CEMENT AND CONCRETE REFERENCE LABORATORY PROFICIENCY SAMPLE PROGRAM

Final Report Pozzolan Proficiency Samples Number 35 and Number 36

November 2004



CEMENT AND CONCRETE REFERENCE LABORATORY

AT THE NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY GAITHERSBURG, MARYLAND 20899 (301) 975-6704

SPONSORED BY COMMITTEE C-1 ON CEMENT COMMITTEE C-9 ON CONCRETE AND CONCRETE AGGREGATES AMERICAN SOCIETY FOR TESTING AND MATERIALS

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November 24, 2004

To: Participants in the CCRL Pozzolan Proficiency Sample Program

SUBJECT: Pozzolan Proficiency Samples No. 35 and No. 36

Enclosed is your copy of the final report on the test results for the CCRL **Pozzolan** Proficiency Samples which were distributed in August 2004.

This report consists of a statistical Summary of Results, a set of general Scatter Diagrams, and associated detailed information. The Table of Results with test results and ratings for your laboratory can be downloaded at our website located at: <u>http://ccrl.us/psp/pspdata.htm</u>.

The CCRL Proficiency Sample Programs are intended for internal use by the laboratory as a tool to identify potential problems in laboratory procedures or test equipment and to initiate remedial actions. These programs are designed to complement the CCRL Laboratory Inspection Program as part of a total quality system. Care should be taken when using this program for any other use.

Additional samples of these two cement and other CCRL samples are available for purchase. These samples may be useful for equipment verification, technician training, and research. Contact CCRL for availability and price.

It is presently anticipated that the next Pozzolan Proficiency Samples will be distributed in August 2005.

Sincerely,

Polin K. Haust

Robin K. Haupt Supervisor, Proficiency Sample Programs Cement and Concrete Reference Laboratory Materials and Construction Research Division Building and Fire Research Laboratory

Enclosure

To: Participants in the CCRL Pozzolan Proficiency Sample Program

FROM: Robin K. Haupt, Supervisor, PSP

SUBJECT: Explanation of Final Report on Results of Tests on Pozzolan Proficiency Samples No. 35 and No. 36

This letter, and the material included with it, constitute the final report and summary of results for the current pair of Pozzolan Proficiency Samples, which were distributed in August 2004. This material includes a Table of Results for individual laboratory data, a statistical Summary of Results, and a set of general Scatter Diagrams. Your unique laboratory number is displayed at the top of the individual Table of Results.

An explanation of the program is contained in the paper: "Statistical Evaluation of Interlaboratory Cement Tests" by J. R. Crandall and R. L. Blaine <u>View document</u>, and "Statistical Aspects of the Cement Testing Program" by W.J. Youden <u>View document</u>, which can be found in Volume 59, Proceedings of the 62nd Annual Meeting of the Society, June 25, 1959, American Society for Testing and Materials.

Laboratory Ratings

Each laboratory receives an individualized Table of Results. The Table of Results shows the test title and the reporting unit in the first two columns. After that it lists in order, the laboratory's results for the odd and even numbered samples, overall averages for the odd and even numbered samples, and the laboratory's ratings for the odd and even samples. (See reverse for an explanation of the scatter diagrams.)

The ratings for the individual laboratory were determined in the manner described by Crandall and Blaine using a rating scale 1 to 5 instead of 0 to 4. The ratings have no valid standing beyond showing the difference between the individual laboratory result and the average for a particular test.

The following table details the relationship between the ratings and the averages.

| Ratings | Range (Number of Standard Deviations) | Number (Per 100) of Laboratories achieving the rating ¹ | | |
|---------|--|--|--|--|
| 5 | Less than 1 | 69 | | |
| 4 | 1 to 1.5 | 18 | | |
| 3 | 1.5 to 2 | 9 | | |
| 2 | 2 to 2.5 | 3 | | |
| 1 | Greater than 2.5 | 1 | | |

The sign of the rating merely shows whether the result reported was greater or less than the average obtained.

