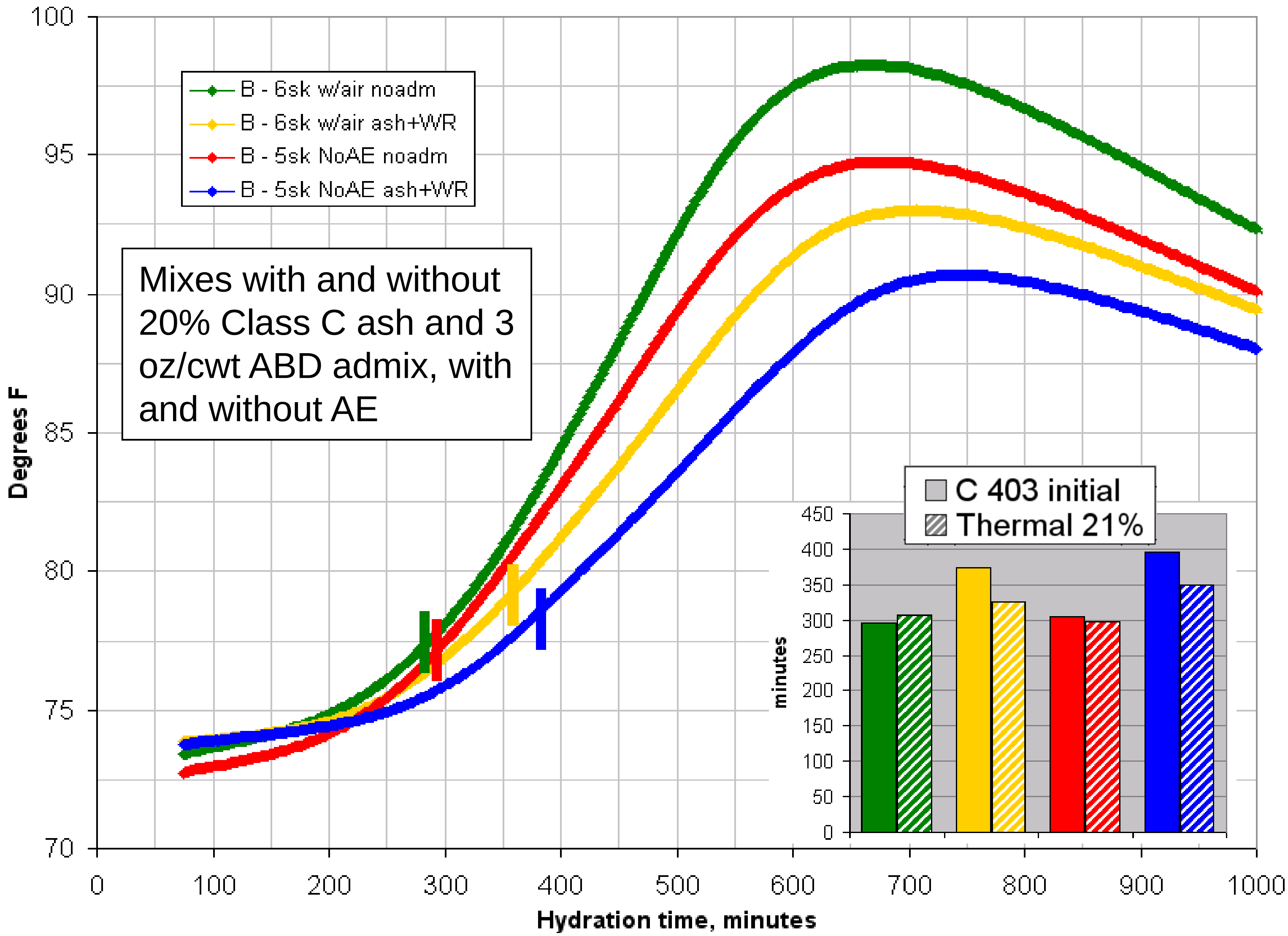
# Lab concrete mix examples:
# C 403 initial set vs. 21% fraction thermal set time



# Lab set time studies using paste mixtures

- Relative time-of-set comparisons can be made using a simple visual indication of the point half-way up the main peak heat rise (50% of peak or 50% "fraction").
- Works well for lab paste mixtures – 50% fraction thermal set indication is typically about 150% of C 403 initial set in parallel concrete mixtures
  - 50% fraction values can be spreadsheet-calculated or estimated (visually)
  - Influence trends of lab paste mixtures track quite well with the same trends in field concrete mixtures
  - Best approach for quick evaluation of a number of variables in multiple mixtures

# Lab set time studies using paste mixtures

*Six mixtures (parallel concrete and paste) used for set time comparisons*

| Mix Designation: | 100-0-0 No admix | 75-0-25C Type A/D | 75-0-25C Mid-Range | 75-0-25F Type A/D | 75-0-25F Mid-Range | 50-35-15C Mid-Range |
|---|---|---|---|---|---|---|
| SCM(s) | ---- | 25% C ash | 25% C ash | 25% F ash | 25% F ash | 35% GGBFS +15% C ash |
| Type A/D admixture | ---- | 3 fl oz/100 lb (195 ml/100 kg) | ---- | 3 fl oz/100 lb (195 ml/100 kg) | ---- | ---- |
| Mid-Range admixture | ---- | ---- | 4 fl oz/100 lb (260 ml/100 kg) | ---- | 4 fl oz/100 lb (260 ml/100 kg) | 4 fl oz/100 lb (260 ml/100 kg) |
| w/cm | 0.54 | 0.51 | 0.51 | 0.51 | 0.51 | 0.51 |
| Slump | 4 in. (100 mm) | 6 in. (150 mm) | 5.25 in. (135 mm) | 5 in. (125 mm) | 5.75 in. (145 mm) | 5.75 in. (145 mm) |
| Air content | 1.3% | 1.3% | 1.8% | 2.4% | 1.7% | 2.4% |
| Initial temp. | 69° F (21° C) | 69° F (21° C) | 70° F (21° C) | 69° F (21° C) | 70° F (21° C) | 68° F (20° C) |

