

ALKALI CONTENT OF FLY ASH: MEASURING AND TESTING STRATEGIES FOR COMPLIANCE

**Final Report
April 2015**



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IOWA STATE UNIVERSITY
Materials Analysis and Research Laboratory

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16. Abstract <p>Sodium and potassium are the common alkalis present in fly ash. Excessive amounts of fly ash alkalis can cause efflorescence problems in concrete products and raise concern about the effectiveness of the fly ash to mitigate alkali-silica reaction (ASR). The available alkali test, which is commonly used to measure fly ash alkali, takes approximately 35 days for execution and reporting. Hence, in many instances the fly ash has already been incorporated into concrete before the test results are available. This complicates the job of the fly ash marketing agencies and it leads to disputes with fly ash users who often are concerned with accepting projects that contain materials that fail to meet specification limits.</p> <p>The research project consisted of a lab study and a field study. The lab study focused on the available alkali test and how fly ash alkali content impacts common performance tests (mortar-bar expansion tests). Twenty-one fly ash samples were evaluated during the testing. The field study focused on the inspection and testing of selected, well documented pavement sites that contained moderately reactive fine aggregate and high-alkali fly ash. A total of nine pavement sites were evaluated. Two of the sites were control sites that did not contain fly ash.</p> <p>The results of the lab study indicated that the available alkali test is prone to experimental errors that cause poor agreement between testing labs. A strong (linear) relationship was observed between available alkali content and total alkali content of Class C fly ash. This relationship can be used to provide a quicker, more precise method of estimating the available alkali content. The results of the field study failed to link the use of high-alkali fly ash with the occurrence of ASR in the various concrete sites. Petrographic examination of the pavement cores indicated that Wayland sand is an ASR-sensitive aggregate. This was in good agreement with Iowa DOT field service records. It was recommended that preventative measures should be used when this source of sand is used in concrete mixtures.</p>			
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EXECUTIVE SUMMARY

Sodium and potassium are the common alkalis present in fly ash. Excessive amounts of fly ash alkalis can cause efflorescence problems in concrete products and raise concern about the effectiveness of the fly ash to mitigate alkali-silica reaction (ASR). Fly ash marketing agencies occasionally provide materials that just miss the criteria for alkali content given in Iowa DOT IM 491.17. Since usage is only from an approved list (certified sources) this leads to disputes that can be difficult to resolve. This is especially problematic when the alkali content of a given source of fly ash only changes by a small amount, but the change causes the source to cross a specification limit. This immediately prompts the marketing agency to question the precision of the alkali determinations and/or the validity of the specification limit. In addition, some power stations add small amounts of various chemicals to the raw coal or flue gas in an attempt to comply with EPA emission regulations. Some of these chemicals can have a significant impact on the alkali content of the fly ash.

The research plan consisted of a literature survey, a lab study and a field study. The literature survey researched upcoming changes in environmental regulations and how they might impact the use of fly ash. The lab study focused on the available alkalis test and how fly ash alkali content impacts common performance tests (mortar bar expansion tests). The field study focused on the inspection and testing of selected, well documented pavement sites that contained moderately reactive fine aggregate and high-alkali fly ash from Ottumwa Generating Station (OGS).

The literature survey indicated that several new (or proposed) environmental regulations will influence how coal-fired power plants operate in the United States. The major impacts will be due to the implementation of the Mercury and Air Toxics Standards (MATS) and the Clean Power Plan (CPP). MATS retrofits are currently in progress and will be completed by about 2016. The future of the CPP rule is currently uncertain, but the rule is expected to be finalized in June 2015. However, legal challenges are expected and this could delay the effective date of the rule.

The lab study found that the available alkalis test was prone to several experimental errors that lead to poor agreement between testing labs. The time required for obtaining test results also seriously restricts the applicability of the test when trying to control the quality of materials going to jobsites. The Iowa DOT has already dealt with these issues and uses a total alkali model to predict the available alkalis. The validity of the Iowa Department of Transportation (Iowa DOT) model was checked using 32 Class C fly ashes because no standard reference materials for available alkali content currently exist. The model predicted values were $\pm 0.2\%$ of the measured available alkali contents for samples that ranged from 1 to 2.5% (as Na_2O_e). The model was within $\pm 0.4\%$ for values that ranged from 2.6 to 5% (as Na_2O_e); however, data was very limited in this range.

Mortar-bar expansion tests conducted for the lab study indicated that performance can be improved (lower measured expansions) by using more fly ash (higher replacement levels), a fly ash containing a higher value of the sum-of-the-oxides (more siliceous), and blended

cements. In this study, the Type IP cement performed the best of the three cements that were studied. The available alkali content of the fly ash generally did not produce the best correlations to measured expansions; this was especially true if one was allowed to change fly ash replacement level.

The field study focused on the evaluation of nine pavement sites for the presence of ASR-related distress. The field sites were selected to study a variety of concrete material combinations. Prior Iowa DOT testing had indicated that some of the sites contained materials that could be considered ASR-sensitive (sites 6 and 7). Each site was subjected to a shoulder survey to evaluate surface distress and pick core locations. Cores were extracted from the sites and subjected to petrographic examination. The findings can be summarized as follows.

- Pavement site 1 (I-80 weigh station, Jasper County) did not exhibit significant distress. The site had a low entrained-air content but an adequate spacing factor. Pop outs were noted on many slabs and this cracking was also noted in the petrographic examinations. However, the shale particles that caused the pop outs were not considered active ASR sites. Hence, this control section looked good after 22 years of service.
- Pavement site 2 (Highway 163, Polk County) exhibited sporadic joint distress that required some patching. The site had a low entrained-air content and a poor spacing factor. Pop outs were noted on many slabs and this feature was also noted in the petrographic examinations. The shale particles that caused the pop outs were not considered active sites of ASR. The joint distress noted at this site was most probably caused by freeze-thaw attack.
- Pavement site 3 (Highway 92, Warren County) exhibited frequent joint distress that required some patching. The site had a marginal entrained-air content and a marginal spacing factor. In addition, the coarse aggregate (Ames Mine limestone) contained some brown particles and some anomalous chert particles that exhibited cracking. The brown particles were enriched in dolomite, and the cracking appeared to be much more prevalent in the cores extracted from the joints. The fine aggregate also contained some ASR sensitive particles (shale, hydrous chert) and ASR gel was noted lining some voids and cracks. However, there were few observed particles that exhibited active ASR (i.e., cracked particles with active cracks extending significantly into the paste). Hence, the distress noted at this site was most probably initiated by freeze-thaw attack and now the process has become more complex (ASR contributing as long as moisture is present).
- Pavement site 4 (Highway 61, Lee County, south-bound at MP 15) appeared to be a region of poor concrete that lasted about 1000 yards. The site had a good entrained air content and a good spacing factor. The cores exhibited signs of poor consolidation. Also, distress was noted in a few specific fine aggregate particles (shale and chert) with active cracks extending into the paste. The site is experiencing cracking caused by ASR; however, the deterioration seems too sporadic to only be attributed to this distress mechanism. The pavement had only been in service for 13 years, so it appears that something else is also playing a role at this site.

- Pavement site 5 (Highway 61, Lee County, north-bound at MP 17) was a section of pavement that contained OGS fly ash, continental cement and Wayland sand. The site had a marginal entrained-air content but a good spacing factor. No significant distress was noted at this site; however, reactive aggregate particles were noted in the sand (shale and chert). Since the site had been in service for 15 years, distress from ASR was expected but not observed at this site.
- Pavement site 6 (Highway 61, Lee County, north-bound at MP 5) was a section of pavement that had been overlaid with asphalt in 2006 because of distress. This site was considered a control site because it did not contain fly ash. The site had a good entrained-air content and a good spacing factor. The coarse aggregate contained an anomalous hydrous chert particle (may have been oversize from the sand) and the fine aggregate contained many reactive particles. The cracking patterns noted in the cores suggested a mixed mode of distress. The fine aggregate exhibited ASR distress (and was producing large amounts of gel); however, the distress also appeared to have a freeze-thaw component (sub-horizontal, simply extending cracks near the top of the core).
- Pavement site 7 (Highway 61, Lee County, north-bound at MP 7) was a section of pavement that had been overlaid with asphalt in 2006 because of distress. This site contained Louisa fly ash in addition to the same materials used in site 6. The site had a good entrained-air content and a good spacing factor. The coarse aggregate was sound, but the fine aggregate contained many reactive particles. The cracking patterns noted in the cores were different from those noted in site 6. The fine aggregate exhibited ASR distress (and was producing large amounts of gel near the reactive particles nearly throughout the depth of the sample); however, the sub-horizontal cracking was not noted at this site. Therefore, the most probable explanation for distress at this site is ASR-induced cracking.
- Pavement site 8 (Highway 61, Lee County, south-bound at station 538) was a section of pavement that was selected because of distress (spall near joint). The site had a marginal entrained-air content and a marginal spacing factor. The coarse aggregate was sound, but the fine aggregate contained many reactive particles. This site has distress related to freeze-thaw and ASR.
- Pavement site 9 (Highway 61, Lee County, north-bound at station 536) was a section of pavement that was selected because it contained OGS fly ash. No surface distress was noted at this site. The site had a low entrained-air content and a marginal spacing factor. The coarse aggregate was sound, but the fine aggregate contained many reactive particles. The cracking pattern noted in the cores was primarily sub-horizontal to the top of the core and very near the top surface. This cracking pattern is common to freeze-thaw distress. The fine aggregate exhibited ASR distress and was also producing gel near the top of the sample. Hence, the distress was primarily related to freeze-thaw attack.

INTRODUCTION

Problem Statement

Sodium and potassium are the common alkalis present in fly ash. Excessive amounts of fly ash alkalis can cause efflorescence problems in concrete products and raise concern about the effectiveness of the fly ash to mitigate alkali-silica reaction (ASR). Fly ash marketing agencies occasionally provide materials that just miss the criteria for alkali content given in Iowa DOT IM 491.17. Since usage is only from an approved list (certified sources) this leads to disputes that can be difficult to resolve. This is especially problematic when the alkali content of a given source of fly ash only changes by a small amount but the change causes the source to cross a specification limit. This immediately prompts the marketing agency to question the precision of the alkali determinations and/or the validity of the specification limit. In addition, some power stations add small amounts of various chemicals to the raw coal or flue gas in an attempt to comply with EPA emission regulations. Some of these chemicals can have a significant impact on the alkali content of the fly ash.

Background

Excessive amounts of alkalis in cementitious materials often correlate to poor field performance of concrete products. Cement alkali content has a profound impact on the performance of concrete products and cement producers make extraordinary efforts to control alkali content within specific ranges at any specific cement plant. This is done by controlling raw materials and manufacturing processes in an attempt to control the amount of alkalis in the finished cement. In contrast, fly ash is not a material that is manufactured within specific limits; rather, it is a by-product from the burning of pulverized coal in coal-fired power stations. Hence, the alkali content of fly ash is not controlled (it is what it is) and fly ash marketing agencies attempt to measure and document the alkali content using quality assurance (QA) programs. Unfortunately, the alkali content can only be controlled by changing to a source of fly ash that has lower (or higher) alkali content.

The available alkali test is commonly used to estimate the amount of fly ash alkali that will be “available” for reacting with concrete aggregates. The test can be classified as a “reactive” rather than a “proactive” test because it takes approximately 35 days for execution and reporting. Hence, in many instances the fly ash has already been incorporated into concrete before the test results are available. This complicates the job of the fly ash marketing agencies and it leads to disputes with fly ash users who often are concerned with accepting projects that contain materials that fail to meet specification limits. Part of the confusion arises from the poor between-lab precision of the available alkali test method (Schlorholtz 2006). For critical projects interlaboratory tests may be needed to provide a better estimate of the allowable discrepancy between test results. This leads to additional work and, because of the 35 day lag between sampling and reporting, it still fails to catch any rapid changes in the alkali content of the fly ash caused by changes in power plant operating conditions.

Fly ash from Ottumwa Generating Station (OGS) has been subjected to several studies since that plant started producing ash in the early 1980's (Bergeson et.al. 1988; Schlorholtz and Bergeson 1993). The power station routinely adds sodium carbonate to the raw coal feed to meet EPA opacity regulations. It has also run full scale co-combustion burns that employed switchgrass as the supplementary fuel (Schlorholtz and Stapp 2005). An example of the alkali content (available alkali content, expressed in terms of Na_2O_e) is given in Figure 1. Figure 1 illustrates that OGS commonly produces a fly ash with an available alkali content exceeding the 1.5% limit (AASHTO) and may, on occasion, exceed the 2.5% limit (Iowa DOT).

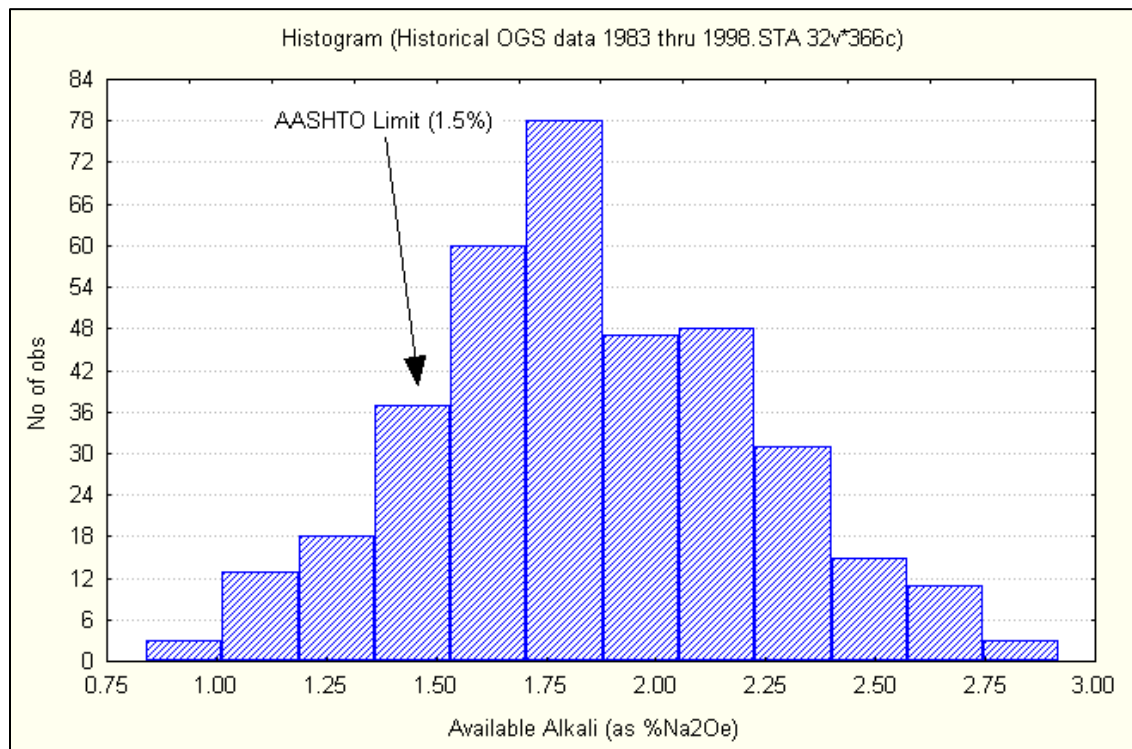


Figure 1. Histogram of available alkali content of OGS fly ash from 1983 to 1999.

Objectives

The objectives of this research project were:

- Determine if and at what content level fly ash with soda dosing has increased potential for alkali silica reactivity (ASR) and any other potential performance impacts in the concrete; both during mixing and placing as well as long-term.
- Evaluate field concrete containing high-alkali fly ash and moderately reactive fine aggregate to see if ASR-related distress has occurred.
- Determine a better method for determining available alkali in fly ashes with soda dosing. The method should be relatively simple and rapid. A specification content limit or performance level should be determined and recommended.

- Perform a literature review and/or a survey to determine if there are other materials and methods for emission control that may impact Midwest power plants and their fly ash chemistry.

Research Plan and Scope

The research plan consisted of a lab study and a field study. The lab study focused on the available alkali test and how fly ash alkali content impacts common performance tests (mortar bar expansion tests). The field study focused on the inspection and testing of selected, well documented pavement sites that contained moderately reactive fine aggregate and high-alkali fly ash from OGS.

The scope of the project was limited to the examination of fly ash sources that are commonly used by the Iowa DOT. Special attention was given to OGS fly ash. Occasionally data from earlier reports or the literature was also used to help extend arguments about how fly ash alkali influences the performance of concrete materials.

Three cements and a single source of slag were primarily used during this project. This was done to limit the physical testing to a testing matrix that could be accommodated by the size of the research project. Briefly, the first cement was a Type I/II cement. The second cement was used for ASR mortar-bar tests (total alkali content of 0.75% as per Iowa DOT IM 491.17), this was a mixture of two type I cements. The third cement was a blended cement (Type IP).

Mortar-bar expansion tests were conducted in accordance with Iowa DOT IM 491.17 (Pyrex glass aggregate). After all of the fly ashes were tested with the ASR cement, a subset of eight fly ashes was selected for testing with the blended cements. In addition, limited studies were conducted using mortar-bar expansion tests containing Wayland sand aggregate (potentially ASR-sensitive). These tests included the rapid mortar-bar expansion test (ASTM C 1567) and the standard mortar-bar expansion test (ASTM C 227).

Nine pavement sites were evaluated for ASR distress. Pavement shoulder surveys were conducted and cores were extracted from the sites. Materials incorporated in specific pavements should have exhibited some ASR reaction (based on Iowa DOT testing or prior field service records). Two pavement sites were selected that did not contain fly ash, these acted as control sections.

LITERATURE REVIEW

Fly ash has been used in concrete construction projects for many years; and hence, the literature is extensive on this topic (Davis et al. 1937; Abdun-Nur 1961; Lea 1970). In contrast, the literature review conducted for this study was quite limited. It focused on four basis topics pertaining to ASR mitigation by fly ash. First, it investigated the test methods that are currently used for evaluating fly ash performance – basically the history and scope of the test methods were of primary importance. Second, it investigated the creation of specification limits for the test results (evaluated for both Iowa and nationally). Third, it quickly detailed the continuing conundrum of prescriptive limits versus performance limits. Finally, it summarized a quick review of how impending legislation may impact the use of fly ash in portland cement concrete products.

Test Methods Available for Fly Ash

The main test methods used for fly ash are given in ASTM C 311, Standard Methods for Sampling and Testing Fly Ash and Natural Pozzolans for Use in Portland-Cement Concrete. The primary test methods of interest to this project included the “Available Alkali” test and the “Effectiveness of Fly Ash or Natural Pozzolan in Controlling Alkali-Silica Reactions.”

The test methods summarized in ASTM C 114, Test Methods for Chemical Analysis of Hydraulic Cement, are commonly used to determine the alkali content of cement. The method for total alkalis is predicated on the very high solubility of cement in mineral acids. This allows for a quick and effective method of putting all of the alkalis into solution for analysis using flame emission techniques (ICP, atomic absorption, etc.). As other rapid methods of chemical analysis improved their sensitivity to light elements (for example, X-ray fluorescence (XRF)), they have also been used to determine the total alkali content of cement. The test method determines the total amount of sodium (expressed as Na_2O) and potassium (expressed as K_2O) present, and then, for the sake of simplicity, the results are commonly expressed in terms of *equivalent* sodium oxide ($\text{Na}_2\text{O}_e = \% \text{Na}_2\text{O} + 0.658 \times \% \text{K}_2\text{O}$). An additional test procedure is also available for quantifying the amount of water-soluble alkalis in cement.

Fly ash also contains alkalis. However, the acid solubility of the alkalis is significantly different than portland cement. Hence, additional test methods have been constructed to measure the alkali content of fly ash. For fly ash one can determine the total alkali, available alkali and soluble alkali contents. The test methods for available alkali and total alkali are given in ASTM C 311. At the present time there is not a standard test method for determining the water-soluble alkali content of fly ash. The details of the various alkali tests are described below. Some of the sections contain a rather large amount of synthesis; however, they were included in this review section because the information has been available for some time (decades). Hence, the information is not new it simply is presented so that the reader has a firm understanding of the test methods and potential problems with the test methods.

The Available Alkali Test

The available alkali test dates back to the 1940's. Information is sketchy but it appears that the method was developed at the US Bureau of Reclamation (Moran and Gilliland 1950; Mielenz 1967). The test procedure was created to measure the rate of release of alkali from pozzolans (and fly ashes). The method was eventually adopted by ASTM to estimate the amount of alkali in pozzolans that was "available" for contributing to ASR. The test method is briefly described below (see Table 1). The current test method (as per ASTM C 311) is also included in the table. The two test methods are similar but they do contain some significant differences. First, the pozzolan/lime ratio is higher (2.5 versus 2.0) in the ASTM C 311 method. This has an impact on the amount of alkali that will be released from the pozzolan (Buttler et.al., 1981); and hence, one would expect that the ASTM method would solubilize less alkali. Secondly, the basic details (glassware requirements or dilutions) have changed by about a factor of two (original method produced more concentrated filtrate than the C 311 method). This discrepancy may be related to improvements in the sensitivity of the measuring system (flame photometers). Do note that the procedural details listed in ASTM 311 have remained relatively constant since the method was adopted in 1957. In addition, the calibration standards and general calibration procedure have also remained relatively constant since that time.

Table 1. Summary of the test procedures for determining available alkali

Method as per Moran & Gilliland	Method as per ASTM C 311-11
Weigh 5.0 grams of pozzolan	Weigh 5.0 grams of pozzolan
Weigh 2.5 grams of calcium hydroxide	Weigh 2.0 grams of calcium hydroxide
Add enough water to fill vial (14mL vial)	Add 10.0mL water (25mL plastic vial)
Mix thoroughly	Mix thoroughly
Cure @ 100°F (38°C) for 28 days	Cure @ 100°F (38°C) for 28 days
Transfer to 250mL beaker	Transfer to 250mL casserole
	Grind to a fine paste
Add 100mL deionized water	Adjust total volume to 200mL with water
Allow to stand for 30 minutes	Allow to stand for 60 minutes
Stir occasionally	Stir frequently
Filter/wash into 250mL volumetric flask	Filter into 500mL volumetric flask
	Wash 8 to 10 times with hot water
	Titrate filtrate with 1:3 HCl to 8.2 endpoint
Acidify filtrate (10mL of 1:10 HCl)	Acidify filtrate (5mL of 1:3 HCl)
Analyze filtrate with flame photometer	Analyze filtrate with flame photometer (or more modern method such as ICP or AA)
	Standards prepared as per ASTM C 114 but with 504ppm CaO in matrix

A formal precision and bias statement for the available alkali test has never been provided in ASTM C 311. Considerable information about the precision of the test method can be obtained from the Cement and Concrete Reference Laboratory Pozzolan Proficiency Sample Program (CCRL-PPSP). The CCRL-PPSP has now completed testing on 56 samples (54 samples of fly ash and a pair of natural pozzolans). The program has provided a great service to labs that test fly ash. Prior to the inception of the CCRL-PPSP, only fly ash marketing agencies and a few large user labs (for example, the Waterways Experiment Station) generally collaborated on fly ash testing. Today about 60 labs routinely participate in the program.

The CCRL-PPSP is a two-sample interlaboratory testing program (Youden 1959). Hence, it produces information about variability of test results between labs ($1s_b$). However, with a few assumptions the test results can also be used to estimate the within-lab variability ($1s$) for the various tests that are conducted in fly ash labs (Youden 1959b, 1961). The determinations of interest to this study pertain to the available alkali test. As was described earlier in this report, the available alkali test result is calculated by combining two separate measurements: available sodium (as %Na₂O) and available potassium (as %K₂O). To provide a single number on a uniform basis the potassium is converted to an equivalent amount of sodium by multiplying by the appropriate gravimetric factor (0.658). Recent results for samples 55 and 56 (both Class C fly ashes) of the CCRL-PPSP will be used to illustrate typical test results.

The scatter plots for the available potassium, available sodium and available alkali are given in figures 2, 3 and 4, respectively. All three plots indicate that systematic errors (bias) are present in the determinations because the test results plot in an elongated ellipse along the 45-degree diagonal (most points in the upper-right or bottom-left quadrants). This indicates that labs that produce high test results on one sample are prone to producing high test results on both samples. Likewise, labs that produce low test results tend to produce low test results on both samples. This is bias in the test method and it makes it difficult to create specification limits for the test method that are concise and meaningful. If random error were to dominate the determinations then the scatter plot would have had a circular shape (roughly equal points in all four quadrants). These plots suggest that it may be a very difficult task to provide robust and meaningful precision estimates for the available alkali test method. Because the problems could be associated with this particular set of samples a larger number of samples was evaluated. For that reason, all of the CCRL-PPSP available alkali test results were tabulated and a subset was extracted for further analysis.

The entire table of available alkali test results is given in Appendix A. The samples from the last 14 years (2001 through 2014) were extracted from the main table and analyzed separately because this allows direct comparison to another test method (total alkali as sodium and potassium, see the next section). This gave a total of 14 sample pairs (for a total of 28 samples). The final subset was relatively balanced and contained 11 Class C fly ashes, 15 Class F fly ashes and two natural pozzolans. Two of the Class F fly ashes were retested with different cements (see CCRL 41 and 42 from 2007, which were repeated as CCRL 43 and 44 in 2008). The precision statement was drafted as per ASTM C 670, Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials.

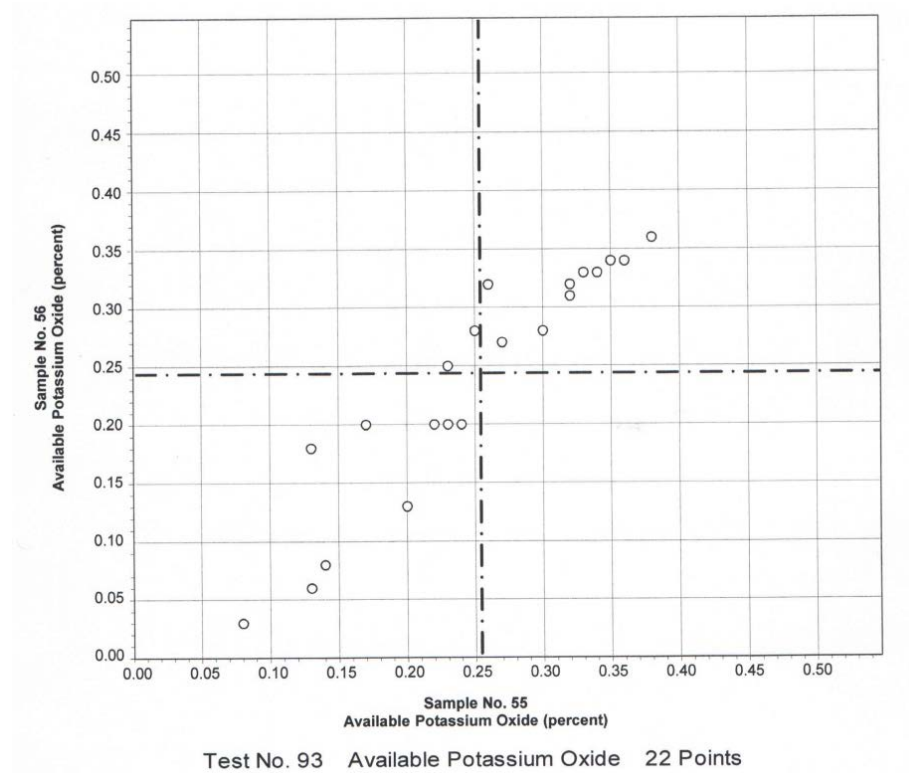


Figure 2. Available potassium content for CCRL 55 and 56 (Haupt 2014)

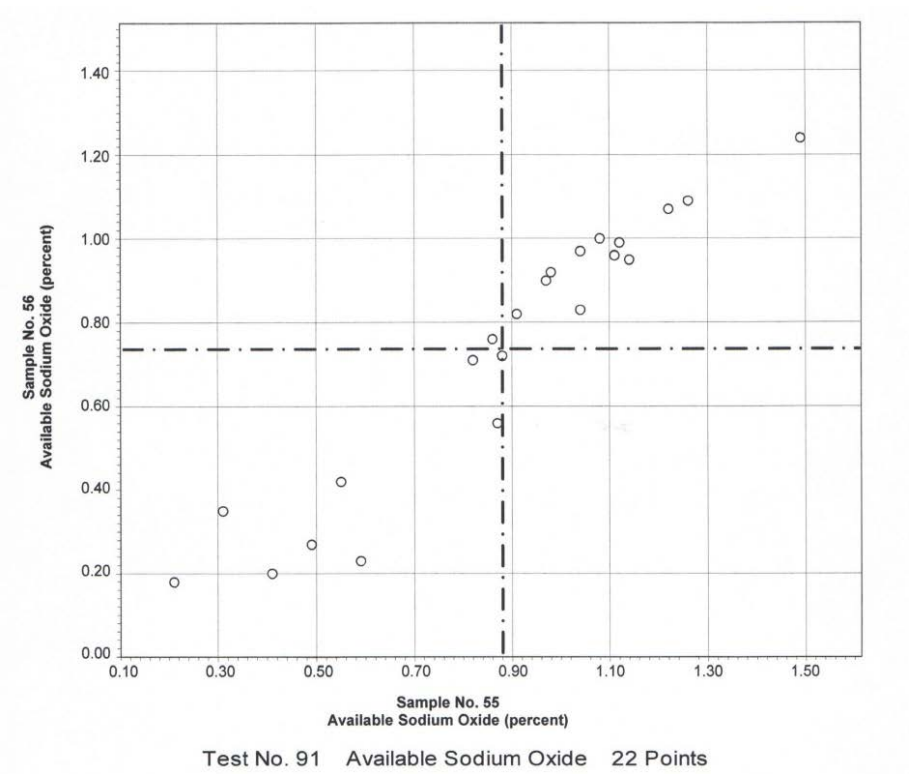


Figure 3. Available sodium content for CCRL 55 and 56 (Haupt 2014)

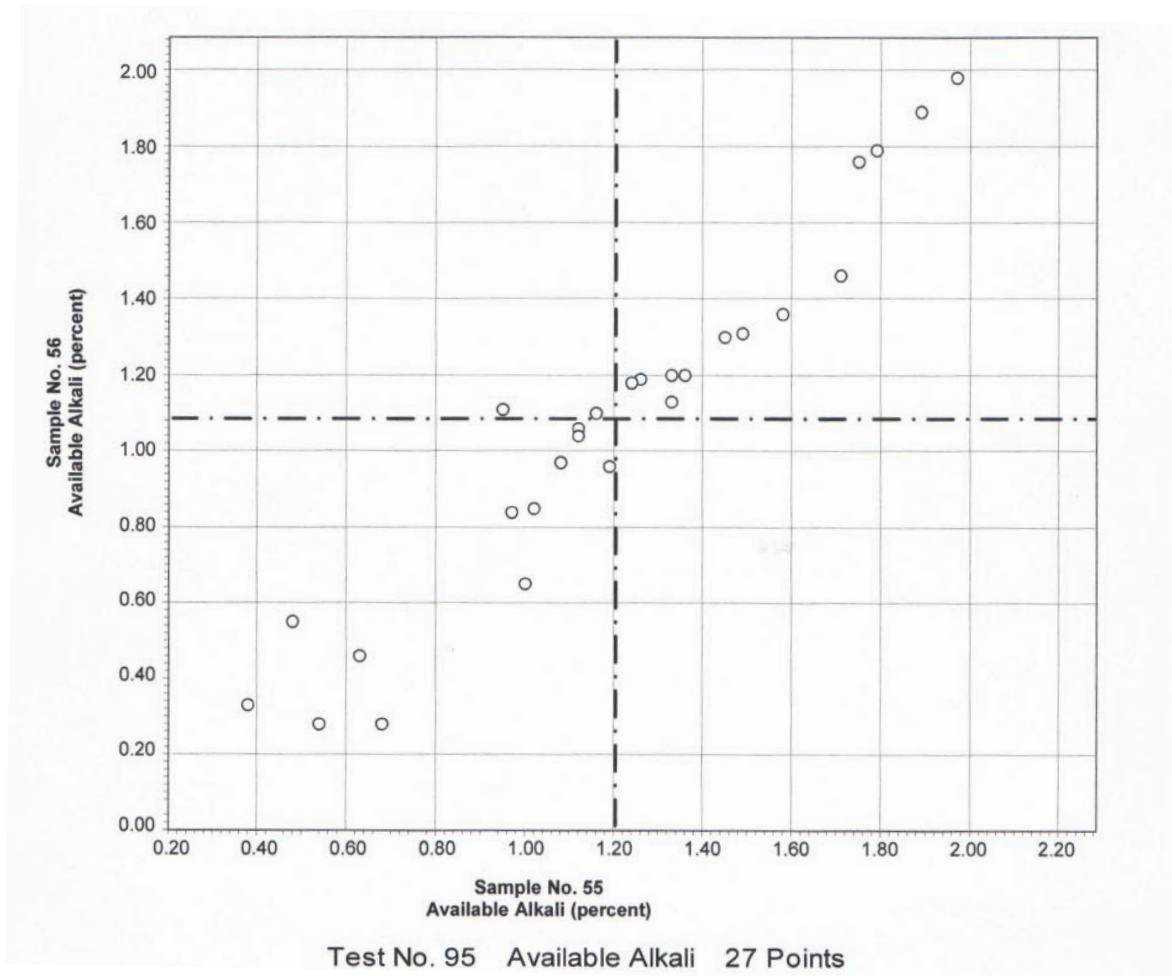


Figure 4. Available alkali content for CCRL 55 and 56 (Haupt 2014)

Preliminary calculations revealed that the standard deviation of the test results was poorly behaved. This is illustrated in figures 5 and 6. The top half of each plot indicates that the variation of Na (or K) measurements increased with increasing concentration of Na (or K). This suggests that the relative standard deviation (coefficient of variation, $1s\% = 100 \text{ times the standard deviation divided by the mean}$) is a more appropriate measure of variability since it is less dependent on concentration (see the bottom half of each figure). Do note that the mathematical transform did not solve the variability problem; it simply made it possible to express the precision of the test method using a single value. However, due to the large range of values shown in the figures, it would be hard to produce a robust (pooled) estimate of the relative standard deviation for the available alkali test method. In fact, it indicated that there were significant problems with the test method that need to be fixed and this needs to be done before a meaningful specification limit can be established. How was the 1.5% maximum limit for the test method established? This will be discussed in another section of this report.

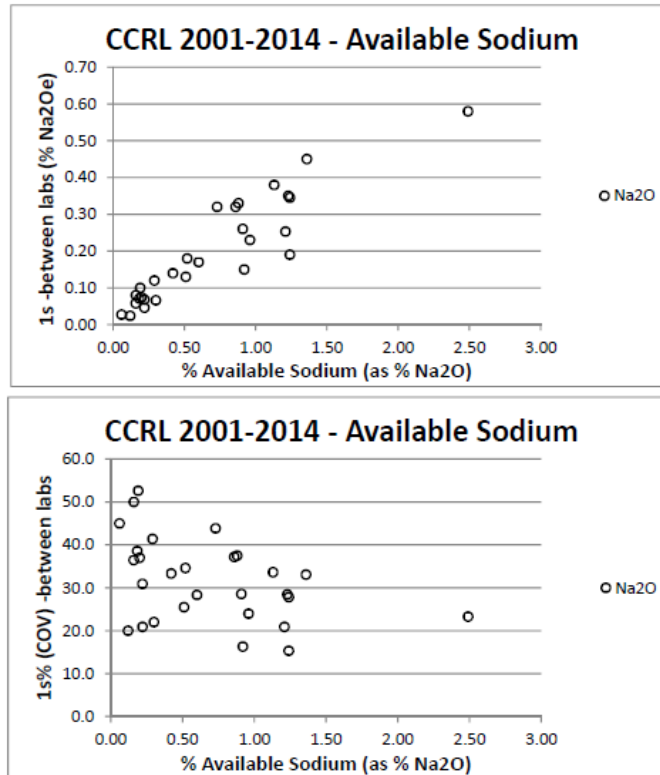


Figure 5. Precision of CCRL available sodium oxide determinations

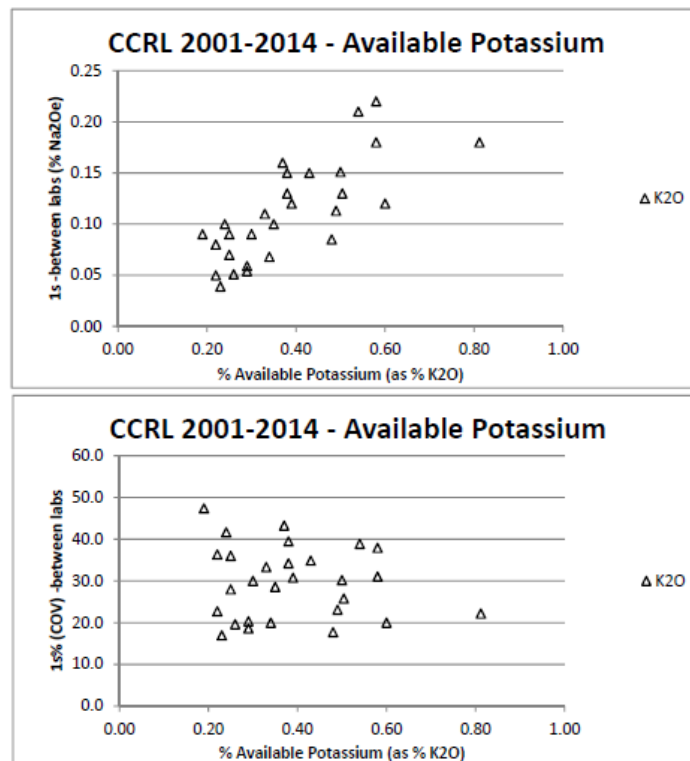


Figure 6. Precision of CCRL available potassium oxide determinations

As an example of the problem that we have encountered let us assume that the precision of the method (1s%) is 20%; this is roughly a lower-bound to the points shown in the bottom half of figures 5 and 6. Quick calculations produce the values given in Table 2. The poor between-lab precision of the test method makes it difficult to use the results of the available alkali test to accept or reject fly ash. The test simply leads to disagreement between testing labs and this leads to additional testing.

