TECH**BRIEF**

Protocol to Identify Incompatible Combinations of Concrete Materials

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This TechBrief summarizes the findings in FHWA-HRT-06-080 *Identifying Incompatible Combinations of Concrete Materials, Volume II—Test Protocol*.

The Concrete Pavement Technology Program (CPTP) is an integrated, national effort to improve the long-term performance and cost-effectiveness of concrete pavements. Managed by the Federal Highway Administration through partnerships with State highway agencies, industry, and academia, the CPTP's primary goals are to reduce congestion, reduce costs, improve performance, and foster innovation. The program is designed to produce user-friendly software, procedures, methods, guidelines, and other tools for use in materials selection, mix design, pavement design, construction, and rehabilitation of concrete pavements.

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What Is Incompatibility?

For this project, "incompatibility" of concrete materials is defined as interactions between acceptable materials that result in unexpected or unacceptable performance. The most common problems are associated with premature stiffening (rapid slump loss) and erratic setting of concrete mixtures (flash set, false set, or delayed setting and strength gain), along with increased risk of cracking and unacceptable air void systems. Proper consolidation, finishing, texturing, and curing can also be disrupted.

Uncontrolled stiffening and setting of concrete can cause serious problems with concrete pavement construction and with other types of flatwork and structures (bridge decks, for example) where timing of finishing and texturing is critical to performance. These problems may not be noticed with formed concrete structural elements as long as the concrete is workable enough to be consolidated in place; however, for both pavements and structures, rapid stiffening may lead to honeycombing and incomplete consolidation.

The aim of this project was to develop a protocol that enables users to assess whether a given combination of materials used to make concrete for pavements is likely to exhibit such incompatibility in a given environment.

Many mechanisms and effects contribute to incompatibility. The mechanisms are complex and interrelated, and often they are temperature related. This means there is no simple method of reliably measuring the risk of incompatibility. Some test methods are suitable for indicating the risk of problems in the first 30 minutes because of aluminate and sulfate balance issues. Other tests are suitable for detecting later silicate hydration problems, while still other tests are useful for assessing other signs of distress. No test method is ideal for everything.

This protocol has been developed to provide as much information as possible during the preconstruction phase, including calibration of the more sensitive central laboratory tests with the equivalent field tests using materials likely to be used in the field and under environmental conditions likely to be experienced in the field. The work may also include preparing alternative mix proportions and practices to accommodate changes in either the environment or in the source of the materials. Field tests could be based on the more rugged tests that are regularly conducted, primarily to monitor the uniformity of the materials and the final mixture.

Most of the tests conducted in this work have some value; the extent of preconstruction and field testing need to be based on equipment availability and the relative cost of testing compared to the cost from the risk of failures. A typical example is with determining the setting time, which can be measured by up to six different techniques, any of which are acceptable. Selecting from among these different techniques should be based on other project requirements and conditions.

A relatively simple suite of field tests, conducted regularly, can provide reassurance that the concrete mixture is performing satisfactorily or warn of undesirable variability or potential incompatibility. The following tests make up this protocol:

- Foam index.
- Foam drainage.
- Unit weight.
- Slump loss.
- Semiadiabatic temperature monitoring.
- Setting time.
- Chemistry of reactive materials.

What Has Changed?

Concrete systems, those used for paving mixtures as well as structural concrete, are progressively more complex and capable of greater performance. More types of materials, more complex material combinations, and tighter deadlines and tolerances, all at extremes of temperature, can mean that concrete mixtures are less forgiving.

What Is Happening?

The chemistry of concrete systems is complex, and a basic understanding of the reactions occurring in the systems is essential in applying the protocol. Hydraulic cementitious systems stiffen, set, and harden by a process called hydration, which is a series of nonreversible chemical reactions with water.

Two aluminate compounds, C_3A and C_4AF, are present in portland cement. C_4AF does not contribute significantly to system performance; however, C_3A reacts rapidly when mixed with water and generates a large amount of heat (figure 1) unless the reaction is controlled by the presence of sulfate. If the reaction of C_3A with water is uncontrolled because there is insufficient sulfate in solution for the amount of C_3A involved, then flash (or permanent) set can occur.