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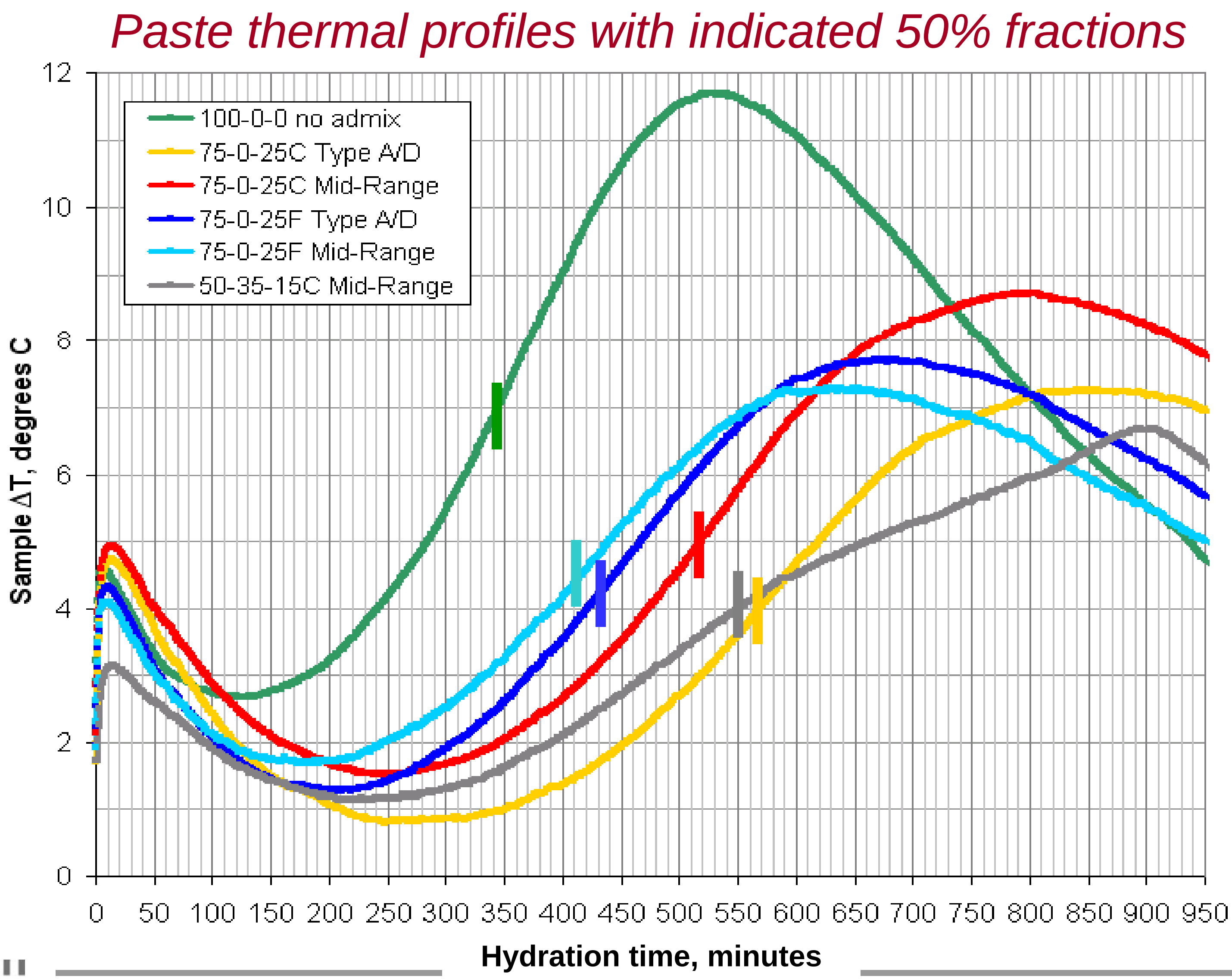
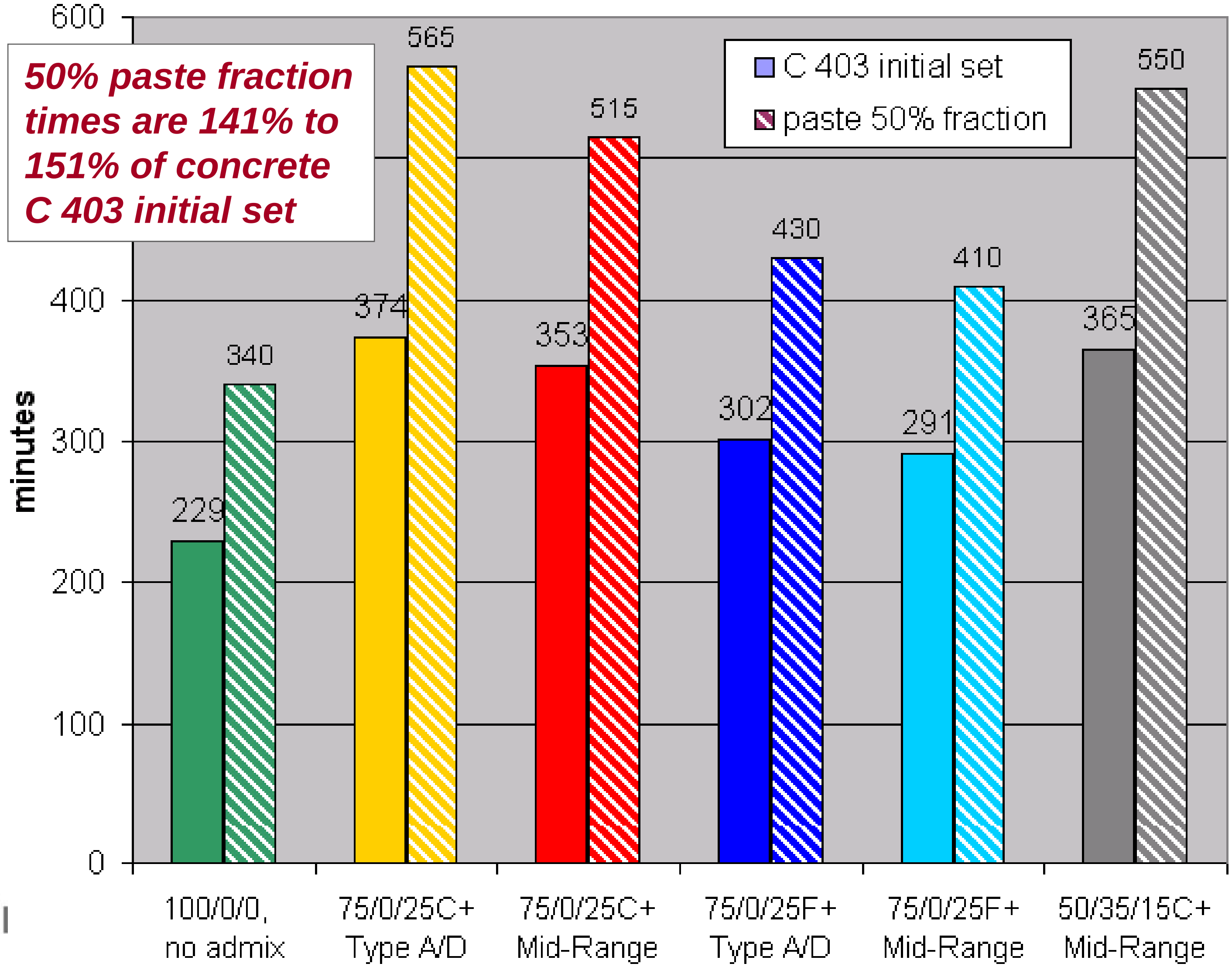
# Lab set time studies using paste mixtures



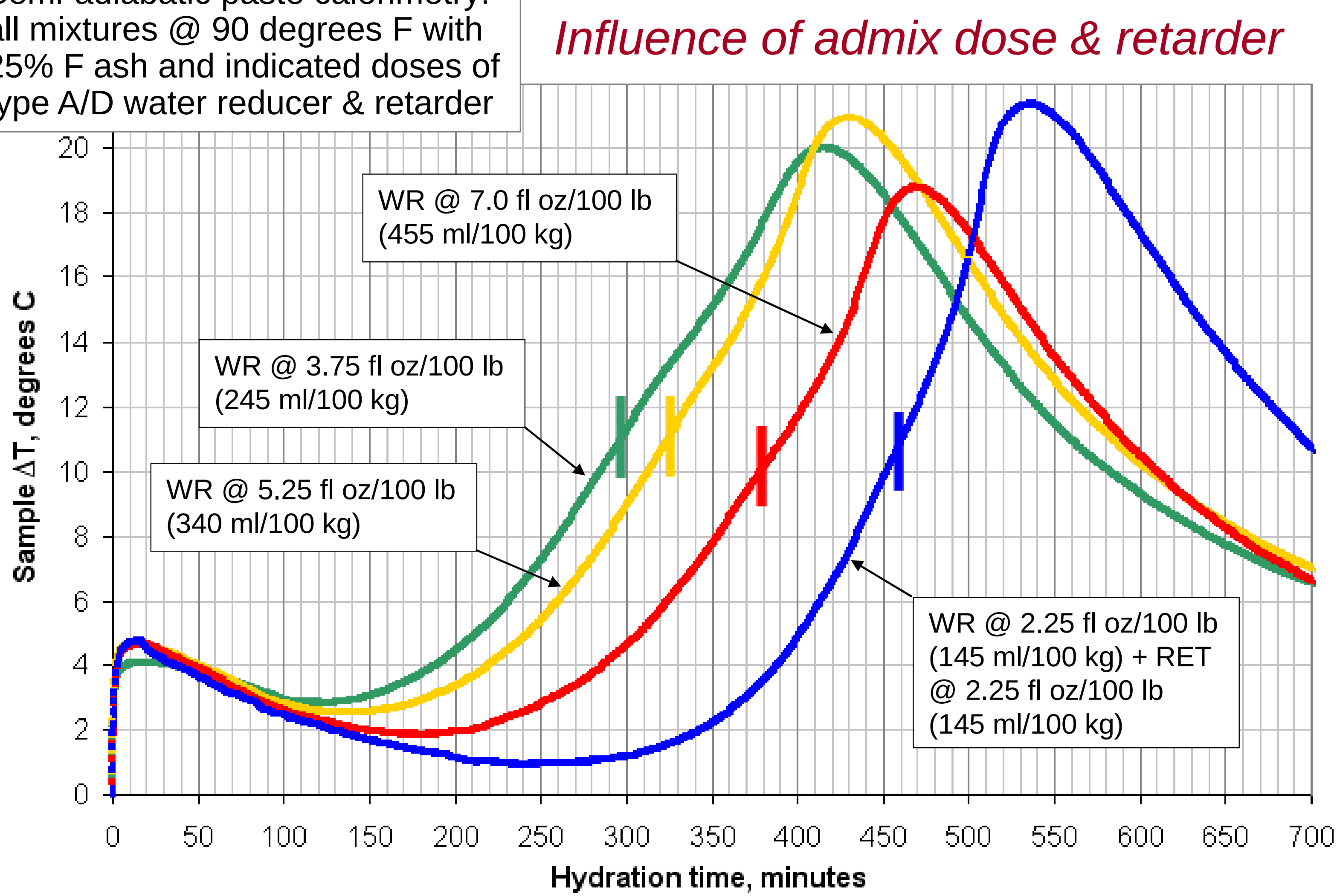
# Lab set time studies using paste mixtures

*C 403 concrete initial set times vs. 50% paste fractions*



# Relative set time example with paste SAC

Semi-adiabatic paste calorimetry: all mixtures @ 90 degrees F with 25% F ash and indicated doses of type A/D water reducer & retarder