Many users have adopted the available alkali test method and take the results at “face value.” The complexity of the method, the amount of time required for curing (28-days) and the labor-intensive final extraction period are often ignored. Miller (1974) researched ways to obtain quicker test results by increasing the curing temperature and decreasing the curing time. The study was very limited (four natural pozzolans and two fly ashes (Class F)) so additional testing would have been required to ensure the validity of the changes. However, his report went unnoticed and it had no impact in changing the procedure given in ASTM C 311.

ASTM Subcommittee C09.24 spent considerable time discussing how to fix the test method. A short list of potential concerns is listed below. However, as was mentioned earlier in this section, the test method is nearly unchanged from the method published in 1957. The Subcommittee reached a partial resolution to the issue by adding new test methods for total alkali and a new specification limit for the mortar bar expansion test (Schlorholtz 2006).

- Test takes too long to complete
- Poor agreement between labs
- Calibration standards do not match test samples
- Alkali release continues past the 28-day curing period

Other researchers have also expressed concerns about the available alkali test. Some indicate that the measured alkali values are too high and indicate that this may be due to the washing process in the test procedure (Duchesne and Berube 1994). They suggest a pore-squeezing method may help provide a better estimate of “available alkali.” Likewise, Butler (1982) has described inconsistencies in different alkali test methods.

Table 2. Example of available alkali precision problems (between-lab estimates)

%Na₂O_e	1s%	1s	2s
0.5	20%	±0.1	±0.2
1.0	20%	±0.2	±0.4
1.5	20%	±0.3	±0.6
2.0	20%	±0.4	±0.8
2.5	20%	±0.5	±1.0

The Total Alkali Test

ASTM C 311 was updated with new verbiage for total alkali determinations (both sodium and potassium) in 2001. This change did not provide a detailed method, rather it allowed labs to use any method and supplied a note suggesting a couple of methods that had been used successfully. The hope was that eventually the available alkali test could be retired (Schlorholtz 2006). It is important that the reader understands that the statistics calculated in this section are very rough because the sodium and potassium determinations have been conducted in different labs using different methods (XRF fused disks, XRF pressed pellets, flame photometry, and atomic absorption spectroscopy to name a few of the common methods). In this section all statistics have been calculated using the tabulated CCRL PPSP summaries (see Table 2, Appendix A) and no attempt has been made to differentiate between different analytical methods. From a practical standpoint this greatly simplifies the calculations; however, one would expect that the precision estimates that are calculated will be larger than those based only on a single technique. Since the goal of the calculations was only to “ball park” the precision of the total alkali determinations and give results that can be compared to the precision estimates for the available alkali test this was deemed adequate for the needs of this research project.

Continuing with the analysis that was used in the prior section, the scatter grams for total sodium and total potassium oxides are given in Figures 7 and 8, respectively. The scatter grams contain almost twice as many data points than the scatter plots for available alkali. This is because more labs measure the total alkali content during their bulk chemical assays. The diagrams still show considerable bias because the large majority of the data points plot in the lower-left and upper-right quadrants of the graph. However, the range of reported values has decreased significantly from the plots for available alkali (refer back to Figures 2 and 3). Also, the points exhibit a very noticeable departure from the main diagonal of the graph (this represents the within-lab variability).

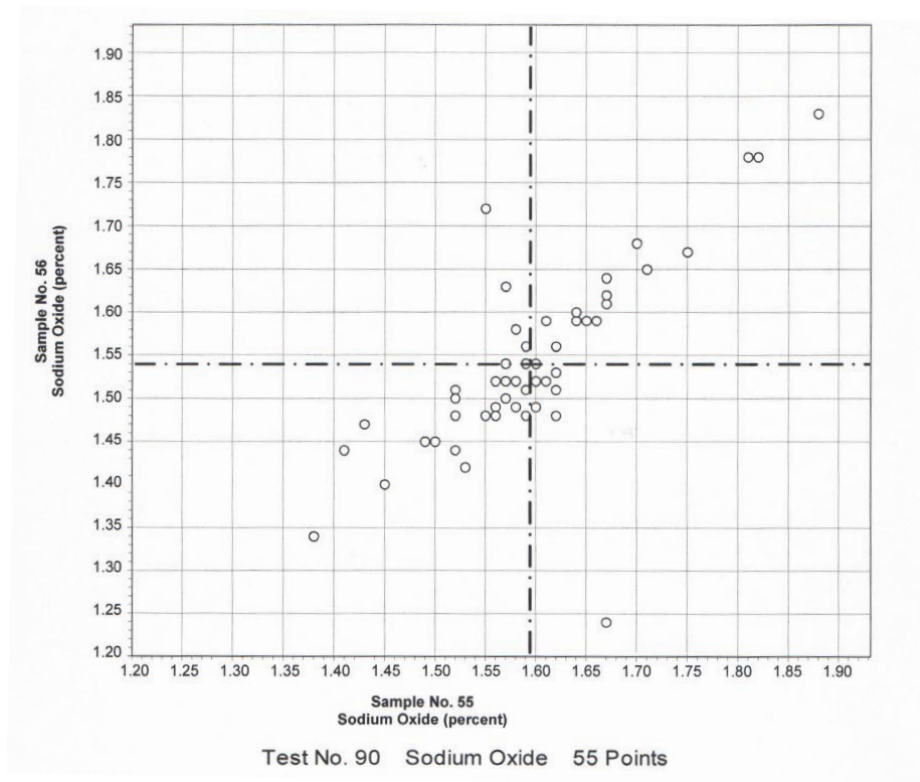


Figure 7. Total sodium content for CCRL 55 and 56 (Haupt 2014)

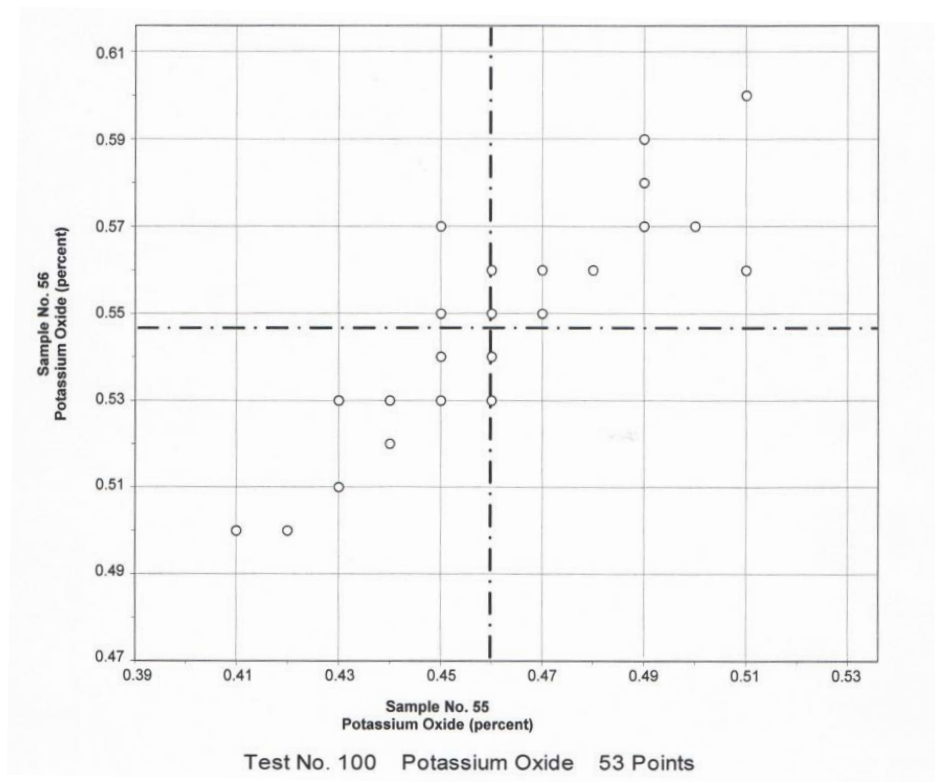


Figure 8. Total potassium content for CCRL 55 and 56 (Haupt 2014)

The sodium data still exhibits a trend of increasing standard deviation with increasing concentration (see Figure 9). The trend is less severe than was noted for the available alkali test. Four data points plotted considerably above a 1s value of 0.25% Na₂O and this had a large impact on the precision of the test (refer to the top half of the figure). When the data is transformed to 1s%, the behavior is better. However, now it is evident that the error of the total sodium determinations increases with decreasing concentration; this is commonly encountered with fixed counting time strategies in XRF analysis. In addition, it could suggest that there is some background compensation or line-overlap issues that need to be resolved. Keep in mind that there is no single specified procedure that has been used for the determinations so the problem could have a simple fix. It is still evident in the figure that the average precision (1s%) for values above about 1% sodium oxide is about 10%. This is much better (by roughly a factor of 2) than was obtained for the available sodium test.

The potassium data also exhibits a trend of increasing standard deviation with increasing concentration (see Figure 10). The trend is less severe than was noted for the available alkali test. If the data is transformed to 1s%, then it indicates that the precision is about 5%. Again, this is much better than the precision of the available potassium determinations (by roughly a factor of 4).

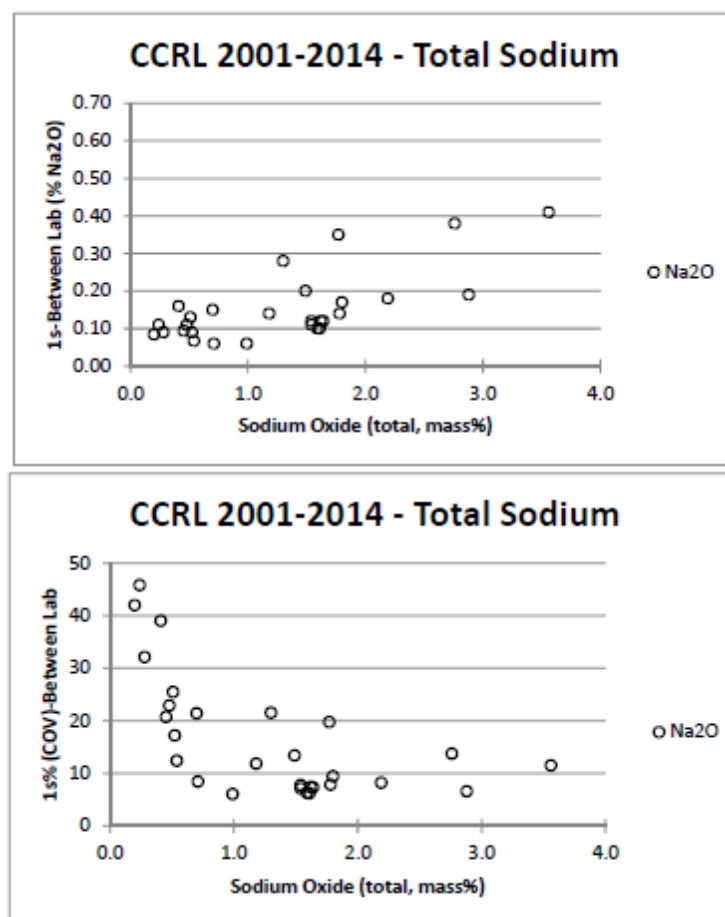


Figure 9. Precision of CCRL total sodium oxide determinations

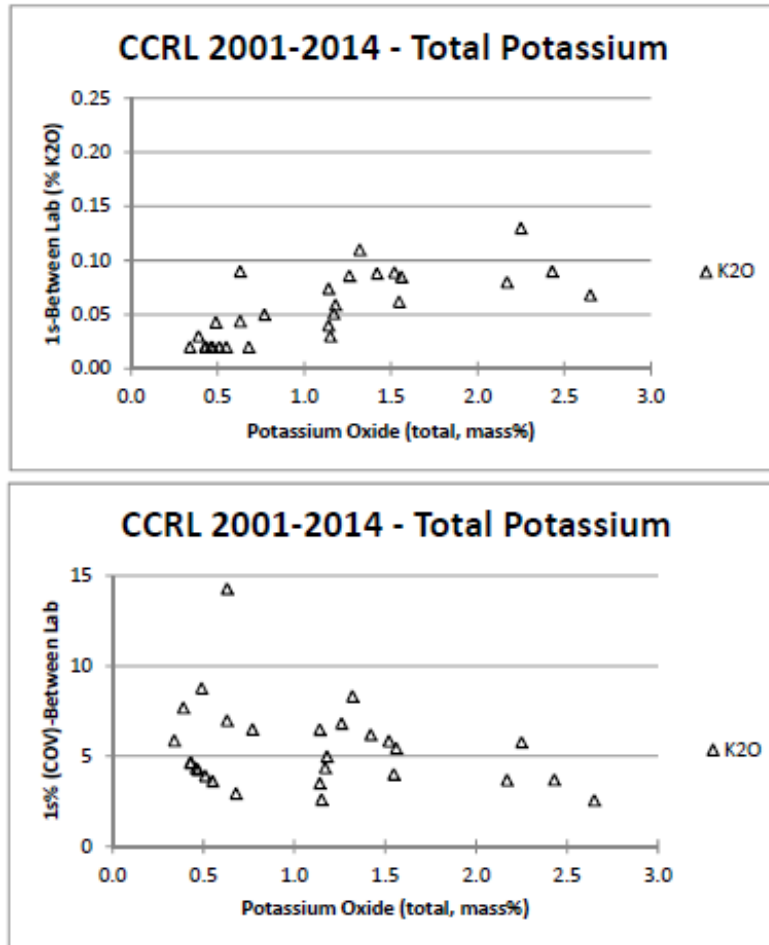


Figure 10. Precision of CCRL total potassium oxide determinations

The Soluble Alkali Test

ASTM C 311 does not give a procedure for determining the water-soluble alkali content of fly ash. Several procedures were found in the literature; however, some methods were vague (Moran and Gilliland 1950) while others were very tedious (Brink and Halstead 1956). Some researchers have used the test method for cement (Pepper and Mather 1959) but this procedure could lead to potential problems with Class C fly ashes because highly reactive ones can set in about 10 minutes. Hence, a procedure that was developed during a prior study will be used (Sutter et.al. 2013). Most of the aforementioned researchers indicated that the soluble alkali content of fly ash varied from about 0.1% to 1% (expressed as %Na₂O_e). No precision information was available for the test methods so that information will be developed later in this study.

Summary of the Alkali Tests

The discussion of the previous three sections is summarized in Table 3. The major deficiency of the current state of fly ash analysis is the lack of good certified reference materials (CRMs). The existing standards are now approaching 20 years of age. In addition, they are only useful for checking the bulk assay determinations (but not for all specified elements). The CCRL test results have helped improve the total oxide determinations but can only be used to roughly judge the available alkali test results. The water-soluble alkali determinations have no certified standards available to check the measurements. However, this could be rectified by mixing known amounts of soluble alkali compounds with bulk ashes that exhibit negligible soluble-alkali content.

Table 3. Strengths and weaknesses of the various alkali tests

	Alkali Test Method		
	Available Alkali	Total Alkali	Water Soluble Alkali
Time for test result	28-day cure + about 3 hrs	Less than 1 hour	Less than 2 hours
CRMs available	No	Yes	No
CCRL results available	Yes	Yes	No
Within-lab precision	OK (but variable)*	OK*	OK*
Between-lab precision	Poor (roughly 1s% >20%)	Better than AA test (roughly 1s% = 10%)	Unknown

*estimated in this project

Specifications for Fly Ash (National)

The main specification limits for fly ash are given in ASTM C 618 (or AASHTO M 295). These two specifications are very similar in most requirements. They both contain performance limits for mortar-bar expansion tests conducted in accordance with ASTM C 311 (using Pyrex glass aggregate). The specified limit is a maximum expansion of 100% of a low-alkali control cement (14 day test criterion). The appendix of C 311 describes how the fly ash replacement level may have to be increased to meet the performance limit. The AASHTO specification also lists an optional chemical requirement for the available alkali test (1.5% maximum). This requirement was removed from C 618 in 2001. The 1.5% limit was actually removed from the AASHTO document by about 2005; however, it was inserted back into the specification when users voiced concerns. Recently, researchers (Sutter et al. 2013) have recommended the removal of the 1.5% available alkali limit, better reporting of the total alkali content and replacing the current performance test (similar to C 441) with the rapid mortar bar expansion test (ASTM C 1567). It is currently not known if these recommended changes will be adopted by AASHTO.

Specifications for Fly Ash (Iowa DOT)

The main specifications for the use of fly ash in the State of Iowa are summarized in Iowa DOT Materials I.M. 491.17. The IM is entitled “INSPECTION AND ACCEPTANCE OF FLY ASH FOR USE IN PORTLAND CEMENT CONCRETE, BASES AND SUBBASES.” As denoted in the title, this document, and associated appendices, details the requirements for fly ash used in state projects.

The acceptance procedure is based on chemical and physical testing. When the available alkali content of the fly ash is less than 1.50% Na_2O_e no physical testing is required. If the available alkali content of the fly ash is greater than 1.50% and less than 2.50%, the fly ash needs to be tested using a mortar bar expansion test with Pyrex glass aggregate. The tests are conducted with a cement containing 0.70 to 0.80% alkali (as % Na_2O_e) and the test mixtures need to exhibit expansions no more than 10% greater than the control specimens. No mention is made about what can be done with fly ashes that have available alkali contents exceeding 2.5%; it is inferred that such ashes cannot be used. Typically, few power plants produce fly ash with available alkali contents above 2.5%. The exact rationale for the 1.5% and 2.5% specification limits has been hard to locate. The specification limits were implemented in August, 1992. Minutes and letters pertaining to the limit have been placed in Appendix B as this may help future researchers to track down the origin of the limits. One can speculate that the 2.5% limit was derived from the fact that at 20% replacement (of fly ash for an equal mass of cement), the cement and the fly ash would provide nearly equal amounts of alkali to the cementitious mixture. This speculation assumes a maximum cement alkali content of 0.6% (low-alkali cement) and that the available alkali test result reflects the true amount of alkali that the fly ash contributes to the pore solution.

Since about 2000, the Iowa DOT has been using a mathematical model for estimating the available alkali content of different sources of fly ash (Cheng 2000). This was done in an effort to reduce the amount of time required to evaluate the compliance of any given sample of fly ash. It was also used because it represented a significant time savings for Iowa DOT chemical staff. The model is based on an empirical relationship between the total alkali and available alkali content of fly ash. This relationship will be discussed later in this report.

Alkali Content – Prescriptive Limits or Performance Limits

There has been an on-going argument about whether specifications should contain prescriptive limits or performance limits. The argument has lasted for over 50 years and still has not been fully resolved. Fly ash characterization and classification is a good example of the prescriptive-performance problem. Figures 11 and 12 illustrate the test response of fly ash for the available alkali test and the mortar-bar expansion test, respectively. One is a chemical test while the other is a physical test. The figures clearly indicate that the test response increases as a function of time. Sometimes the test response increases significantly with time. The goal of each test is to predict how fly ash impacts ASR, and, to simplify that evaluation, specific test criteria have been invoked. The criteria are normally set using interlaboratory studies so that the estimates are robust. However, all such limits are only empirical facts.

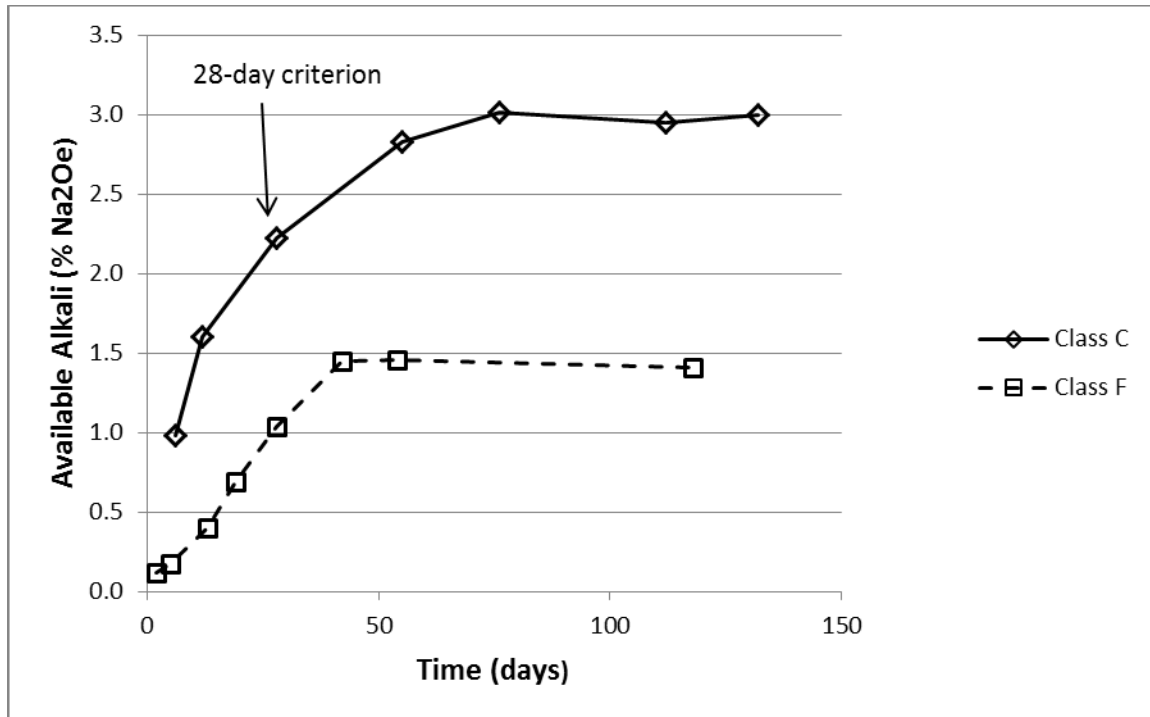


Figure 11. Typical test results from the available alkali test (versus time of extraction)

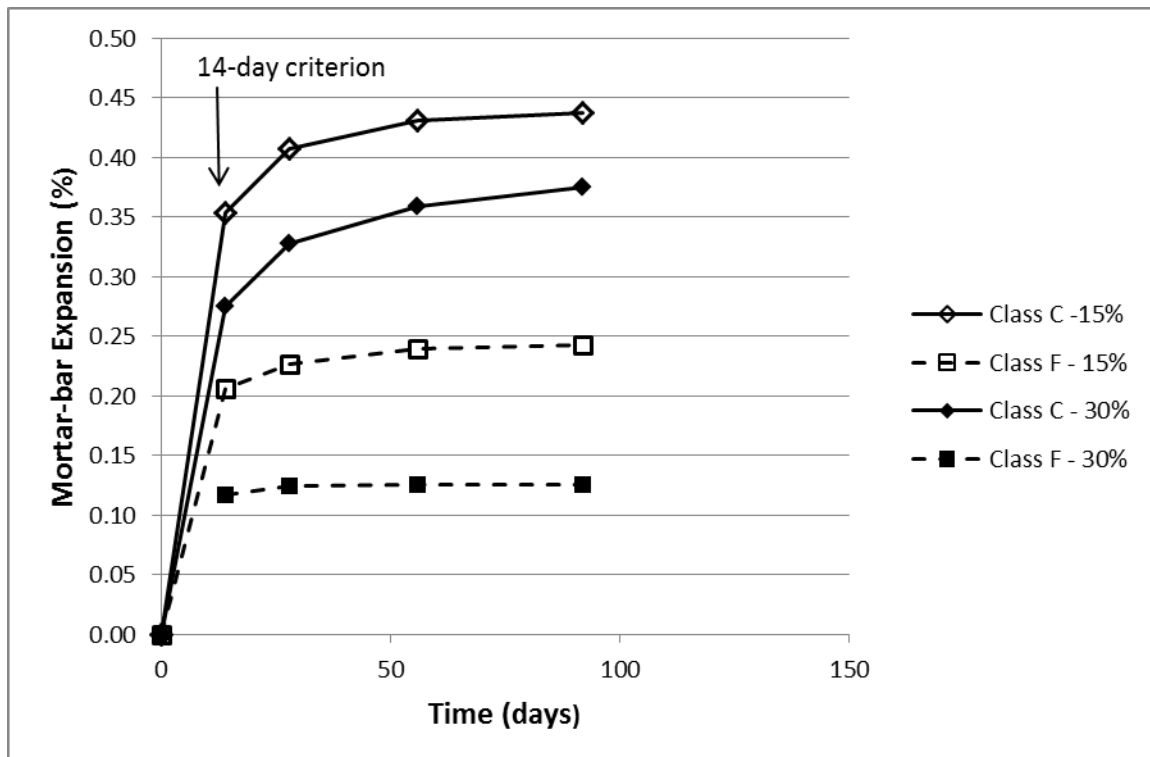


Figure 12. Typical test results from the ASR mortar-bar test with pyrex glass

Unfortunately, empirical facts only pertain to the range of materials that were present in the research program. Attempting to extrapolate the relationships to a wider range of materials is almost always problematic. Fly ash suffers from this problem. Any outsider looking at Figures 11 or 12 would immediately predict that the test methods would probably exhibit poor precision (due to the rapidly increasing test response at the criterion point).

Figure 13 depicts a table extracted from Minnick (1954). This table predates the first release of fly ash specification limits in the United States. The circled values reflect a chemical test limit (1.5% for exchangeable alkalis – now called available alkali) and a physical test limit (60% reduction). The limits were to protect from using fly ash that was not effective in mitigating ASR. However, as given in the far right-hand column, one can easily surmise that the limits are not compatible. Why? Because the two tests have specification limits that fail different samples. Only sample seven shows up on both lists. One would think that the limits would have been reconsidered based on that inconsistency. However, that was not the case.

The rest of this report will present information based on these two test methods. And each time we use the test method we will simplify the information obtained and report a 28-day value (the available alkali) or the mortar-bar expansion at some age (often only 14-days as specified in AASHTO M295). However, the reader needs to understand that in many instances, especially when evaluating Class C fly ash, these metrics leave much to be desired.

TABLE XX.—ANALYSIS OF FLY ASH SPECIMENS USING AS A BASIS THE REQUIREMENTS THAT HAVE BEEN PROPOSED AS A SPECIFICATION FOR FLY ASH FOR USE AS ADMIXTURE FOR PORTLAND CEMENT CONCRETE.

	Proposed Limits	Specimens Not Passing Limits
CHEMICAL REQUIREMENTS		
SiO ₂ (minimum), per cent.	40.0	2, 3, 7, 8, 9, 10
Al ₂ O ₃ (minimum), per cent.	15.0	All pass
SiO ₂ + Al ₂ O ₃ (minimum), per cent.	50.0	All pass
MgO (maximum), per cent.	3.0	All pass
SO ₃ (maximum), per cent.	4.0	All pass
Loss on ignition (maximum) per cent.	12.0	2, 3, 5, 9
Moisture (maximum), per cent.	3.0	All pass
Exchangeable alkalis expressed as Na ₂ O, (maximum), per cent.	1.5	5, 7, 12, 13, 15, 16, 17, 18
PHYSICAL REQUIREMENTS		
Blaine fineness (minimum), sq cm per g.	2800	All pass
Contribution to compressive strength used as a replacement of standard Ottawa sand, 25 per cent by weight of the Standard Ottawa sand:		
Per cent of control at 7 days.	115.0	2, 3, 5, 6, 9, 18
Per cent of control at 28 days.	125.0	2, 3, 5, 6, 9, 18
Pozzolanic strength index		
Per cent of control at 28 days (minimum).	85.0	1, 2, 3, 5, 6, 9, 10, 11, 18
Per cent of 28-day strength, at 90 days (minimum).	100	3
Drying shrinkage (maximum), per cent.	0.10	All pass
Reduction of mortar expansion (minimum), per cent.	60.0	1, 2, 3, 4, 7, 9, 11
Autoclave expansion (minimum), per cent.	0.50	Not tested
Water requirement (maximum), per cent of control.	103.0	2, 3, 5, 9, 12
Air entraining agent required		
Number of times the amount of admixture required for control (ASTM Method C 185 mortar)	5.0	Not tested

Figure 13. Suggested fly ash specification limits (Minnick 1954)

Future of Coal Power Generation

The title of this section points to the fact that it is currently a tough time for power plants that use coal to generate electricity. This is very odd because for the past 50 years coal has played the major role in the electrification of the United States. At its peak, coal provided over 50% of the gross generating capacity of the US and currently coal provides about 37% of the gross generating capacity (EIA 2014). There are several reasons for the decline in generating capacity - they include: power plant age, competing technology and environmental regulations. Obviously, if the number of coal plants decline then the availability of fly ash will be reduced and that could impact the economic viability of fly ash marketing agencies. Ultimately, this would reduce the number of sources of fly ash available for use in concrete construction projects.

Natural gas has replaced coal as the fuel of choice for electrical generation (Trembath et. al. 2014). This is due to the rapid expansion of fracking in the US. The construction cost (in dollars per kilowatt) for a gas-fired power plant is about two-thirds of that of a traditional coal-fired power plant (Dears 2014). Fuel costs are variable but are currently at a low point that may last for years. Unfortunately, natural gas also serves both commercial and residential customers and, as with all commodities, the fuel cost is subject to market fluctuations that can greatly impact the fuel cost.

The average age of a US coal-fired power plant is about 45 years old (EIA 2014). The major Iowa power plants were all built between 1978 and 1983, so they are currently about 35 years old. One would anticipate that a number of new power stations would be in the planning and design phase to alleviate the ultimate retirement of the older stations. Currently there are no plans to build any major coal-fired power plants in the US (EIA 2014). This is due to new regulations that have been recently enacted (or proposed). Figure 14 illustrates the power plant closings and proposed closings over the next decade (GAO 2014). The Midwest region is not heavily impacted by the closings; however, the eastern and south-eastern states will be significantly impacted.

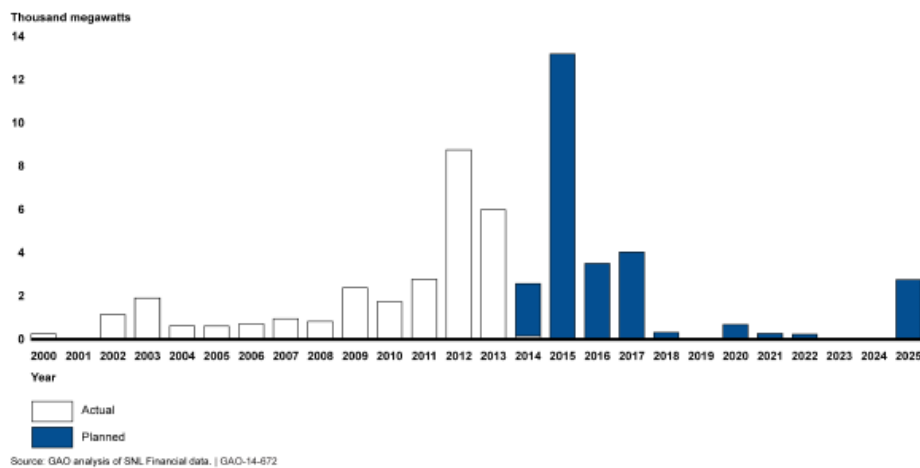


Figure 14. Coal plant closings (actual and planned) from GAO (2014)

Dockter and Jagiella (2005) give an excellent overview of both federal and state regulations for coal combustion products. No attempt will be made to summarize the contorted history of these regulations. Rather, an attempt will be made to describe recent regulations that will impact coal burning and/or fly ash usage. Roughly speaking, the same rules apply today as they did in the past - coal ash is treated as a solid waste (Subtitle D) under the Resource Conservation and Recovery Act. The states are charged with constructing laws to use this potential resource.

The new federal regulations that have been formulated and approved (at least tentatively) have been summarized in Table 4. The first three regulations should have limited impact on power plant closures. However, the MATS and CCP regulations have had (or will have) major impacts.

The disposal of coal combustion residuals rule sets minimum federal requirements for new and existing fly ash landfills or impoundments (EPA 2014a). It attempts to reduce the risk of catastrophic failure of surface impoundments and also protect groundwater resources. It places considerable cost of compliance and implementation with owners and states. The rule also details the requirements for closing a landfill or surface impoundment. The rule has already come under the scrutiny of Republicans and claims have been made that the rule potentially could allow coal ash to be reclassified as hazardous at a later date (Cama 2015).

The Mercury and Air Toxics Standards (MATS) is an attempt to set national standards to reduce mercury, non-mercury metals, dioxins, furans, acid gases and sulfur dioxide pollution from power plants (EPA 2014b). The rule applies to all power plants that generate more than 25 megawatts of power. Hence, this rule applies to all of the power plants that were included in this study. Activated carbon injection into the flue-gas stream is the proposed solution for most power plants. However, depending on age of the power station and the coal source that is burned, additional retrofits may be required. The additional retrofits could include dry sorbent injection (DSI), upgrading the electrostatic precipitators or the addition of a scrubber. North Omaha station is a power plant in this study that will be closed because it was deemed uneconomical to retrofit the plant to meet the new regulations (MATS rule). Actually, many of the planned closures shown in Figure 14 are related to this rule.

Table 4. A summary of recent regulations that impact coal-fired power plants

Name of Regulation	Date Finalized	Date Effective
Cross State Air Pollution Rule (CSAPR)	July 6, 2011	2015
Cooling Water and Air Intake Structures	April 20, 2011	October 14, 2014
Disposal of Coal Combustion Residuals	December 19, 2014	2015-2016
Mercury and Air Toxics Standards (MATS)	December 16, 2011	2015-2016
Clean Power Plan (CCP)	Tentatively June 2015	unknown

The Clean Power Plan (CCP) is by far the most controversial of the regulations listed in Table 4. This rule has not been finalized yet, but the plan is to reduce carbon dioxide emissions from existing power stations by 30% by 2030 (relative to 2005 emissions). This would mean that significant reductions would be required by 2020 and the EPA estimated that roughly 108 to 134 GW of generating capacity would be retired to meet that timeline (EPA 2015). The North American Electric Reliability Corporation (NERC) provided an initial review of the proposed plan in late 2014 (NERC 2014). They estimated that rule impacts about 65% of the total existing power plants (nameplate capacity) in the US, and they indicated that the timeline was too short to ensure that the bulk power system (BPS) would operate reliably. The NERC also indicated that additional analysis would be required to demonstrate that the assumptions made to construct the CCP are feasible. They provided a range of reliability evaluations that would need to be conducted prior to moving forward. Other agencies have been more critical of the proposed rule. For example, the Institute for Energy Research indicated (IER 2014):

“NERC was very diplomatic in its approach to the EPA’s grid takeover via the CPP. However, what NERC is not saying is that EPA’s proposed “Clean Power Plan” to run the nation’s electricity production and delivery system will be costly, confusing and disruptive to all users of electricity. It could lead to severe shortages of electrical capacity. It is a disaster waiting to happen.”