Calcium sulfate is added to cement as gypsum ($C\bar{S}H_2$) during grinding to control the initial reaction of C_3A. During grinding, some of the gypsum is dehydrated to form plaster ($C\bar{S}H_{1/2}$). The amount of dehydration is controlled by the manufacturer to provide optimum performance of the cement; however, if the amount of dehydration is incorrect, then false (temporary) set can occur.

Use of a fly ash containing C_3A may result in flash set or rapid stiffening because of insufficient sulfate to control its hydration.

Some type A water-reducing admixtures also may influence the balance between C_3A and sulfates because they tend to accelerate C_3A hydration. Likewise, increasing temperatures accelerate the chemical reactions and also increase the risk of uncontrolled stiffening if marginally balanced materials are in use. Other contributors to potential risks are very finely ground cements, high alkali content in the system, and very low water-to-cementitious materials ratios.

All of these reactions and changes occur within the first 15 to 30 minutes after mixing, which has implications for concrete paving that uses nonagitating transporters. Even when agitators or truck mixers carry the concrete to the paver, the delivery time may be so short that there may not be an opportunity to work through a false set if it occurs. On longer deliveries for structures or flatwork, early stiffening may be less evident, but it may cause the addition of excessive water to the concrete delivered in a truck mixer.

One of the hydration products of the silicates (C_2S and C_3S) in cement is calcium silicate hydrate (CSH), which is the primary contributor to concrete strength, durability, and the heat of hydration (figure 1). The silicates start to react 2 to 4 hours after mixing, when calcium reaches supersaturation in the mix solution. These reactions lead to setting and strength gain. If too much calcium has been consumed during earlier, uncontrolled C_3A reactions, then setting may be delayed. In addition, the same type A water-reducing admixtures that accelerate C_3A reactions may retard silicate reactions, also potentially adding to the delay. Low temperatures also will slow the hydration process.

It is possible that both accelerated C_3A (uncontrolled stiffening) and delayed silicate reactions (delayed setting) can occur in the same mix.

The effects of rapid stiffening on paving will be a mixture that may be delivered with an acceptable workability, but will stiffen up in the paving machine, leading to poor consolidation and difficulties with finishing and texturing.