¹Youden, W.J., "Statistical Aspects of the Cement Testing Program", Volume 59, *Proceedings of the 62nd Annual Meeting of the Society, June 25, 1959, American Society for Testing and Materials.*

In cases where some laboratories' results are eliminated, averages, standard deviations, coefficients of variation and the ratings of the other laboratories' results, are recalculated using the data remaining after the elimination. Since the laboratory ratings given are the results from this one series of tests, you need not attach too much significance to a single low rating, or pair of ratings, from this one series. A continuing tendency to get low ratings on several pairs of samples should lead a laboratory to consider the types of error, systematic and random, which contribute to ratings that are low. Systematic error, which is indicated by low ratings with the same signs on each pair of samples, means a consistent error is occurring in equipment and/or test procedures. One indication of random error is low ratings on both samples with different signs. Since systematic error occurs with more regularity, its cause is generally easier to find than the cause of random error.

Summary of Results

Usually, averages, standard deviations, and coefficients of variation are given with all test results reported, and then with one or more outlying test results omitted. Sometimes, two or more recalculations with laboratories omitted, have been performed for the same test. In these cases, all of the laboratories omitted in previous recalculations are also omitted in subsequent ones. Results omitted are values that are more than three standard deviations from the mean of one or both samples. Often, elimination of these outlying results has little effect on the average, but may have a more pronounced effect on the standard deviation and coefficient of variation.

Scatter Diagrams

General scatter diagrams are supplied with this report. Crandall and Blaine describe the manner of preparing scatter diagrams, and their interpretation, in the paper published in the 1959 ASTM Proceedings. Each laboratory will receive a complete set of diagrams according to their participation in chemical and/or physical tests.

Using the results received from each laboratory, a scatter diagram is generated for each test method by plotting the value for the odd numbered samples on the *X*, or horizontal axis, against the value for the even numbered samples on the *Y*, or vertical axis. To find your point, just plot as you would when plotting any scatter diagram. Vertical and horizontal dashed lines, which divide the diagrams into four sections or quadrants, place the average values for the odd and even numbered samples, respectively. The first line of print under the diagram includes the test number, as given on the data sheet, the test title, and the number of data points on the diagrams. The number of plotted points may not agree with the total number of data pairs included in the analysis because a few points may be off the diagram, and some points may represent several data pairs, which are identical. Laboratories whose points are off the diagram will have a rating of ± 1 for that particular test.

As described in Crandall and Blaine, a tight circular pattern of points around the intersection of the median lines is the ideal situation. Stretching out of the pattern into the first (upper right) and third (lower left) quadrants, suggests some kind of bias, or tendency for laboratories to get high or low results on both samples. Examination of the scatter diagrams may indicate strong evidence of bias in many cases.

CCRL PROFICIENCY SAMPLE PROGRAM Pozzolan Proficiency Samples No. 33 and No. 34 Final Report - Chemical Results November 24, 2004