The only way for pulverized coal power stations to meet the Clean Power Plan requirements is to incorporate carbon sequestration and storage (CCS) technology. However, to date (October 2014), only a single pulverized-coal power station has been retrofitted with CCS (Johnstone 2014), and that power plant was not located in the United States. In addition, the retrofit was performed on a small power plant (162 MW) and the CCS unit needed 34 MW to operate (a loss of 21%, not including final transportation). Recently, both Europe and America have scrapped major CCS projects because of cost issues [Doyle (2015), Wolfgang (2015)]. The energy industry feels great risk because CCS is not a proven, economical; and hence, viable retrofit for existing power plants. Many power plants may eventually close because of this rule.

In summary, it is only possible to comment on how the MATS rule will impact fly ash sources used by the Iowa DOT. The impact of the CPP rule is highly uncertain at this time. The probable MATS-related retrofit for most power plants will be the injection of a small amount of activated carbon into the flue-gas stream. This will cause a small increase in the loss-on-ignition (LOI) of the fly ash; however, due to the high activity of the carbon, it has the potential to cause air-entrainment problems. Since the Iowa DOT is already highly cognizant of concrete air issues, the author does not believe this will cause significant problems. It may require the use of more air-entraining solution but the issue can be solved. The use of DSI to meet the MATS rule will need to be dealt with on a plant-by-plant basis. Common DSI materials include trona, sodium salts (carbonate, bicarbonate, etc.), calcium hydroxide and sometimes proprietary chemicals. The Iowa DOT already has experience using fly ashes treated with similar materials (for example, OGS commonly uses trona) so the impact should not be severe. However, due diligence is required to make sure that the additive is known and monitored.

DETAILS - EXPERIMENTAL PROCEDURES

This section describes the materials and test procedures that were used in this study. As described in earlier sections of this report, the primary interest was in the alkali content of fly ash and the corresponding expansion noted in mortar bar expansion tests.

Materials

Fly Ash Sources

Twenty-one fly ash samples were obtained for this research project. The samples represented fourteen different sources (power stations). Eight samples of fly ash from Ottumwa Generation station (OGS) were obtained for this study. The sample names, power plant designation and marketing agency are summarized in Table 5. The power plants were primarily located in Iowa and have been used in many projects over the last few decades. However, fly ash samples from Missouri, Nebraska, North Dakota and Wisconsin were also included in the study. Most of the samples were grab samples and the sampling date has been embedded in the sample name (as MMddYY). Composite samples from Nebraska City, North Omaha and Ottumwa Generating Station were also included in the study.

Table 5. Summary of fly ashes obtained for this study

Sample Name	Power Plant	Marketing Agency
HAV051112	Havana, IL	Headwaters
CBF051512	Council Bluffs Unit#3, IA	Headwaters
PN4051412	Port Neal Unit#4, IA	Headwaters
COA051512	Coal Creek, ND	Headwaters
OGS051512	Ottumwa Generating Station, IA	Headwaters
CLI051412	M.L. Kapp, IA	Headwaters
COL072712	Columbia Unit#1, WI	Lafarge NA
PLP072312	Pleasant Prairie, WI	Lafarge NA
EDG072712	Edgewater Unit#5, WI	Lafarge NA
OGS073012	Ottumwa Generating Station, IA	Headwaters
IAT080812	Iatan, MO	Lafarge NA
OGS081312	Ottumwa Generating Station, IA	Headwaters
OGS083012L-1	Ottumwa Generating Station, IA	Headwaters
OGS083012L-2	Ottumwa Generating Station, IA	Headwaters
OGS083012H-1	Ottumwa Generating Station, IA	Headwaters
OGS083012H-2	Ottumwa Generating Station, IA	Headwaters
RUS092012	Rush Island, MO	MRT/CEMEX
LAB092012	Labadie, MO	MRT/CEMEX
NBCCOMP7-12	Nebraska City, NE	Nebraska Ash
NOSCOMP5-12	North Omaha, NE	Nebraska Ash
OTTCOMP12-98*	Ottumwa Generating Station, IA	Midwest Fly Ash

*This old sample of OGS fly ash was used to obtain a sample closer to the 1.5% available alkali limit

Hydraulic Cement Sources

Cement materials were limited to four cements and a single source of slag. The cements consisted of two low-alkali cements from Lafarge, high-alkali cement from St. Marys, and a blended cement (Type IP).

The majority of the testing for this project was conducted using the Lafarge Type I/II, low alkali cement from Davenport, IA. This cement was used to check the 21 fly ash samples for compliance to ASTM C 618 specification limits. It was also used for the ASTM C 1567 testing.

The other two cements, Lafarge and St. Marys, were used to proportion the ASR cement as per Iowa DOT Materials I.M. 491.17 (total alkali content between 0.70 and 0.80%, as Na_2O_e). Appropriate amounts of cement were combined and thoroughly mixed to provide the designated alkali content. The alkali content of each batch was verified by chemical analysis.

Blended cements consisted of a Type IP cement obtained from Ash Grove in Kansas City, MO and a physical mixture of Lafarge slag (grade 120) and Lafarge Type I/II cement (20% slag with 80% portland cement). The blended cement mixtures were used to see how high-alkali fly ash would impact the ASR expansion of specific mortar-bar test specimens.

Aggregates Used in this Study

Three different fine aggregates were used during this study. They consisted of graded standard sand, pyrex glass and Wayland sand.

Standard cube test sand was used for the fly ash testing. The sand was purchased from US Silica and it met the gradation requirements for graded sand specified in ASTM C 778, Standard Specification for Standard Sand.

Pyrex glass was obtained from VSI Products LLC in Nampa, ID. The glass was purchased in specific size fractions that were recombined to meet the gradation specified in ASTM C 441, Test Methods for Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction.

Wayland sand was provided by the Iowa DOT. The bulk sand was sieved into specific size fractions for ASR testing. The material retained on the #4 sieve was scalped from the sample and no attempt was made to grind it and recombine it with the bulk sample. Then the individual sieve fractions were washed and dried prior to being recombined to meet the gradation specified in ASTM C 441.

Other Materials

Several additional materials were used throughout this study. Calcium hydroxide (for available alkali tests), US Silica Sil-Co-Sil 100 (a finely ground quartz sand that was used as an “inert” material in specific ASR tests), reagent grade calcium carbonate, sodium chloride and potassium chloride (for making available alkali standards), and Sigma 12:22 lithium borate flux (for fused disks) were all used during this study.

Chemical Testing Methods

A variety of different chemical tests were conducted during this study. All efforts were made to use standard test methods when they were available; however, in certain instances new methods had to be developed. The major chemical tests that were conducted consisted of bulk chemical analysis via x-ray fluorescence spectroscopy (XRF) and alkali determinations (both available and water-soluble) via flame photometry. The equipment used for this study was checked during the most recent CCRL Inspection of the laboratory (June 2012) and Pozzolan Proficiency Sample Program (summer/fall of 2011, 2012, 2013 and 2014).

XRF

All chemical assays for this study were conducted using a Panalytical PW 2404 XRF spectrometer. The spectrometer is equipped with a rhodium x-ray tube that was operated at 3600 watts. The Materials Analysis and Research Lab standard operating procedure (SOP) was used for this study. Briefly, this consists of fusing fly ash samples into glass disks using a LECO FX-503 fluxer. The basic fusion recipe used 1-part sample to 5-parts Sigma flux. The spectrometer was calibrated using synthetic standards proportioned using a variety of different certified reference materials. The spectrometer was calibrated for major, minor and selected trace elements common to fly ash, this included Na, Mg, Al, Si, P, S, K, Ca, Ti, Mn, Fe, Sr and Ba. The calibration for each element was verified using four fly ash standards (NIST 1633b, NIST 2689, NIST 2690 and NIST 2691); however, it is important to note that these particular standards may not have been certified for all of the listed elements (Ba and S being common issues).

Alkali Determinations

A Beckman DU-2 spectrometer equipped with a flame attachment was used to determine the concentration of sodium (Na), potassium (K) and calcium (Ca) in solutions that were extracted for this study. The spectrometer employed a hydrogen-oxygen flame for all determinations. The spectrometer was calibrated prior to each analysis using standard solutions prepared in accordance with ASTM C 114. Reagent grade calcium carbonate, sodium chloride and potassium chloride were used to make the standard solutions. The working range for sodium was 0 to 80 ppm (6 standard points), 0 to 40 ppm for potassium (5 standard points), and 0 to 600 ppm for calcium (5 standard points). All sodium and potassium standards contained 504 ppm calcium as noted in ASTM C 311. The same standard solutions

were used to calibrate for both available alkali and soluble alkali samples; however, the working range for potassium was greatly reduced for soluble alkali tests (0 to 10 ppm working range). Samples were typically diluted into the working ranges for the various determinations.

Physical Testing Methods

A series of different physical test methods was used in this study. The equipment used for the test methods was checked during the most recent CCRL Inspection of the laboratory (June 2012) and Pozzolan Proficiency Sample Program (summer/fall of 2011, 2012 and 2013).

Fly Ash – Mandatory Requirements

The fly ash samples were subjected to the physical testing required to check compliance to ASTM C 618 (and AASHTO M 295). Briefly, this consisted of determining the moisture content, loss-on-ignition, density, fineness, strength activity index with portland cement at 7-days (SAI-7) and 28-days (SAI-28), water requirement and soundness (via the autoclave expansion test).

ASR Mortar Bar Tests

In addition to the mandatory tests, all fly ash samples were subjected to ASR testing as specified in Iowa DOT Materials I.M. 491.17. The majority were mortar bar expansion tests using pyrex glass as the reactive aggregate. Two blended cements (Type IP and Type I/II + 20% slag) were also used in this study. Specific tests were also conducted with Wayland sand to check to see if it was a reactive aggregate. These tests were conducted in accordance with ASTM C 1567 and ASTM C 227.

Pavement Core Samples

Core samples were cut and polished using a standard operating procedure that has been described in earlier investigations (Schlorholtz 2000). The procedure produced polished cores sections that were used for standard petrographic examinations. Two different microscopes were used during the petrographic examinations. Bulk or polished sections were viewed using an Olympus BH reflected light microscope and/or an Olympus SZH stereo microscope. The magnification used for this study normally ranged from about 10x to 400x.

Pavement Core Sites and Project Information

Field service record was evaluated by extracting cores from specific pavement sites and looking for ASR-related distress. Fifteen pavement sites were evaluated by shoulder surveys. The Technical Advisory Committee (TAC) for the project reviewed a presentation of observations noted at the potential sites and helped to select nine sites for subsequent coring. The selected sites are summarized in Table 6. The sites were heavily weighted to evaluate pavements containing Wayland sand (all of the Lee County sites) and fly ash (OGS and Louisa). Two control sections (no fly ash) were also evaluated.

The Iowa DOT coring crew extracted cores for this project in early 2014. They extracted two cores from the joint region and one core from the mid-panel region of each pavement site. Cores were delivered to the researcher in mid-April for analysis. The pavement cores were logged and then subjected to petrographic examination. Due to the preliminary findings a second opinion was desired. Hence, one core from each site was sent to American Petrographic Services (APS, St. Paul, MN) for additional testing. APS conducted standard petrographic testing that included linear traverse for air content and spacing factor, and thin sections for inspection of paste properties, aggregate-related issues (like ASR), and an estimate of the water-cement ratio of the concrete mix.

Materials properties from the various pavement sites are summarized in Table 7. The information was gathered from Iowa DOT records from the various projects. Occasionally

Table 6. Pavement sites selected for coring

Site	Project#	Year Paved	Hwy-County	Comments/Location
1	IR-80-5(132)151	1992	I-80 -Jasper	Control section/WB driving lane, STA128
2	RP-163-1(50)	1992	Hwy 163 -Polk	EB driving lane near MP13.80
3	HES-92-5(27)--2H-91	1993	Hwy 92 - Warren	EB driving lane near MP 133.4
4	NHS-61-1(77)--19-56	2001	Hwy 61 - Lee	Distressed area/SB driving lane just after MP 15.0
5	NHS-61-1(77)--19-56	1999	Hwy 61 - Lee	NB driving lane, MP 17.2, STA 383
6	DE-RP-518-1(10)	1992	Hwy 61 - Lee	Control section/NB driving lane, MP5, STA 350
7	DE-RP-518-1(10)	1992	Hwy 61 - Lee	NB driving lane, MP 7, STA 470
8	BRF-RP-61-1(56)--35-56	1994	Hwy 61 - Lee	Joint spall/SB driving lane, STA 538
9	BRF-RP-61-1(56)--35-56	1993	Hwy 61 - Lee	NB driving lane, STA 536

the project information was rather vague but every attempt was made to track down the materials incorporated into the specific pavement location that was cored. Mix designs were rather similar, basically C-3 or C-4 mix designs with a single instance of a C-6 mix design (on Hwy 92 in Warren County). Water-reducer was used in all the mixtures and seven of the nine mixtures contained fly ash (OGS or Louisa fly ash at 15% or 20% replacement). Site 4 was supposed to contain 20% fly ash but the exact batch records denoting the source of the fly ash could not be located.

Table 7 also contains the results for P-214 (now designated as ASTM C1260) testing that was conducted at the Iowa DOT on the sand proposed for use in the projects. The central Iowa sands all exhibited large expansions (>0.20%) which is denoted as “potentially deleteriously reactive.” It was very odd to note that the Wayland sand produced the lowest test result (this sand is being investigated for reactivity via this project). Anecdotal information for this source suggests that the sand is sensitive to ASR.

Table 7. Mix and materials details for the pavement sites selected for coring

Site	Mix#	Cement	Fly Ash	P-214 (%)	Aggregate Used	
					Coarse	Fine
1	C-3WR	Lafarge	None	0.29	Sully	Van Dussel
2	C-4WR-C	Holnam	OGS	0.25	Sully	Van Dussel
3	C-6WR-C	Lafarge	OGS	0.37	Ames Mine	Hallet-DSM
4	C-3WR-C20	Continental	??	-	Huntington	Wayland
5	C-3WR-C20	Continental	OGS	-	Huntington	Wayland
6	C-3WR	Lafarge	None	-	Huntington	Wayland
7	C-3WR-C20	Lafarge	Louisa	-	Huntington	Wayland
8	C-4WR-C15	Holnam	Louisa	-	Huntington	Wayland
9	C-4WR-C15	Continental	OGS	0.13	Huntington	Wayland

RESULTS AND DISCUSSION

The main thrust of this project was to measure the alkali content of fly ash and how it impacted the expansion of mortar bar specimens containing fly ash. For completeness, the fly ash samples used in this project were also analyzed for mandatory chemical and physical requirements listed in AASHTO M295. This included determination of moisture content, loss-on-ignition, bulk chemistry (via XRF), density, fineness, strength activity index, soundness and water requirement. In addition, field cores were obtained to see if ASR had occurred in actual pavement sites containing similar material combinations.

Materials Properties

The results of chemical analysis of the fly ash samples are summarized in Tables 8, 9 and 10. The sum-of-the-oxides ($SUM = \%SiO_2 + \%Al_2O_3 + \%Fe_2O_3$) ranged from about 50% to 67% for the Class C fly ashes. Only a single Class F fly ash (COA051512) was included in the study and this tended to produce unbalanced experiments. Occasionally, an additional Class F fly ash (FA-I) from a recent NCHRP project (Sutter et al. 2013), was added to some experiments to help expand the range of composition.

Table 8. Results of chemical analysis of fly ash (major oxides and LOI).

Fly Ash	SiO ₂ , %	Al ₂ O ₃ , %	Fe ₂ O ₃ , %	SUM, %	CaO, %	LOI, %
HAV051112	40.4	20.7	6.20	67	21.4	0.24
CBF051512	34.5	19.0	5.72	59	27.6	0.23
PN4051412	33.6	17.8	5.99	57	28.4	0.34
COA051512	52.7	16.1	6.31	75	13.1	0.09
OGS051512	31.1	16.5	6.48	54	28.4	0.33
CLI051412	36.8	19.9	5.35	62	25.3	0.40
COL072712	35.3	19.4	5.76	60	26.6	0.23
PLP072312	39.8	21.8	5.42	67	21.3	0.54
EDG072712	35.3	19.0	5.83	60	27.0	0.35
OGS073012	29.9	16.5	6.93	53	29.0	0.34
IAT080812	37.1	19.3	6.23	63	25.3	0.38
OGS081312	27.5	16.7	6.35	51	29.3	0.48
OGS083012L-1	29.7	15.1	6.75	52	28.8	0.35
OGS083012L-2	30.2	15.5	6.66	52	28.7	0.34
OGS083012H-1	29.0	15.2	6.80	51	29.1	0.31
OGS083012H-2	29.0	15.2	6.76	51	29.0	0.32
RUS092012	36.3	17.9	6.27	60	25.7	0.30
LAB092012	37.8	20.1	6.37	64	22.5	0.56
NBCCOMP7-12	34.9	18.5	5.78	59	26.2	0.45
NOSCOMP5-12	36.5	19.7	5.91	62	25.1	0.25
OTTCOMP12-98	36.9	21.6	5.87	64	22.4	0.54
FA-I	62.4	20.1	6.95	89	1.81	2.19

Test results that did not meet the specification limits given in AASHTO M 295 are underlined in Table 9. Two samples of OGS fly ash tested over 5% for sulfur trioxide (SO₃). These two samples were taken on a single day in quick succession (designated H-1 and H-2) near the maximum trona dosage rate at OGS. The two samples tested nearly identical and they were retained in the study to act as natural duplicates. Two additional OGS samples, denoted as L-1 and L-2, were obtained in a similar manner but earlier in the day. These two samples were measurably different from each other; this is in contrast to the two H samples. These four samples represented OGS near the high limit of the dosage schedule that usually precedes a power plant maintenance outage (Schlorholtz and Bergeson 1993).

Overall, the alkali content of the samples ranged from approximately 2 to 5% (total alkali) and about 1 to 3% (available alkali). A plot of the available alkali content versus the total alkali content of the fly ash samples is given in Figure 15. Most of the fly ash samples had available alkali contents less than the specification limit (1.5%); however, all of the OGS samples tested above the specification limit. Four of the OGS samples tested above the 2.5% Iowa DOT specification limit. Only one Class F fly ash was originally included in this study; however, FA-I was included to expand the lower limit of the available alkali content to less than 1%.

Table 9. Results of chemical analysis of fly ash (minor oxides and available alkali).

Fly Ash	MgO, %	SO ₃ , %	Na ₂ O, %	K ₂ O, %	Total alkali (Na ₂ O _e , %)	Available alkali (Na ₂ O _e , %)
HAV051112	4.73	1.14	1.45	0.61	1.85	1.10
CBF051512	4.69	1.81	1.77	0.58	2.15	1.34
PN4051412	5.89	2.89	1.74	0.30	1.94	1.07
COA051512	4.50	0.60	2.11	2.47	3.74	1.04
OGS051512	6.69	3.71	3.54	0.29	3.73	<u>2.09</u>
CLI051412	4.63	1.73	1.71	0.47	2.02	1.43
COL072712	5.24	1.79	1.86	0.49	2.18	1.44
PLP072312	4.06	1.66	1.67	0.57	2.05	1.20
EDG072712	5.15	1.91	1.75	0.44	2.04	1.27
OGS073012	6.89	4.04	3.50	0.31	3.70	<u>2.22</u>
IAT080812	5.35	1.15	1.70	0.53	2.05	1.22
OGS081312	7.07	4.93	3.86	0.30	4.06	<u>2.68</u>
OGS083012L-1	6.83	4.91	4.73	0.34	4.95	<u>2.81</u>
OGS083012L-2	6.87	4.64	4.49	0.31	4.70	<u>2.65</u>
OGS083012H-1	6.91	<u>5.68</u>	4.54	0.33	4.75	<u>2.71</u>
OGS083012H-2	6.89	<u>5.66</u>	4.52	0.33	4.74	<u>2.83</u>
RUS092012	5.70	1.44	2.16	0.39	2.42	1.36
LAB092012	4.54	1.81	1.66	0.60	2.05	1.16
NBCCOMP7-12	4.77	2.68	1.75	0.36	1.99	1.13
NOSCOMP5-12	4.88	1.72	1.70	0.45	1.99	1.38
OTTCOMP12-98	4.42	1.27	2.36	0.48	2.68	<u>1.68</u>
FA-I	1.33	0.33	0.89	1.93	2.16	0.81

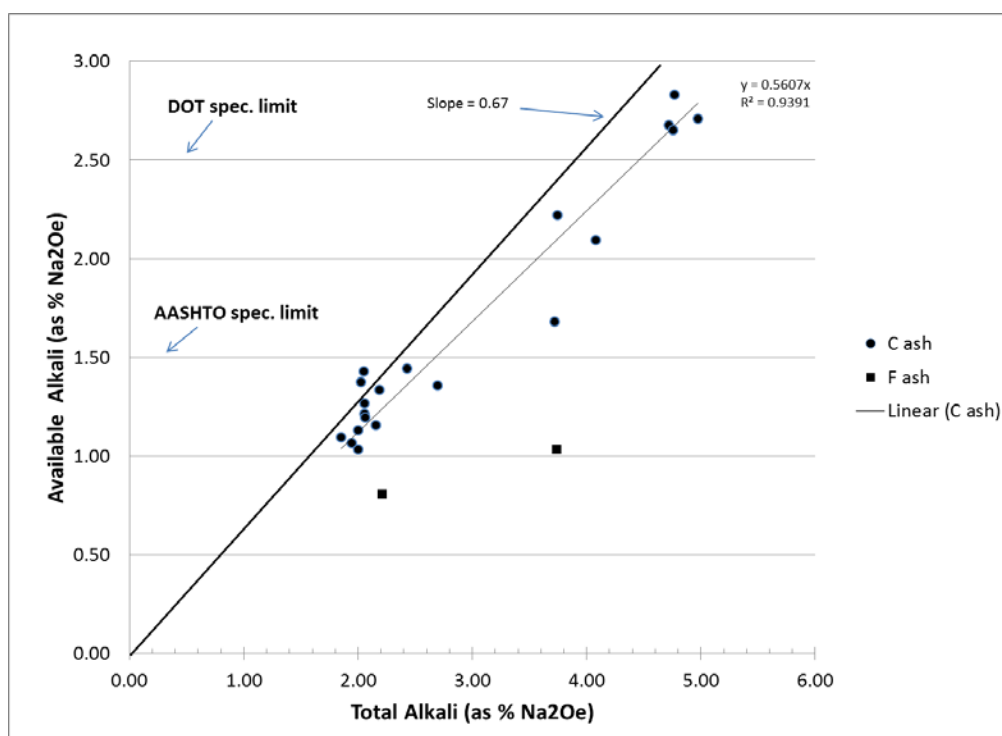


Figure 15. A plot of available alkali versus total alkali for the samples in this study

Table 10. Results of chemical analysis of fly ash (other oxides and moisture content).

Fly Ash	P ₂ O ₅ , %	TiO ₂ , %	SrO, %	Mn ₂ O ₃ , %	BaO, %	Moisture, %
HAV051112	0.99	1.50	0.32	0.03	0.69	0.02
CBF051512	1.35	1.59	0.47	0.03	0.85	0.07
PN4051412	0.74	1.67	0.42	0.03	0.80	0.00
COA051512	0.19	0.63	0.26	0.07	0.57	0.00
OGS051512	0.78	1.23	0.53	0.03	0.87	0.04
CLI051412	1.06	1.60	0.45	0.03	0.78	0.00
COL072712	1.10	1.62	0.43	0.03	0.81	0.03
PLP072312	1.07	1.46	0.44	0.03	0.68	0.01
EDG072712	0.82	1.61	0.41	0.05	0.77	0.03
OGS073012	0.62	1.40	0.50	0.02	0.84	0.00
IAT080812	1.12	1.50	0.39	0.04	0.80	0.10
OGS081312	1.02	1.24	0.56	0.03	0.98	0.06
OGS083012L-1	0.58	1.03	0.50	0.03	0.81	0.00
OGS083012L-2	0.60	1.10	0.50	0.03	0.81	0.03
OGS083012H-1	0.66	1.04	0.52	0.03	0.86	0.01
OGS083012H-2	0.67	1.04	0.52	0.03	0.86	0.00
RUS092012	1.43	1.33	0.49	0.01	0.91	0.08
LAB092012	1.14	1.47	0.35	0.03	0.74	0.27
NBCCOMP7-12	1.67	1.51	0.46	0.02	0.95	0.09
NOSCOMP5-12	1.23	1.52	0.42	0.03	0.80	0.02
OTTCOMP12-98	1.58	1.60	0.44	0.03	0.83	0.09

Table 11. Fly ash alkali content information (water soluble and available alkali).

Fly Ash	Water-soluble (Na ₂ O, %)	Water-soluble (K ₂ O, %)	Water-soluble (as Na ₂ O _e , %)	Available alkali (Na ₂ O, %)	Available alkali (K ₂ O, %)	Available alkali (as Na ₂ O _e , %)
HAV051112	0.05	<0.01	0.05	0.90	0.30	1.10
CBF051512	0.10	0.01	0.10	1.13	0.31	1.34
PN4051412	0.12	<0.01	0.13	0.98	0.14	1.07
COA051512	0.07	0.01	0.08	0.64	0.60	1.04
OGS051512	0.71	0.01	0.72	1.99	0.16	2.09
CLI051412	0.09	<0.01	0.09	1.24	0.29	1.43
COL072712	0.15	<0.01	0.15	1.28	0.28	1.46
PLP072312	0.10	0.01	0.11	1.02	0.26	1.20
EDG072712	0.08	<0.01	0.08	1.10	0.25	1.27
OGS073012	0.76	0.01	0.77	2.08	0.21	2.22
IAT080812	0.07	0.01	0.08	1.04	0.27	1.22
OGS081312	1.00	0.01	1.01	2.60	0.22	2.74
OGS083012L-1	1.15	0.02	1.16	2.68	0.21	2.81
OGS083012L-2	1.09	0.03	1.11	2.53	0.18	2.65
OGS083012H-1	1.39	0.03	1.41	2.58	0.19	2.71
OGS083012H-2	1.35	0.03	1.37	2.68	0.24	2.83
RUS092012	0.08	<0.01	0.08	1.22	0.21	1.36
LAB092012	0.09	<0.01	0.09	0.96	0.30	1.16
NBCCOMP7-12	0.07	0.01	0.08	1.01	0.19	1.13
NOSCOMP5-12	0.07	<0.01	0.07	1.19	0.29	1.38
OTTCOMP12-98	0.08	<0.01	0.08	1.51	0.26	1.68

An interesting trend was noted between the total alkali (sodium only) and the soluble alkali content of the fly ash samples (see Figure 16). All of the samples, with the exception of the OGS samples, had low water-soluble alkali contents. Only one OGS sample (OTTCOMP12-98) had a low water-soluble alkali content. There appears to be a significant gap in the data which can probably be attributed to the limited time period that the OGS samples were taken (basically from May through August). This suggests that when the OGS power plant is adding trona at low rates the alkali (primarily sodium) can be incorporated into fly ash particles; however, as the rate increases, it eventually only attaches weakly (as a surface condensate) on the ash particles. A similar trend was noted between water-soluble alkali and available alkali test results; however, that was expected because they are not independent measurements.

The results of the physical tests are summarized in Table 12. All of the fly ashes easily met the requirements specified in AASHTO M 295. One trend that was noticed was that the high-alkali ashes from OGS tended to have higher 7-day strength activity index results than the 28-day values. The number of samples is too small to be statistically valid and the differences push the precision limit of the test method, but it did seem odd that most of the OGS samples would exhibit this behavior.

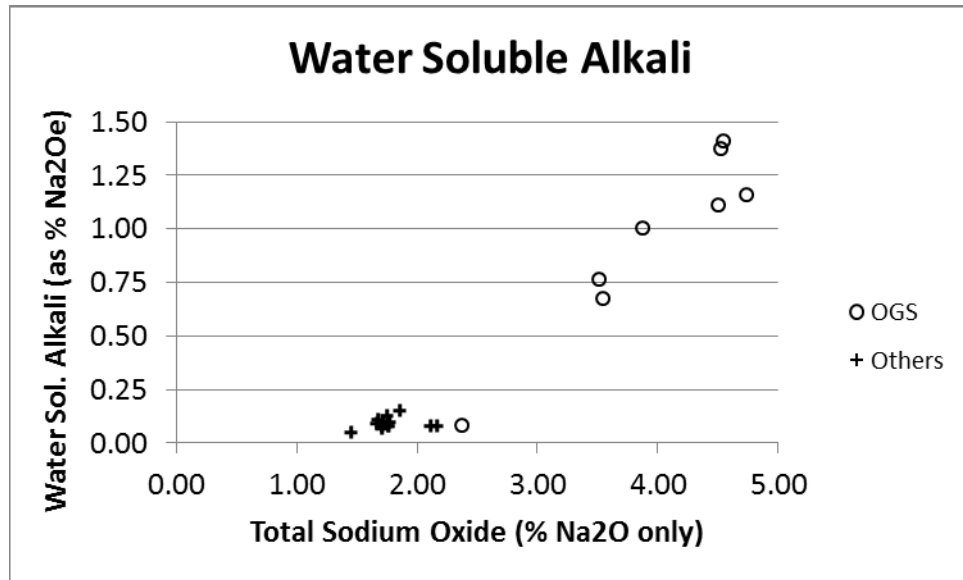


Figure 16. Water soluble alkali versus total alkali for the samples in this study

Table 12. Results of physical tests conducted on fly ash.

Fly Ash	Density (g/cm ³)	Autoclave Expansion (%)	Fineness (% retained)	SAI-7d (% control)	SAI-28d (% control)	Water req'd (% control)
HAV051112	2.60	0.03	13.9	94	103	95
CBF051512	2.67	0.05	12.5	92	101	95
PN4051412	2.72	0.08	22.6	92	99	95
COA051512	2.45	0.01	22.7	81	91	95
OGS051512	2.78	0.11	14.6	94	96	95
CLI051412	2.70	0.04	12.1	101	110	95
COL072712	2.72	0.04	13.3	95	99	95
PLP072312	2.50	0.02	19.0	92	95	95
EDG072712	2.74	0.06	16.2	99	97	95
OGS073012	2.80	0.12	19.0	102	96	95
IAT080812	2.64	0.04	14.7	96	97	95
OGS081312	2.82	0.11	7.2	105	104	95
OGS083012L-1	2.76	0.17	15.9	100	91	95
OGS083012L-2	2.75	0.17	17.4	101	87	95
OGS083012H-1	2.78	0.15	18.8	99	94	95
OGS083012H-2	2.79	0.14	18.7	103	95	95
RUS092012	2.73	0.06	19.2	99	97	95
LAB092012	2.68	0.03	11.2	91	100	95
NBCCOMP7-12	2.60	0.05	12.9	93	100	95
NOSCOMP5-12	2.67	0.05	12.1	90	105	95
OTTCOMP12-98	2.61	0.04	15.4	92	101	95

The results of chemical analysis on the cements and slag that were used in this study are summarized in Table 13. The cement denoted as “ASR” was the material that was used for the mortar-bar expansion tests conducted in accordance with Iowa DOT IM 491.17. That column represents the average of four individual batches (all batches had equivalent alkali contents within $\pm 0.02\%$ of the average).

Table 13. Results of chemical tests conducted on cements and slag.

Oxide (%)	Lafarge I/II	ASR	St Marys	Lafarge I	Ash Grove IP	Slag
SiO ₂	19.8	20.1	19.7	20.5	30.0	37.0
Al ₂ O ₃	4.6	4.9	5.0	4.7	8.6	11.2
Fe ₂ O ₃	3.0	2.9	3.0	2.9	3.9	0.6
CaO	62.5	62.9	62.3	64.0	47.8	39.9
MgO	2.9	2.9	3.7	2.3	3.0	10.8
SO ₃	3.4	2.8	3.3	2.4	3.5	not meas'd
S	not meas'd	not meas'd	not meas'd	not meas'd	not meas'd	1.06
Na ₂ O	0.1	0.2	0.2	0.2	0.3	0.4
K ₂ O	0.7	0.85	1.3	0.5	0.7	0.5
Equiv. Alkali (as % Na ₂ O _e)	0.54	0.74	1.04	0.51	0.75	0.73
SrO	0.05	0.06	0.04	not meas'd	0.15	0.05
TiO ₂	0.23	0.25	0.26	not meas'd	0.5	0.5
P ₂ O ₅	0.13	0.10	0.07	not meas'd	0.12	< 0.05
Mn ₂ O ₃	0.50	0.11	0.08	not meas'd	0.07	0.3
LOI	2.0	1.9	1.3	2.0	1.3	not meas'd

Precision of the Chemical Measurements

Pooled estimates of precision were used when possible because this tends to provide more robust estimates (Taylor 1990). Briefly, this consists of testing duplicate samples and evaluating whether the test response is stable over the range of interest. If the difference between duplicates is well-behaved [see Figures 17 and 18, data from Sutter et al. 2013)] then the sums-of-squares of the differences are pooled to estimate the standard deviation (1s). This is similar to the method described in ASTM C 670 where the evaluation is made using the standard deviation as a function of test response.

The repeatability estimates for the chemical tests are tabulated in this section (see Tables 14 and 15). Some of the precision estimates are extracted from a recent study (Sutter et al. 2013) because the standard operating procedures for the tests were identical to that study.

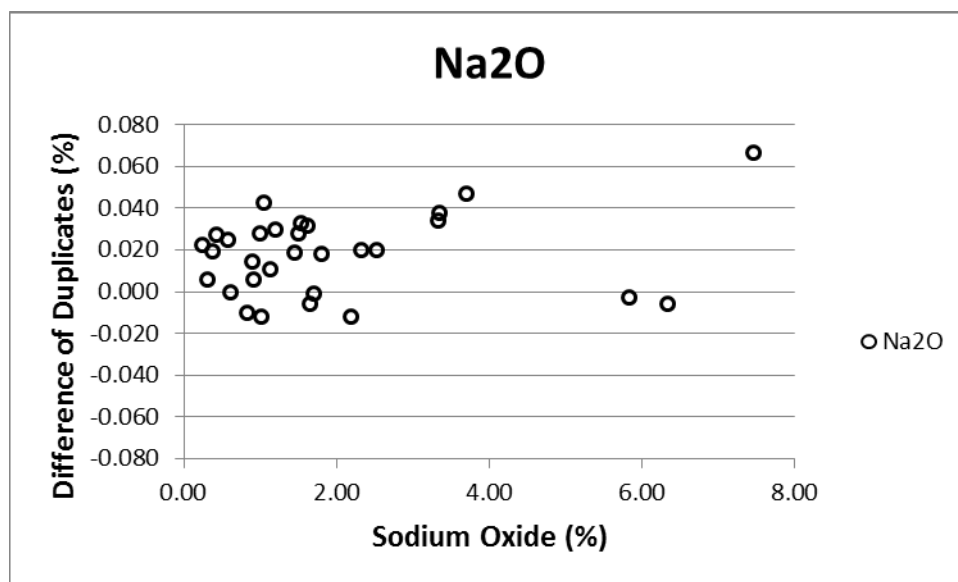


Figure 17. Difference between duplicate fused disks versus concentration (sodium)

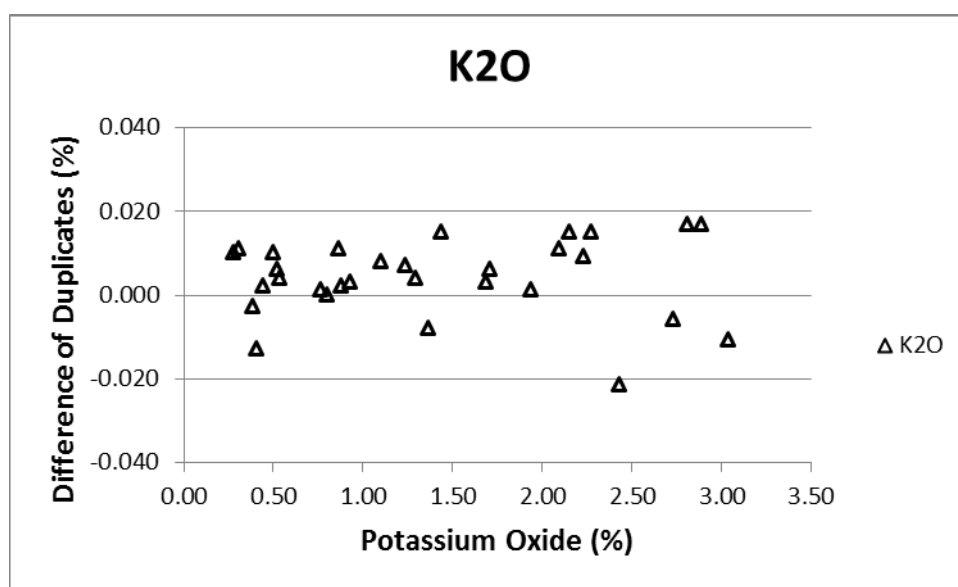


Figure 18. Difference between duplicate fused disks versus concentration (potassium)

An extra decimal place was used to express the precision data. This was done to allow readers to perform additional calculations without producing significant rounding errors. In general, most chemical test results should only be reported to two decimal places (or less in many instances, judging by the statistics listed in the tables). Note that C311 does not list precision information for either sodium or potassium (see Table 14). This is unfortunate. However, all of the precision information is rather dated and really needs to be considered for updating by the appropriate ASTM Subcommittee (C09.24 in this instance).