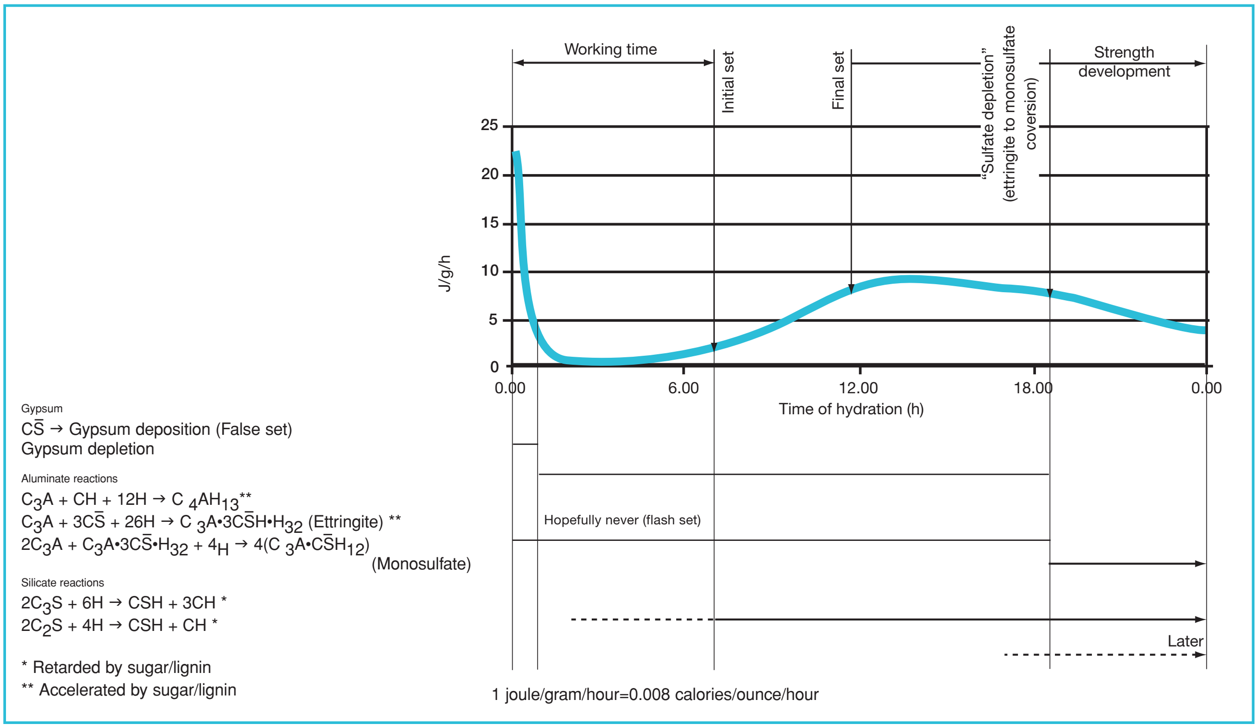


Figure 1: Reactions that occur in hydrating cement, the times they occur, the heat they generate, and the effects on stiffening and setting.

Delayed setting significantly increases the risk of plastic shrinkage cracking, and makes it difficult to get the saw-cutting of joints completed at the right time. For concrete delivered in truck mixers, more water may have to be added before discharge for either paving or structural applications.

How Do I Prevent These Problems?

Although there is no silver bullet for preventing these problems, careful evaluation of the materials before construction starts will indicate potential problems and help develop guidelines on what to change if problems occur. The evaluation tests should be conducted over the range of likely temperatures and using the range of likely materials quantities and dosage rates of admixtures.

The aim of conducting preconstruction tests is to evaluate the sensitivity of the proposed system to variations in materials composition and in the environment. This will allow for selection of alternative materials in advance or for preparation of action plans to be implemented if such changes are observed in the field. The work also will provide calibration between field- and laboratory-based tests, and give guidance on the limits appropriate for the materials likely to be used and conditions likely to be encountered.

Before any physical tests are conducted, a review of the chemistry of the reactive materials is recommended. Fine cementitious materials with high C_3A or low sulfate contents, or both, may be at risk, as will fly ashes with high calcium oxide contents. Sugar and triethanolamine-based water-reducing admixtures may increase the risk of problems, especially if the concrete is to be placed at elevated temperatures.

Paste- and mortar-based laboratory tests, including the minislump test and the American Society for Testing and Materials (ASTM) C 359 mortar stiffening test, indicate whether aluminate-based incompatibilities are occurring. Tests flagging silicate reaction problems in paste and mortar include parallel-plate rheology, setting time, and isothermal calorimetry. If the paste and mortar tests indicate potential problems, then concrete mixtures should be made and tested for slump loss, semiadiabatic temperature curve, and setting time.