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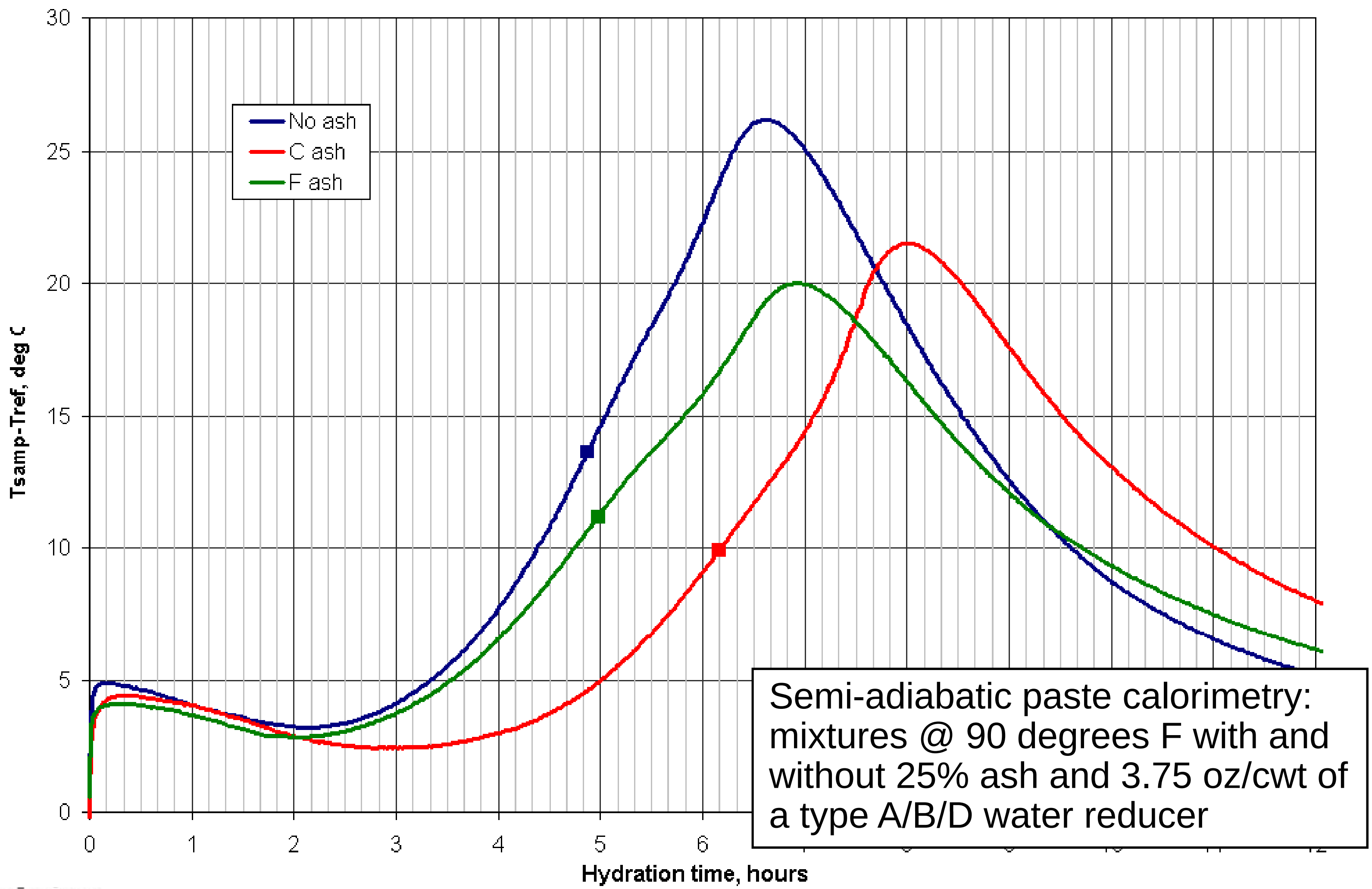
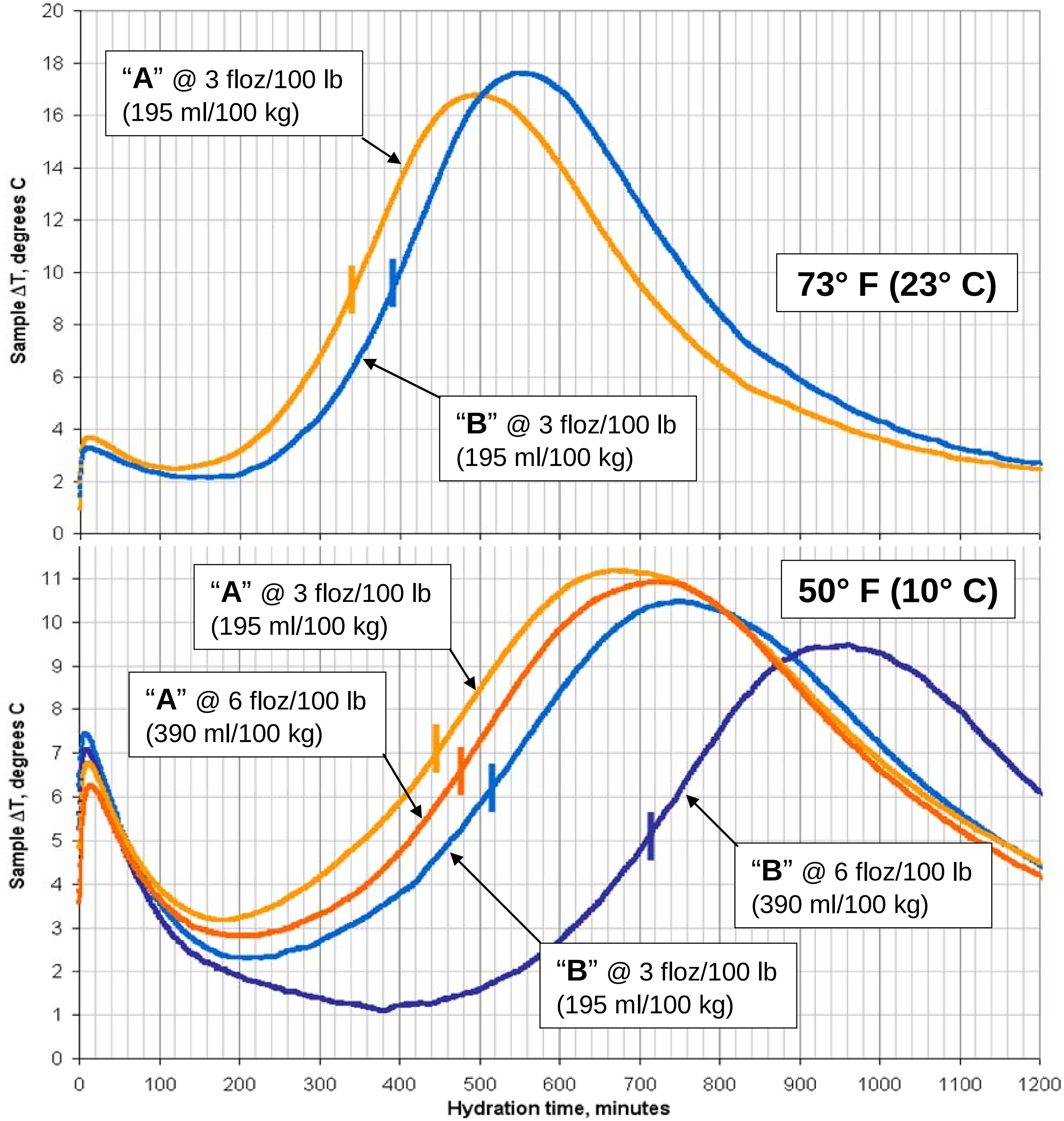
# Relative set time example with paste SAC

*Influence of Class C and Class F fly ash*




Semi-adiabatic paste calorimetry: mixtures @ 90 degrees F with and without 25% ash and 3.75 oz/cwt of a type A/B/D water reducer




# Relative set time example with paste SAC

*Two admixtures compared for temperature & dosage effects*

Semi-adiabatic paste calorimetry: 0.40 w/c cement-only mixtures @ 73 or 50 deg F, with 3 or 6 oz/cwt of 2 different candidate mid-range admixtures

# Lab paste study - admixture influences

- A series of paste mixtures done in the lab to compare the influences of 4 different water reducing admixtures
  - A single cement sample
  - 20% Class C fly ash, all mixes except controls
  - 3 different temperatures (50°, 73°, 95° F)
  - One type A/B/D, three "set neutral" mid-range admixtures:
    - Baseline dosages chosen for normal range (5-6%) water reduction
    - Higher dosages (2x baseline) included in testing, all well within dosage range recommendations

| Admixture | C 494 spec types met | contains chlorides? | recommended dosage range |
|---|---|---|---|
| ABD | A, B, D | no | 3-7 oz/cwt |
| MR1 | A | yes | 3-9 oz/cwt |
| MR2 | A | no | 3-15 oz/cwt |
| MR3 | A, F | no | 3-12 oz/cwt |