Comparing the statistics summarized in the tables indicates that the precision of the fused disk method for XRF is much better than the available alkali test. Typically, the d2s values for sodium and potassium via XRF are about the same as the 1s value for the available alkali test. This is important to remember when one desires to estimate the error in the total alkali content that is calculated from the independent determinations of sodium and potassium. The precision of the soluble alkali content is dominated by the sodium content.

Table 14. Summary of the precision of the XRF assays versus C 311 (within-lab)

	Na ₂ O (%)	MgO (%)	Al ₂ O ₃ (%)	SiO ₂ (%)	SO ₃ (%)	K ₂ O (%)	CaO (%)	Fe ₂ O ₃ (%)
1s (pooled)	0.018	0.013	0.063	0.18	0.022	0.007	0.058	0.046
d2s	0.05	0.04	0.18	0.50	0.06	0.02	0.16	0.13
C 311 -within lab precision								
1s	not given	0.20	1.46	0.62	0.10	not given	0.63	0.12
d2s	not given	0.55	4.12	1.75	0.28	not given	1.79	0.34

Table 15. Precision of available alkali and soluble alkali test methods (within-lab).

Available Alkali test	This Study (n=7 duplicates)			NCHRP Study (n=29 duplicates)		
	Na ₂ O, %	K ₂ O, %	Total alkali (Na ₂ O _e , %)	Na ₂ O, %	K ₂ O, %	Total alkali (Na ₂ O _e , %)
1s (pooled)	0.043	0.014	0.048	0.033	0.025	0.039
1s% (COV)	2.4	5.7	2.4	4.2	4.8	3.5
d2s	0.12	0.04	0.13	0.09	0.07	0.11
Range of values	1 to 2.6	0.16 to 0.31	1.1 to 2.8	-	-	0.5 to 2.3
Soluble Alkali Test (n=6 duplicates)						
1s (pooled)	0.03	0.008	0.03	-	-	-
1s% (COV)	4.8	30	4.8	-	-	-
d2s	0.09	0.02	0.09	-	-	-
Range of values	0.08 to 1.4	0.001 to 0.03	0.08 to 1.4	-	-	-

Checking the Iowa DOT Available Alkali Model

The Iowa DOT quickly noted the need for a fast estimate of the available alkali content of fly ash (Cheng 2000). As was noted earlier in this report, this has been a major practical problem for over 40 years. Since the DOT had access to bulk chemical information via XRF (fused disk technique) they used the total alkali information to estimate the available alkali content. The model for making this calculation is shown in Figure 19 and additional details are listed in Table 16. The model was constructed via linear regression and it has been “tweaked” a couple of times to achieve better agreement. A quick look back at Table 2 indicates that the “tweaks” have had little real impact on the available alkali results predicted by the model because of the poor precision inherent in the test method. The models are diverging at high available alkali contents and that is a concern. However, it is pertinent to repeat the purpose of the model. The model is simply used to estimate the available alkali content. If the available alkali content is greater than 1.5% (the low limit) and less than 2.5% (the high limit) then the fly ash needs to be subjected to further testing for approval (mortar-bar testing). When the estimated available alkali content of the fly ash exceeds the high limit, the Iowa DOT normally requests that the marketing agency perform the available alkali test. If the actual test result exceeds 2.5%, then the fly ash cannot be used in state projects. At first thought this seems rather harsh; however, it actually provides significant time saving for the DOT Central Lab and avoids the many arguments that arise with fly ashes near the 1.5% limit. The biggest problem with the strategy is that no CRMs are currently available to validate the available alkali test method and the 2.5% limit. Therefore, the models were checked using data collected during this study (20 samples of Class C fly ash) and a recent NCHRP fly ash project (Sutter et al. 2013) that analyzed 12 samples of Class C fly ash.

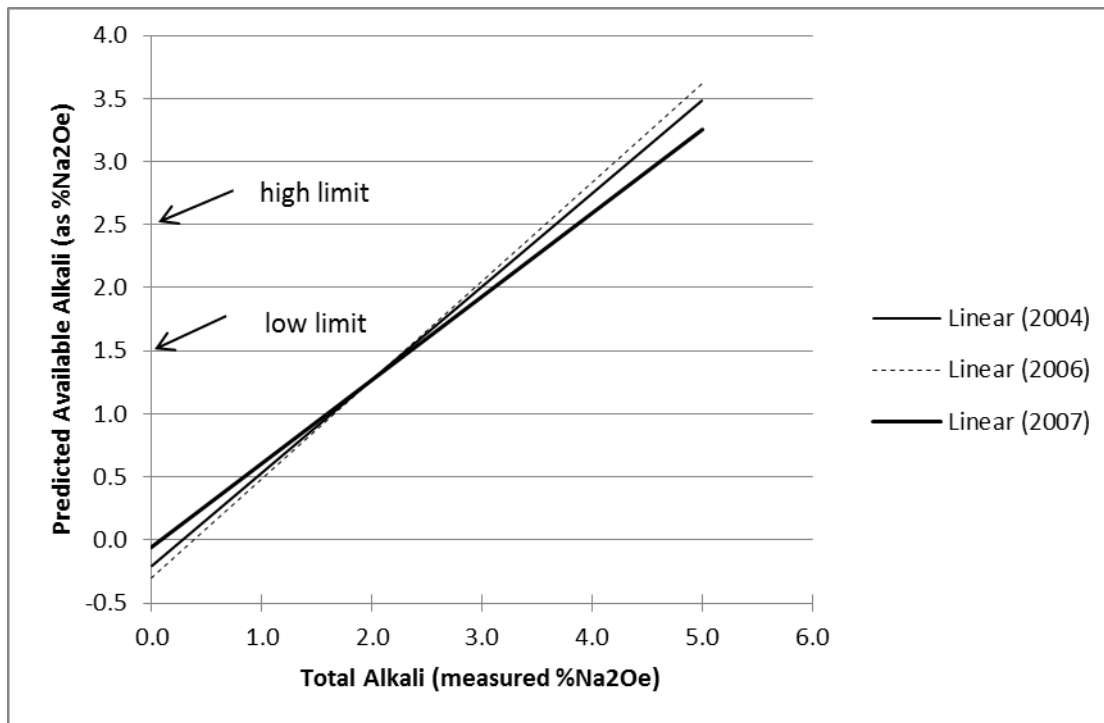


Figure 19. Iowa DOT model for predicting available alkali

Table 16. Summary of DOT models for predicting available alkali

Total Alkali (%Na ₂ O _e)	Predicted Available Alkali (as % Na ₂ O _e)			Iowa DOT criteria
	2004 (slope=0.74)	2006 (slope=0.78)	2007 (slope=0.66)	
0.0	-0.21	-0.30	-0.06	
1.0	0.53	0.48	0.60	
1.5	0.90	0.88	0.93	
2.0	1.27	1.27	1.26	
2.36	1.53	1.55	1.50	Low limit
3.0	2.00	2.05	1.93	
3.86	2.64	2.73	2.50	High limit
5.0	3.48	3.62	3.25	

The metric researchers really desire to estimate at this point is the accuracy of the model; however, that is not possible due to the lack of certified standards. Hence, we will have to be satisfied with being in good agreement with companion labs that perform the same test method. The results of comparing the DOT model (only the model denoted as 2007) and the measured available alkali content are shown in Figure 20. The range of interest is from about zero to 3% available alkali, and the DOT model does a reasonable job over that range (roughly $\pm 0.4\%$ absolute). It is important to note that the comparison is based on between-lab test results and at a 2.5% available alkali content, the precision is about $\pm 0.5\%$ (1s). The CCRL data were not used for this direct comparison because of the uncertainty issues described earlier in this report. Even so, the calculated values are in reasonable agreement with the actual measured values for Class C fly ash (see Figure 21).

The precision of an individual total alkali determination can be calculated using the precision values given in Table 14. Combining the sodium and potassium variances indicates that the precision of calculating the total alkali content is about $\pm 0.02\%$ (1s). Hence, for practical purposes the error in the calculation can be ignored. This is especially true when considering that the accuracy of the estimate is going to dominate the final reported test result.

In summary, this section evaluated the Iowa DOT model for calculating the available alkali content of fly ash. The model is based on an empirical relationship between the available alkali and total alkali content of Class C fly ash. The model is very precise (good to better than $\pm 0.05\%$ (2s)) when the total alkali determinations met the precision indices given in this report. The accuracy of the model was evaluated by comparing predicted values with measured values. This suggested that the accuracy was good over the range of interest. The model could be extended to provide estimates for all fly ash samples by using a relationship similar to that shown in Figure 21; this will be discussed later in this report.

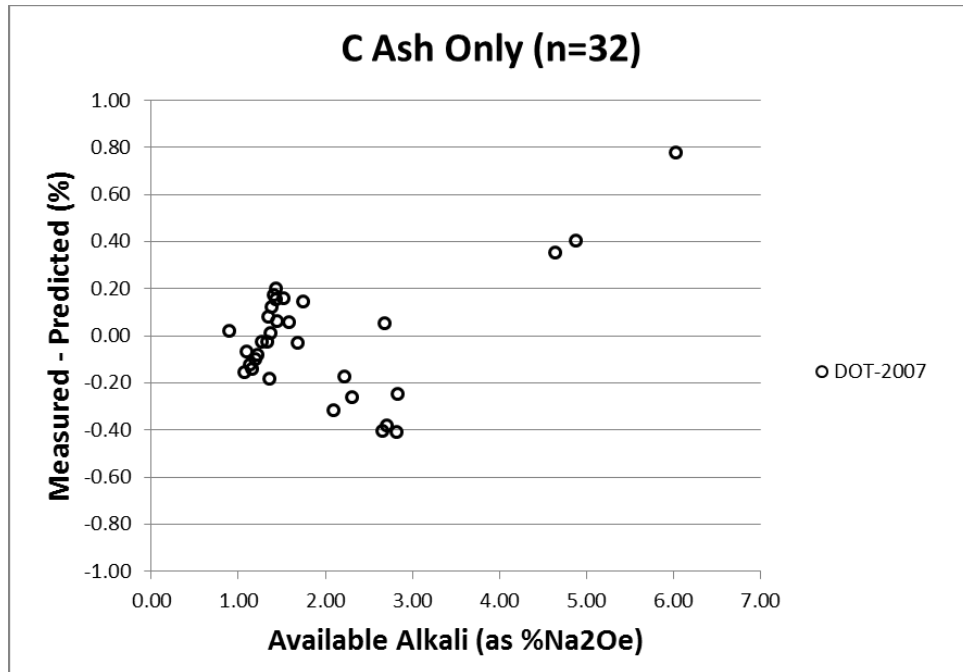


Figure 20. Difference of measured versus predicted available alkali for this study

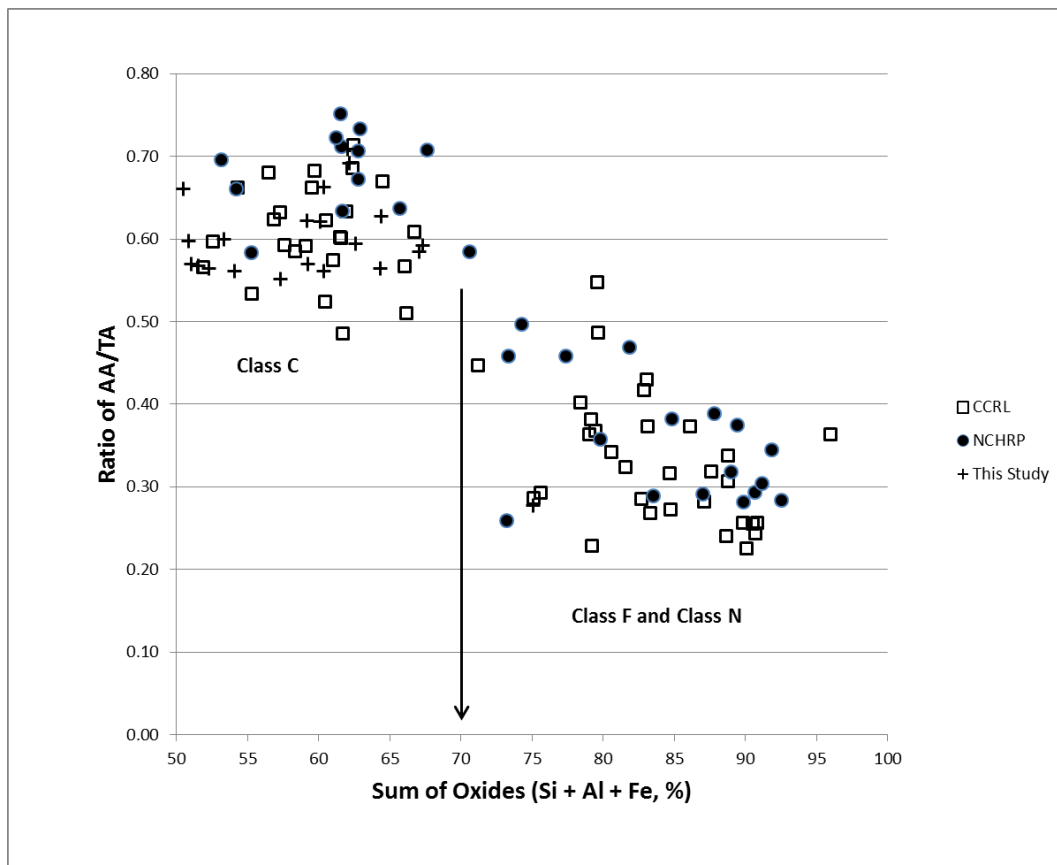


Figure 21. Ratio of available alkali to total alkali versus fly ash Class.

Several attempts were made to construct more complex statistical models (basically linear regression) from the chemical information. This yielded some interesting models, but in nearly all instances the models' ability to make accurate predictions did not improve much from the very simple statistical model currently used by the Iowa DOT. After much work, it became apparent that a major part of the difficulty was probably caused by multicollinearity in the data. This simply means that the choice of independent variable is difficult because of very significant cross correlations with other variables. This is illustrated in Table 17, which shows a partial correlation matrix for fly ash composition and other tests of interest to this study. This matrix is normally symmetric about the main diagonal; however many columns have been removed to save space. The circled values show highly significant correlations, and the bold values denote significance at the 5% level.

Since the new models did not do significantly better than the existing model they were rejected. A better strategy for building a robust model would consist of selecting a few physical properties that impact performance. This would help eliminate the many variables that have strong statistical associations but no real physical meaning.

Table 17. Partial correlation matrix for fly ash chemistry and other tests.

	Na ₂ O	MgO	SiO ₂	SO ₃	CaO	Sum	Avail Alk
Na ₂ O	1.000	0.882	-0.672	0.915	0.534	-0.788	0.975
MgO	0.882	1.000	-0.788	0.895	0.727	-0.892	0.867
Al ₂ O ₃	-0.824	-0.844	0.451	-0.773	-0.419	0.659	-0.729
SiO ₂	-0.672	-0.788	1.000	-0.826	-0.964	0.968	-0.756
P ₂ O ₅	-0.515	-0.491	-0.005	-0.418	0.039	0.179	-0.408
SO ₃	0.915	0.895	-0.826	1.000	0.722	-0.909	0.919
K ₂ O	-0.261	-0.420	0.846	-0.467	-0.853	0.705	-0.379
CaO	0.534	0.727	-0.964	0.722	1.000	-0.935	0.615
TiO ₂	-0.653	-0.452	-0.074	-0.440	0.194	0.141	-0.525
Fe ₂ O ₃	0.788	0.797	-0.436	0.685	0.319	-0.558	0.708
SrO	0.683	0.719	-0.887	0.760	0.838	-0.881	0.753
Mn ₂ O ₃	-0.187	-0.276	0.636	-0.340	-0.606	0.511	-0.274
BaO	0.374	0.529	-0.782	0.520	0.781	-0.742	0.456
Sum	-0.788	-0.892	0.968	-0.909	-0.935	1.000	-0.833
Avail Alk	0.975	0.867	-0.756	0.919	0.615	-0.833	1.000
Water Sol Alk	0.975	0.882	-0.709	0.957	0.578	-0.815	0.966
15% rep @56-d	0.728	0.856	-0.899	0.800	0.915	-0.942	0.770
30% rep@56-d	0.411	0.663	-0.819	0.536	0.899	-0.795	0.486

bold are significant at p=0.05

Mortar-Bar Expansion Testing

The results of mortar-bar expansion tests are presented in this section. The tests consisted of mixtures containing ASR cement (Iowa DOT specification) and tests containing blended cements (slag and Type IP). These mixtures contained Pyrex glass as the aggregate with fly ash alkali content considered as a major variable. In addition, two different test methods were conducted that utilized Wayland sand as the aggregate (ASTM C227 and C1567) to evaluate its potential for expansion due to ASR.

The results of the mortar-bar expansion tests conducted with ASR cement are given in Table 18. These tests were conducted to compare to the Iowa DOT specification limit. Typically, only the fly ash samples that exhibited available alkali contents above 1.5% are subjected to mortar-bar expansion testing (this would have only tested the eight OGS fly ash samples). However, in this study all fly ashes were subjected to mortar-bar expansion tests using replacement (equal mass of fly ash for cement) levels of 15% and 30%. An inert material (Sil-Co-Sil 100) was used to estimate the dilution effect of removing cement from the mix.

Table 18. Mortar-bar expansion test results, fly ash samples (increasing alkali content).

Sample	15% Replacement			30% Replacement		
	14-days, %	28-days, %	56-days, %	14-days, %	28-days, %	56-days, %
FA-I	0.182	0.199	0.203	0.102	0.101	0.104
HAV051112-1	0.282	0.299	0.322	0.249	0.270	0.278
PN4051412-1	0.349	0.391	0.418	0.304	0.337	0.355
COA051512-1	0.206	0.227	0.240	0.117	0.125	0.126
NBCCOMP7-12	0.332	0.349	0.373	0.249	0.267	0.280
NOSCOMP5-12	0.333	0.352	0.381	0.263	0.287	0.305
CLI051412-1	0.331	0.358	0.385	0.270	0.287	0.304
EDG072712	0.354	0.387	0.417	0.263	0.286	0.308
IAT080812	0.331	0.354	0.384	0.292	0.313	0.329
PLP072312	0.256	0.276	0.290	0.213	0.233	0.241
LAB092012	0.246	0.267	0.282	0.175	0.193	0.201
CBF051512-1	0.314	0.364	0.393	0.285	0.306	0.323
COL072712	0.342	0.363	0.391	0.282	0.305	0.325
RUS092012	0.335	0.356	0.387	0.309	0.333	0.354
OTTCOMP12-98	0.289	0.314	0.339	0.214	0.231	0.244
OGS073012	0.353	0.408	0.431	0.275	0.328	0.359
OGS051512-1	0.372	0.423	0.449	0.254	0.301	0.322
OGS081312	0.366	0.432	0.458	0.241	0.300	0.332
OGS083012L-2	0.379	0.474	0.496	0.251	0.311	0.335
OGS083012H-2	0.336	0.425	0.450	0.253	0.316	0.342
OGS083012H-1	0.346	0.429	0.458	0.238	0.288	0.311
OGS083012L-1	0.377	0.454	0.485	0.272	0.342	0.371
Sil-Co-Sil (inert)	0.375	0.409	0.441	0.292	0.316	0.330

Precision of the Mortar-bar Measurements

The precision estimates for the control mixture are given in Table 19. The estimate was made because of a slight change from the normal procedure described in ASTM C311. These test mixtures consisted of two specimens rather than the three described in C311. This was done to increase the throughput of the experiment. The precision of the control mixture (ASR cement and Pyrex glass, no fly ash) was calculated using seven individual mixtures (of two bars each) mixed on different days, over the duration of the experiment (many months). The test results in Table 19 gave slightly better precision values than those obtained from a recent study conducted in this lab (Sutter et al. 2013). In that study, the 1s values ranged from 0.03% to 0.05% and d2s values ranged from 0.09% to 0.14% (for 56-day expansion values, depending on the alkali content of the cement that was tested). It was concluded that the use of duplicate bars was slightly more precise than using triplicate bars.

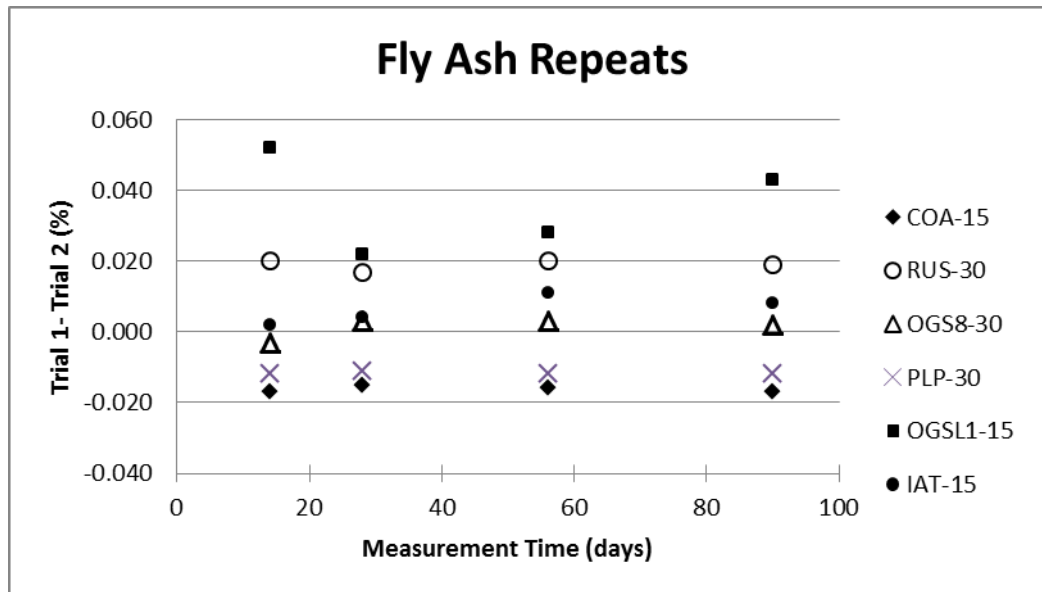
Table 19. Test method precision calculations, mortar-bar expansion tests, ASR cement.

Trial	14-days, %	28-days, %	56-days, %	90-days, %
1	0.434	0.472	0.514	not meas'd
2	0.365	0.429	0.466	not meas'd
3	0.370	0.471	0.493	not meas'd
4	0.403	0.478	0.512	0.516
5	0.372	0.480	0.505	0.508
6	0.363	0.481	0.501	0.504
7	0.359	0.465	0.491	0.494
Statistics				
average	0.381	0.468	0.497	0.506
1s	0.028	0.018	0.016	0.009
COV, %	7.2	3.9	3.3	1.8
d2s	0.08	0.05	0.05	0.03

The precision estimates for the test mixtures are given in Table 20. The estimates were made by pooling the test results of duplicate determinations made on six different fly ash samples that covered the range of expansions observed in this study. The experiment was balanced by using an equal number of replicates containing 15% and 30% replacement. The trends are illustrated in Figure 22. Most of the trends are relatively flat as a function of time and test response so the pooled estimate appears to be adequate. The very high-alkali fly ash (OGS083012-L1) did exhibit more variation than the other five samples of fly ash. The estimate was made because of a slight change from the normal procedure described in ASTM C311. These test mixtures consisted of two specimens rather than the three (as was described earlier). The test results in Table 20 gave slightly better precision values than those obtained from a recent study conducted in this lab (Sutter et. al. 2013). In that study, the 1s value was 0.02% and the d2s value was 0.06% (for 56-day expansion values and mixtures containing high-alkali cement). These precision indices were assumed to pertain to the blended cement mixtures. A d2s value of about 0.04 to 0.05% seems to be a good compromise for the observed precision of the control specimens and test specimens containing fly ash.

Table 20. Precision of fly ash repeats, mortar-bar expansion tests, ASR cement.

Fly Ash	% Replacement	Difference Between Trial 1 and Trial 2			
		14-days, %	28-days, %	56-days, %	90-days, %
COA051512-1	15	-0.017	-0.015	-0.016	-0.017
RUS092012	30	0.020	0.017	0.020	0.019
OGS081312	30	-0.003	0.003	0.003	0.002
PLP072312	30	-0.012	-0.011	-0.012	-0.012
OGS083012L-1	15	0.052	0.022	0.028	0.043
IAT080812	15	0.002	0.004	0.011	0.008
Statistics					
1s (pooled)		0.017	0.010	0.012	0.015
d2s		0.05	0.03	0.03	0.04

**Figure 22. Difference between duplicate mixtures for fly ash specimens***Influence of fly ash composition and dosage*

The mortar-bar expansion tests containing fly ash exhibited interesting trends with composition, fly ash replacement and measurement time. Tables 18 and 19 clearly indicated that expansion was strongly related to measurement time, both for the specimens containing fly ash and the control specimens. This trend, shown back in Figure 12, is also illustrated in a slightly different manner in Figure 23. The major compositional variable (available alkali content) is plotted on the x-axis of Figure 23. The duration of time increases from the bottom (14-days) to the top graph in the figure (56-days). The expansion of the control specimens are plotted as an “X” on the y-axis (except at 56-days it was 0.50% so it would be at the top of the y-axis). At early time periods, the test response was nearly flat for the different Class C

fly ash samples. As time increased, the data appeared to spread apart so that differences between fly ashes could be observed. Hence, the test response really had to be evaluated after 28 days (or later) rather than the 14 days recommended by the test method.

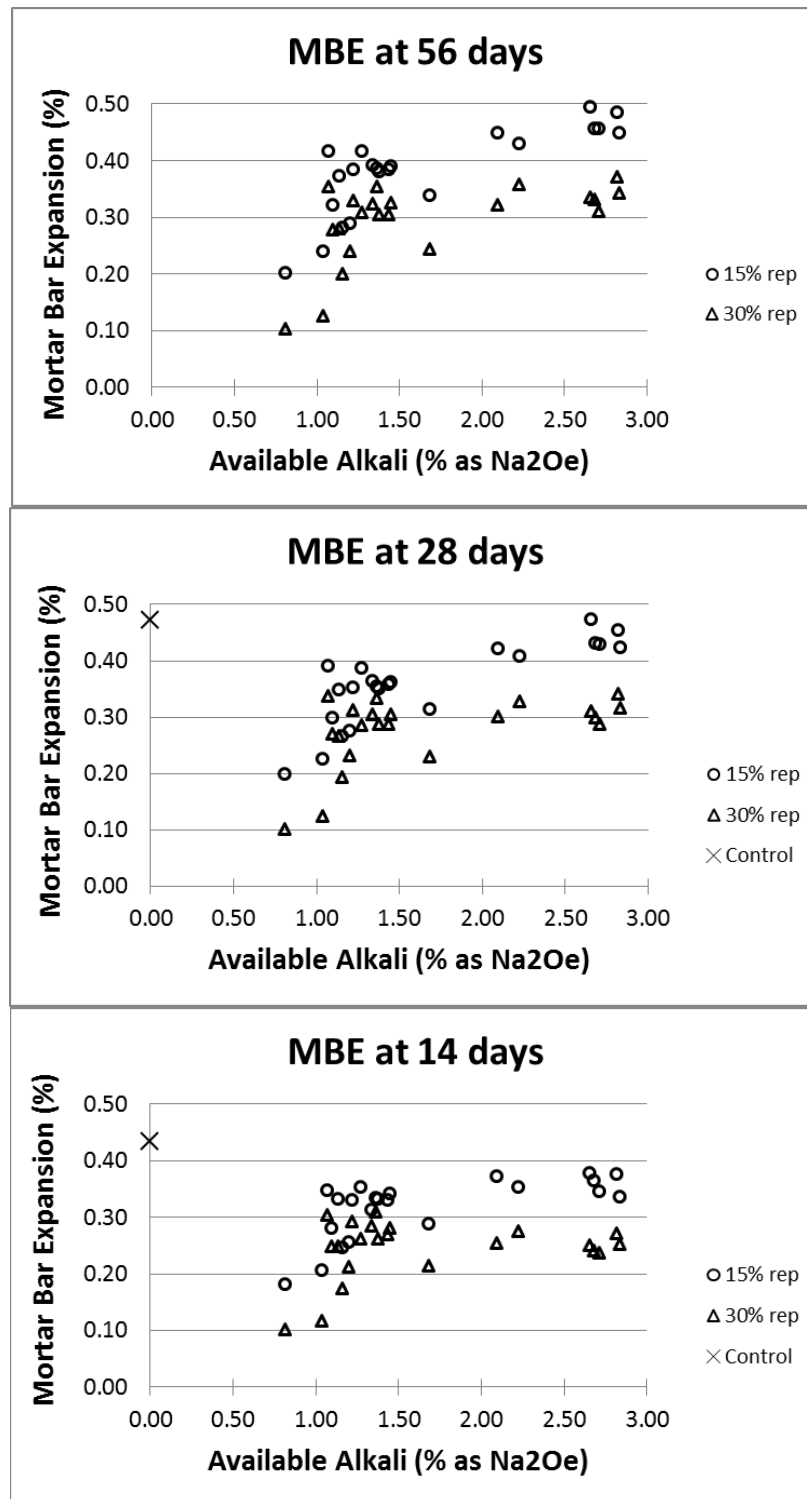


Figure 23. Influence of time and available alkali content on the mortar-bar tests

Better renditions of Figure 23 are given in Figures 24 and 25. Now the high and low available alkali limits have been placed on the graphs as per the Iowa DOT requirements. Also, the test results for mortar bars containing an inert sand (rather than fly ash) have also been placed on the graphs. The figures show a relationship between mortar-bar expansion and available alkali content when used at a 15% replacement. This relationship was not evident when the replacement was increased to 30%. Mortar bars containing the Class F fly ashes performed the best (lowest expansion values at both 15% and 30% replacement). The Class C fly ashes generally did not perform much better than the inert material when used at similar replacement levels; however, there were exceptions. The exceptions did not appear

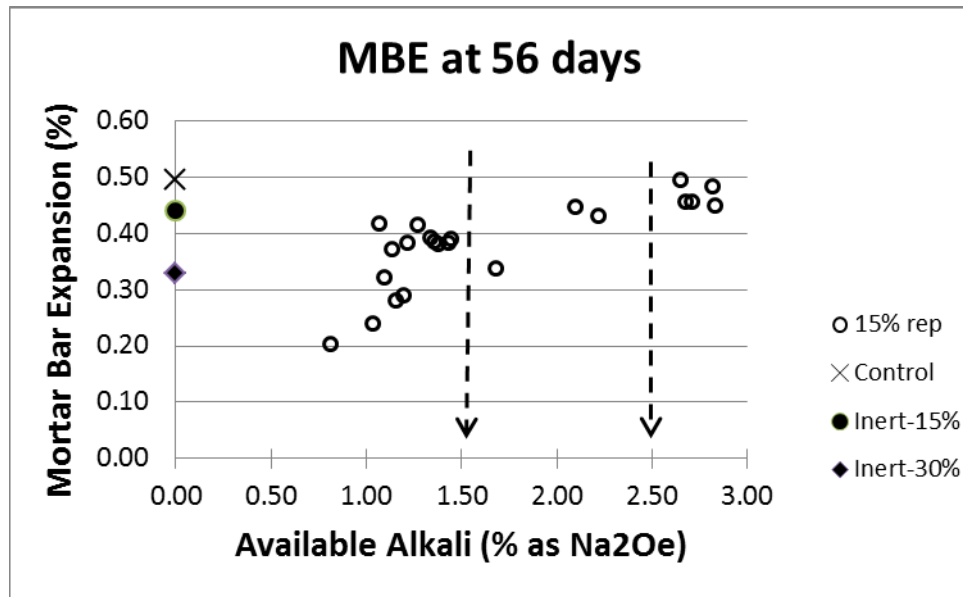


Figure 24. Mortar-bar expansion (56-d) versus available alkali content (15% ash)

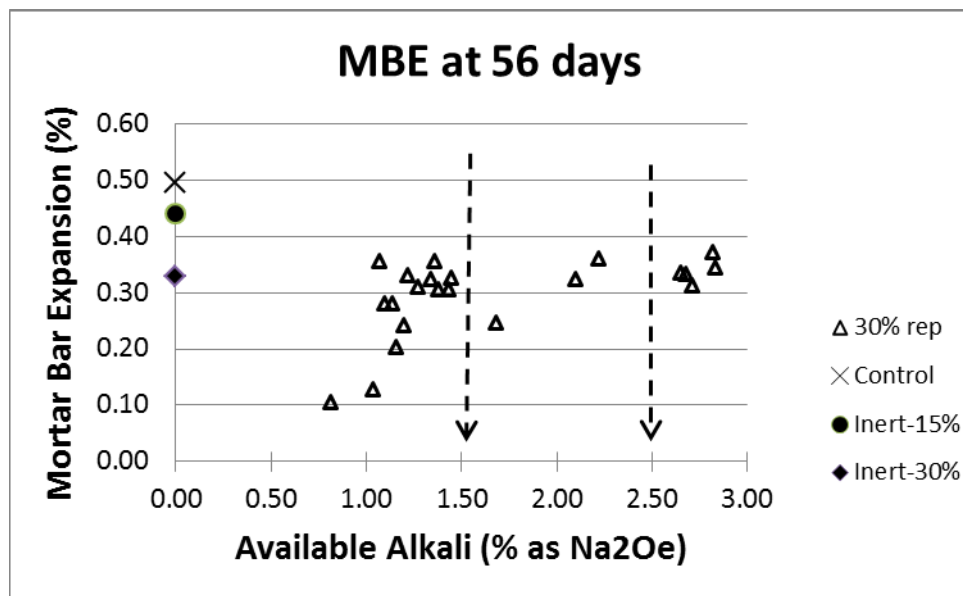


Figure 25. Mortar-bar expansion (56-d) versus available alkali content (30% ash)

to be strongly related to available alkali content. Instead, the siliceous nature of the fly ash appeared to play a greater role. Fly ash replacement level seems to have a much larger impact on performance than available alkali content. Hence, only the test results shown in Figure 24 support the current available alkali testing strategy used by the Iowa DOT. Even in that figure, it is hard to say that the fly ashes over 2.5% available alkali perform significantly better than those that test between 1.5 and 2.5% (assuming the test statistic of $d_2s=0.03\%$ is relevant and ignoring OTTCOMP12-98 which tests below many of the ashes with available alkali contents under 1.5%). Perhaps looking at performance limits will help clarify the situation.

Comparison to performance limits

As was mentioned earlier in this report, there is no single criterion for performance when testing fly ash for mitigating ASR. It would be ideal if a single criterion could be devised. The purpose of this section is to point out the strengths and weaknesses of the various limits. The performance limits of interest include the current Iowa DOT limits (see Figure 26), the limits currently given in AASHTO M 295 (14-day limit), and finally, the old ASTM performance limits (see Figure 28, this 14-day limit was suggested but never implemented).