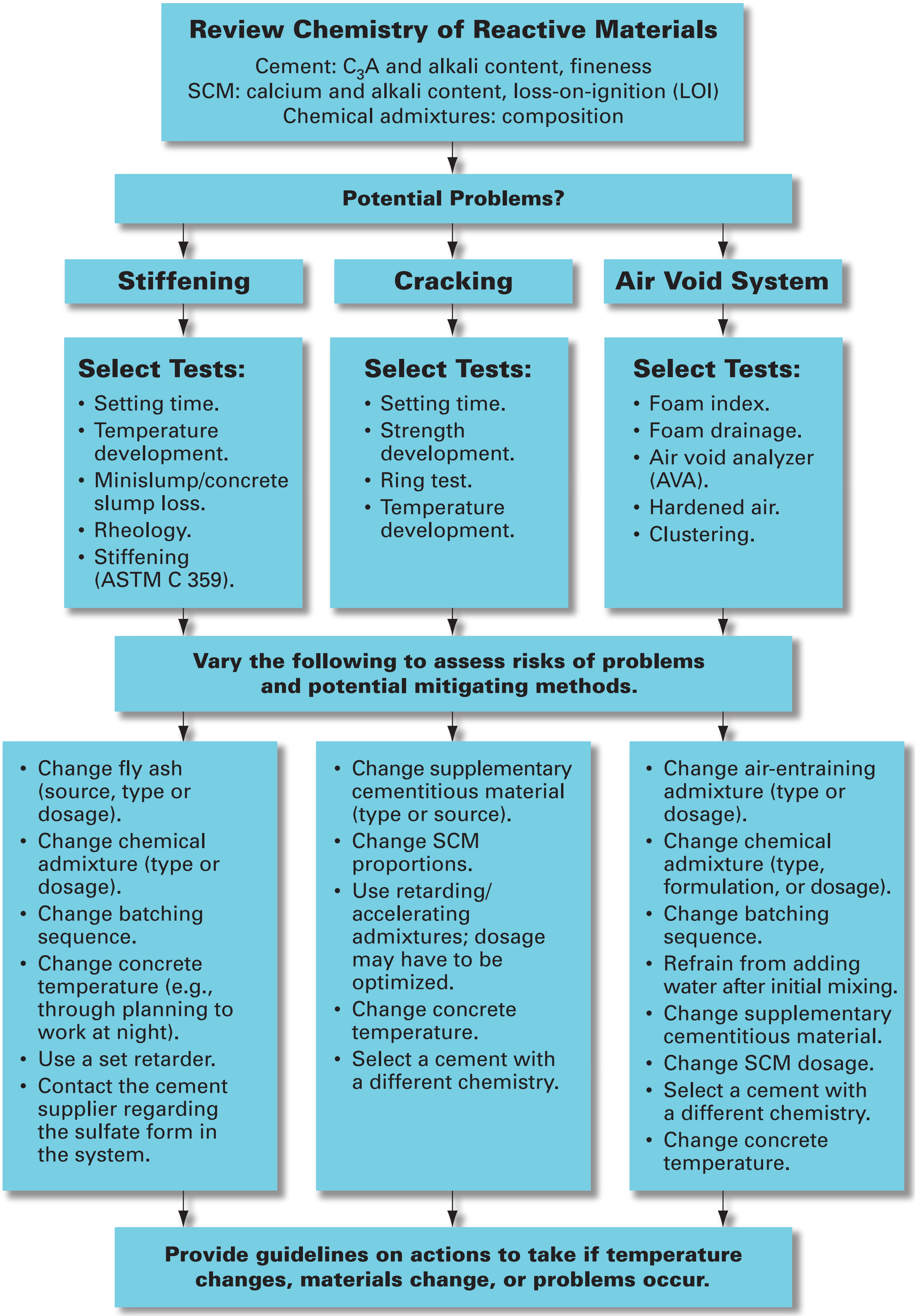
If problems are still likely in the field, then adjust any of the following: supplementary cementitious material (SCM) type, source, or quantity; chemical admixture type or dosage; batching sequence; and mix temperature. If time and budget allow, a series of mixtures can be run to indicate the range of variability that can be accommodated. The best corrective action can then be implemented when field problems occur or appear likely.

Field tests during construction should aim to confirm that the materials being delivered are uniform and similar to those used in the preconstruction tests. Significant variations as indicated by control charts should flag that the mixture is performing in the same way that it has previously, and that changes to mix proportions or construction practices may be necessary.

Field tests would include monitoring chemical reports for the delivered reactive materials, measuring and tracking for concrete slump, slump loss at different times after mixing, semiadiabatic temperature curve, and setting time. These results then can be compared with the preconstruction data and monitored for drift.

Not all of these tests are available in all laboratories, and some are more expensive than others. The decision about which suite of tests to run is largely governed by the balance of costs and risks. A large, high-profile project with significant penalties is going to require more tests than a small urban repair.

The protocol is summarized in the following flow charts (figures 2 and 3). The charts also address potential incompatibilities that may be exhibited as excessive cracking, or problems with the air void system.



Review Chemistry of Reactive Materials

Cement: C_3A and alkali content, fineness
SCM: calcium and alkali content, loss-on-ignition (LOI)
Chemical admixtures: composition

Potential Problems?

Stiffening

Select Tests:

- Setting time.
- Temperature development.
- Minislump/concrete slump loss.
- Rheology.
- Stiffening (ASTM C 359).

Cracking

Select Tests:

- Setting time.
- Strength development.
- Ring test.
- Temperature development.

Air Void System

Select Tests:

- Foam index.
- Foam drainage.
- Air void analyzer (AVA).
- Hardened air.
- Clustering.

Vary the following to assess risks of problems and potential mitigating methods.

- Change fly ash (source, type or dosage).
- Change chemical admixture (type or dosage).
- Change batching sequence.
- Change concrete temperature (e.g., through planning to work at night).
- Use a set retarder.
- Contact the cement supplier regarding the sulfate form in the system.