SUMMARY OF RESULTS

| | | | | Sample | e No. 35 | Sample No. 36 | | | |
|--|-------------------|-----|----|-------------|------------|---------------|---------|-------|------|
| Test | #Labs | | | Average | S.D. | C.V. | Average | S.D. | C.V. |
| Moisture Content | prent | | 55 | 0.13 | 0.070 | 52.8 | 0.11 | 0.053 | 47.6 |
| Moisture Content | prent | * | 53 | 0.12 | 0.046 | 37.4 | 0.10 | 0.041 | 39.1 |
| Silicon Dioxide | prent | | 49 | 56.10 | 4.4 | 7.77 | 52.75 | 2.9 | 5.47 |
| Silicon Dioxide | prent | * | 45 | 56.76 | 1.9 | 3.43 | 53.00 | 1.5 | 2.87 |
| Al ₂ O ₃ w/minor ¹ | prent | | 24 | 29.55 | 3.1 | 10.4 | 25.18 | 3.0 | 11.9 |
| $Al_2O_3^2$ w/minor ¹ (P ₂ O ₃ & TiO ₂ ir | prent ncluded) | * | 22 | 30.06 | 1.8 | 5.91 | 25.33 | 1.6 | 6.39 |
| Al_2O_2 wo/minor ² | prent | | 38 | 27.29 | 3.1 | 11.22 | 23.20 | 2.1 | 9.02 |
| Al_2O_3 wo/minor ² | prent | * | 36 | 27.87 | 1.17 | 4.19 | 23.51 | 0.94 | 4.00 |
| 2 (P ₂ O ₃ & TiO ₂ n | ot inclu | ded |) | | | | | | |
| Ferric Oxide | prent | | 47 | 5.26 | 0.74 | 14.1 | 4.02 | 0.42 | 10.5 |
| Ferric Oxide | prent | * | 43 | 5.42 | 0.45 | 8.38 | 4.05 | 0.31 | 7.67 |
| Calcium Oxide | prent | | 48 | 0.80 | 0.26 | 32.77 | 12.11 | 0.57 | 4.69 |
| Calcium Oxide | prent | * | 46 | 0.82 | 0.23 | 28.51 | 12.16 | 0.51 | 4.16 |
| | | | | CONTINUED (| ON NEXT PA | AGE | | | |

* ELIMINATED LABS: Data over three S.D. from the mean

| Moisture Content | 126 1251 |
|---|---------------|
| Silicon Dioxide | 15 23 205 930 |
| Al ₂ O ₃ w/minor oxides | 45 930 |
| Al_2O_3 wo/minor oxides | 23 25 |
| Ferric Oxide | 25 29 50 2150 |
| Calcium Oxide | 23 930 |

CCRL PROFICIENCY SAMPLE PROGRAM Pozzolan Proficiency Samples No. 35 and No. 36 Final Report - Chemical Results November 24, 2004

SUMMARY OF RESULTS

| | | | | Sample | e No. 35 | Sample No. 36 | | | |
|--|----------------------------------|----|----------------------|------------------------------|---------------------------------|------------------------------|------------------------------|---------------------------------|----------------------|
| Test | | #I | Labs | Average | S.D. | C.V. | Average | S.D. | C.V. |
| Magnesium Oxide | prcnt | | 48 | 0.82 | 0.40 | 48.4 | 2.07 | 0.25 | 12.2 |
| Magnesium Oxide | prent | * | 46 | 0.76 | 0.16 | 20.7 | 2.04 | 0.21 | 10.4 |
| Sulfur Trioxide | prent | | 50 | 0.17 | 0.094 | 53.9 | 0.59 | 0.126 | 21.5 |
| Sulfur Trioxide | prent | * | 48 | 0.17 | 0.090 | 53.1 | 0.56 | 0.071 | 12.6 |
| Loss on Ignition | prent | | 58 | 2.51 | 0.12 | 4.77 | 0.64 | 0.14 | 22.29 |
| Loss on Ignition | prent | * | 54 | 2.49 | 0.077 | 3.11 | 0.61 | 0.089 | 14.61 |
| Sodium Oxide | prent | | 44 | 0.20 | 0.084 | 41.9 | 0.28 | 0.090 | 31.6 |
| Potassium Oxide | prent | | 42 | 2.25 | 0.22 | 9.95 | 0.79 | 0.15 | 19.32 |
| Potassium Oxide | prent | * | 36 | 2.25 | 0.130 | 5.78 | 0.77 | 0.050 | 6.51 |
| Available Na ₂ O | prent | | 27 | 0.09 | 0.12 | 132.8 | 0.15 | 0.12 | 82.2 |
| Available Na_2^2O | prent | * | 23 | 0.06 | 0.027 | 44.5 | 0.12 | 0.024 | 20.2 |
| Available K ₂ O | prcnt | | 27 | 0.49 | 0.177 | 36.2 | 0.24 | 0.081 | 33.3 |
| Available K_2O | prent | * | 25 | 0.48 | 0.085 | 17.9 | 0.23 | 0.039 | 17.3 |
| Available Alkali | prent | | 27 | 0.43 | 0.20 | 47.4 | 0.32 | 0.17 | 52.8 |
| Available Alkali | prent | * | 25 | 0.38 | 0.091 | 24.2 | 0.27 | 0.047 | 17.3 |
| Available K ₂ O Available K ₂ O Available Alkali Available Alkali | prent prent prent prent | * | 27 25 27 25 | 0.49 0.48 0.43 0.38 | 0.177 0.085 0.20 0.091 | 36.2 17.9 47.4 24.2 | 0.24 0.23 0.32 0.27 | 0.081 0.039 0.17 0.047 | 33 17 52 17 |