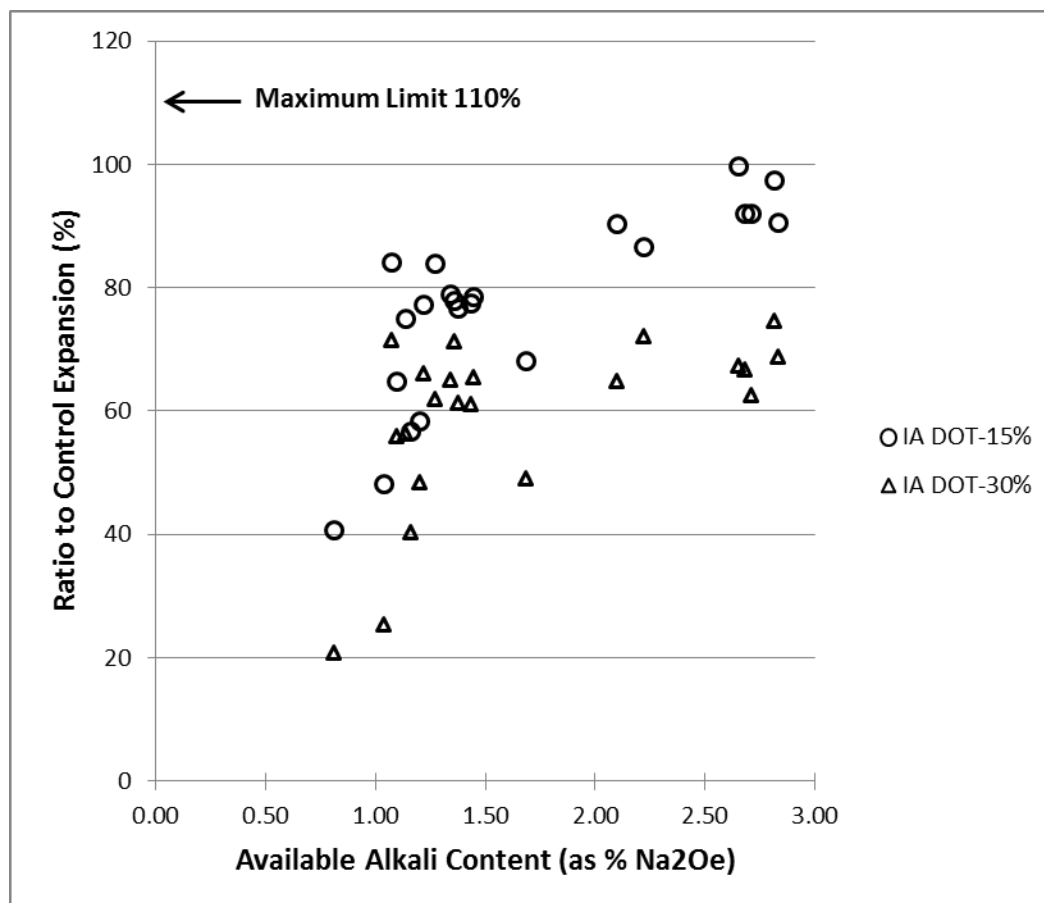


Figure 26. Comparison to Iowa DOT performance limits (110% max. @ 56-d)

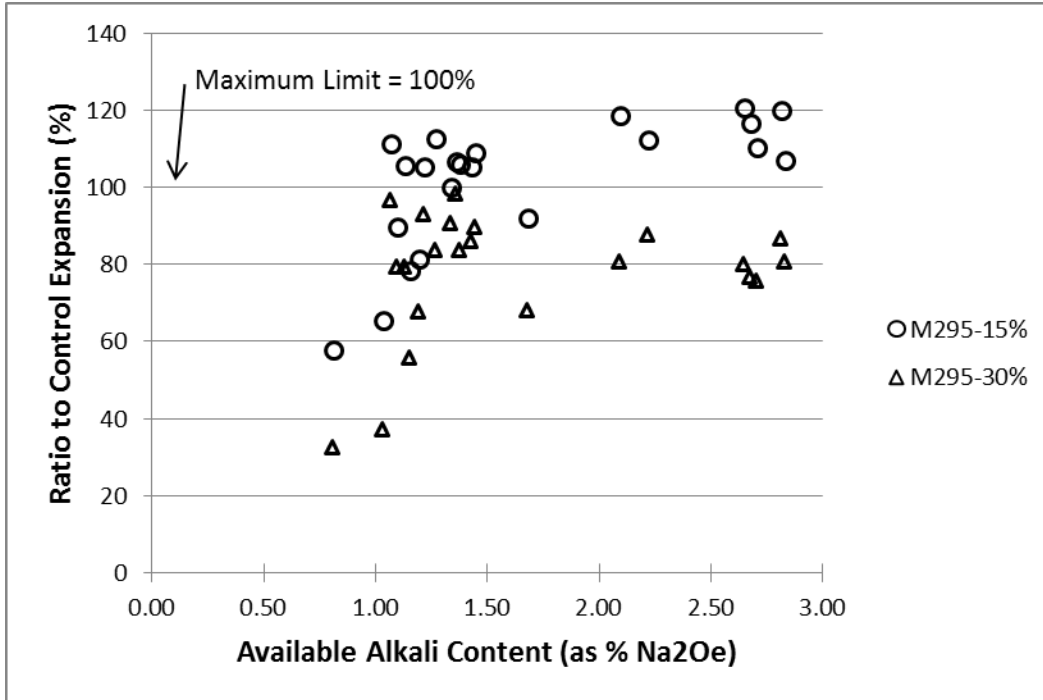


Figure 27. Comparison to AASHTO M295 performance limit (14-day limit)

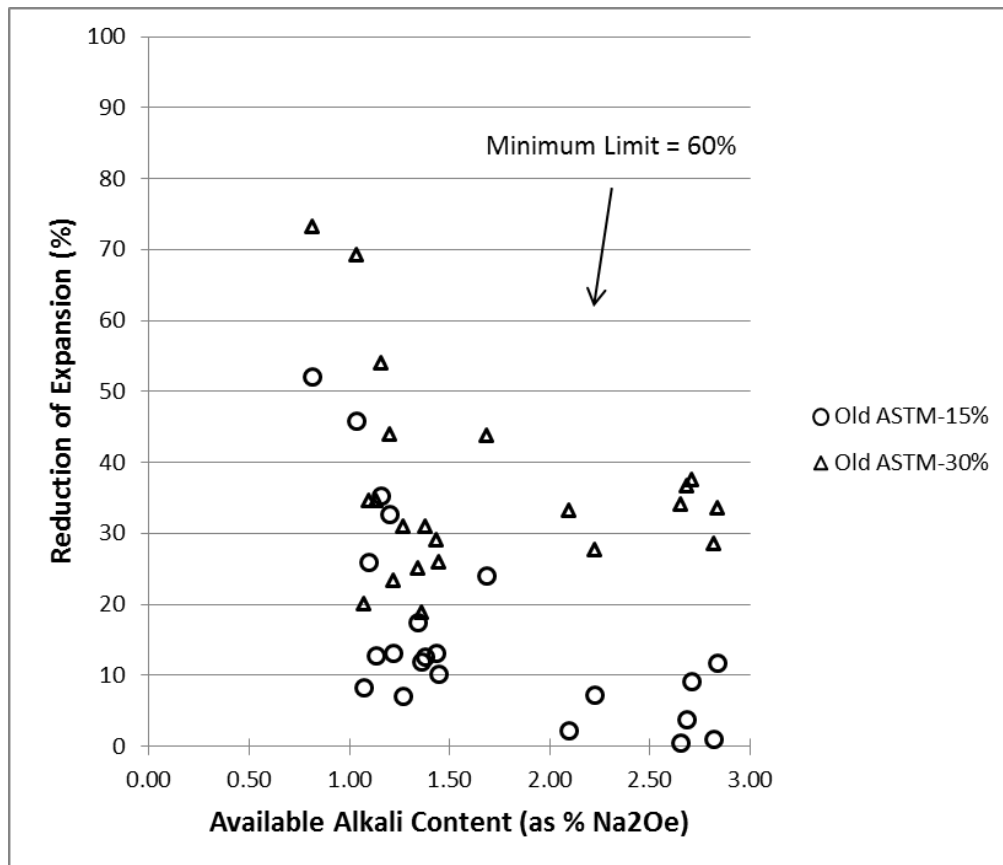


Figure 28. Comparison to old (proposed) ASTM performance limit (14-day limit)

The general trend in each figure is roughly the same (although one has to think in reverse for Figure 28 because it is expressed in percent reduction). Fly ash class has a major impact on the observed performance. In addition, fly ash dosage also plays a major role (with 30% replacement consistently producing better performance). However, the failure rate is very different using the different failure criteria. It is important to point out the performance limit shown in Figure 28 was never implemented; rather, an absolute expansion of 0.02% at 14-days was finally approved (none of the fly ashes in this study could meet that limit).

The final failure tally from using the different criteria is given in Table 21. Keep in mind that the chemical testing indicated that eight of the samples would have been flagged as being above the 1.5% available alkali limit; and four of those eight samples exceeded the 2.5% limit. The Iowa DOT method does not fail a single fly ash based on performance of mortar-bar specimens. The current AASHTO method fails 68% of the mortar-bar specimens containing 15% fly ash and none of the test specimens containing 30% fly ash. And finally, the Old ASTM method fails all of the specimens containing 15% fly ash and 86% of the specimens containing 30% fly ash (only the two Class F fly ashes passed). It is very evident that the old specification limits were much harder to meet, especially for Class C fly ash.

The poor correlation between performance and available alkali content was also evident in Figures 26 through 28. This was anticipated because it was observed in earlier figures; however, it was hoped that one of the numerical transforms would have helped things. Instead, we can note that the link with available alkali content at the 15% replacement level, basically falls apart when the fly ash replacement is increased to 30%. It could also indicate that another variable plays a stronger role in the performance test than the available alkali content.

Referring back to the correlation matrix (Table 17), it is observed that several variables exhibit strong correlations to the expansion values. The strongest correlation was noted between sum-of-the-oxides and expansion (at 15% replacement). This trend is illustrated in Figure 29, where it is evident that the experimental program lacked sufficient Class F fly ash samples to produce a balanced dataset. Strong correlations were also noted between expansion and CaO, MgO, SiO₂ and SO₃. However, as was noted earlier in this report, many of the composition variables were strongly correlated with each other, and this made it difficult to select additional variables for construction of a robust multi-variable model for mortar-bar expansion. Other researchers have noted similar problems building ASR expansion models, and they also reached the conclusion that sum-of-the-oxides is an important compositional variable (Malvern 2009).

Table 21. Failure tally for the ASR mortar-bar tests using different failure criteria

	DOT@15%	DOT@30%	AASHTO @15%	AASHTO @30%	Old ASTM @15%	Old ASTM @30%	Available Alkali
Number failed	0	0	15	0	22	19	8
% failed	0	0	68	0	100	86	36

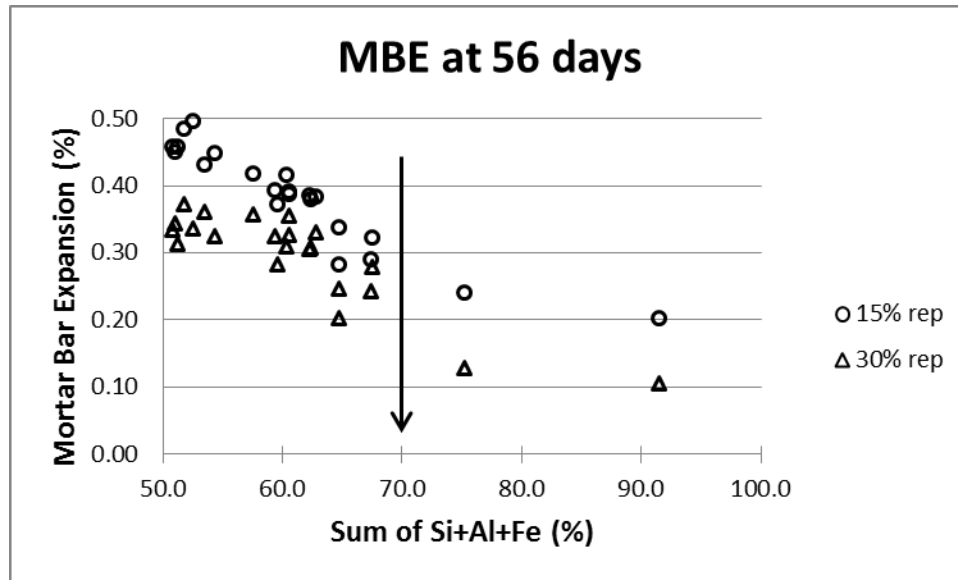


Figure 29. Expansion versus the sum of the oxides for the various fly ashes in this study

This section has indicated that the current Iowa DOT failure limit listed in IM 491.17 needs to be adjusted to come into better agreement with other metrics. All of the other metrics indicated that some of the Class C fly ashes simply do not perform well in mitigating ASR, especially at the 15% replacement level. This has also been observed in past studies using fly ash from these same power plants (Schlorholtz and Bergeson 1993). It is also in general agreement with other literature (Thomas 2013), which clearly indicates that fly ash replacement levels for ASR mitigation are normally above 30% (even for many Class F fly ashes). Many of the earlier figures have suggested that fly ash alkali content is only part of the explanation. We also need to consider the amount of siliceous material (as per Figure 29) added to the mixture if we want to set an appropriate limit. In addition, since the Iowa DOT routinely uses blended cements in many situations, it seems wise to evaluate how those materials perform before changing the performance limit for the IM.

ASTM C 441 Test Results

Mortar bar expansion tests containing pyrex glass were also conducted using blended cements. These mixtures were composed of lab-mixed slag cement (containing 20% slag) and Type IP cement. Eight fly ash samples were included in the study. Fly ashes were selected based on the level of expansion that was observed using the normal mixtures (see the preceding section), and it was assumed that four OGS samples would be included so that the whole range of alkali content could be studied. Fly ash replacement was fixed at a single level (20%). The results for the slag cement are given in Table 22 and the results for the Type IP cement are given in Table 23, respectively. Note that Table 22 also summarizes results for a few mixtures containing lab cement (LAFDAV12), 20% slag and 20% fly ash. This was done to check how cement alkali content impacted the expansion of mortars containing the high-alkali OGS fly ash.

Table 22. Results of mortar-bar expansion tests, slag cement (20% slag)

Sample	Cement Used	20% Replacement		
		14-days, %	28-days, %	56-days, %
Control	ASR	0.199	0.237	0.260
PN4051412	ASR	0.128	0.146	0.161
COA051512	ASR	0.071	0.078	0.085
IAT080812	ASR	0.119	0.137	0.146
PLP072312	ASR	0.082	0.095	0.096
OTTCOMP12-98	ASR	0.099	0.120	0.133
OGS073012	ASR	0.190	0.222	0.248
OGS081312	ASR	0.179	0.223	0.256
OGS083012L-1	ASR	0.185	0.217	0.247
Control	LAFDAV12	0.131	0.149	0.166
PLP072312	LAFDAV12	0.041	0.048	0.052
OTTCOMP12-98	LAFDAV12	0.050	0.061	0.071
OGS073012	LAFDAV12	0.161	0.184	0.209
OGS081312	LAFDAV12	0.175	0.204	0.231
OGS083012L-1	LAFDAV12	0.178	0.205	0.230

Table 23. Results of mortar-bar expansion tests, Type IP cement

Sample	Cement Used	20% Replacement		
		14-days, %	28-days, %	56-days, %
Control	ASG-IP	0.022	0.023	0.026
PN4051412-1	ASG-IP	0.064	0.075	0.080
COA051512-1	ASG-IP	0.008	0.009	0.012
IAT080812	ASG-IP	0.046	0.056	0.063
PLP072312	ASG-IP	0.029	0.036	0.040
OTTCOMP12-98	ASG-IP	0.037	0.048	0.054
OGS073012	ASG-IP	0.105	0.114	0.120
OGS081312	ASG-IP	0.095	0.105	0.116
OGS083012L-1	ASG-IP	0.099	0.109	0.116
Sil-Co-Sil (inert)	ASG-IP	0.014	0.014	0.015
Lafarge Slag	ASG-IP	0.025	0.032	0.036

The test results are plotted versus fly ash available alkali content in Figure 30. This compares the performance of the blended cements versus the portland cement (ASR cement only) mixtures containing the various fly ashes. Again, the mortar-bar expansion at 56 days is given on the vertical axis. Note that the fly ash replacement level was 15 or 30% for the ASR

cement but was held constant at 20% replacement in the blended cement mixtures. Hence, the test results are being compared across a variety of different mixtures. To some this may seem to be an “apples-to-oranges” comparison and that should be kept in-mind during the evaluation. However, all of the mixtures (with the exception of the 30% fly ash replacement) represent materials combinations that a contractor could choose to combine for a given paving project.

Again, the observed expansion is relatively poorly correlated to the available alkali content of the fly ash. However, it is very easy to see that at high available alkali contents, the blended cements exhibited less expansion than the mixtures that only contained fly ash. This trend disappeared when the available alkali content was less than 1.5%. The control mixtures had 56-day expansion values of 0.03% (Type IP), 0.26% (ASR+slag) and 0.50% (ASR cement). So again it was noted that most of the high-alkali OGS samples did little to reduce the expansion of the test specimens significantly below that of the control specimens.

Figure 31 illustrates the mortar-bar expansion values plotted against the sum-of-the-oxides. This actually produces relatively well-behaved trends over the range of samples included in the study. The trends fail (become flat) when the sum-of-the-oxides values approach the 50% lower limit for Class C fly ash. Lowest expansions were observed with the Type IP cement, followed by the slag cement, followed by 30% fly ash replacement and then 15% fly ash replacement. This is basically the same performance rating that was evident in the high-alkali portion of Figure 30; however, the relationship is better behaved because the trends remain relatively well-spaced over the majority of the graph.

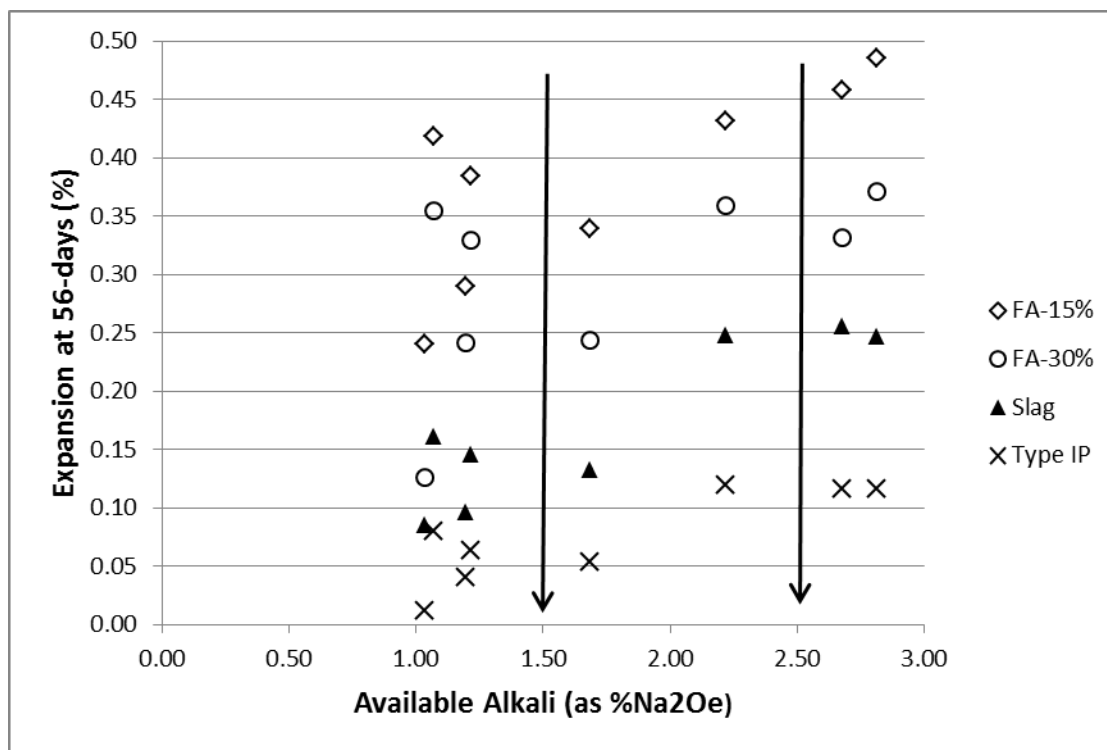


Figure 30. Mortar-bar expansion versus available alkali content

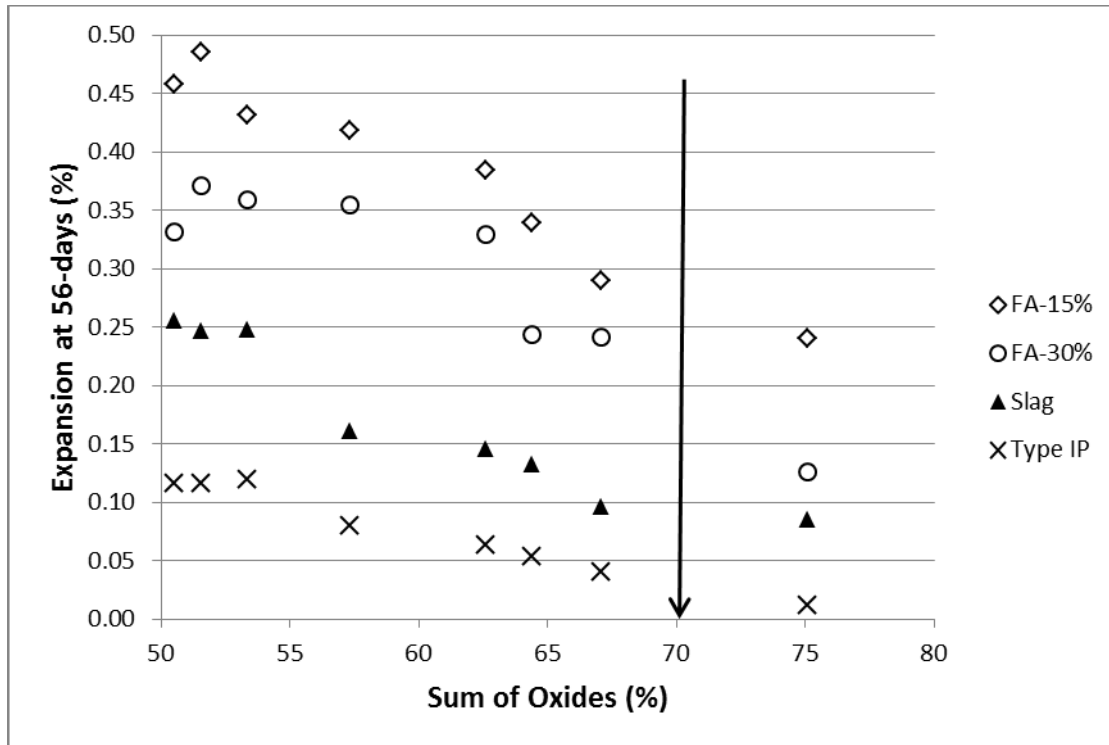


Figure 31. Mortar-bar expansion versus SUM

Expansion tests containing Wayland sand

This section summarizes the mortar-bar expansion tests that were conducted using Wayland sand aggregate. These tests consisted of ASTM C1567 tests that utilize high temperature and strongly alkaline solutions to accelerate ASR expansion (see Table 24). All of these mixtures were made using the lab cement because the test method is supposed to be insensitive to cement alkali content. In addition, traditional ASTM C227 tests were conducted using three different cements (alkali contents ranged from about 0.5 to over 1.0% as Na_2O_e) and a couple OGS fly ash samples (see Table 25, these values are reported to an extra decimal place for comparison purposes). The OGS fly ashes represented a “best case” and “worst case” of OGS samples that were obtained for this project (available alkali contents of 1.7% and 2.8% as Na_2O_e , respectively).

The “suggested” specification limit for C 1567 is 0.10% (though some still say a 0.08% limit is better). After running this test many times and noting the problems (normally between batches), the author does not think that those two limits are significantly different on a within-lab precision basis (1s was determined to be 0.01% for these experiments). The two limits are certainly not significant when between-labs precision is considered because the 1s% value given in C1567 is 15%, that corresponds to a value of $d2s=0.04\%$ at the 0.10% specification limit. This is another example of a test method that has poor between-lab precision (much like the problem that was noted for the available alkali test).

Table 24. Results of ASTM C 1567 (rapid mortar-bar) tests with Wayland sand

Fly Ash	Cement	Slag	14-day Expansion, %	Status
None (control)	LAFDAV12	None	0.23	Failed
PLP072312	LAFDAV12	None	0.14	Failed
OTTCOMP12-98	LAFDAV12	None	0.13	Failed
OGS073012	LAFDAV12	None	0.21	Failed
OGS081312	LAFDAV12	None	0.22	Failed
OGS083012L-1	LAFDAV12	None	0.23	Failed
None (control)	LAFDAV12	20% LAF120	0.18	Failed
PLP072312	LAFDAV12	20% LAF120	0.03	passed
OTTCOMP12-98	LAFDAV12	20% LAF120	0.04	passed
OGS073012	LAFDAV12	20% LAF120	0.09	passed
OGS081312	LAFDAV12	20% LAF120	0.08	passed
OGS083012L-1	LAFDAV12	20% LAF120	0.12	Failed
None (control)	AG-IP	None	0.02	passed
OGS083012L-1	AG-IP	None	0.02	passed

Table 25. Results of ASTM C 227 tests (Wayland sand aggregate)

Fly Ash	Cement	Slag	% Expansion at time in days				
			14	56	90	183	259
None (control)	SM	None	0.013	0.024	0.026	0.030	0.033
None - repeat	SM	None	0.012	0.022	0.027	0.030	not meas'd
OTTCOMP12-98	SM	None	0.013	0.023	0.028	0.032	not meas'd
OGS083012L-1	SM	None	0.016	0.027	0.036	0.044	not meas'd
None (control)	ASR	None	0.006	0.013	0.013	0.016	0.019
None (control)	LAFDAV12	None	0.005	0.013	0.014	0.017	0.019
None (control)	LAFDAV12	20%	0.008	0.014	0.016	0.019	not meas'd
OTTCOMP12-98	LAFDAV12	20%	0.011	0.016	0.016	0.019	not meas'd
OGS083012L-1	LAFDAV12	20%	0.015	0.023	0.025	0.033	not meas'd

The last column of Table 24 tabulates the status of the tests. This is also illustrated in Figure 32. All of the mixtures containing cement or cement plus 20% fly ash failed the test (i.e., exhibited expansion values exceeding 0.10%). This suggests that the Wayland sand is a reactive aggregate; however, the specification cautions that additional testing (concrete prism testing) is required to make this designation. This additional testing was not conducted because field service information was obtained via coring and subsequent petrographic examination. The observed expansion values are in reasonable agreement with those reported

by Dockter (2009) for similar levels of fly ash replacement. That study indicated that replacements levels of about 30% to 40% are often required to meet the 0.10% criterion.

The slag mixtures (20% slag and 20% fly ash) did considerably better in the C1567 tests, with only the control and the highest alkali OGS sample failing (note that the highest alkali OGS sample was within experimental error of the 0.10% limit). The use of slag alone did not mitigate the ASR reactivity of the Wayland sand.

The Type IP samples did not exhibit much expansion in this test (maximum of 0.02% for both the control and the high-alkali OGS sample). In fact, when the experimental program was started, it was unfortunate that the testing began with the Type IP cement. Many of the mixtures produced almost zero expansion (plus some negative values of course) which did not make much sense at the time. The method was practiced, an oven was changed to explicitly follow the procedure, but still there were problems with repeatability. In the end it was evident that this was just variation in the test method. This method normally produces large expansions and that is required due to the rapid length change of the test specimens during the measurement process (measuring something at 80°C is difficult and it is rapidly cooling). It seems very reasonable that this contributes to the poor between-lab precision exhibited by this test method.

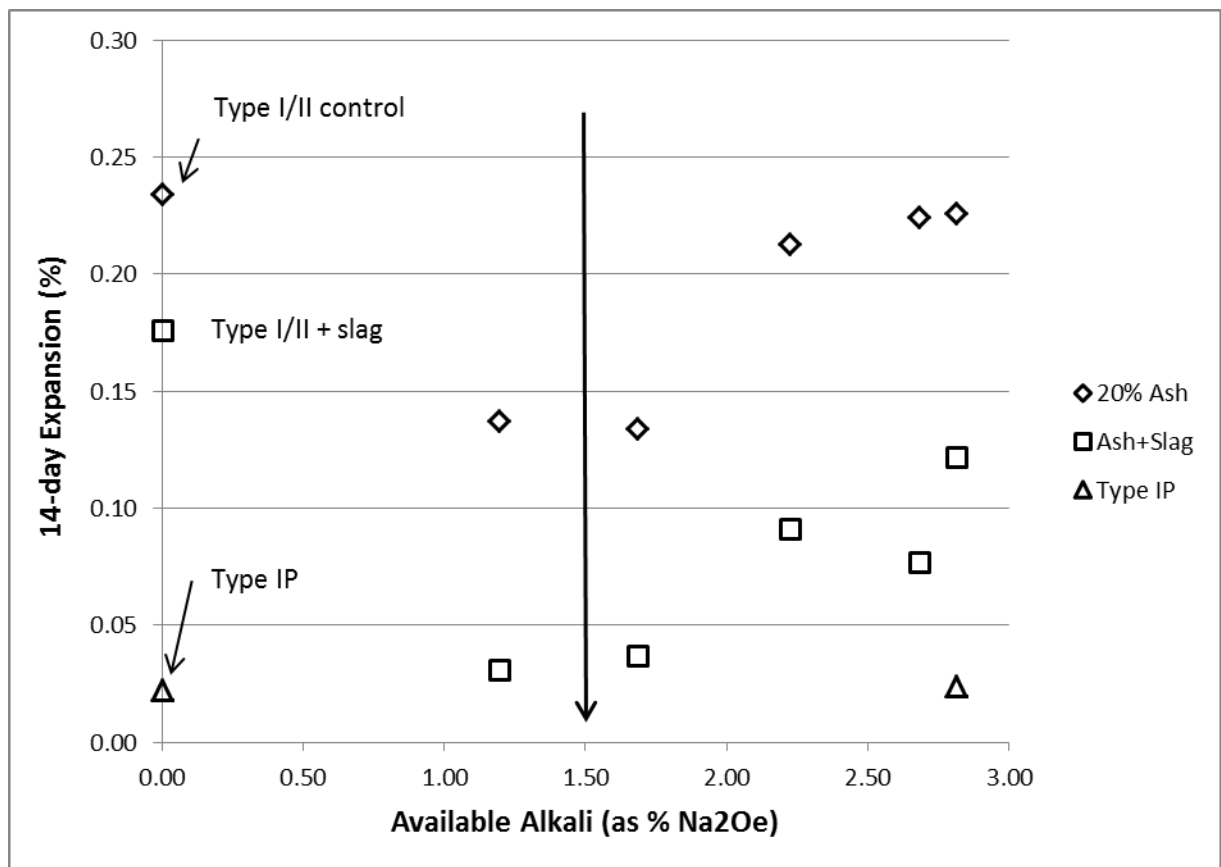


Figure 32. Results of ASTM C1567 tests with Wayland sand

The C227 test results (refer back to Table 25) did not agree with the C1567 test results. All of the different cements that were used to check the Wayland sand did not exceed the maximum expansion limits of 0.05% at three months, or 0.10% at six months (as per ASTM C 33). Some of the specimens were measured to nearly nine months, but the expansion values were only increasing very slowly so the tests were terminated. Hence, these measurements indicated that the Wayland sand is not a reactive aggregate. In addition, 20% of the high-alkali OGS samples had only a small impact on the measured expansions (expansion was still less than 0.05% at six months).

Summary of the Mortar-bar Expansion Tests

The mortar bar expansion tests produced some interesting results. The performance of all fly ash mixtures appeared to be better related to the sum-of-the-oxides than it was to the available alkali content. This applied to the standard Iowa DOT tests and the mixtures containing blended cement. This does not mean that fly ash alkali content is not important – it just means that the relationship is more complex than can be attributed to a single variable. The fixation on available alkali content was initially placed on fly ash by people who were more familiar with portland cement materials and they misunderstood the complexity of fly ash chemistry and how it related to alkali solubility. Fly ash replacement level had a strong impact on the observed expansion; however, other constraints tend to limit the replacement level to about 20% for Iowa DOT concrete mixtures.

The ASTM C 1567 tests indicated that the Wayland sand could be considered as moderately ASR-sensitive. The use of fly ash (20% replacement) did little to help reduce the expansion of the test specimens. Both blended cements (20% slag and Type IP) generally reduced the observed expansion below 0.10%. Only a single OGS fly ash sample failed to meet the 0.10% criterion when used with 20% slag. That same fly ash sample easily met the requirement when it was used with the Type IP cement. Hence, blended cements seem to have a higher tolerance to high-alkali fly ash.

The ASTM C 227 tests indicated that the Wayland sand was not sensitive to ASR. The use of fly ash (20% replacement) did not increase the observed expansion (i.e., exacerbate the ASR problem).

The information provided by the different standard tests has again provided facts that do not appear to be consistent. However, before final judgment is proclaimed, it is a good idea to consider the field observations because they pertain to real pavements.

Summary of Field Surveys and Petrographic Studies

This section describes the field portion of the research project. It contains a description of each field site, along with the general appearance and condition of the core specimens that were provided for petrographic examination. A summary of the field surveys and core logs is provided in Table 26. The detailed core logs are given in Appendix C. A summary of the hardened air content, spacing factor and estimated w/c ratio is given in Table 27. Finally, a summary of the petrographic examinations of the core specimens is provided in Table 28.

Table 26. Summary of field inspections and core logs

Site	Road	Description of Pavement	Description of Distress in Cores
1	I-80	No distress noted other than shale pop outs on many slabs	No distress noted except for some shale pop outs (mostly noted in one core)
2	Hwy 163	Slight pattern cracking at joints, shale pop outs	Shale pop outs noted
3	Hwy 92	Pattern cracking at joints, shale pop outs	Shale pop outs; one core turned off at about 6.5"
4	Hwy 61, SB	Distress over about 15 panels	Consolidation problem evident in cores
5	Hwy 61, NB	No distress noted	No distress noted; core from midpanel not well compacted
6	Hwy 61, NB	Surface not visible, asphalt overlay	White residue near fine aggregate particles and in voids
7	Hwy 61, NB	Surface not visible, asphalt overlay	White residue near fine aggregate particles and in voids
8	Hwy 61, SB	Slight spall near joint	White residue near fine aggregate particles; also many iron-bearing fine agg particles
9	Hwy 61, NB	No distress noted	Some cracked fine aggregate particles noted

Site 1: I-80 weigh station

The first site was a weigh station on westbound I-80 in Jasper County. The site was chosen as a control section (no fly ash) to mimic the cement and aggregates that were present in Site 2. The shoulder survey indicated the pavement was generally in good shape. Few spalls were noted and no pattern cracking was observed near the pavement joints. Shale pop outs were common on many slabs, and there were many instances of joint-sealant failures. The pavement had been in service for 22 years at the time it was cored.

Petrographic examination of the cores from the site showed little evidence of distress. The site had a low hardened-air content (2.9%,) but an acceptable spacing factor (0.005 inches). No macrocracking was noted in the cores. The only microcracking that was observed appeared to be related to shrinkage or was near the very bottom of the core (bottom 0.25"). Cracked shale particles were noted in several cores; however, the cracking did not extend into the paste so ASR-related distress due to these reactive particles appeared to be minimal.

ASR gel was noted in the voids adjacent to the reactive shale particles. Ettringite was also found in some air voids – sometimes it tended to fill the very smallest voids. No fly ash particles were noted in the core.

Site 2: Highway 163 – East Bound Driving Lane

The second site was a four-lane highway passing through Polk County. The site was selected because of joint distress that was noted during the shoulder survey. The distress manifested itself as corner-breaks, most commonly observed near the pavement centerline. Some of the distressed areas had already been patched. Shale pop outs were also noted on many slabs. The joint sealant normally appeared to be in good condition. The materials used at this site consisted of OGS fly ash, Holnam cement, Sully coarse aggregate and Van Dussel fine aggregate. The pavement had been in service for 22 years at the time it was cored.

Petrographic examination of the cores from the site showed little distress. The site had a low hardened-air content (3.6% total air but only 2.3% entrained) and a poor spacing factor (0.012 inches); and hence, by modern standards one would expect to find freeze-thaw distress at this site. No macrocracking was noted in the cores and the only microcracking that was observed appeared to be related to shrinkage at the top, or moisture near the very bottom of the core (bottom 0.25”). Cracked shale particles were noted in several cores; however, the cracking did not extend into the paste so ASR-related distress due to these reactive particles appeared to be minimal. ASR gel was noted in the voids adjacent to the reactive shale particles. Ettringite was also found in some of the air voids – sometimes it tended to completely fill the smallest voids. Fly ash particles were noted in the cores.

Table 27. Summary of air-void parameters and estimated water/cement ratio

Site	Mix Design	Total Air (%)	Entrained Air (%)	Specific Surface (1/inch)	Spacing Factor (inches)	w/c ratio
1	C-3WR	2.9	2.7	1130	0.005	0.40 to 0.45
2	C-4WR-C	3.6	2.3	460	<u>0.012</u>	0.40 to 0.45
3	C-6WR-C	5.9	4.0	550	0.007	0.40 to 0.45
4	C-3WR-C20	7.6	4.8	570	0.005	0.40 to 0.45
5	C-3WR-C20	4.7	3.8	1030	0.004	0.40 to 0.45
6	C-3WR	7.8	6.2	710	0.003	0.40 to 0.45
7	C-3WR-C20	7.1	5.2	470	0.006	0.40 to 0.45
8	C-4WR-C15	4.8	3.9	640	0.007	0.40 to 0.45
9	C-4WR-C15	4.9	3.2	490	<u>0.009</u>	0.40 to 0.45

Site 3: Highway 92 – East Bound

This site was a two-lane highway in Warren County, just east of Indianola. The site was selected because of joint distress that was noted during the shoulder survey. The distress was not widespread, but there were some regions that had been patched. The joint sealant

normally looked poor; however, the joints did not appear to be closing. The materials used at this site consisted of OGS fly ash, Lafarge cement, Ames Mine coarse aggregate and Hallett (Des Moines) fine aggregate. This pavement used a mix design with a high fraction of fine aggregate (C-6 mix). The pavement had been in service for 21 years at the time it was cored.