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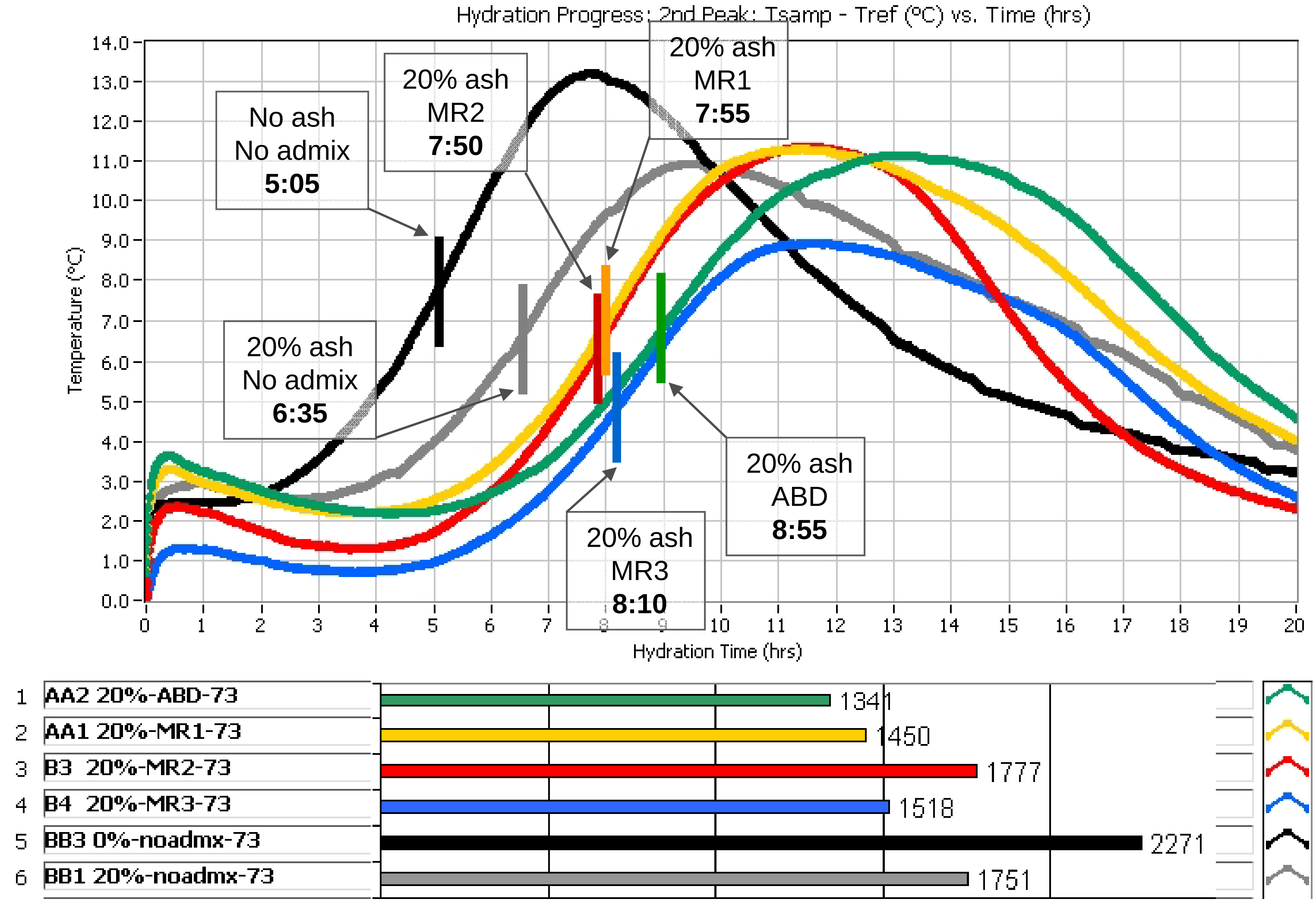
# Lab paste study - admixture influences

**Test plan - SAC paste mixtures, 3" x 4.75" cyls**

| Channel | Temp, deg F | Mix ID | w/cm | cement wt. (g) | SCM material | SCM % | SCM wt (g) | water (tap) | Admixture product | Admixture oz/cwt | Admixture ml | record mix time |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A1 | 50 | 20%-MR1-50 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR1 | 4.5 | 2.05 | |
| A2 | 50 | 20%-ABD-50 | 0.46 | 560 | C ash | 20% | 140 | 322 | ABD | 3.5 | 1.59 | |
| A3 | 50 | 20%-MR2-50 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR2 | 6 | 2.73 | |
| A4 | 50 | 20%-MR3-50 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR3 | 4 | 1.82 | |
| A5 | 50 | 20%-MR1x2-50 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR1 | 9 | 4.10 | |
| A6 | 50 | 20%-ABDx2-50 | 0.46 | 560 | C ash | 20% | 140 | 322 | ABD | 7 | 3.19 | |
| A7 | 50 | 20%-MR2x2-50 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR2 | 12 | 5.46 | |
| A8 | 50 | 50 degree reference | sand + water | | | | | | | | | |
| B1 | 50 | 20%-MR3x2-50 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR3 | 8 | 3.64 | |
| B3 | 50 | 20%-noadmx-50 | 0.46 | 560 | C ash | 20% | 140 | 322 | | | | |
| B5 | 50 | 0%-noadmx-50 | 0.46 | 700 | | | | 322 | | | | |
| AA1 | 73 | 20%-MR1-73 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR1 | 4.5 | 2.05 | |
| AA2 | 73 | 20%-ABD-73 | 0.46 | 560 | C ash | 20% | 140 | 322 | ABD | 3.5 | 1.59 | |
| B3 | 73 | 20%-MR2-73 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR2 | 6 | 2.73 | |
| B4 | 73 | 20%-MR3-73 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR3 | 4 | 1.82 | |
| AA3 | 73 | 20%-MR1x2-73 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR1 | 9 | 4.10 | |
| AA4 | 73 | 20%-ABDx2-73 | 0.46 | 560 | C ash | 20% | 140 | 322 | ABD | 7 | 3.19 | |
| AA5 | 73 | 20%-MR2x2-73 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR2 | 12 | 5.46 | |
| AA6 | 73 | 20%-MR3x2-73 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR3 | 8 | 3.64 | |
| AA8 | 73 | 73 degree reference | sand + water | | | | | | | | | |
| BB1 | 73 | 20%-noadmx-73 | 0.46 | 560 | C ash | 20% | 140 | 322 | | | | |
| BB3 | 73 | 0%-noadmx-73 | 0.46 | 700 | | | | 322 | | | | |
| AA1 | 95 | 20%-MR1-95 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR1 | 5 | 2.28 | |
| AA2 | 95 | 20%-ABD-95 | 0.46 | 560 | C ash | 20% | 140 | 322 | ABD | 4 | 1.82 | |
| AA7 | 95 | 20%-MR2-95 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR2 | 6 | 2.73 | |
| BB1 | 95 | 20%-MR3-95 | 0.46 | 560 | C ash | 20% | 140 | 322 | MR3 | 4 | 1.82 | |
| AA8 | 95 | 95 degree reference | sand + water | | | | | | | | | |