* ELIMINATED LABS: Data over three S.D. from the mean

| Magnesium Oxide | 40 205 |
|------------------------------|----------------------|
| Sulfur Trioxide | 47 1940 |
| Loss on Ignition | 19 52 126 158 |
| Potassium Oxide | 25 40 52 46 205 2116 |
| Available Na ₂ O | 44 52 46 2522 |
| Available $K_2 \overline{O}$ | 40 44 |
| Available Alkali | 44 2522 |

CCRL PROFICIENCY SAMPLE PROGRAM Moisture Content POZZOLAN SAMPLES NO. 35 & NO. 36



LABS ELIMINATED 126 1251

CCRL PROFICIENCY SAMPLE PROGRAM Silicon Dioxide POZZOLAN SAMPLES NO. 35 & NO. 36



LABS OFF DIAGRAM 2150



SAMPLE NO. 36 AVE 25.33 S.D. 1.6 C.V. 6.39 LABS ELIMINATED 45 930

CCRL PROFICIENCY SAMPLE PROGRAM Aluminum Oxide (minor oxides excluded) POZZOLAN SAMPLES NO. 35 & NO. 36



LABS ELIMINATED 23 25

CCRL PROFICIENCY SAMPLE PROGRAM Ferric Oxide POZZOLAN SAMPLES NO. 35 & NO. 36



LABS ELIMINATED 25 29 50 2150



LABS OFF DIAGRAM 1

CCRL PROFICIENCY SAMPLE PROGRAM





LABS OFF DIAGRAM 45





SAMPLE NO. 36 AVE 0.565 S.D. 0.071 C.V. 12.6 LABS ELIMINATED 47 1940

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LABS ELIMINATED 19 52 126 158





CCRL PROFICIENCY SAMPLE PROGRAM Potassium Oxide POZZOLAN SAMPLES NO. 35 & NO. 36



LABS ELIMINATED 25 40 52 46 205 2116

CCRL PROFICIENCY SAMPLE PROGRAM Available Sodium Oxide POZZOLAN SAMPLES NO. 35 & NO. 36



LABS ELIMINATED 44 52 46 2522

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CCRL PROFICIENCY SAMPLE PROGRAM Available Potassium Oxide POZZOLAN SAMPLES NO. 35 & NO. 36



SAMPLE NO. 36 AVE 0.2280 S.D. 0.039 C.V. 17.3 LABS ELIMINATED 40 44

CCRL PROFICIENCY SAMPLE PROGRAM Available Alkali POZZOLAN SAMPLES NO. 35 & NO. 36



CCRL PROFICIENCY SAMPLE PROGRAM Pozzolan Proficency Sample No. 35 and No. 36 Final Report - Physical Results November 24, 2004