Petrographic examination of the cores indicated significant distress. One of the cores broke off during extraction (at about 6.5"). The site had a moderate hardened-air content (5.9% total air but only 4.0% entrained) and a marginal spacing factor (0.007 inches). Macrocracking was noted in the core containing the dowel bar (joint region). The epoxy-coated steel was not corroded. The macrocracks were at depths of about three to five inches below the surface of the core. The macrocracks were subparallel to the top of the core and propagated across the diameter of the core. Some of the macrocracks were lined with ASR gel. A few fine subvertical microcracks were noted in the top half-inch of the core (probably shrinkage). Sub-horizontal microcracks were noted in the core at depths of about 1.5 to 5 inches. The microcracking passed through cement paste, some coarse aggregate particles (an anomalous chert particle and several dark brown particles) and several fine aggregate particles (shale and hydrous chert). The microcracks traversed across much of the diameter of the cores extracted from the joints. Sometimes the cracks were lined with ASR gel (near the shale or hydrous chert particles). Extensive microcracking was noted in the brown coarse aggregate particles that tended to extend into the paste – no ASR gel was noted in the cracks. However, little distress was noted in the brown coarse aggregate particles observed in the core from the mid-panel of the pavement. Ettringite was also found in some the air voids near the very bottom of the core. Fly ash particles were noted in the cores.

Sites four through nine all were selected along sections of Iowa Highway 61. Highway 61 is a four-lane highway that runs north-south through Lee County. South of Keokuk, the highway passes over a bridge (Des Moines River) into Missouri. All five of the sites contain Wayland sand as fine aggregate. This particular sand contains a wide variety of particles that could be problematic (shale, chert, hydrous chert, greywacke, meta-granite, silicified limestone and iron stone or iron oxide). The Iowa DOT had previously cored sites six and seven, and the purpose of including the sites in this study was to evaluate how far the deterioration had progressed since the prior study. All of the cores were extracted from the driving lane of the pavement.

Site 4: Highway 61 – South Bound-MP 15

This site was selected because of joint distress that was noted during the shoulder survey. The distressed section of pavement was noted from about milepost 15.45 to 14.95. The distress was not evident south of milepost 14.95. Walking from the north to south, eight spalls were noted along with many additional patches. The joint sealant normally looked intact. The fly ash used at this site was not documented; however, OGS or Louisa would be the closest sources. The pavement had been in service for 13 years at the time it was cored.

Petrographic examination of the cores did not indicate significant distress. The site had a good hardened-air content (7.6% total air but only 4.8% entrained) and a good spacing factor

(0.005 inches). There was a considerable amount of entrapped air voids noted and this suggests a plastic-concrete issue or a consolidation problem (or both because they are often observed in unison). A few fine sub-vertical microcracks were noted in the top half-inch of the core (probably shrinkage). Microcracking, was noted sporadically in several fine aggregate particles (chert and shale). The microcracks proceeded into the paste (active ASR sites). Some ASR gel sites were noted that could not be attributed to a specific aggregate particle. Ettringite was found in air voids below the top two-inches of the core (small voids were nearly filled). Fly ash particles were noted in the cores.

Site 5: Highway 61 – North Bound-MP 17

This site was selected because it was paved using OGS fly ash and Wayland sand. The pavement was evaluated from about milepost 16.8 to 17.3. No distress was noted at this site. The pavement had been in service for 15 years at the time it was cored.

Petrographic examination of the cores did not indicate significant distress. The site had a low hardened-air content (4.7% total air but only 3.8% entrained) and a good spacing factor (0.004 inches). A few fine sub-vertical microcracks were noted in the top inch of the core (probably shrinkage). Microcracking related to reactive particles in the fine aggregate (shale and chert) was not noted at this site. Ettringite was found in air voids, especially near the bottom of the core (small voids were nearly filled). Fly ash particles were noted in the cores.

Site 6: Highway 61 – North Bound-MP 5

This site was selected because the section was paved using Wayland sand and no fly ash. The pavement was difficult to evaluate because it had been overlaid with asphalt in 2006. The pavement had been in service for 22 years at the time it was cored.

Petrographic examination of the cores found significant distress. The site had a good hardened-air content (7.8% total air and 6.2% entrained) and a good spacing factor (0.003 inches). The limestone coarse aggregate contained a few anomalous hydrous chert particles (actually these were probably oversize particles from the sand). The fine aggregate contained some ASR sensitive particles (shale, hydrous chert, hard chert, siltstone and some silicified limestone). No subvertical microcracks (shrinkage) were noted at this site. Nearly continuous sub-horizontal microcracks proceeded across the core at depths from 1” to 5” below the top of the core. The microcracks passed through the paste, coarse aggregate particles and fine aggregate particles. ASR-active cracks were also noted in several reactive fine aggregate particles. The microcracks were sometimes lined with ASR gel. Voids near the reactive particles were often filled with ASR gel. Ettringite was found lining air voids (small voids were nearly filled). No fly ash particles were noted in the cores.

Site 7: Highway 61 – North Bound-MP 7 Driving Lane

This site was selected because the section was paved using Wayland sand and Louisa fly ash. The pavement was difficult to evaluate because it had been overlaid with asphalt in 2006. The pavement had been in service for 22 years at the time it was cored.

Petrographic examination of the cores found significant distress. The site had a good hardened-air content (7.1% total air and 5.2% entrained) and a good spacing factor (0.006 inches). The limestone coarse aggregate was sound. The fine aggregate contained some ASR-sensitive particles (shale and hydrous chert). Sub-vertical microcracks (shrinkage) were noted at this site (to a depth of about 0.75"). Microcracks passed through several ASR reactive fine aggregate particles. The microcracks usually propagated into the paste (active ASR sites). Voids near the reactive particles were often lined or filled with ASR gel, this was commonly observed throughout much of the depth of the core. Ettringite was found lining air voids (small voids were nearly filled near the bottom of the core). Fly ash particles were noted in the cores.

Site 8: Highway 61 – South Bound Station 538

This site was selected because it was paved using Louisa fly ash and Wayland sand. The pavement had minor spalls but generally looked to be in good shape. The joint sealant was in good shape. The pavement had been in service for 20 years at the time it was cored.

Petrographic examination of the cores did not indicate significant distress. The site had a low hardened-air content (4.8% total air but only 3.9% entrained) and a marginal spacing factor (0.007 inches). A few fine subvertical microcracks were noted in the top half-inch of the core (probably shrinkage). Sub-horizontal microcracks proceeded across the core at depths from 0.25" to 1.25" below the surface of the core. These microcracks proceeded through paste, coarse aggregate particles and fine aggregate particles. Microcracks were also noted in the ASR-sensitive fine aggregate particles (shale, chert and hydrous chert). Some ASR gel sites were noted that could not be attributed to a specific aggregate particle. The gel was noted in voids throughout much of the depth of the core. Ettringite was found in air voids below the surface of the core (small voids were nearly filled at the bottom of the core). Fly ash particles were noted in the cores.

Site 9: Highway 61 – North Bound Station 536

This site was selected because it was paved using OGS fly ash and Wayland sand. The pavement exhibited no visible distress and the joint sealant was in good shape. The pavement had been in service for 21 years at the time it was cored.

Petrographic examination of the cores did not indicate significant distress. The site had a low hardened-air content (4.9% total air but only 3.9% entrained) and a marginal-to-poor spacing factor (0.009 inches). The limestone coarse aggregate was sound. The fine aggregate

Table 28. Summary of petrographic examinations

Site	Road	Aggregates	Voids	Cracking	Comments
1	I-80	Limestone CA* sound; Sand sound except for shale particles	Ettringite lines voids; ASR gel lines voids near shale particles	No macrocracks Microcracks 0.1 to 0.2" deep (shrinkage)	No fly ash observed Carbonated depth about 0 to 0.1"
2	Hwy 163	Limestone CA sound; Sand sound except for shale particles	Ettringite lines voids; ASR gel lines voids near shale particles	No macrocracks Microcracks 0.1 to 0.5" (shrinkage) also near bottom of core;	Fly ash observed Carbonated depth about 0 to 0.15"
3	Hwy 92	Limestone CA contains brown particles and anomalous hydrous chert; Sand contains shale and hydrous chert particles	Ettringite lines voids; ASR gel lines voids near shale and hydrous chert particles	Macrocracks near steel; Microcracks due to shrinkage, ASR (shale) and freeze- thaw	Fly ash observed Carbonated depth about 0 to 0.2"
4	Hwy 61 SB MP 15	Limestone CA sound; Sand contains reactive shale and chert particles	Poor consolidation; Ettringite lines voids; ASR gel lines or fills voids scattered throughout core	No macrocracks; Microcracks due to shrinkage and some ASR (chert)	Fly ash observed Carbonated depth about 0 to 0.25"
5	Hwy 61 NB MP 17	Limestone CA sound; Sand contains shale and chert particles	Ettringite lines voids; ASR gel lines some voids in top 3.5" of core	No macrocracks Microcracks 0 to 0.7" deep (shrinkage)	Fly ash observed Carbonated depth about 0 to 0.4"
6	Hwy 61 NB MP 5	Limestone CA sound except an anomalous hydrous chert particle; Sand contains shale, hydrous chert, siltstone, partially silicified limestone and chert particles	Ettringite lines voids; ASR gel fills or partially fills voids near reactive particles	No macrocracks; Microcracks due to ASR (many cracks filled with gel) from 1 to 5" below top of core	No fly ash observed Carbonated depth about 0 to 0.3"
7	Hwy 61 NB MP 7	Limestone CA sound; Sand contains shale and hydrous chert particles	Ettringite lines voids; ASR gel fills or partially fills voids from 1" to 9" deep in core	No macrocracks; Microcracks due to ASR (hydrous chert) scattered throughout core	Fly ash observed Carbonated depth about 0 to 0.5"
8	Hwy 61 SB	Limestone CA sound; Sand contains shale, hydrous chert and chert particles	Ettringite lines voids; ASR gel fills or partially fills some voids from 0 to 1" deep in core	No macrocracks; Microcracks due to ASR (hydrous chert, chert and shale) scattered throughout core; Microcracks in the top inch of core could be freeze-thaw	Fly ash observed Carbonated depth about 0 to 0.25"
9	Hwy 61 NB	Limestone CA sound; Sand contains shale, hydrous chert, chert plus some other reactive particles	Ettringite lines voids; ASR gel fills or partially fills some voids from 0 to 1" deep in core	No macrocracks; Microcracks in the top inch of core appear to be freeze-thaw but are filling with ASR gel	Fly ash observed Carbonated depth about 0 to 0.25"

CA* = coarse aggregate

contained particles that were potentially ASR-sensitive (shale, chert, hydrous chert, greywacke and meta-granite). A few fine sub-vertical microcracks were noted in roughly the top inch of the core (probably shrinkage). Sub-horizontal microcracks were noted in the top 0.5" to 1.25" of the core. The microcracks proceeded across the diameter of the core and passed through paste, coarse aggregate particles and fine aggregate particles. ASR gel lined or filled the cracks near the top of the core. The ASR gel was primarily observed in the top 1.25" of the core. Ettringite was found lining air voids in the core, nearly filling the small voids. Fly ash particles were noted in the cores.

ADDITIONAL DISCUSSION

The alkali content of OGS fly ash and how it impacted ASR was a main topic of this research program. The laboratory study indicated that the available alkali content of the OGS samples varied by about a factor of 2. This makes it sound like the test results vary greatly but this is an oversimplification. Figure 33 illustrates the alkali solubility curves for the eight OGS fly ash samples that were used in this study. The test results were obtained by conducting available alkali tests but extracting the samples after different curing times. Only the results of the sodium determinations are shown in the figure. The plots of all eight OGS show similar trends of increasing sodium content until about 50 to 75 days, after that time they tend to flatten out. If the water-soluble alkali content of each fly ash is subtracted from the available alkali content, then the trends shown in Figure 34 are obtained. Now the range of sodium concentration between the various OGS samples has been reduced significantly. Hence, the water-soluble alkalis can be a large fraction of the measured available alkali content. It is important to realize that the available alkali test result is actually comprised of two components. One component is obtained immediately (water-soluble) and the second component requires time for the calcium hydroxide to react with the fly ash particles and liberate the alkali. The water-soluble alkalis can impact the efficiency of some air-entraining agents and can cause efflorescence problems.

Earlier in this report it was noted that there was an interesting relationship between the total alkali and the water-soluble alkali (refer back to Figure 16). However, the OGS samples were spaced too closely in time to provide a good depiction of the trend. Data from a continuous suite of OGS samples from 1998 is shown in Figure 35. This provides a better illustration of the general trend (gradual versus abrupt) that occurs during long-term trona addition at OGS.

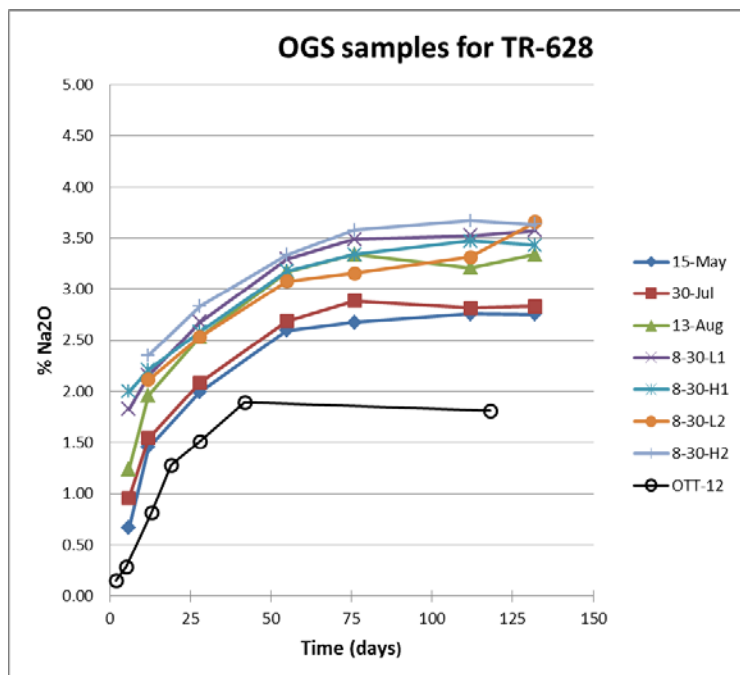


Figure 33. Alkali solution rate curves for OGS fly ash samples in this study

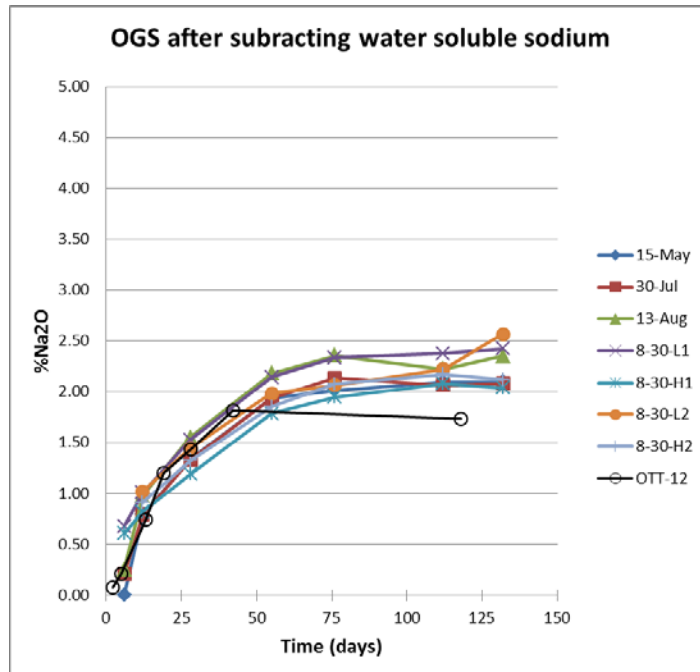


Figure 34. OGS solution rate curves after subtracting the water-soluble fraction

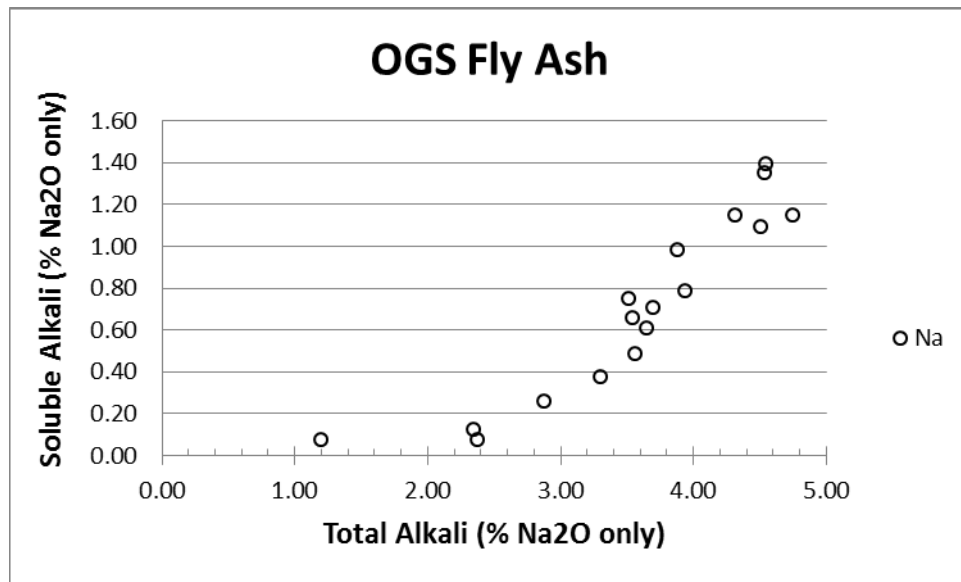


Figure 35. Water-soluble alkali in OGS fly ash samples from 1998

Implementation of recent environmental rules may mandate changes to operating procedures at existing coal power plants. If specific plants need to resort to dry-sorbent injection (DSI) of alkaline materials, then the water-soluble alkali test is a quick test that can be used to monitor the fly ash for changes in alkali content. Luckily, most plants burning Powder River Basin coal do not have problems that require DSI for mitigation.

In an earlier section (refer to Table 15), the within-lab precision of the available alkali test was estimated at about 0.11 to 0.13% Na_2O_e (d2s statistic). These estimates are valid statistics calculated from repeated experiments; however, they fail to contain all the variation that can exist in the available alkali test method. To look at all of the variability one really needs to consider how the calcium hydroxide impacts the long-term test results.

An example of the long-term trends observed in the available alkali determinations are illustrated in Figures 36 and 37. The figures show how available sodium and potassium vary as a function of time. The two samples that were studied consisted of Class C fly ashes and the study spanned several years. These graphs are basically control charts that were used to check how the test was performing. Each graph consists of over 100 independent observations that were collected at a rate of about 30 to 40 tests per year (about 4 tests per month). The graphs generally show flat trends interrupted by significant deviations. The magnitude of the deviations was most pronounced in the sodium determinations; however, close inspection shows that the potassium measurements were also impacted. Most of the deviations were traced back to the calcium hydroxide that was used in the experiments (the calcium hydroxide had carbonated). Special precautions were taken over the duration of the experiment to avoid carbonation of the calcium hydroxide – yet it was a tough problem to avoid. The special precautions consisted of verification tests conducted on each new bottle of calcium hydroxide that was purchased. The verification tests consisted of measuring the level of carbonation and level of sodium and potassium contamination in the bulk sample. A new bottle of calcium hydroxide is normally purchased every year. After the verification tests, the calcium hydroxide was subdivided into smaller bottles for daily use.

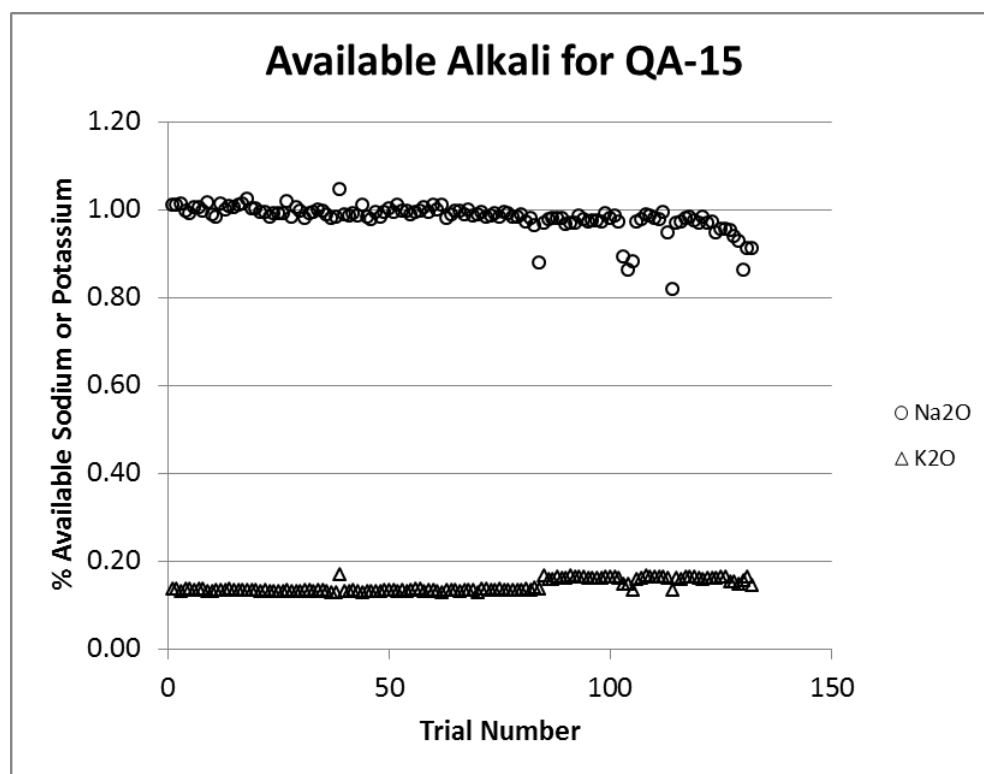


Figure 36. Time series of available sodium and potassium determinations for QA-15

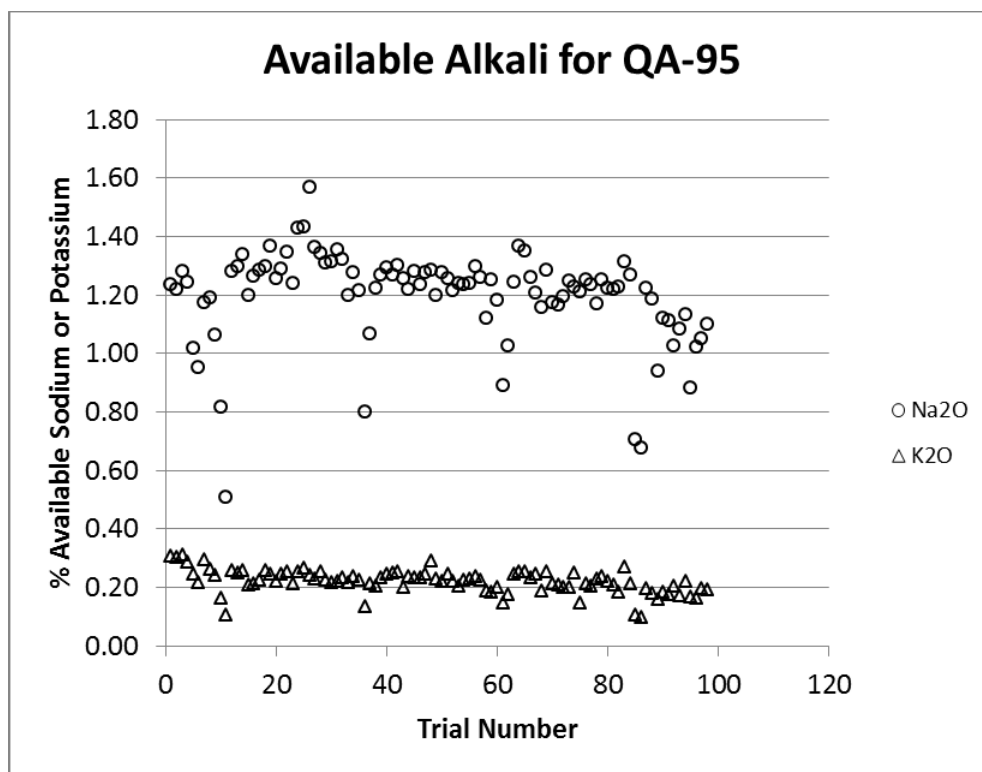


Figure 37. Time series of available sodium and potassium determinations for QA-95

The smaller bottles allow the calcium hydroxide to be used over a longer period of time by restricting the number of times that any single bottle is opened (and hence, the potential for excessive carbonation). Experience has shown that each smaller bottle can be opened three times before the material carbonates to a level of about 6%. Each smaller bottle is verified before being put into service, and generally a single batch of calcium hydroxide can be used for a year without significant carbonation. However, as is indicated in two time series, sometimes things still do not go as desired. Carbonation of the calcium hydroxide is probably a major reason why the between-lab precision of the available alkali test method is so poor.

If one selects an arbitrary construction year of data from the test data, then additional estimates of precision can be calculated (see Table 29). The calculations give a d_{2s} value of about 0.16 to 0.21, depending on the fly ash used. These estimates are considerably larger than those given in Table 15). If all test values are used, then the calculations give a d_{2s} value of about 0.1 to 0.5, depending on the fly ash used. These statistics suggest that the two fly ashes produce different estimates for the precision of the method (even though they are only about 0.4% apart in test response). This behavior is currently unexplained.

To contrast the precision of total alkali determinations (via XRF) and available alkali determinations, a series of nine fused-disk specimens from a single sample were made over the course of a year. The test statistics calculated from the experiment are given in Table 30. The long-term stability of the XRF method is very good when compared to the available alkali method. The alkali determinations and calculation of total alkali equivalent vary only

in the second decimal place of the test results (d2s statistic). This is another strong argument for replacing the available alkali test with the total alkali test. However, one must not forget that the available alkali test provides something that the total alkali test cannot, and that is an estimate of the mobility of the fly ash alkalis. The total alkali test simply measures all of the sodium and potassium in the fly ash. The available alkali test does something more than that and for research purposes that is very important. This was mentioned earlier in this report (refer back to Figures 15 and 21), and these observations will now be developed in more detail.

Table 29. Precision of single samples made in 9 different months (AA).

Trial#	Sample QA-15			Sample QA-95		
	Na ₂ O, %	K ₂ O, %	Avail. Alkali (Na ₂ O _e , %)	Na ₂ O, %	K ₂ O, %	Avail. Alkali (Na ₂ O _e , %)
February	0.987	0.130	1.072	1.294	0.244	1.455
March	0.983	0.133	1.071	1.219	0.237	1.375
April	0.990	0.131	1.076	1.275	0.244	1.436
May	0.984	0.131	1.070	1.258	0.247	1.420
June	0.969	0.165	1.077	1.240	0.230	1.391
July	0.971	0.160	1.076	1.261	0.224	1.408
August	0.989	0.163	1.075	1.184	0.199	1.314
September	0.819	0.132	0.906	1.368	0.253	1.534
October	0.970	0.158	1.045	1.158	0.189	1.283
Average	0.96	0.14	1.05	1.25	0.23	1.40
1s	0.05	0.02	0.06	0.06	0.02	0.07
1s% (COV)	5.64	10.97	5.29	4.94	9.63	5.32
d2s	0.15	0.04	0.16	0.17	0.06	0.21

Table 30. Precision of a single sample (fused disk) made in 9 different months (XRF).

Disk Made	SiO ₂ , %	Al ₂ O ₃ , %	Fe ₂ O ₃ , %	CaO, %	Na ₂ O, %	K ₂ O, %	Total alkali (Na ₂ O _e , %)
February	36.86	19.09	7.38	23.43	2.49	0.56	2.85
March	36.93	19.04	7.41	23.44	2.49	0.56	2.85
April	36.99	19.07	7.40	23.47	2.49	0.56	2.86
May	36.92	19.09	7.41	23.48	2.49	0.56	2.86
June	36.74	19.04	7.37	23.37	2.48	0.55	2.84
July	36.87	19.06	7.38	23.36	2.49	0.55	2.86
August	36.96	19.05	7.41	23.52	2.50	0.56	2.86
September	37.09	19.03	7.43	23.62	2.48	0.55	2.85
October	36.81	18.91	7.38	23.41	2.50	0.56	2.86
Average	36.91	19.04	7.40	23.46	2.49	0.56	2.86
1s	0.10	0.05	0.02	0.08	0.006	0.003	0.007
d2s	0.29	0.15	0.06	0.23	0.02	0.01	0.02

Earlier in this report, it was shown that there is a good correlation between available alkali and total alkali in fly ash. That was the basis for the Iowa DOT model that is currently used to predict available alkali. In addition, the observed correlations appeared to depend on fly ash class (Class F versus Class C). This information is presented in Figure 38. Data from additional studies has also been placed on the graph, and the graph has been truncated at 6% total alkali to better illustrate the region of interest for current fly ash utilization. Plots like this have been reported by Smith (1988). In that paper, he indicated the available alkali test is not the best method to predict if fly ash will help control ASR. His reasoning was based on the observation that nearly all fly ashes can be used to reduce expansion in laboratory mixes if the correct mix proportions are selected (and this fact cannot be deduced from an available alkali test result). He also indicated that ASR performance was more closely related to the sum-of-the-oxides than the available alkali content; however, his study was limited to only 15 fly ashes. No explanation was given for the observed behavior. However, making some assumptions and then performing calculations using available alkali test results provides a tentative explanation for the behavior.

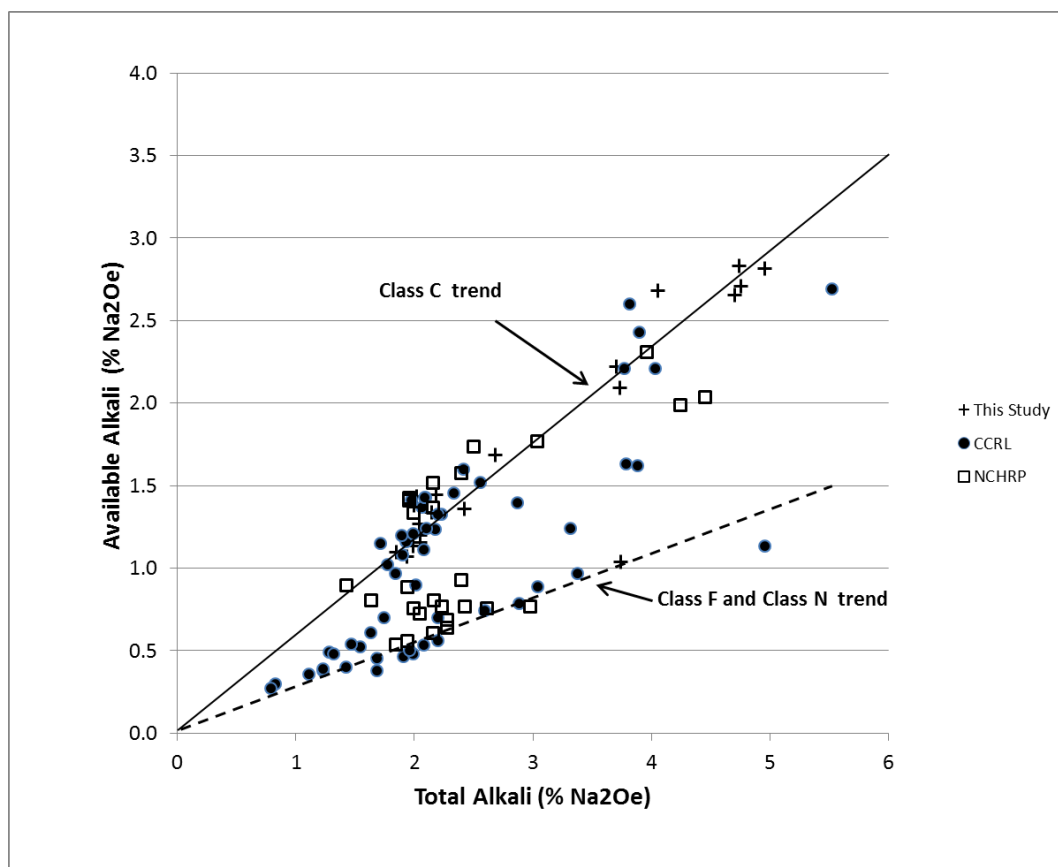


Figure 38. Available alkali versus total alkali for a variety of different studies

First, assume that all the bulk silica and calcium oxides in the fly ash are available for reaction with the calcium hydroxide used for the available alkali test. This will only be roughly true because of the different mineral phases and glass fractions present in fly ash. In addition, assume that the reaction has proceeded to an extent that it is reaching completion (and chemical products dominate the system). This assumption is also only roughly valid because the alkali solution curves (see Fig. 33) are not flat at 28-days. However, bearing these rather rough approximations in mind, one can calculate the mole ratio of $\text{CaO}:\text{SiO}_2$ and see how it impacts the relative alkali mobility (the ratio of available alkali to total alkali, AA/TA). This plot (see Figure 39) clearly shows two rather distinct regions with high point density. The upper right region contains all the Class C fly ashes while the bottom left contains all the Class F fly ashes. This type of behavior has been discussed by other researchers (Rayment 1982; Glasser et al. 1987) and it has been attributed to different levels of alkali binding by the solid phase (calcium-silicate-hydrate, CSH). Since the available alkali system is primarily composed of fly ash with some of calcium hydroxide (no portland cement is present) then one may also speculate that the concentration of CSH may also impact the test results. Or it could be due to a rather complex function of glass type (Mather 1984). These details could be probed by additional experimentation. However, the result that we desire from the graph is the average mobility for the two different regions so that we can calculate the expected available alkalis if we measure only the total alkalis.