# Profiles & 1-day strengths at 73° F




Approx. 1-day paste compressive strengths, psi

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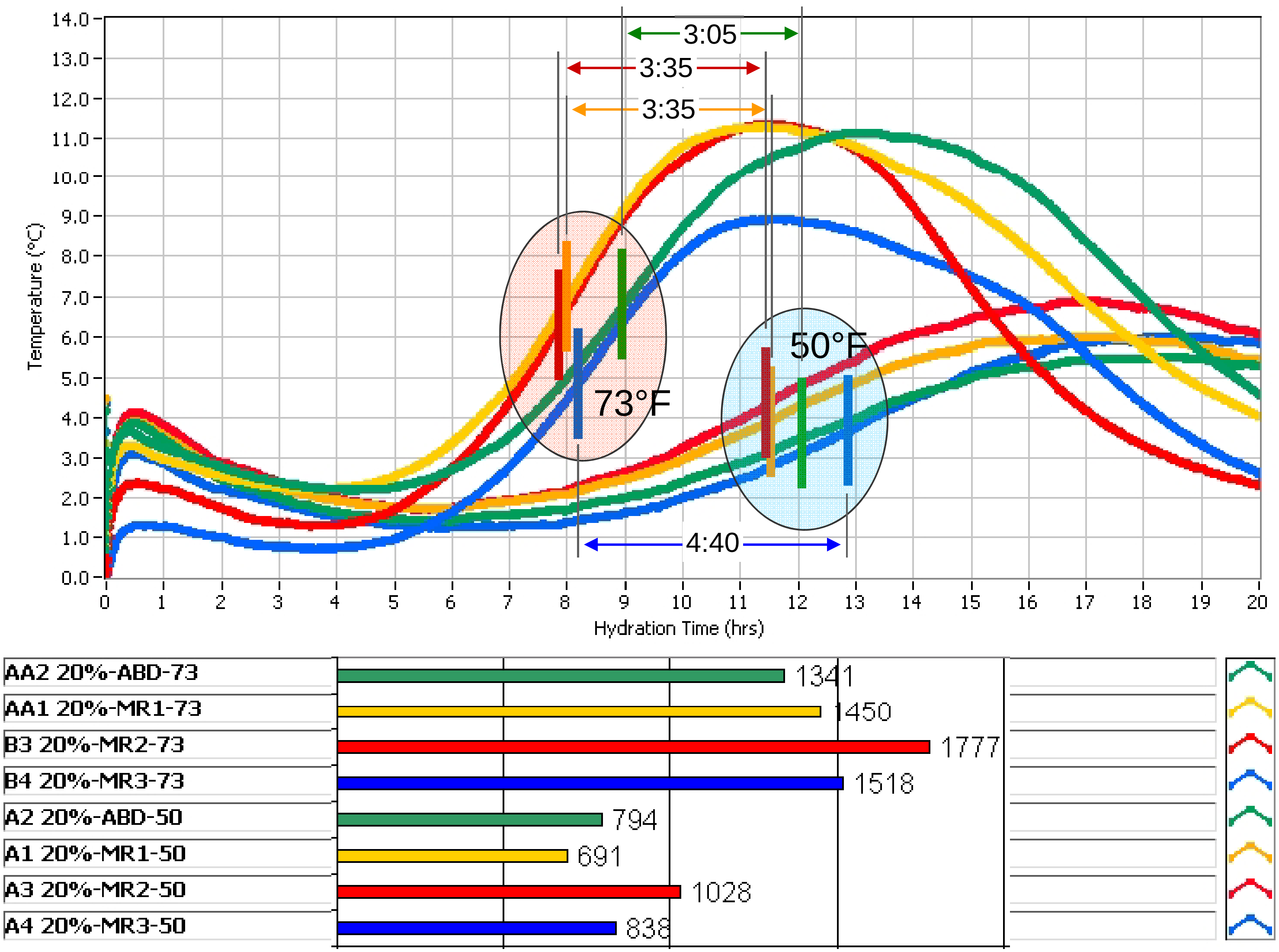
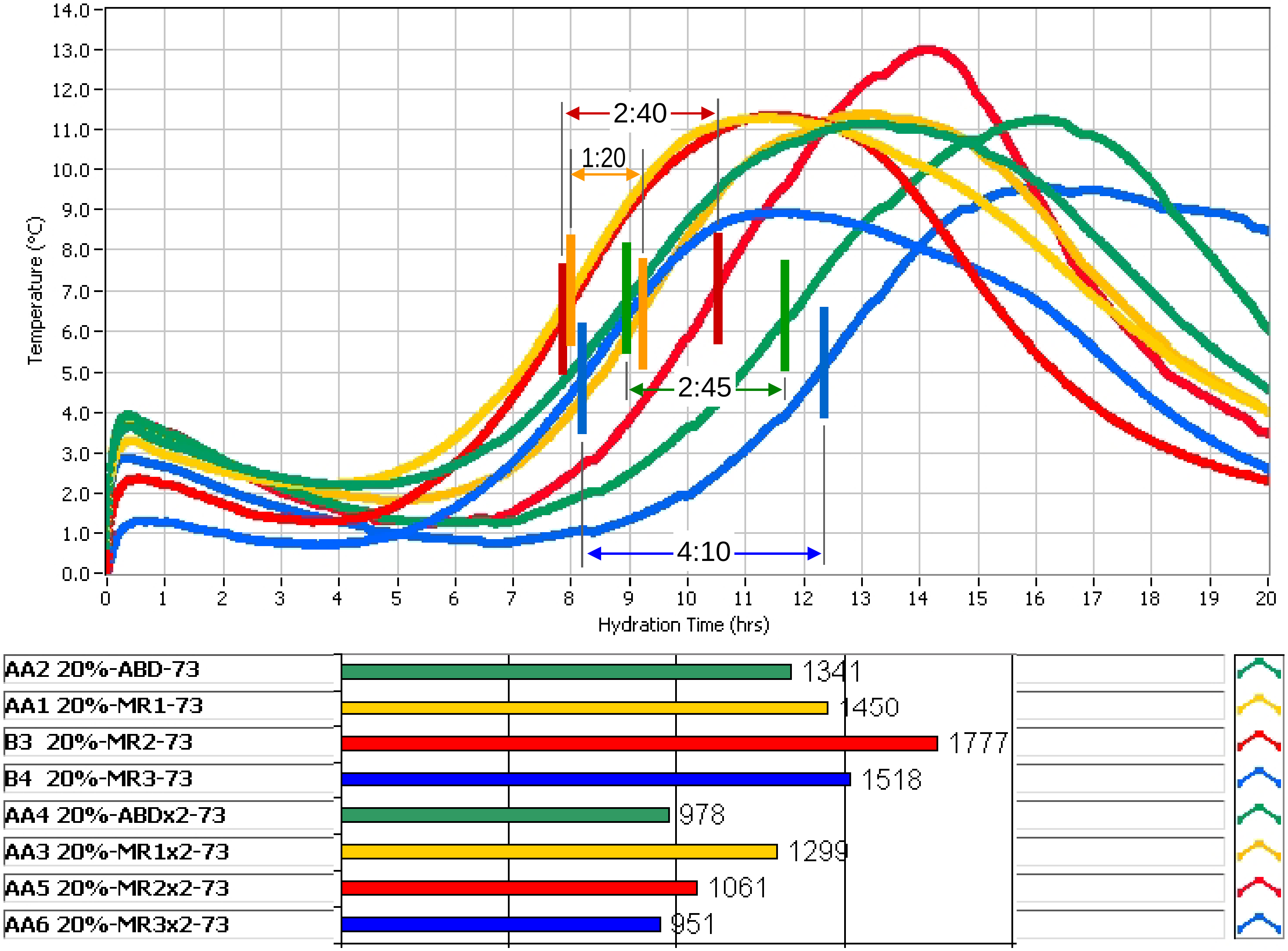
# Admixture effects compared – 73° vs. 50° F




Approx. 1-day paste compressive strengths, psi

# 2x dosage rate effects compared @ 73°




Approx. 1-day paste compressive strengths, psi

# Abnormal behavior / sulfate balance issues

## *"Incompatibility" of concrete materials*

- Responsible for certain concrete performance issues
- Cement $SO_3$ content is spec limited and typically optimized using procedures that do not account for other influences.
- Other materials and conditions affect demand for sulfates:
  - Admixtures
  - Some SCM's, especially Class C fly ash
  - Hot weather
- If early $C_3A$ hydration becomes uncontrolled due to the depletion of soluble sulfates, erratic behavior results:
  - Unpredictable (sometimes extreme) set effects and / or slump loss
  - Interrupted silicate hydration & strength gain

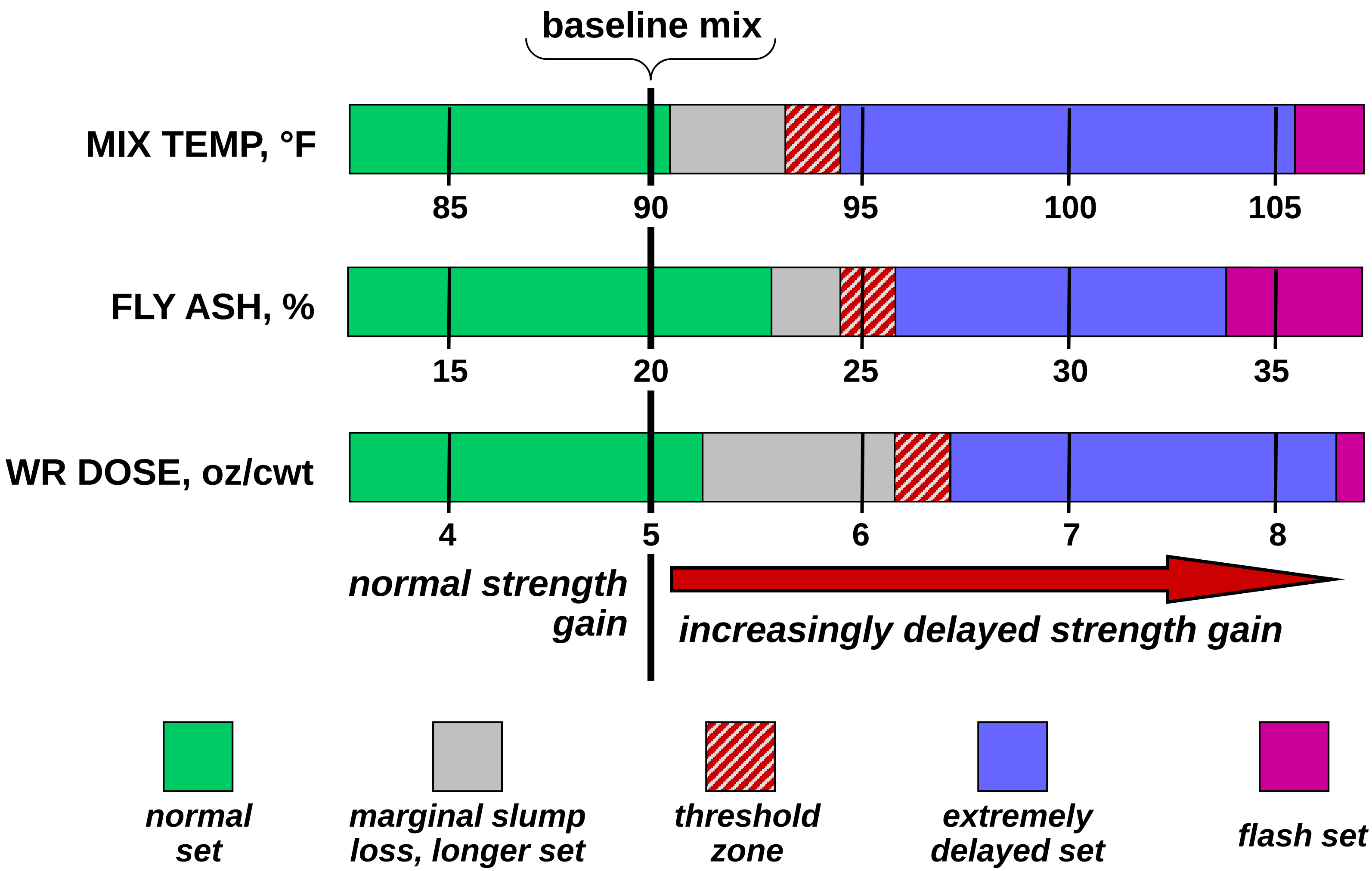
# Abnormal behavior / sulfate balance issues

*Related concrete behavior:*

- Mild influences - typically with higher temps:
  - Some increase in slump loss
  - Slight extending of set time
  - Unexpected responses to admixtures
- Nearer the incompatibility threshold:
  - Sluggish strength gain
  - More severe slump loss, extended set
- True incompatibility behavior:
  - No normal set, 24 to 48 hours or longer, or…
  - Flash set (extreme cases)
  - Interrupted strength gain for several days