SUMMARY OF RESULTS

| | | | | Sample No. 35 | | | Sample No. 36 | | |
|------------------------------------|--|-----|-----------|-------------------|-----------------|--------------------------|----------------------------|---------------------------|--------------|
| Test | | #L | abs | Average | S.D. | C.V. | Average | S.D. | C.V. |
| Density Density | g/cm ³ g/cm ³ | * | 57 55 | 2.26 2.23 | 0.14 0.037 | 6.21 1.67 | 2.34 2.32 | 0.15 0.047 | 6.56 2.02 |
| 45μm Sieve 45μm Sieve | prent prent | * | 64 60 | 31.02 31.55 | 5.1 2.8 | 16.6 9.05 | 20.58 21.02 | 4.2 1.7 | 20.4 8.19 |
| Drying Shrinkage | prent | | 13 | 0.016 | 0.039 | 240 | 0.009 | 0.045 | 503 |
| Autoclave Expan Autoclave Expan | prent prent | * | 48 45 | 0.02 0.02 | 0.035 0.018 | 149 78.4 | 0.01 0.02 | 0.063 0.019 | 491 88.7 |
| N.C. Water | prcnt | | 50 | 26.2 | 0.82 | 3.14 | 24.9 | 0.82 | 3.30 |
| Air Entrainment | prent | | 7 | 0.347 | 0.82 | 236 | 0.352 | 0.83 | 235 |
| STRENGTH ACTIV | TTY INI | DEX | (SAI |) with Porti | LAND CEMI | ENT | | | |
| SAI 7 day | prent | * | 52 | 77 | 5.7 | 7.43 | 85 | 4.8 | 5.58 |
| SAI 28 day SAI 28 day | prent prent | * | 49 47 | 84 83 | 8.3 4.7 | 9.86 5.66 | 98 96 | 8.9 4.8 | 9.11 4.99 |
| SAI Water SAI Water | prent prent | * | 54 52 | 98 100 | 9.9 2.1 | 10.13 2.14 | 96 98 | 9.4 1.8 | 9.85 1.88 |
| EFFECTIVENESS C Reduction Expan | of Mini prent | ERA | L AD 9 | MIXTURES IN 56 | Controll 9.8 | ing Alkal 17.6 | 1-SILICA REAC 60 | TIONS (ASR 13.6 |) 22.7 |

* ELIMINATED LABS: Data over three S.D. from the mean

| Density | 33 47 |
|---------------------|-------------|
| 45µm Sieve | 15 24 26 33 |
| Autoclave Expansion | 23 47 126 |
| SAI 7 day | 15 1251 |
| SAI 28 day | 15 1251 |
| SAI Water | 158 1251 |

CCRL PROFICIENCY SAMPLE PROGRAM Density POZZOLAN SAMPLES NO. 35 & NO. 36



LABS ELIMINATED 33 47

CCRL PROFICIENCY SAMPLE PROGRAM Fineness - 45 micron Sieve Retained POZZOLAN SAMPLES NO. 35 & NO. 36



SAMPLE NO. 35 AVE 31.55 S.D. 2.8 C.V. 9.05 SAMPLE NO. 36 AVE 21.02 S.D. 1.7 C.V. 8.19 LABS ELIMINATED 15 24 26 33 LABS OFF DIAGRAM 1773

CCRL PROFICIENCY SAMPLE PROGRAM Drying Shrinkage POZZOLAN SAMPLES NO. 35 & NO. 36



CCRL PROFICIENCY SAMPLE PROGRAM Autoclave Expansion POZZOLAN SAMPLES NO. 35 & NO. 36



LABS OFF DIAGRAM 40 2295

CCRL PROFICIENCY SAMPLE PROGRAM Normal Consistency Water POZZOLAN SAMPLES NO. 35 & NO. 36

CCRL PROFICIENCY SAMPLE PROGRAM Air Entrainment POZZOLAN SAMPLES NO. 35 & NO. 36

LABS ELIMINATED 15 1251

IMINATED 15 1251

SAMPLE NO. 35 AVE 82.91 S.D. 4.7 C.V. 5.66 SAMPLE NO. 36 AVE 96.00 S.D. 4.8 C.V. 4.99 LABS ELIMINATED 15 1251

CCRL PROFICIENCY SAMPLE PROGRAM SAI Water Requirement POZZOLAN SAMPLES NO. 35 & NO. 36

LABS OFF DIAGRAM 1940

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CCRL PROFICIENCY SAMPLE PROGRAM Alkali-Silica Reaction - Reduction of Expansion POZZOLAN SAMPLES NO. 35 & NO. 36