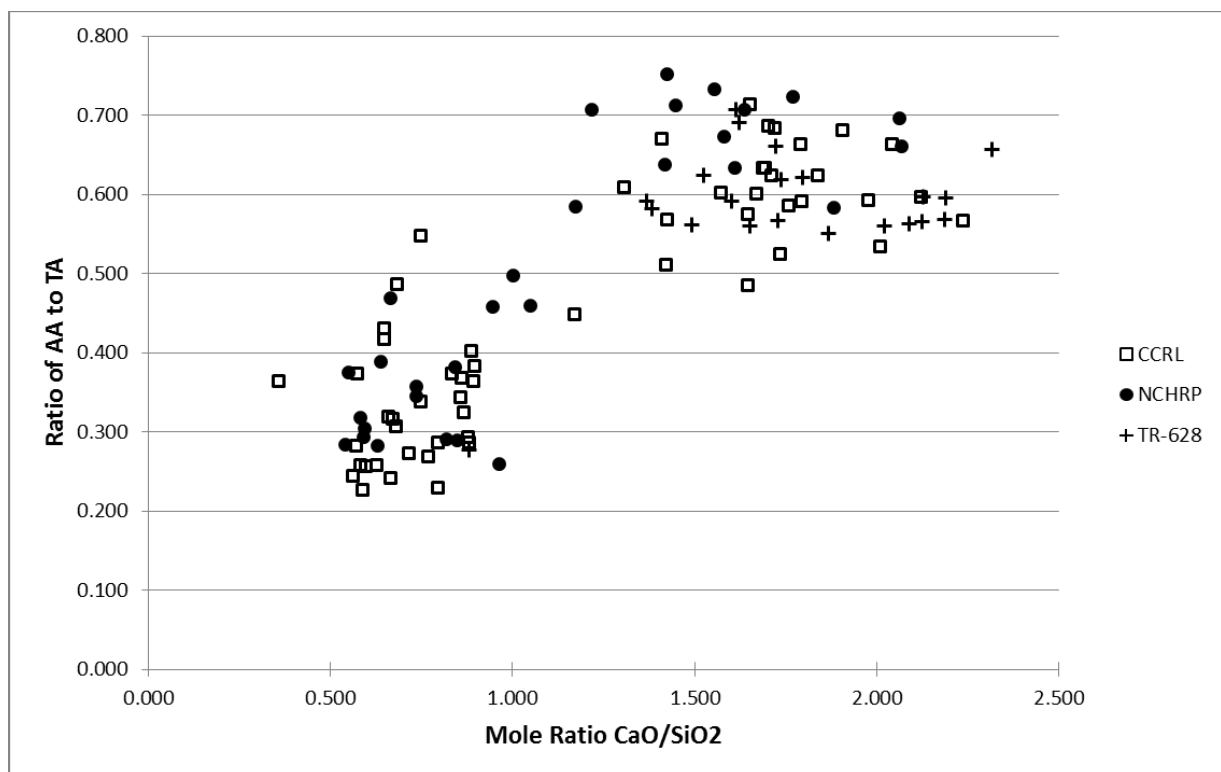


Figure 39. An illustration for the difference in alkali mobility for fly ash classes

Figure 39 shows that if the $\text{CaO}:\text{SiO}_2$ ratio is greater than about 1.1 then the AA/TA ratio is about 0.6 (average value, ranges from about 0.50 to 0.75). In contrast, if the $\text{CaO}:\text{SiO}_2$ ratio is less than about 1.1 then the AA/TA ratio is about 0.35 (average, ranges from about 0.25 to 0.55). There is some overlap (only about four or five points); however, with all the assumptions that were made only general agreement was expected. Especially since the results were from different studies conducted in many different years. Now the available alkali values can be calculated from the total alkali content across the full range of fly ash composition. The final results can be improved if one is willing put forth the effort of tweaking in the AA/TA ratio with a few experiments for each source of fly ash (actually mine source for the coal that the power plant is burning). However, when considering the poor agreement between labs that is typical of the available alkali test, the rough values given above are probably adequate for routine work. Do note, that if it is not desired to calculate the Ca:Si mole ratio as described above, the relationship shown in Figure 21, between AA/TA ratio and the sum-of-the-oxides, can simply be used. In this author's opinion, that relationship is not quite as concise as the molar ratio but it does provide reasonable values for estimating the available alkali content of fly ash. The fact to keep-in-mind is that the available alkali content of a fly ash is only a single point on an alkali-solubility curve (refer back to Figures 11 or 33). As was discussed earlier, this is not a special metric, rather it is both arbitrary and sensitive to many experimental variables. In most cases it appears to have little physical significance or any concise relationship to overall performance.

SUMMARY AND CONCLUSIONS

In summary, a detailed study has been conducted on how fly ash alkalis are measured and how the measured alkali content impacts the behavior of common ASR-performance tests. The goal of the project was to provide a quicker and more reliable way to test fly ash for use in portland cement concrete. The study contained a laboratory component and a field component.

The lab component consisted of examining test methods that are commonly used to measure the alkali content of fly ash (chemical tests). This included evaluating a model used by the Iowa DOT for predicting available alkali content. Special emphasis was placed on determining if the model could accurately predict the available alkali content of high-alkali fly ash. The lab study also evaluated performance tests that are often performed in unison with the chemical tests, to accept or reject fly ash. And finally, a quick literature review was conducted to locate the rationale behind the current testing procedures and specification limits used for fly ash. In addition, the literature was searched to assess the impact of the new environmental rules on the use of fly ash.

The field study was focused on the evaluation of nine pavement sites for the presence of ASR-related distress. The field sites were selected to study a variety of concrete material combinations. Prior Iowa DOT testing had indicated that some of the sites contained materials that could be considered ASR-sensitive.

Conclusions – Field Study

1. Pavement site 1 (I-80 weigh station, Jasper County) did not exhibit significant distress. The site had a low entrained-air content but an adequate spacing factor. Pop outs were noted on many slabs and this cracking was also noted in the petrographic examinations. However, the shale particles that caused the pop outs were not considered active ASR sites. This control section looked good after 22 years of service.
2. Pavement site 2 (Highway 163, Polk County) exhibited sporadic joint distress that required some patching. The site had a low entrained-air content and a poor spacing factor. Pop outs were noted on many slabs and this feature was also noted in the petrographic examinations. However, the shale particles that caused the pop outs were not considered active sites of ASR. Hence, the joint distress noted at this site was most probably caused by freeze-thaw attack.
3. Pavement site 3 (Highway 92, Warren County) exhibited frequent joint distress that required some patching. The site had a marginal entrained-air content and a marginal spacing factor. In addition, the coarse aggregate (Ames Mine limestone) contained some brown particles and some anomalous chert particles that exhibited cracking. The brown particles were enriched in dolomite, and the cracking appeared to be much more prevalent in the cores

extracted from the joints. The fine aggregate also contained some ASR sensitive particles (shale, hydrous chert) and ASR gel was noted lining some voids and cracks. However, there were few observed particles that exhibited active ASR (i.e., cracked particles with active cracks extending significantly into the paste). The distress noted at this site was most probably initiated by freeze-thaw attack and now the process has become more complex (with ASR contributing as long as moisture is present).

4. Pavement site 4 (Highway 61, Lee County, south-bound at MP 15) appeared to be a region of poor concrete that lasted about 1000 yards. The site had a good entrained air content and a good spacing factor. The cores exhibited signs of poor consolidation. Also, distress was noted in a few specific fine aggregate particles (shale and chert) with active cracks extending into the paste. Hence, this site is experiencing cracking caused by ASR; however, the deterioration seems too sporadic to be attributed only to this distress mechanism. The pavement had only been in service for 13 years so it appears that something else is also playing a role at this site.
5. Pavement site 5 (Highway 61, Lee County, north-bound at MP 17) was a section of pavement that contained OGS fly ash, continental cement and Wayland sand. The site had a marginal entrained-air content but a good spacing factor (due to a high value of specific surface, which indicates very small entrained-air voids). No significant distress was noted at this site; however, reactive aggregate particles were noted in the sand (shale and chert). Since the site had been in service for 15 years, distress from ASR was expected but not observed at this site.
6. Pavement site 6 (Highway 61, Lee County, north-bound at MP 5) was a section of pavement that had been overlaid with asphalt in 2006 because of distress. This site was considered a control site because it did not contain fly ash. The site had a good entrained-air content and a good spacing factor. The coarse aggregate contained an anomalous hydrous chert particle (may have been oversize from the sand), and the fine aggregate contained many reactive particles. The cracking patterns noted in the cores suggested a mixed mode of distress. The fine aggregate exhibited ASR distress (and was producing large amounts of gel); however, the distress also appeared to have a freeze-thaw component (sub-horizontal, simply extending cracks near the top of the core). This seems reasonable since the pavement has seen 22 years of service.
7. Pavement site 7 (Highway 61, Lee County, north-bound at MP 7) was a section of pavement that had been overlaid with asphalt in 2006 because of distress. This site contained Louisa fly ash in addition to the same materials used in site 6. The site had a good entrained-air content and a good spacing factor. The coarse aggregate was sound, but the fine aggregate contained many reactive particles. The cracking patterns noted in the cores were different from those noted in site 6. The fine aggregate exhibited ASR distress

(and was producing large amounts of gel near the reactive particles nearly throughout the depth of the sample); however, the sub-horizontal cracking was not noted at this site. Hence, the most probable explanation for distress at this site is ASR-induced cracking.

8. Pavement site 8 (Highway 61, Lee County, south-bound at station 538) was a section of pavement that was selected because of distress (spall near joint). The site had a marginal entrained-air content and a marginal spacing factor. The coarse aggregate was sound but the fine aggregate contained many reactive particles. The cracking pattern noted in the cores was primarily sub-horizontal to the top of the core and very near the top surface. This cracking pattern is common to freeze-thaw distress. However, the fine aggregate exhibited ASR distress with some active cracking sites (and was also producing gel near the reactive particles nearly throughout the depth of the sample). This site has distress related to freeze-thaw and ASR.
9. Pavement site 9 (Highway 61, Lee County, north-bound at station 536) was a section of pavement that was selected because it contained OGS fly ash. No surface distress was noted at this site. The site had a low entrained-air content and a marginal spacing factor. The coarse aggregate was sound but the fine aggregate contained many reactive particles. The cracking pattern noted in the cores was primarily sub-horizontal to the top of the core and very near the top surface. This cracking pattern is common to freeze-thaw distress. The fine aggregate exhibited ASR distress and was also producing gel near the top of the sample. Hence, the distress was primarily related to freeze-thaw attack.
10. Service record indicates that Wayland sand is an ASR-sensitive aggregate. Four of the six sites that used this sand exhibited microcracking that could be attributed to specific fine aggregate particles. In the remaining two sites that used Wayland sand, minimal cracking was noted but ASR gel was noted in both cores. The most common reactive components consisted of shale, chert and hydrous chert. Occasionally greywacke and meta-granite were also found near gel deposits.
11. Fly ash from Ottumwa Generating Station was not conclusively linked to ASR problems at any field sites. It was used in pavements that had both good and poor performance. Sites 2, 5 and 9 all contained OGS fly ash but did not exhibit significant ASR-related distress in the petrographic exams. Site 3 contained OGS fly ash and also exhibited ASR-related distress (fine aggregate); however, the interpretation of cause of the cracking at this site was complicated by the presence of the brown carbonate coarse aggregate particles. These coarse aggregate particles exhibited extensive cracking that appeared to be related to freeze-thaw damage.
12. The air content and spacing factor of the concrete from sites 6 and 7 were in good agreement with prior work. The previous Iowa DOT work on Highway

61, near sites 6 and 7 of this study, had indicated that the air content and spacing factor were adequate for freeze-thaw resistance. Iowa DOT air content values ranged from 4.8% to 6.9% and the spacing factor ranged from 0.005 inches to 0.007 inches. This study indicated that the entrained-air content ranged from 5.2% to 6.2% and the spacing factor ranged from 0.003 inches to 0.006 inches.

Conclusions – Lab Study

1. The available alkalis test is prone to several experimental errors that lead to poor agreement between testing labs. The time required for obtaining test results also seriously restricts the applicability of the test when trying to control the quality of materials going to jobsites. The exact rationale for the 1.5% specification limit was not found but the literature study indicated that it pre-dated the approval of the fly ash specification (mid-1950's).
2. The Iowa DOT model for predicting the available alkali content of Class C fly ashes is adequate for general use. The model was validated by comparing the predicted values from 32 Class C fly ash samples to test results from available alkali determinations. The model predicted values were $\pm 0.2\%$ of the measured available alkali contents for samples that ranged from 1 to 2.5% (as Na_2O_e). The model was within $\pm 0.4\%$ for values that ranged from 2.6 to 5% (as Na_2O_e); however, data was very limited in this range.
3. Mortar-bar expansion tests indicated that performance can be improved (lower measured expansions) by using more fly ash (higher replacement levels), a fly ash containing a higher value of the sum-of-the-oxides (more siliceous), and blended cements. In this study, the Type IP cement performed the best of the three cements that were studied. The available alkali content of the fly ash generally did not produce the best correlations to measured expansions; this was especially true if one was allowed to change fly ash replacement level.
4. Mortar-bar expansion tests conducted in accordance with Iowa DOT IM 491.17 did not fail any fly ash samples due to excessive expansion. However, four of these fly ash samples had available alkali contents exceeding 2.5%. Comparison of the Iowa DOT failure criterion with other specifications indicated that it needs to be re-evaluated.
5. When expansion tests were performed with inert material in place of fly ash, it was observed that the measured expansions were similar to those obtained with many Class C fly ashes (at similar levels of replacement). This was in contrast with more siliceous fly ashes that exhibited significant reduction in expansion.
6. Mortar-bar expansion testing using ASTM C1567 indicated that Wayland sand was a moderately-reactive fine aggregate. Similar tests conducted using

ASTM C227 indicated that Wayland sand was not reactive. This apparent contradiction would normally require additional testing (concrete prism tests); however, field service record indicated that the sand was moderately reactive.

7. Mortar-bar expansion testing using ASTM C1567, with Wayland sand and various fly ashes at a 20% replacement level, indicated that it was difficult to meet the 0.10% performance criterion without the use of blended cement. Blended cements appeared to be much more tolerant to the high-alkali content of OGS fly ash. Only the highest alkali content OGS sample failed to meet the 0.10% criterion when used with slag cement (0.12% measured expansion, just exceeding the test limit when considering experimental error in the measurements).
8. Recent changes (or proposed changes) in environmental regulations will impact all coal-burning power plants in the United States of America. The major impacts will be due to the implementation of the Mercury and Air Toxics Standards (MATS) and the Clean Power Plan (CPP). MATS retrofits are currently in progress and will be completed by about 2016. The future of the CPP is currently uncertain, but the rule is expected to be finalized in June 2015. Due to the controversy of the rule, many legal challenges are expected and this could delay the effective date of the rule.

RECOMMENDATIONS

- The available alkali test needs to be replaced because it does not have the between-lab precision required to make concise rejection of materials that fail to conform to standard specifications.
- The total alkali content of fly ash should be used to estimate the available alkali content of Class C fly ash. Note that the main reason for calculating available alkali content is to compare with other testing labs that still perform the available alkali test. Eventually, the available alkali test will be removed from specifications and this step will no longer be required.
- A simpler calibration strategy could be used for the Iowa DOT alkali model. Currently the Iowa DOT uses linear regression to determine the slope and intercept used in the model for predicting available alkalis. It is recommended to set the slope of the calibration curve to 0.66 and the intercept to zero. Then CCRL samples 38 and 47 can be used to evaluate the performance of the model.
- Potential changes in power plant operating procedures could impact the soluble alkali content of fly ash. It is recommended that the Iowa DOT start measuring the water-soluble alkali content of fly ash. Additional research is needed to put meaningful specification limits on the water-soluble alkali test results.

- IM497.17 has served the Iowa DOT for over 20 years. Changes need to be made to that document to bring it up-to-date. However, it may also be wise to postpone any major changes until after the current (and proposed) EPA regulations are implemented because that would avoid making multiple changes that could confuse users. The following changes are recommended in the interim:
 - a) The specification limits in that document need to be changed to reflect the change to total alkali content. This means that the low limit should be changed to 2.3% and the high limit should be changed to 3.8%.
 - b) The Iowa DOT failure criterion for the mortar-bar expansion test needs to be re-evaluated or removed. The test is performed infrequently (less than one time per year), and has never failed any source of fly ash so it could be removed from the IM without much impact.
- Test results indicated that the two blended cements used in this study were able to control the mortar-bar expansion of very high-alkali fly ash. Unfortunately the maximum alkali content that was observed in the research project only reached 5% total alkali (roughly 3% available alkali); and hence, it is recommended that additional work be conducted for verification.
- Wayland sand is an ASR-sensitive aggregate. It is recommended that preventative measures be used when utilizing this aggregate in concrete mixtures.

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Appendix A – CCRL Available Alkali Summary

Table A1. Available alkali data for the CCRL Pozzolan Proficiency sample program

Sample	Class	Count (N)	Available Alkali, %	1s AA	Na ₂ O (%)	1s Na	K ₂ O (%)	1s K
CCRL 1	C	12	0.97	0.28	0.80	0.27	0.26	0.11
CCRL 2	C	12	1.52	0.60	1.35	0.53	0.26	0.13
CCRL 3	F	20	0.56	0.21	0.09	0.03	0.63	0.30
CCRL 4	F	20	0.74	0.35	0.18	0.07	0.75	0.39
CCRL 5	F	27	2.21	0.57	1.90	0.72	0.24	0.06
CCRL 6	F	27	2.69	0.50	2.38	0.75	0.20	0.04
CCRL 7	F	29	0.47	0.14	0.27	0.09	0.30	0.12
CCRL 8	F	29	0.40	0.12	0.22	0.08	0.28	0.09
CCRL 9	C	28	3.77	1.80	3.41	1.50	0.44	0.16
CCRL 10	C	28	6.18	2.70	5.83	2.40	0.45	0.15
CCRL 11	F	27	0.54	0.18	0.10	0.05	0.81	0.61
CCRL 12	F	27	0.48	0.19	0.08	0.04	0.78	0.65
CCRL 13	C	23	1.33	0.25	1.28	0.28	0.14	0.07
CCRL 14	C	23	1.40	0.59	1.29	0.51	0.26	0.14
CCRL 15	F	27	0.50	0.10	0.08	0.04	0.88	0.60
CCRL 16	F	27	0.79	0.19	0.19	0.09	1.34	0.89
CCRL 17	C	29	1.23	0.40	1.16	0.39	0.14	0.05
CCRL 18	C	29	1.11	0.38	1.02	0.37	0.15	0.05
CCRL 19	F	28	1.24	0.32	1.02	0.26	0.32	0.09
CCRL 20	F	28	1.14	0.44	1.08	0.40	0.11	0.05
CCRL 21	C	23	2.43	0.65	2.36	0.64	0.26	0.09
CCRL 22	C	23	2.21	0.81	2.14	0.80	0.28	0.13
CCRL 23	C	25	1.33	0.24	1.14	0.20	0.26	0.08
CCRL 24	C	25	1.24	0.25	1.09	0.22	0.23	0.07
CCRL 25	F	20	0.45	0.10	0.30	0.08	0.21	0.08
CCRL 26	F	20	0.38	0.10	0.15	0.04	0.32	0.12
CCRL 27	C	25	1.37	0.15	1.20	0.12	0.26	0.04
CCRL 28	C	25	1.46	0.15	1.30	0.15	0.23	0.04
CCRL 29	F	20	0.70	0.15	0.16	0.06	0.81	0.18
CCRL 30	F	20	0.52	0.13	0.18	0.07	0.50	0.13
CCRL 31	N	23	0.36	0.13	0.16	0.08	0.33	0.11
CCRL 32	N	23	0.30	0.13	0.19	0.10	0.19	0.09
CCRL 33	C	20	1.15	0.19	0.92	0.15	0.34	0.07
CCRL 34	C	20	1.43	0.22	1.24	0.19	0.29	0.05
CCRL 35	F	25	0.38	0.09	0.06	0.03	0.48	0.09
CCRL 36	F	25	0.27	0.05	0.12	0.02	0.23	0.04
CCRL 37	F	25	0.90	0.25	0.60	0.17	0.50	0.15
CCRL 38	C	25	2.60	0.58	2.49	0.58	0.26	0.05
CCRL 39	F	21	0.39	0.09	0.20	0.07	0.29	0.06
CCRL 40	F	21	0.61	0.14	0.30	0.07	0.49	0.11
CCRL 41	F	27	0.49	0.13	0.22	0.05	0.38	0.13
CCRL 42	F	27	1.63	0.30	1.21	0.25	0.58	0.18
CCRL 43	F	28	0.48	0.16	0.22	0.07	0.38	0.15
CCRL 44	F	28	1.62	0.47	1.24	0.35	0.54	0.21

CCRL 45	C	23	1.02	0.37	0.86	0.32	0.22	0.08
CCRL 46	C	23	1.43	0.30	1.23	0.35	0.30	0.09
CCRL 47	C	21	1.60	0.29	1.36	0.45	0.22	0.05
CCRL 48	C	21	1.41	0.29	1.13	0.38	0.35	0.10
CCRL 49	F	24	0.97	0.23	0.52	0.18	0.58	0.22
CCRL 50	F	24	0.54	0.11	0.29	0.12	0.37	0.16
CCRL 51	C	21	1.16	0.21	0.96	0.23	0.25	0.07
CCRL 52	C	21	1.21	0.22	0.91	0.26	0.39	0.12
CCRL 53	F	25	0.89	0.18	0.51	0.13	0.60	0.12
CCRL 54	F	25	0.70	0.21	0.42	0.14	0.43	0.15
CCRL 55	C	27	1.20	0.43	0.88	0.33	0.25	0.09
CCRL 56	C	27	1.08	0.47	0.73	0.32	0.24	0.10

Table A2. Total alkali data for the CCRL Pozzolan Proficiency sample program

Sample	Class	Count (N)	Na ₂ O (%)	1s Na	K ₂ O (%)	1s K
CCRL 29	F	27	0.45	0.09	2.65	0.07
CCRL 30	F	27	0.52	0.09	1.55	0.06
CCRL 31	N	33	0.24	0.11	1.32	0.11
CCRL 32	N	33	0.41	0.16	0.63	0.09
CCRL 33	C	41	1.30	0.28	0.63	0.04
CCRL 34	C	41	1.77	0.35	0.49	0.04
CCRL 35	F	44	0.20	0.08	2.25	0.13
CCRL 36	F	44	0.28	0.09	0.77	0.05
CCRL 37	F	40	1.18	0.14	1.26	0.09
CCRL 38	C	40	3.56	0.41	0.39	0.03
CCRL 39	F	49	0.48	0.11	1.14	0.07
CCRL 40	F	49	0.70	0.15	1.42	0.09
CCRL 41	F	50	0.51	0.13	1.17	0.05
CCRL 42	F	50	2.76	0.38	1.56	0.09
CCRL 43	F	51	0.54	0.07	1.18	0.06
CCRL 44	F	51	2.88	0.19	1.52	0.09
CCRL 45	C	53	1.49	0.20	0.43	0.02
CCRL 46	C	53	1.80	0.17	0.43	0.02
CCRL 47	C	55	2.19	0.18	0.34	0.02
CCRL 48	C	55	1.64	0.12	0.51	0.02
CCRL 49	F	51	1.78	0.14	2.43	0.09
CCRL 50	F	51	0.71	0.06	1.15	0.03
CCRL 51	C	50	1.62	0.12	0.47	0.02
CCRL 52	C	50	1.54	0.12	0.68	0.02
CCRL 53	F	53	1.61	0.10	2.17	0.08
CCRL 54	F	53	0.99	0.06	1.14	0.04
CCRL 55	C	54	1.59	0.10	0.46	0.02
CCRL 56	C	54	1.54	0.11	0.55	0.02

Appendix B – Iowa DOT correspondence for the available alkali limits

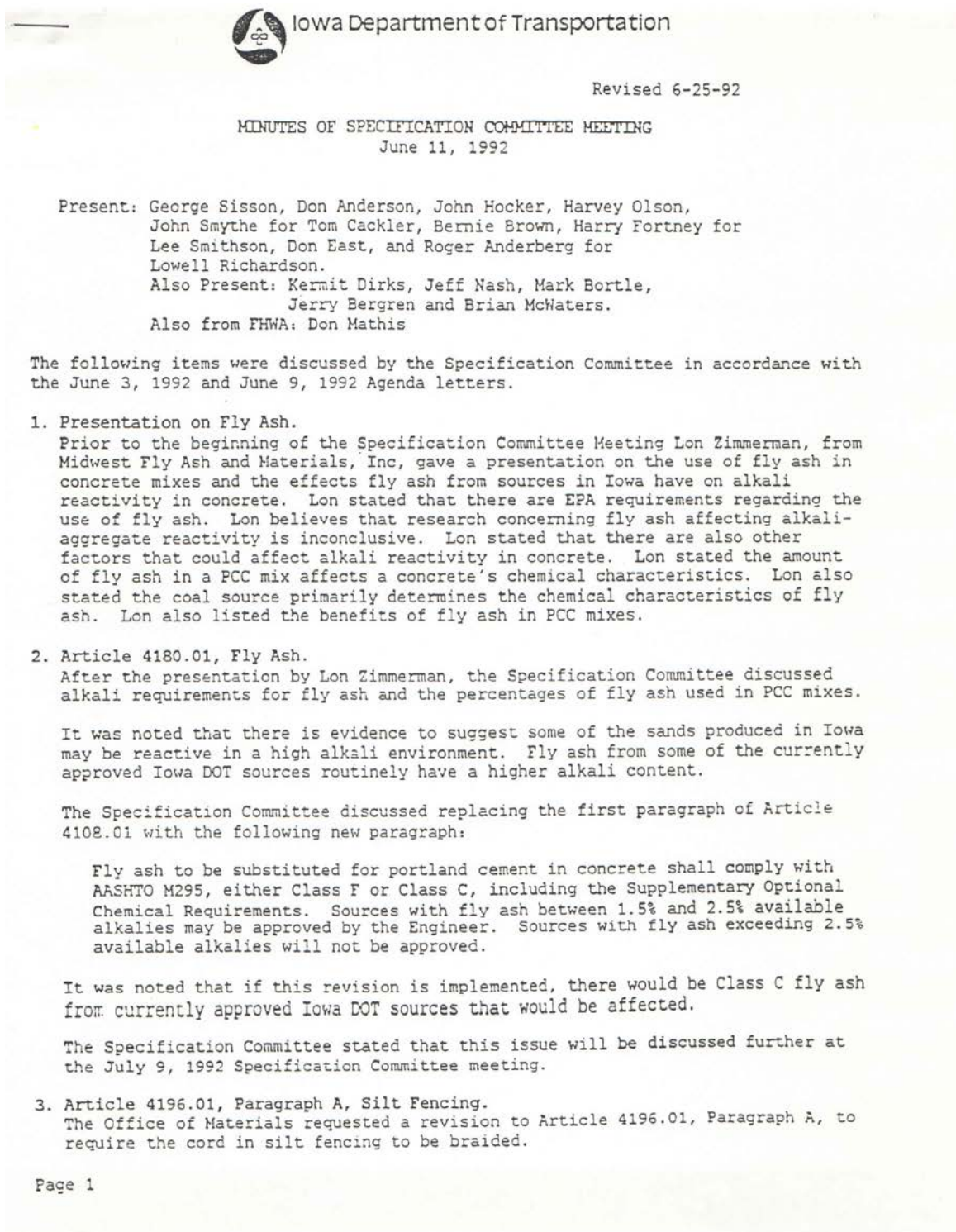


Figure B1. Iowa DOT minutes for the available alkali limits



Iowa Department of Transportation

800 Lincoln Way, Ames, Iowa 50010 515/239-1452

August 18, 1992

Ref. No.: 435.24051

Mr. Lon Zimmerman
President
Midwest Fly Ash and Materials Inc.
2220 Hawkeye Drive
Box 3557
Sioux City, IA 51102

Dear Lon:

Sorry about the delay in responding to your July 27, 1992 letter.

The "approved by the Engineer" caveat will be used to approve those sources that meet expansion criteria when exposed to a sulfate solution as well as when tested for alkali expansion. The specific acceptance criteria will be:

1. Mortar bars made per ASTM C-1012 with 15% and 30% fly ash and Type 1 cement achieve at least 90% of the time period for 0.10% expansion of a control mortar bar without fly ash.
2. Mortar bars made per ASTM C-311 with 15% and 30% fly ash, Type I cement (alkali 0.70 to 0.80% equivalent Na_2O) and pyrex aggregate exhibit not more than 10% expansion over non-fly ash mortar bars.

Mortar made with Council Bluffs fly ash does not meet the above criteria and consequently, this source will be dropped from the approved list.

The Specifications Committee approved the specification change for fly ash at the August 13, 1992 meeting. The change will go into effect with the November 10, 1992 letting and thereafter. Primary and interstate paving projects where Council Bluffs fly ash is being used will be modified by change order disallowing this source.

Figure B2. Iowa DOT letter to Midwest Fly Ash Company, page 1

Mr. Lon Zimmerman
Page 2
August 18, 1992

We will be continuing the same system we currently use regarding fly ash. Acceptance will not be on a project by project basis but rather will be on an approved source basis. It would be desirable to look at specific materials combinations on a project by project basis, however, it doesn't seem practical to do so, at least not now.

The issue of potential alkali reactivity and/or sulphate expansion is not settled. We will be testing more combinations to identify the potential magnitude of the problem. Toward that end we will soon begin ASTM P214 expansion tests on mortar containing 30 sands. As more information becomes available acceptance criteria could be modified.

We will soon be issuing letters to fly ash suppliers concerning the new specification change. We will then detail approval procedures for ashes between 1.5% and 2.5% available alkalies.

Lon, I appreciate your cooperation in this period of change. It is only by working together that we can be assured of durable concrete in the future.

If you have any questions about our new specifications or acceptance procedures feel free to contact me.

Sincerely,



Bernard C. Brown
Materials Engineer

BCB/esb
cc: D. A. Anderson
J. V. Bergren
O. J. Lane
C. L. Narotam

Figure B3. Iowa DOT letter to Midwest Fly Ash Company, page 1

Appendix C – Core Logs

Site 1

1A

Concrete Core Log-In Form

Sample Identification: I-80 Weigh station
Control section

General Observations:

Dimensions: _____

Surface conditions: Tined, 2mm deep

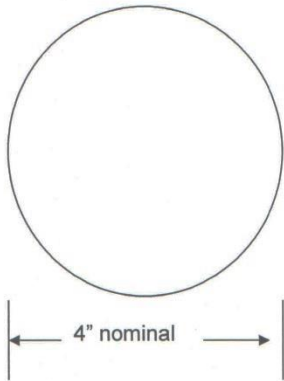
Bottom: turned off due to steel?

Reinforcement: Dowel @ 6 1/2 depth, clear // 3/8" @ bottom rusted
due to no cover

Cracks, comments: NONE

Diagram of core:

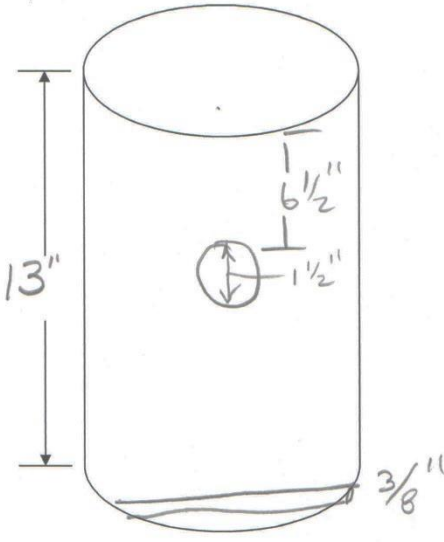
Top view



4" nominal

Mass= 6332

Side View



13"

6 1/2"

1 1/2"

3/8"

Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
334	334	333	331	325	329	330	334	

Figure C1. Core log for site 1, joint

Site 1

1B

Concrete Core Log-In Form

Sample Identification: I-80 Jasper Co

General Observations:

Dimensions: _____

Surface conditions: tined, 2mm depth

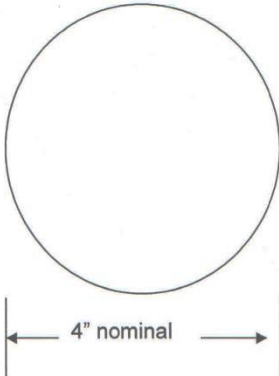
Bottom: crushed stone

Reinforcement: NONE

Cracks, comments: NONE, except for some cracked shale parting

Diagram of core:

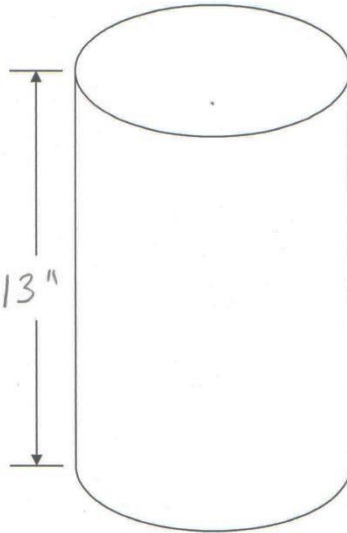
Top view



4" nominal

Mass= 6205g

Side View



13"

Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
330	329	324	330	328	330	331	330	

Figure C2. Core log for site 1, joint

Site 1 IC

Concrete Core Log-In Form

Sample Identification: I-80 Jasper Co

General Observations:

Dimensions: _____

Surface conditions: fined, < 1 mm

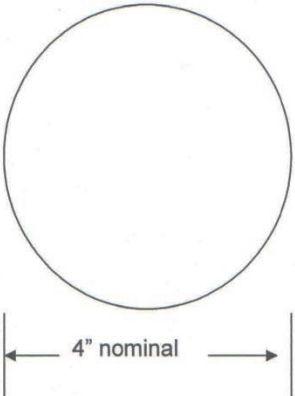
Bottom: crushed stone

Reinforcement: NONE

Cracks, comments: _____

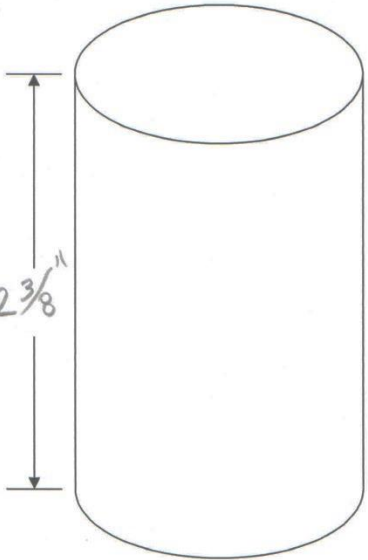
Diagram of core:

Top view



Mass= 5727g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
316	314	315	312	315	314	319	319	

Figure C3. Core log for site 1, mid-panel

Site 2

2A

Concrete Core Log-In Form

Sample Identification: IA 163 EB MP13.80

General Observations:

Dimensions: _____

Surface conditions: fined, about 2mm

Bottom: Not evident - did core turn off?

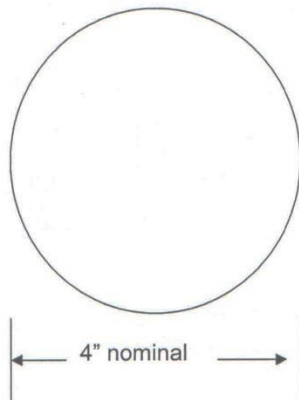
Reinforcement: NONE

Cracks, comments: NONE

Some pop outs noted (g) shale particles

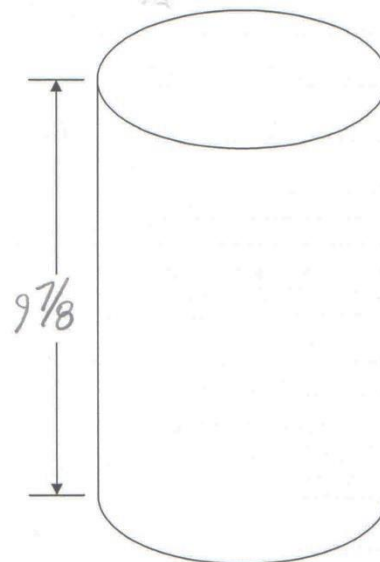
Diagram of core:

Top view



Mass= 4721 g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
253	255	251	252	255	254	253	254	

Figure C4. Core log for site 2, joint

Site 2

2B

Concrete Core Log-In Form

Sample Identification: _____

General Observations:

Dimensions: _____

Surface conditions: tined, not deep ~ 1mm

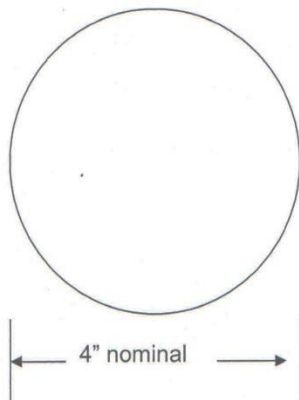
Bottom: crushed stone

Reinforcement: 3/8" bar @ bottom of core

Cracks, comments: NONE

Diagram of core:

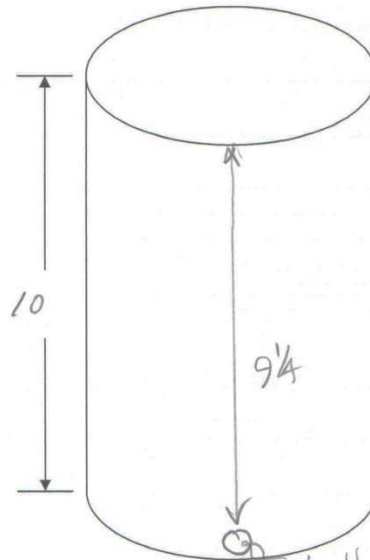
Top view



Mass=

4712

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
252	250	250	249	253	255	253	253	

Figure C5. Core log for site 2, joint

Site 2.