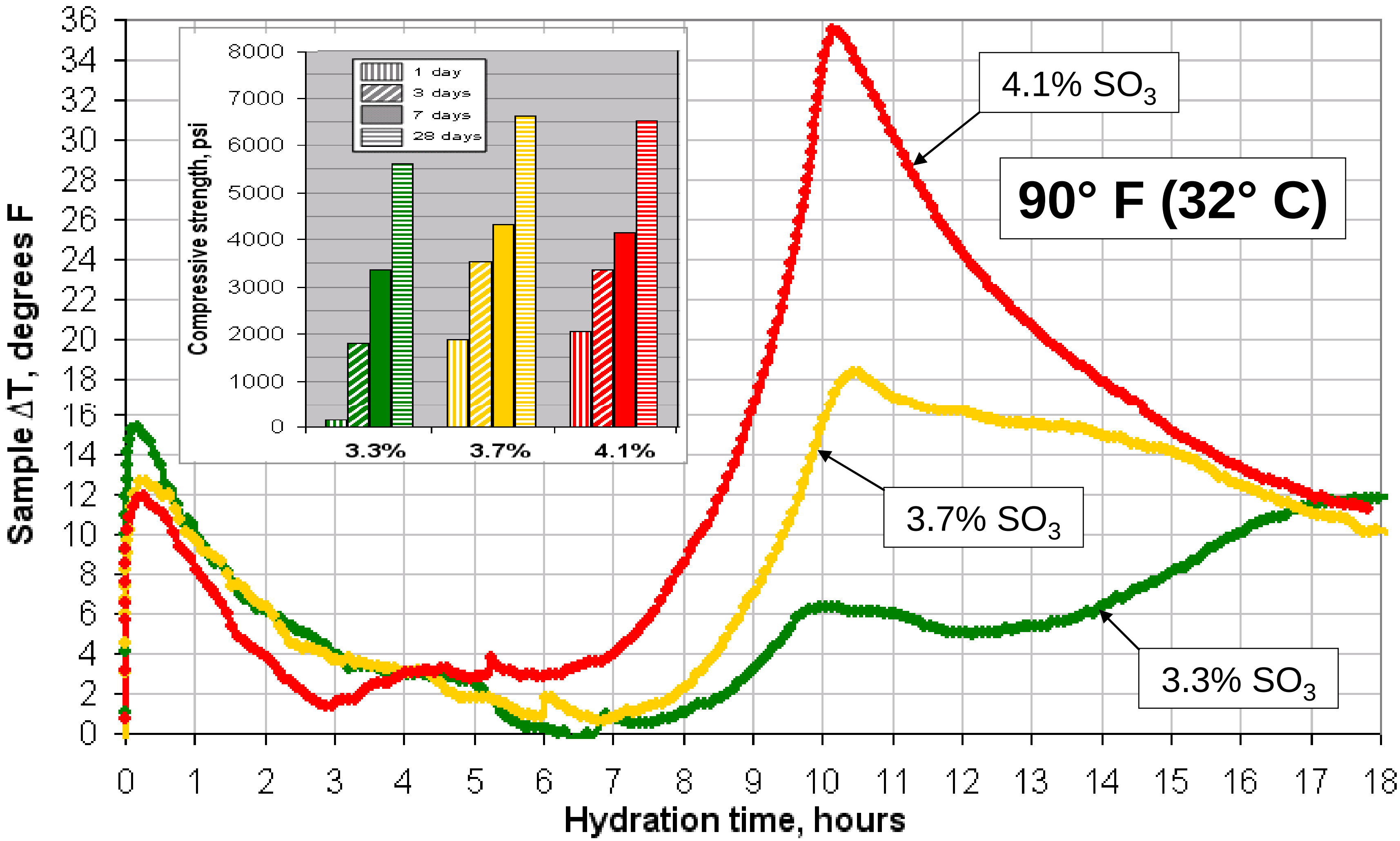
# Example: sulfate balance effects of changes in temperature, fly ash %, or water reducer dosage

“baseline” mix has 20% ash with 5 oz/cwt water reducer at 90°F



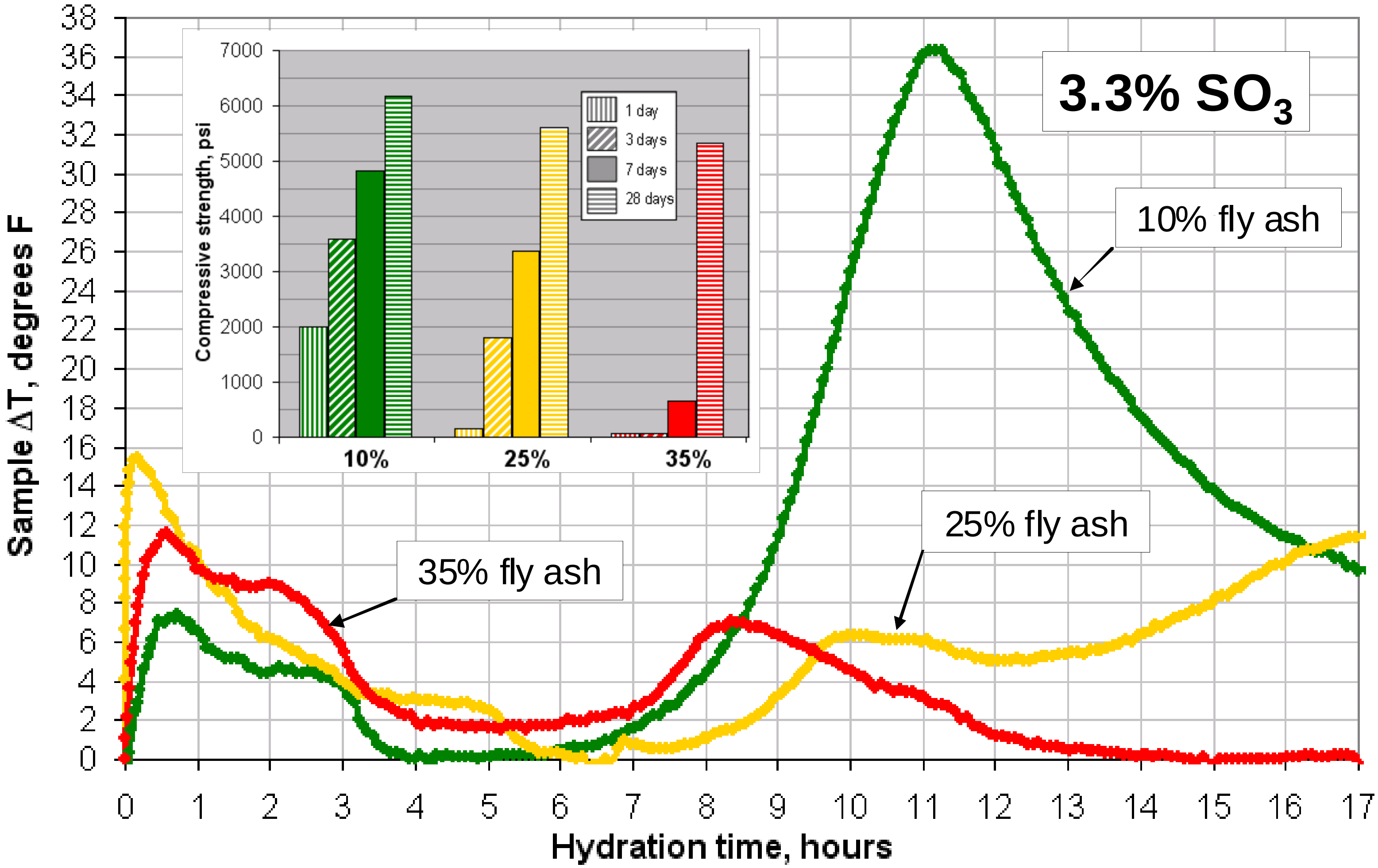
*Thermal profiles and corresponding mortar strengths for similar "on the edge" paste mixtures using 3 different cement $SO_3$ levels*