- Change supplementary cementitious material (type or source).
- Change SCM proportions.
- Use retarding/accelerating admixtures; dosage may have to be optimized.
- Change concrete temperature.
- Select a cement with a different chemistry.

- Change air-entraining admixture (type or dosage).
- Change chemical admixture (type, formulation, or dosage).
- Change batching sequence.
- Refrain from adding water after initial mixing.
- Change supplementary cementitious material.
- Change SCM dosage.
- Select a cement with a different chemistry.
- Change concrete temperature.

Provide guidelines on actions to take if temperature changes, materials change, or problems occur.

Figure 2: Protocol flow chart, preconstruction stage.

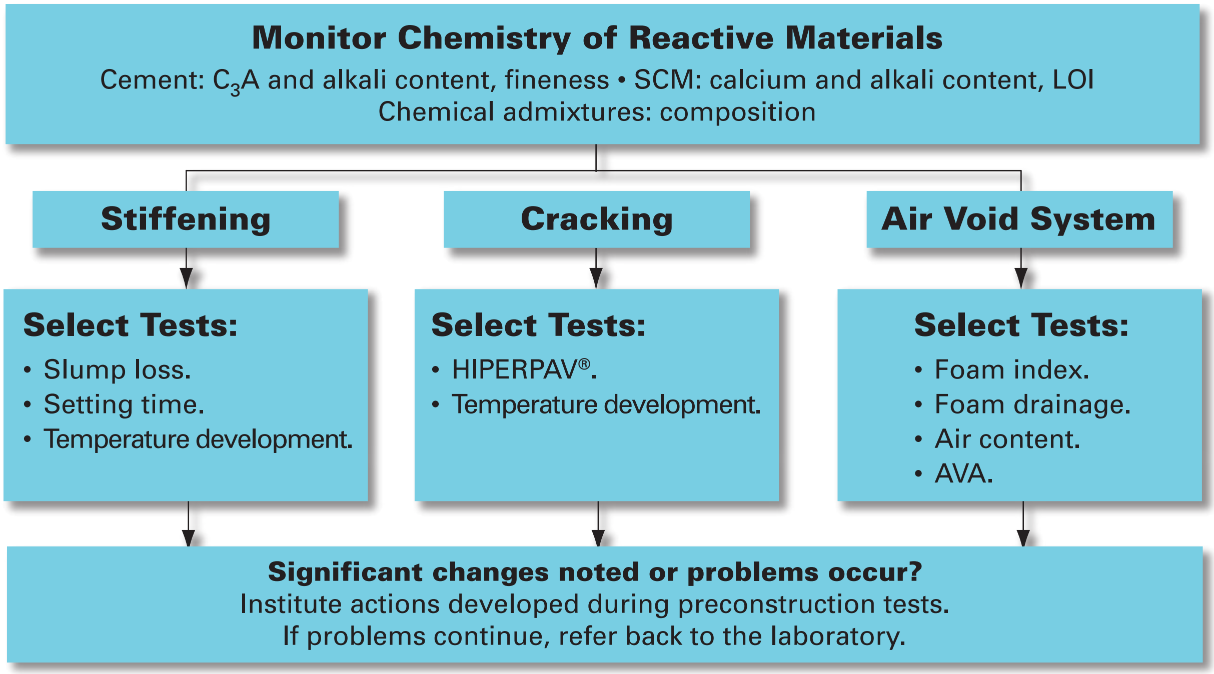


Figure 3: Protocol flow chart, construction stage.

Notes**:**

- Tests should be selected based on what is locally available, the value of the project, and the risk or consequences of failures occurring.
- Setting time may be monitored using any one of a number of techniques including vicat, penetrometer, calorimetry, temperature monitoring, and P-wave.

Researchers—This study was performed by CTLGroup, Skokie, IL.

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Availability—The report *Identifying Incompatible Combinations of Concrete Materials, Volume II—Test Protocol* (FHWA-HRT-06-080), which is the subject of this TechBrief, will be available in July 2006. A printed copy of the report is available at the FHWA Product Distribution Center by e-mail to report.center@fhwa.dot.gov, by fax to 301–577–1421, or by phone to 301–577–0818. An electronic copy is available at the Turner-Fairbank Highway Research Center Web site. To download the report, go to www.tfhrc.gov.

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