2 c

Concrete Core Log-In Form

Sample Identification: _____

General Observations:

Dimensions: _____

Surface conditions: tined, ~2mm deep

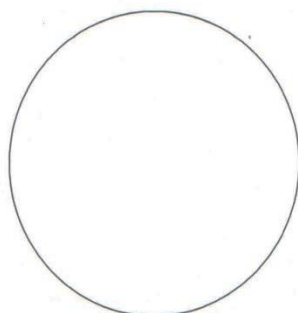
Bottom: crushed stone

Reinforcement: NONE

Cracks, comments: NONE

Diagram of core:

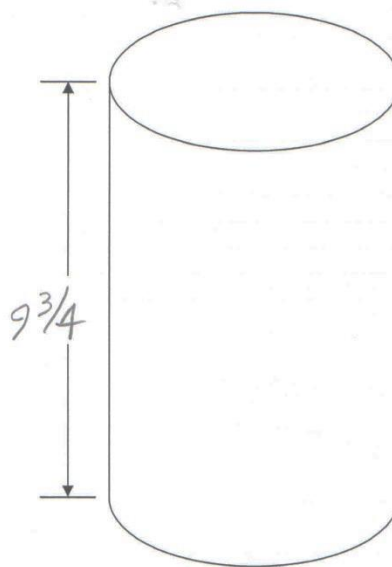
Top view



4" nominal

Mass= 4688g

Side View



Length Measurements (mm)								Ave.
1	2	3	4	5	6	7	8	
250	250	250	247	248	251	249	250	

Figure C6. Core log for site 2, mid-panel

B to A
S
E

Site 3
Core A

Concrete Core Log-In Form

Sample Identification: IA92 (East of Indiarola)

General Observations:

Dimensions: see below

Surface conditions: fine nearly polished smooth

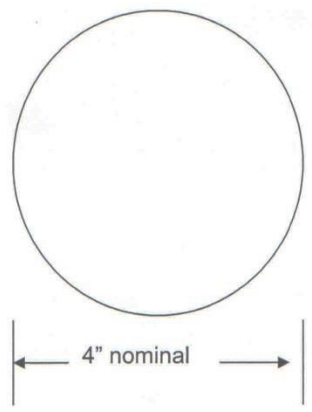
Bottom: cracked stone base

Reinforcement: clipped a dowel @ ~5"

Cracks, comments: Cracks noted in CA near dowel - ? a weak plane?
Mostly well compacted with some shale popouts

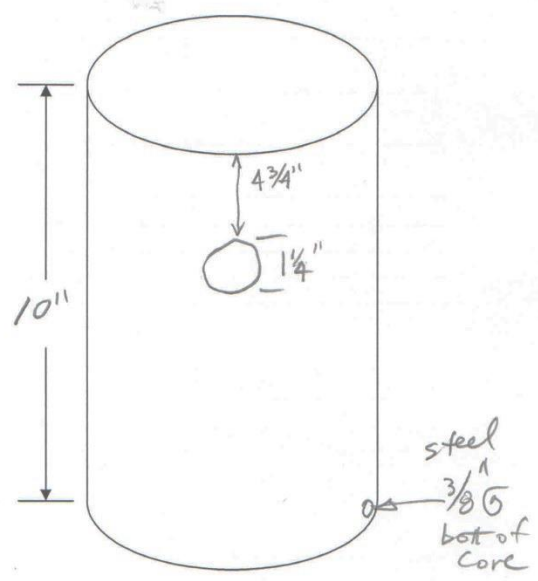
Diagram of core:

Top view



Mass= 4750 g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
255	255	260	255	255	254	256	256	

Figure C7. Core log for site 3, joint

Site 3
Core B

Concrete Core Log-In Form

Sample Identification: Hwy 92 EB

General Observations:

Dimensions: see below

Surface conditions: polished smooth

Bottom: NOT present

Reinforcement: NONE

Cracks, comments: core turned off @ 6 1/2 bottom not extracted

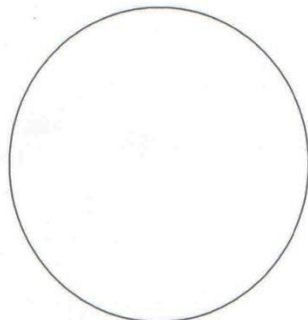
Some pop outs noted - white exudations mostly well compacted

Some discolored areas noted - mostly near top of core - could be due to core bit.

Diagram of core:

Top view

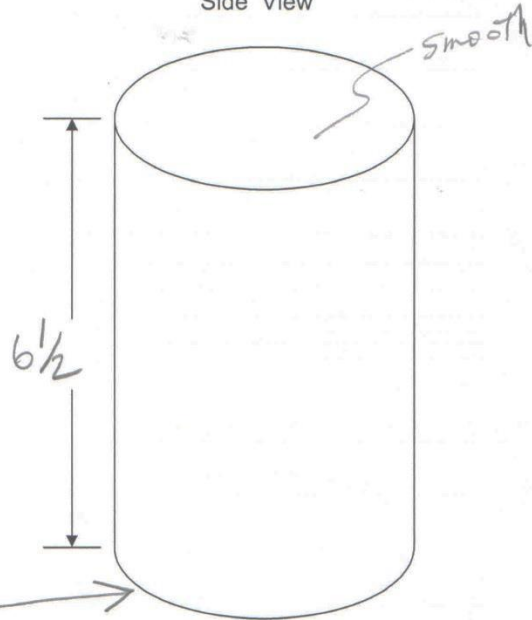
Side View



4" nominal

Mass= 3023

turned off @ 6 1/2



Length Measurements (mm)								Ave.
1	2	3	4	5	6	7	8	
162	165	160	163	168	167	167	163	

Figure C8. Core log for site 3, joint

Site 3
Core C

Concrete Core Log-In Form

Sample Identification: Hwy 92EB

General Observations:

Dimensions: see below

Surface conditions: tired but nearly worn smooth

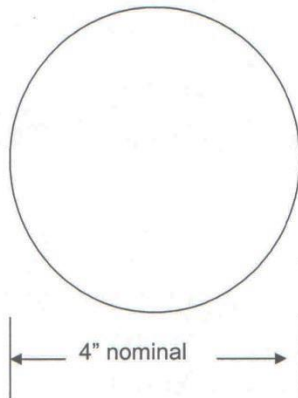
Bottom: crushed rock

Reinforcement: NONE

Cracks, comments: NONE existent except for some pop outs
 near shale particles

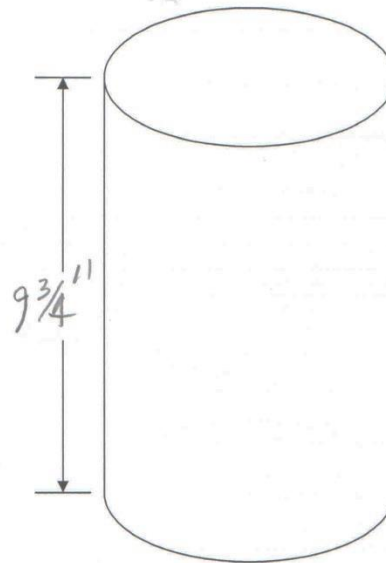
Diagram of core:

Top view



Mass= 4565

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
247	250	247	247	247	248	245	247	

Figure C9. Core log for site 3, mid-panel

Core Site 4 4A

Concrete Core Log-In Form

Sample Identification: HWY 61 SB G MP 15

General Observations:

Dimensions: _____

Surface conditions: no crack evident, tined-shallow

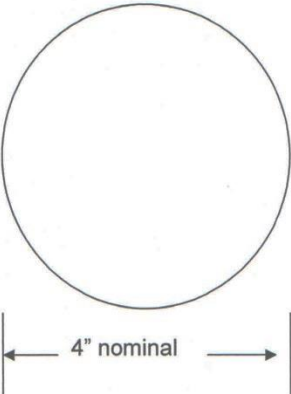
Bottom: crush agg base

Reinforcement: NONE

Cracks, comments: lot of "bugholes" suggest poor compaction

Diagram of core:

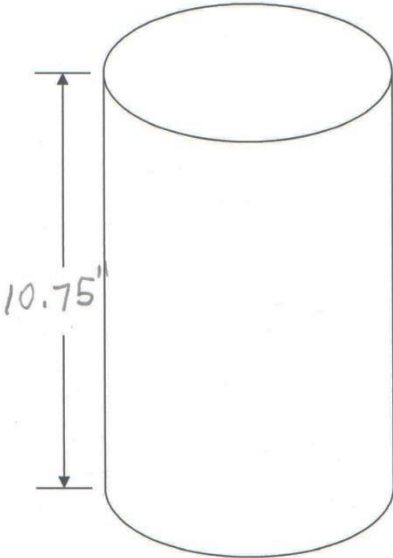
Top view



4" nominal

Mass= 5025.4 g

Side View



10.75"

Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
275	275	272	270	270	269	265	281	

Figure C10. Core log for site 4, joint

Concrete Core Log-In Form

4B

Sample Identification: HWY 61 SB G MP15

General Observations:

Dimensions: _____

Surface conditions: Tined - shallow ~ 1.5 mm

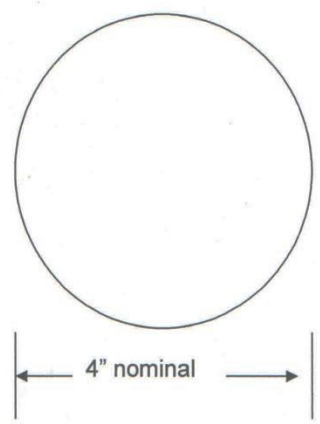
Bottom: crushed agg base

Reinforcement: NONE

Cracks, comments: NONE Evident

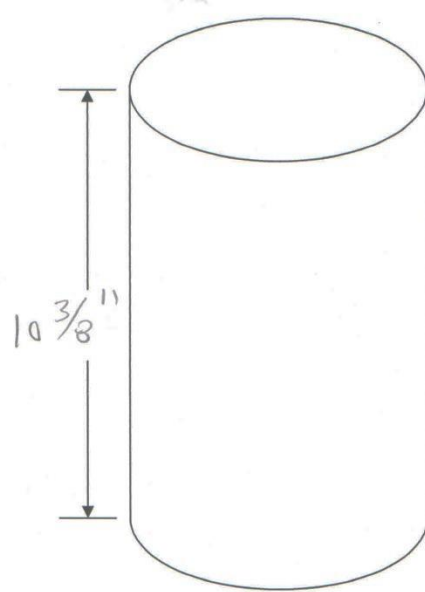
Diagram of core:

Top view



Mass= 4868.2 g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
260	262	262	260	262	262	268	263	

Figure C11. Core log for site 4, joint

AC

Concrete Core Log-In Form

Sample Identification: Hwy 61 SB GMP15

General Observations:

Dimensions: 1

Surface conditions: Tired shallow

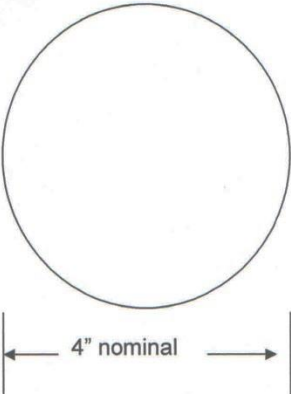
Bottom: Crushed Agg Base

Reinforcement: NONE

Cracks, comments: NONE Evident
some discolored areas - typically
near dark tan/brown agg (fine)
particles

Diagram of core:

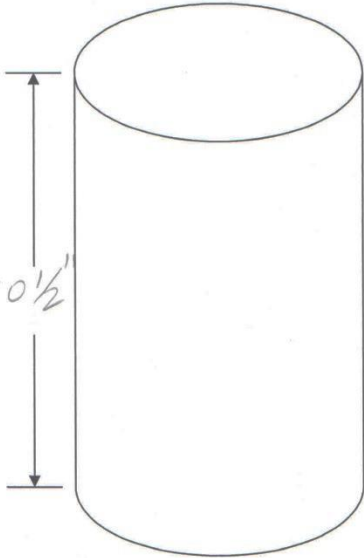
Top view



4" nominal

Mass=

Side View



10 1/2"

Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
265	264	262	265	265	263	262	263	

Figure C12. Core log for site 4, mid-panel

Site 5 (5A)

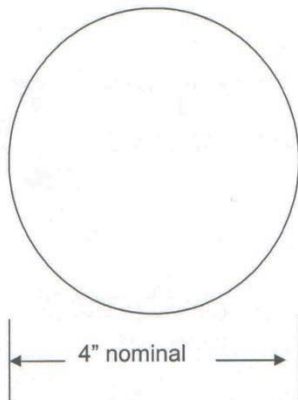
Concrete Core Log-In Form

Sample Identification: LEE Co. Hwy 61 NB
MP 17.2 STA 383

General Observations:
 Dimensions: Tined / Not deeply
 Surface conditions:
 Bottom: crushed rock
 Reinforcement: At bottom of core
 Cracks, comments: None Evident
Core looked good/ No popouts

Diagram of core:

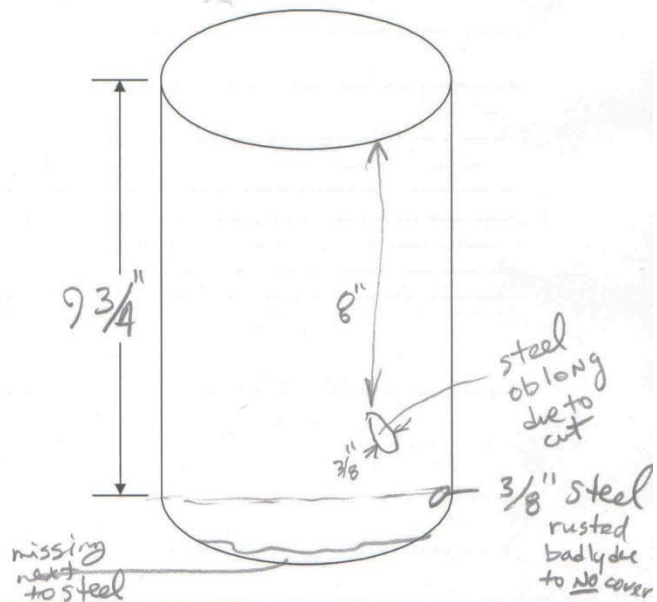
Top view



Mass=

4795g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
254	253	253	255	255	245	247	248	

Figure C13. Core log for site 5, joint

Site 5 (5B)

Concrete Core Log-In Form

Sample Identification: HWT 61NB MP 17.2

General Observations:

Dimensions: see below

Surface conditions: tined / Not deep

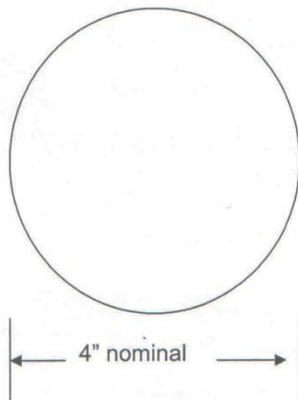
Bottom: crushed rock or ecobase?

Reinforcement: NONE

Cracks, comments: NONE

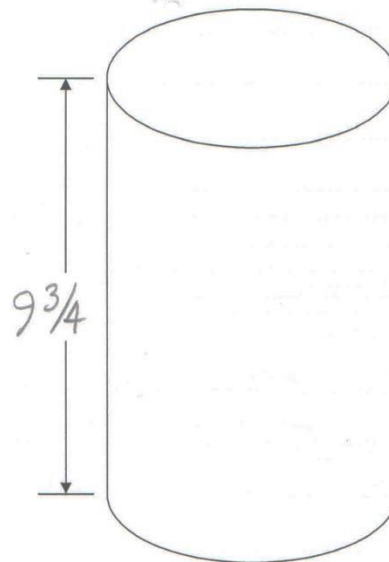
Diagram of core:

Top view



Mass= 4691g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
249	250	248	249	248	250	250	249	

Figure C14. Core log for site 5, joint

Site 5 (5C)

Concrete Core Log-In Form

Sample Identification: Hwy 61NB GMP 17.2

General Observations:

Dimensions: _____

Surface conditions: tined top ~2mm deep

Bottom: crushed stone or ecocrete?

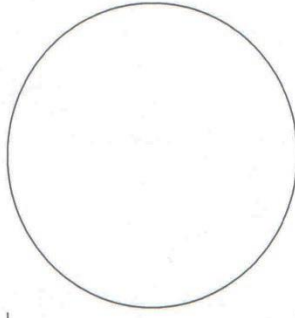
Reinforcement: NONE

Cracks, comments: NONE

Not particularly well compacted

Diagram of core:

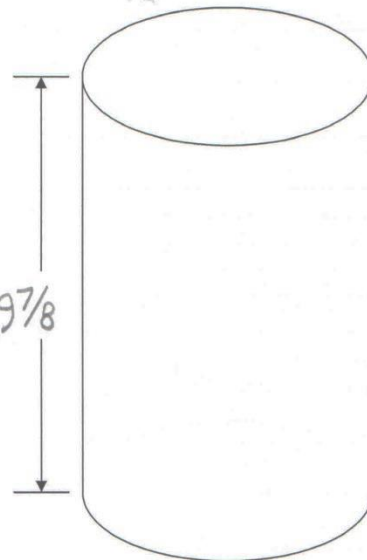
Top view



4" nominal

Mass= 4721g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
250	247	250	252	250	250	251	251	

Figure C15. Core log for site 5, mid-panel

Site 6

6 A

Concrete Core Log-In Form

Sample Identification: Control Section Hwy 61 NB STA 350

General Observations: Asphalt removed from top of core (4")

Dimensions: _____

Surface conditions: Asphalt overlay

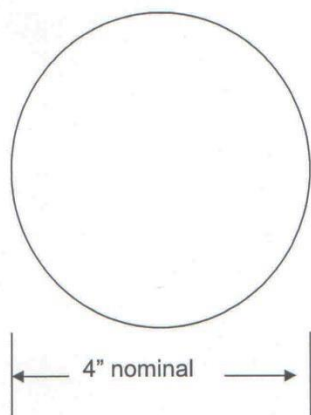
Bottom: Crushed agg base

Reinforcement: NONE

Cracks, comments: No macro cracks evident
many FA particles with white material
so rimmed FA particles
some mottled areas

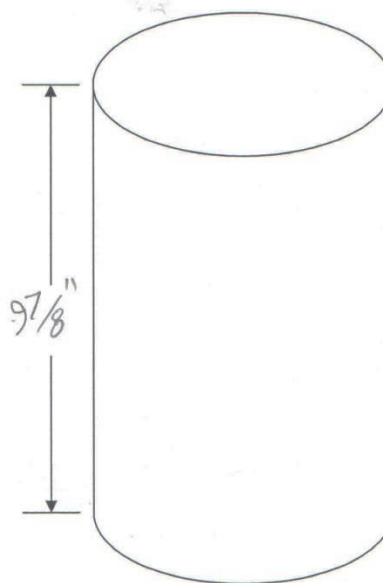
Diagram of core:

Top view



Mass= 4611.4g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
248	248	248	245	245	248	246	250	

Figure C16. Core log for site 6, joint

6B

Concrete Core Log-In Form

Sample Identification: Site 6
HWY 61 NB G STA 350

General Observations: Asphalt top removed from core (4")
 Dimensions: _____

Surface conditions: Asphalt overlay

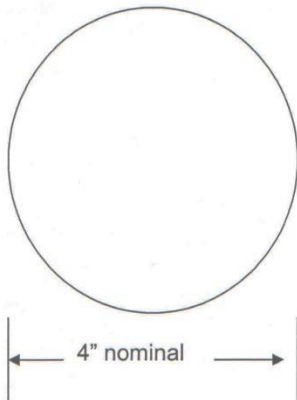
Bottom: crushed Agg Base

Reinforcement: NONE

Cracks, comments: No macro cracks
some white material in voids/near FA
particle (with some rims).
Some mottled areas

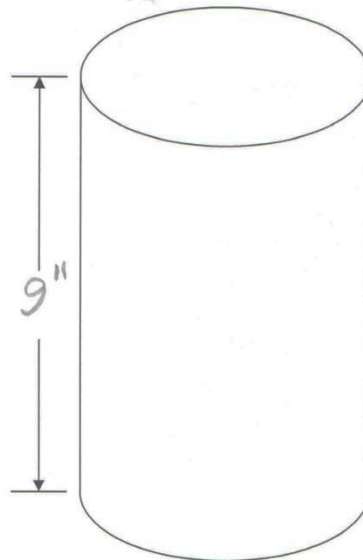
Diagram of core:

Top view



Mass= 4247.1g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
225	226	229	223	226	227	225	225	

Figure C17. Core log for site 6, joint

6C

Concrete Core Log-In Form

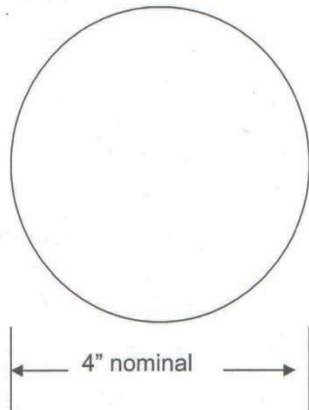
Sample Identification: Site 6 HWY 61 NB STA 350General Observations: Asphalt top removed from core (4")

Dimensions: _____

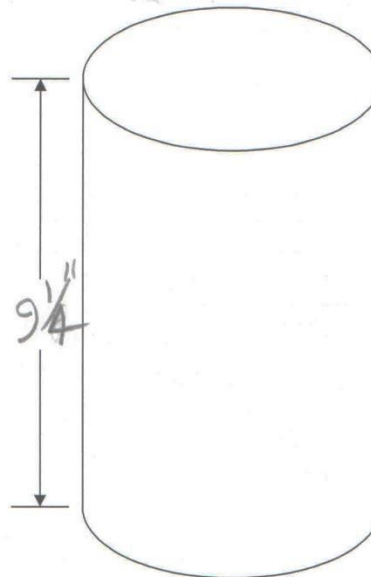
Surface conditions: Asphalt overlayBottom: crushed Agg BaseReinforcement: NONECracks, comments: No macro-cracks
some white material in voids near FA
particlesSome mottled areas

Diagram of core:

Top view

Mass= 4322.1

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
230	232	235	238	240	238	239	232	

Figure C18. Core log for site 6, mid-panel

Concrete Core Log-In Form

7A

Sample Identification: Site 7 Hwy 61 NB STA 470

General Observations: Asphalt overlay removed (4")

Dimensions: _____

Surface conditions: Asphalt overlay

Bottom: ? Crush agg base

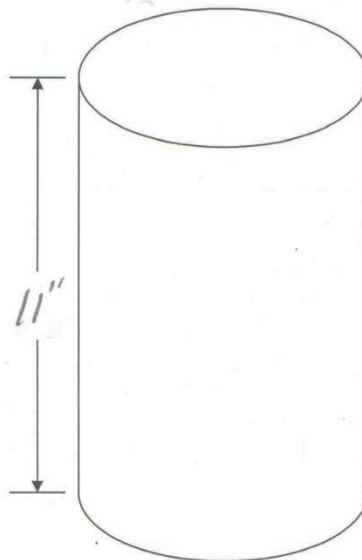
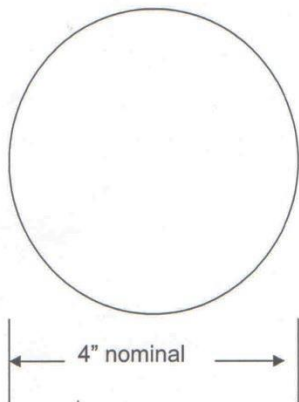
Reinforcement: NONE

Cracks, comments: No macro cracks
Some white residue in voids &
in FA particles
Some mottled areas

Diagram of core:

Top view

Side View



Mass= 5134.4g

Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
275	276	277	276	277	275	277	277	

Figure C19. Core log for site 7, joint

7B

Concrete Core Log-In Form

Sample Identification: Site 7 Hwy 61 NBG STA 470

General Observations: Asphalt removed from core (4")

Dimensions: _____

Surface conditions: ? crush agg base or just rolled?

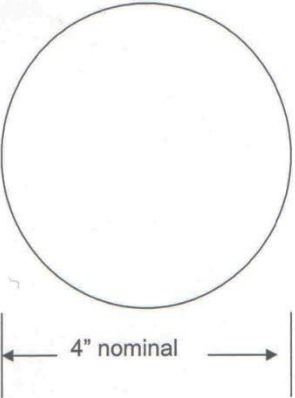
Bottom: →

Reinforcement: NONE

Cracks, comments: No macro cracks
some white material in voids
near FA particles
lots of iron-bearing FA particles
some mottled areas

Diagram of core:

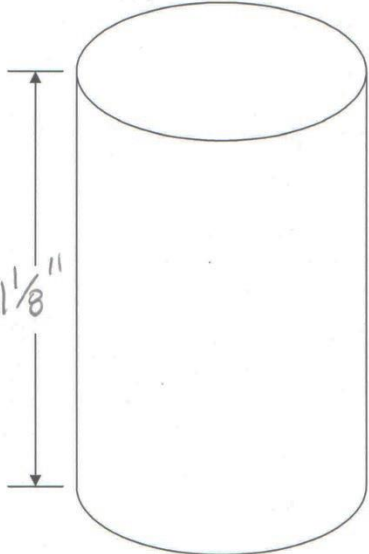
Top view



4" nominal

Mass= 5262.1

Side View



11 1/8"

Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
281	280	279	281	280	280	278	281	

Figure C20. Core log for site 7, joint

Concrete Core Log-In Form

7C

Sample Identification: Site 7 HWY 61 NB G STA 470

General Observations: Asphalt over lay removed from core (4")

Dimensions: _____

Surface conditions: Asphalt overlay

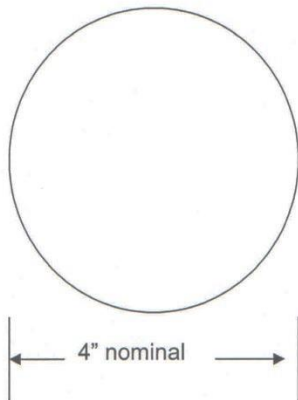
Bottom: ? crushed Agg base or just rolled stone?

Reinforcement: NONE

Cracks, comments: 1 - macro crack S running to fines
penetrates into core about 1"
White residue in voids near #A
some mottling

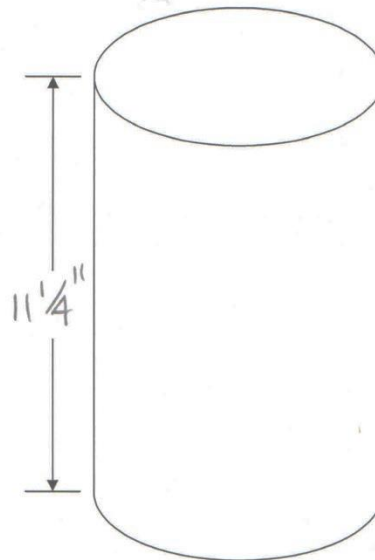
Diagram of core:

Top view



Mass= 5308.0

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
<u>284</u>	<u>284</u>	<u>285</u>	<u>283</u>	<u>283</u>	<u>285</u>	<u>285</u>	<u>285</u>	

Figure C21. Core log for site 7, mid-panel

Site 8
8A

Concrete Core Log-In Form

Sample Identification: Hwy 61 SB STA 538 nearspall

General Observations:

Dimensions: _____

Surface conditions: tined, shallow < 1mm

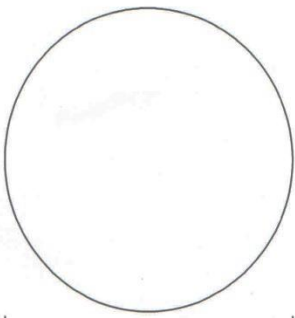
Bottom: crushed Agg base

Reinforcement: NONE

Cracks, comments: No macro cracks
 some white residue near FA particles
 on surface of core

Diagram of core:

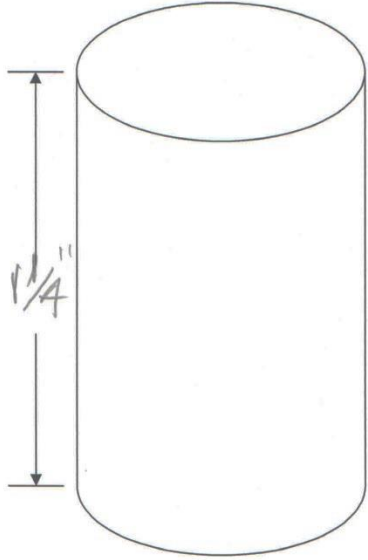
Top view



4" nominal

Mass= 5276.2

Side View



11 1/4"

Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
283	283	286	285	285	285	284	282	

Figure C22. Core log for site 8, joint

Concrete Core Log-In Form

8B

Sample Identification: Site 8 Hwy 61 SB

General Observations:

Dimensions: _____

Surface conditions: tired/shallow

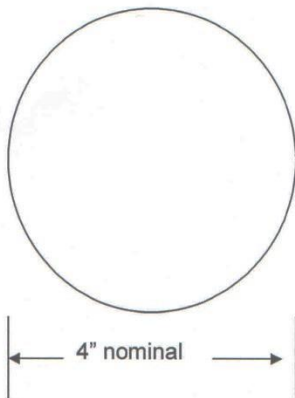
Bottom: crushed Agg base

Reinforcement: NONE

Cracks, comments: No macro cracks
some FA particles rimmed
many Fe-bearing FA particles

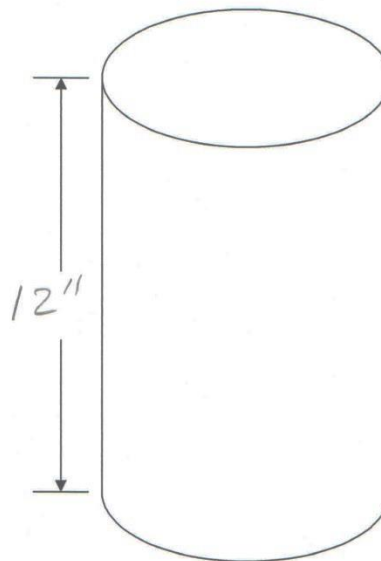
Diagram of core:

Top view



Mass= 5539.3

Side View



Length Measurements (mm)								Ave.
1	2	3	4	5	6	7	8	
300	302	301	301	304	301	299	305	

Figure C23. Core log for site 8, joint

8C

Concrete Core Log-In Form

Sample Identification: Site 8 Hwy 61 SB

General Observations:

Dimensions: _____

Surface conditions: lined, moderate depth

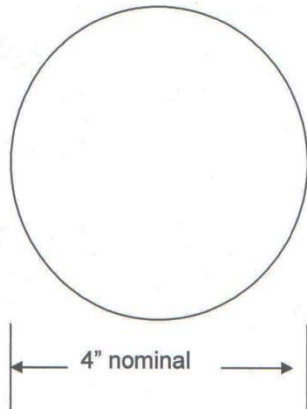
Bottom: crushed Agg base

Reinforcement: NONE

Cracks, comments: No macro cracks
Iron-bearing FA particles noted / some with rings
ICA particle cracked on surface / lower 1/3rd of core

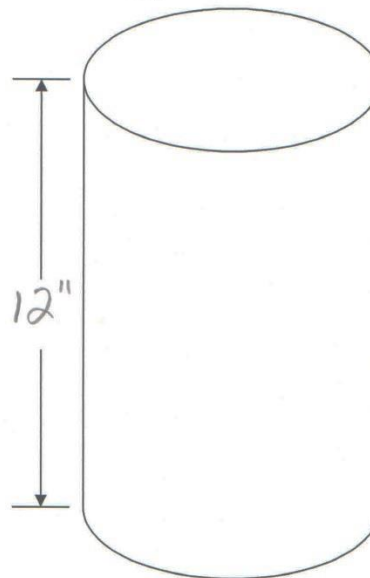
Diagram of core:

Top view



Mass= 5608.7g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
305	306	304	297	295	297	305	308	

Figure C24. Core log for site 8, mid-panel

Site 9

9A

Concrete Core Log-In Form

Sample Identification: Hwy 61 NB G STA 536

General Observations:

Dimensions: _____

Surface conditions: tined, polished, ~1mm deep

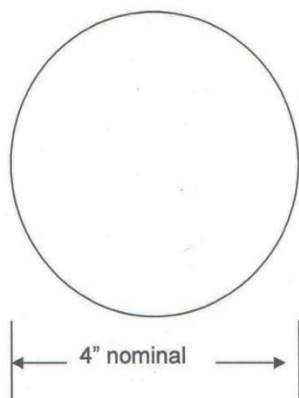
Bottom: crushed stone base

Reinforcement: Rusted steel @ bottom - cover < 1/2"

Cracks, comments: No macro cracks
some FA particles had "moisture rims"

Diagram of core:

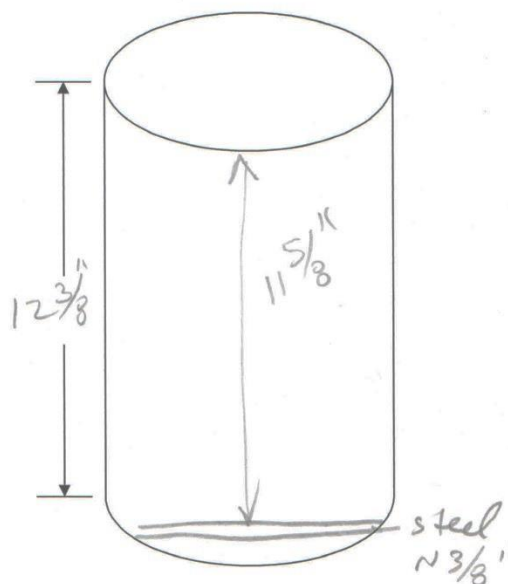
Top view



Mass=

5915g

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
313	315	313	314	315	311	315	316	

Figure C25. Core log for site 9, joint

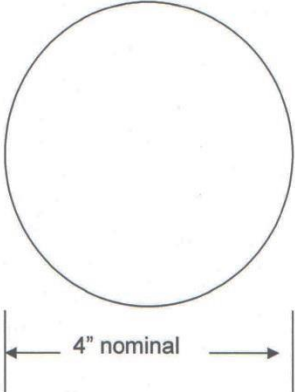
site 9
 9B

Concrete Core Log-In Form

Sample Identification: HWY 61 NB @ STA 536

General Observations:
 Dimensions: _____
 Surface conditions: tired, <1mm depth
 Bottom: crushed stone base
 Reinforcement: small bars @ 7" & 11 depth - generally not rusted
 Cracks, comments: NONE

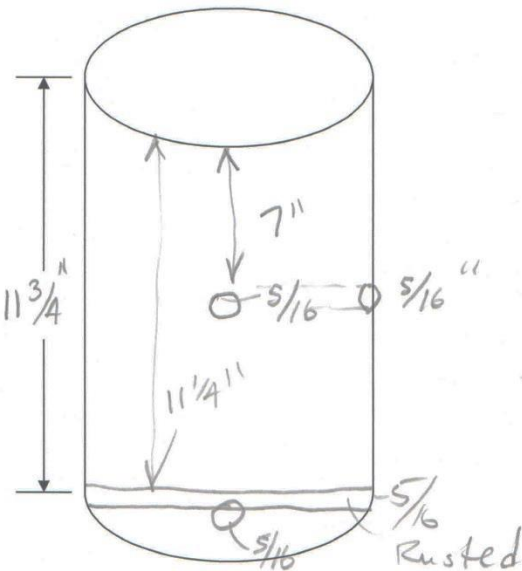
Diagram of core:
Top view



4" nominal

Mass= 5622

Side View



Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
290	300	303	309	300	300	300	305	

Figure C26. Core log for site 9, joint

Site 9
 9C

Concrete Core Log-In Form

Sample Identification: HWY 61 NB G STA 536

General Observations:

Dimensions: _____

Surface conditions: tired, irregular, polished to agg.

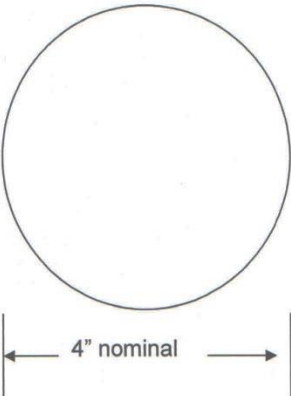
Bottom: crushed rock base

Reinforcement: NONE

Cracks, comments: NONE
several FA particles are cracked

Diagram of core:

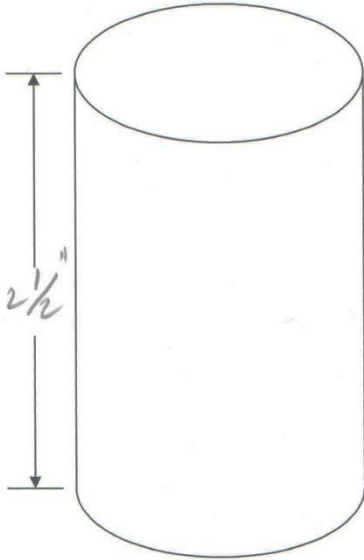
Top view



4" nominal

Mass= 5847

Side View



12 1/2"

Length Measurements (mm)								
1	2	3	4	5	6	7	8	Ave.
310	315	315	315	315	323	321	317	

Figure C27. Core log for site 9, mid-panel