*6 oz/cwt type A/D water reducer dose, 90 degrees F initial and cure temps*



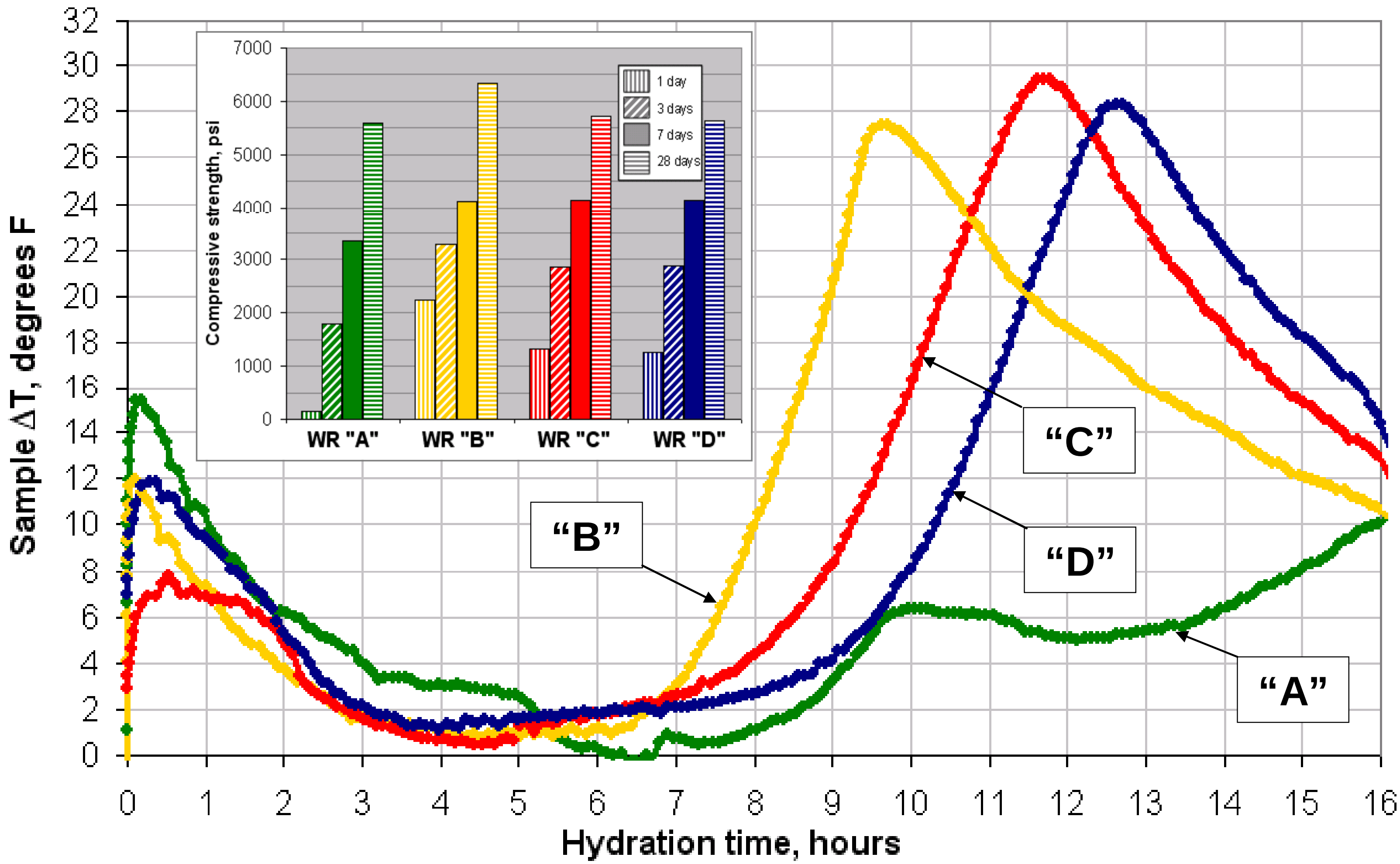
*Thermal profiles and corresponding mortar strengths for similar "on the edge" paste mixtures – fly ash replacement comparison*

*6 oz/cwt type A/D water reducer dose, 90 degrees F initial and cure temps, 3.3% $SO_3$ cement sample, Class C ash at 10%, 25%, and 35%*



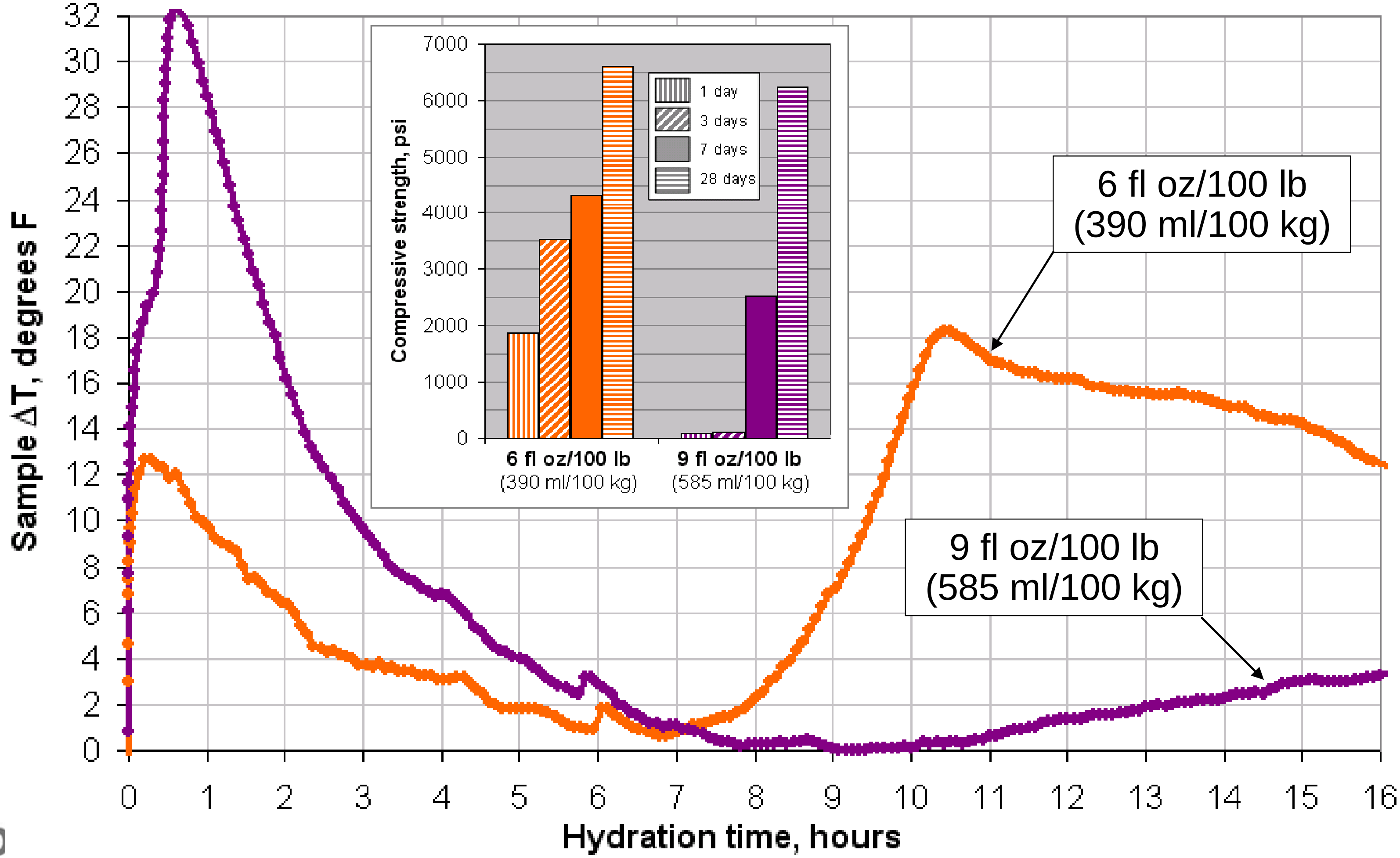
*Thermal profiles and corresponding mortar strengths for similar "on the edge" paste mixtures with 4 different type A/D admixtures*

*25% Class C fly ash replacement, 90 degrees F initial and cure temps, 3.3% $SO_3$ cement sample, admix dose @ top of recommended range*



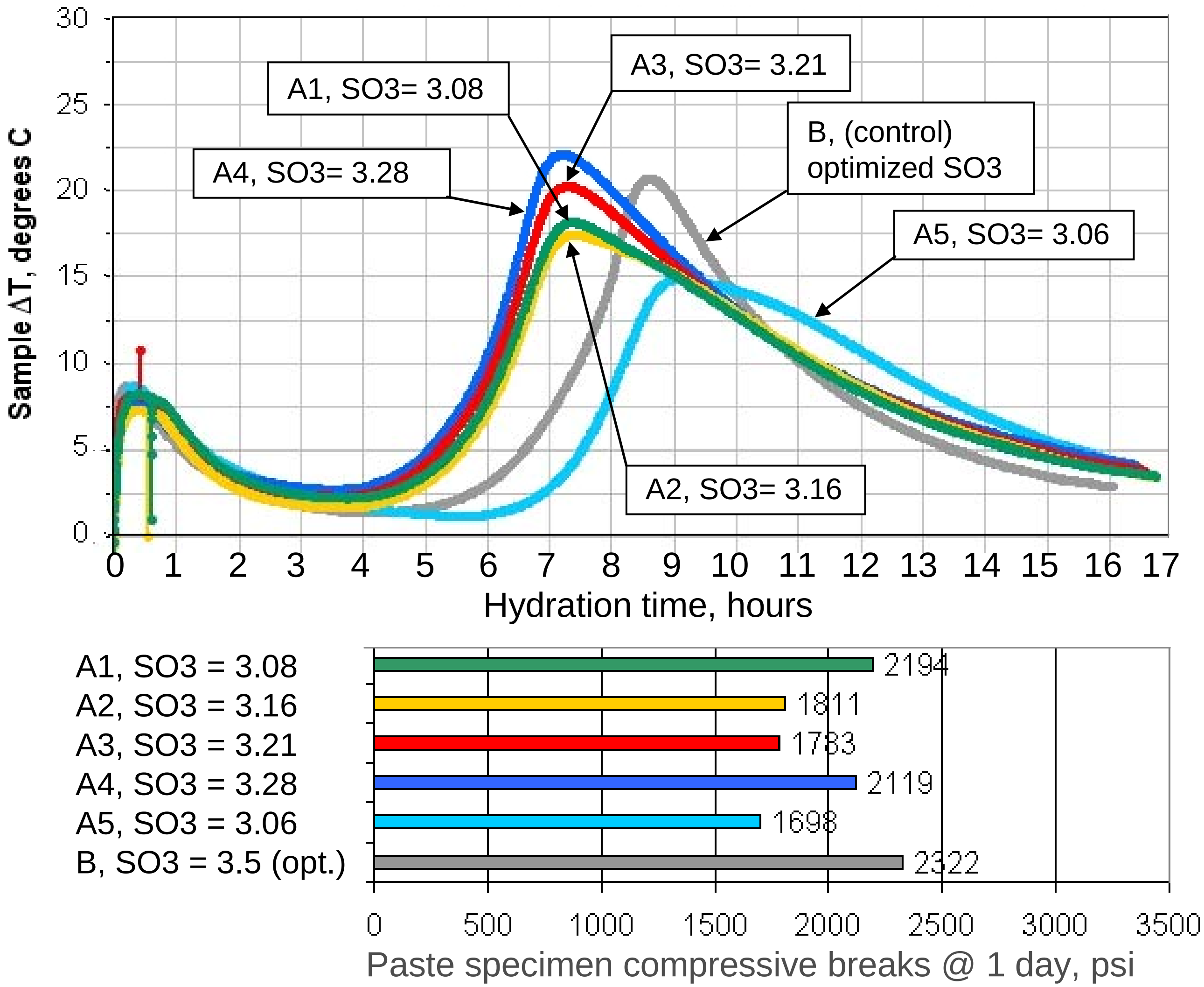
*Thermal profiles and corresponding mortar strengths for similar "on the edge" paste mixtures - admix dosage rate comparison*

*25% Class C fly ash replacement, 90 degrees F initial and cure temps, 3.7% $SO_3$ cement sample, Type A/D admixture "A" used at 6 or 9 oz/cwt*



## *Troubleshooting a field Issue – high strength building columns: 15% C ash mix, multiple admixtures, low 1-day strengths with increasing summer temps – sulfate balance issue?*

*90° F paste mixes, 4 oz/cwt admix dose, varying $SO_3$ cement samples:*

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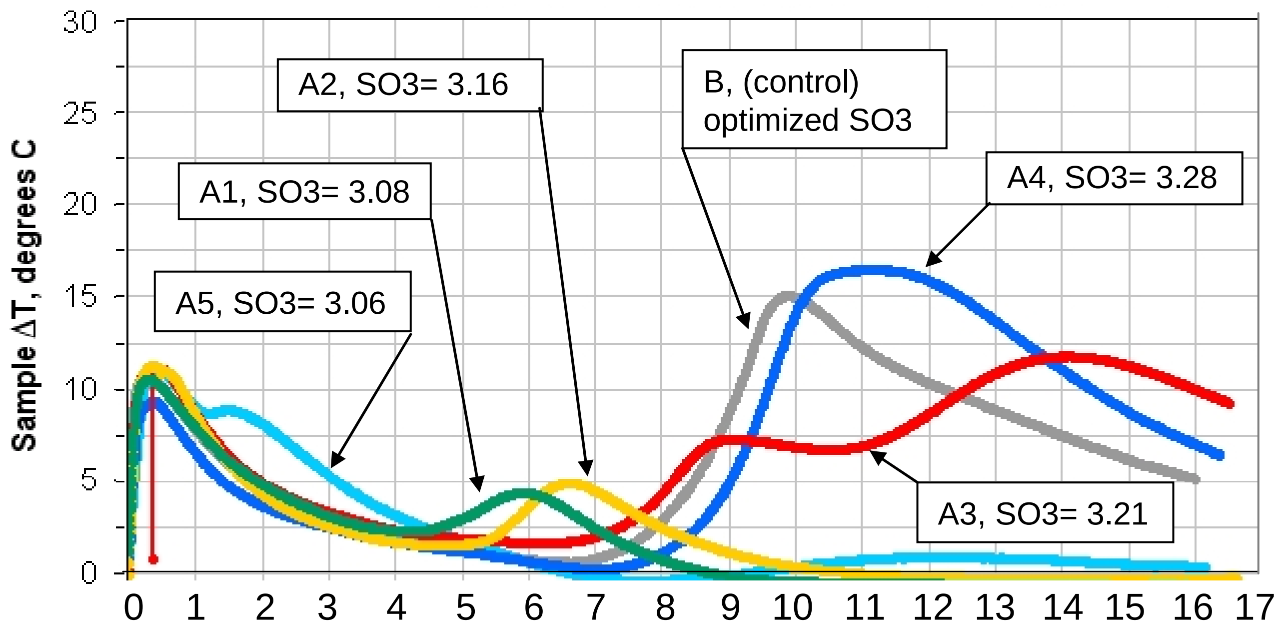
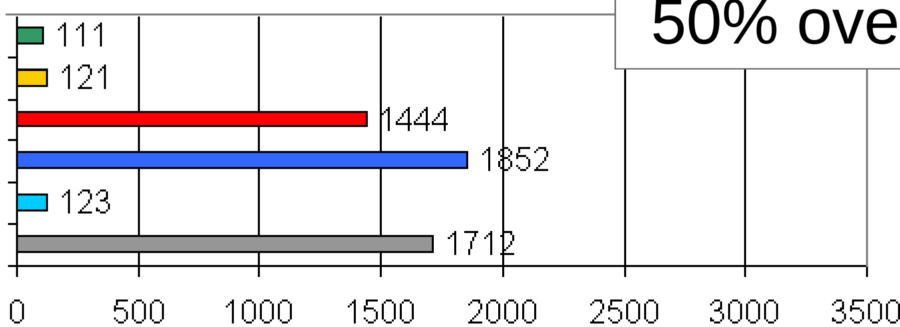
# *Troubleshooting a field Issue – high strength building columns: 15% C ash mix, multiple admixtures, low 1-day strengths with increasing summer temps – sulfate balance issue?*

*90° F paste mixes, 8 oz/cwt admix dose, varying $SO_3$ cement samples:*




***Conclusion: At the lower $SO_3$ levels, cement source "A" may be under-sulfated for use in aggressive mixes at higher temperatures.***




50% overdose

# Resolution process - sulfate balance issues

- Confirm sulfates influences with SAC mixes
  - Incremental sulfate demand approach (overdose admixtures, increase SAC %, higher mix temps)
  - Incremental sulfate supply approach (different cement samples at varied $SO_3$, sulfate additions to mixtures)
- Change one or more of the key influences:
  - Replacement rate of Class C fly ash
  - Admixture dosage or type
  - Review / evaluate retardation strategy
  - Cement $SO_3$ level
  - Mix temperatures
- Re-evaluate in the lab under the most extreme field conditions envisioned

# ASTM methods for calorimetry in the works

**Designation: X XXXX-XX**

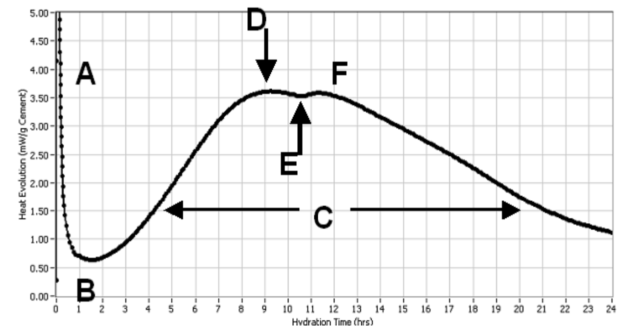
Standard practice for

**Measuring hydration kinetics of hydraulic cementitious mixtures using isothermal calorimetry**

**1. Scope**

1.1 This practice describes the apparatus and proce
hydration kinetics of hydraulic cementitious mixtures,
including those containing admixtures, various suppler
and other fine materials, by measuring the thermal pow

NOTE 1- Paste specimens are often preferred for m
individual reaction peaks are important or for particula
specimens may give results that have better correlation




X XXXX    **DRAFT**    6-23-08

Standard practice for

**Measuring hydration kinetics of hydraulic cementitious mixtures using semi-adiabatic calorimetry**

apparatus and procedure for measuring relative differences in
nentitious mixtures in paste, mortar, or concrete (Note 1),
ures, various supplementary cementitious materials (SCM),
ing temperature change over time using a semi-adiabatic
ecording equipment.

often preferred for mechanistic research when details of
rtant or for particular calorimetry configurations. Mortar or
ts that have better correlation with concrete setting and early
n preferred to evaluate different mixture proportions for

**Designation: X XXXX-XX**    **Ballot Item #1**

Standard Test Method for
**Determination of Setting Time of Concrete by the Temperature Method**[1]

This standard is issued under the fixed designation X XXXX; 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 time of setting of concrete by means of monitoring the temperature change of a concrete specimen from a representative concrete mixture

# SAC for producer QC – when to test?

- Evaluate unfamiliar materials and the proposed mix(es) under all extremes of possible project temperatures
  - Compare against controls (materials from other projects)
  - Check set time and main peak variability with temperature
- Check sensitivities of proposed materials to incompatibility
  - Test at highest expected mix temp
  - Include overdoses of admixtures and SCM's
  - Compare against known mixtures with familiar materials
- Test regularly to gage materials variability
- Test when materials sources are changed or a new mix design is first used
- Troubleshoot unexplained trends in set time, slump loss, early strengths

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# *Questions?*

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