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Certification of SRM 114q: Part I

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Abstract

The standard reference material (SRM) for fineness of cement, SRM 114, is an integral part of the calibration material routinely used in the cement industry to qualify cements. Being a powder, the main physical properties of cement, prior to hydration, are its surface area and particle size distribution (PSD). Since 1934, NIST has provided SRM 114 for cement fineness and it will continue to do so as long as the industry requires it. Different lots of SRM 114 are designated by a unique letter suffix to the SRM number, e.g., 114a, 114b, ..., 114q. A certificate that gives the values obtained using ASTM C204 (Blaine), C115 (Wagner) and C430 (45 μm sieve residue) is included with each lot of the material. The supply of SRM 114p, which was released in 1994, was depleted in 2004.

Therefore, a new batch of SRM 114 needed to be developed. This process included selection of the cement, packaging the cement in small vials, and determination of the values for the ASTM tests reported. In this case, the Blaine, Wagner and 45 μm sieve residue were the tests used. Later the particle size distribution (PSD) will be added.

The purpose of this report is to provide a detailed description of the process used to package and certify SRM 114q. All measurements used for the certifications are provided along with descriptions of the statistical analyses.

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1 Introduction

A standard reference material (SRM) is a material that has been well characterized with regard to its chemical composition, physical properties, or both. National Institute of Standards and Technology (NIST) provides over 1300 different SRMs to industry and academia. These materials are used in quality assurance programs, for calibration, and to verify the accuracy of experimental procedures. Every NIST SRM is provided with a certificate of analysis that gives the official characterization of the material's properties. In addition, supplementary documentation, such as this report, that describes the development, analysis and use of SRMs is also often published by NIST to provide the context necessary for effective use of these materials.

There are several SRMs related to cement (<http://ts.nist.gov/ts/htdocs/230/232/232.htm>). SRM 114 is related to the fineness of cement, as measured by various indirect methods giving its surface area and by passing the material through a fine sieve. This SRM is an integral part of the calibration material routinely used in the cement industry to qualify a cement. Being a powder, the main physical properties of cement are its surface area and particle size distribution (PSD). Since 1934, NIST has provided SRM 114 for cement fineness and it will continue to do so as long as the industry requires it. Different lots of SRM 114 are designated by a unique letter suffix to the SRM number. A certificate that gives the values obtained using ASTM C 204 (Blaine) [1], C 115 (Wagner) [2] and C 430 (45- μm residue) [3] is included with each lot of the material.

In 1934, only the Wagner test and the 45 μm residue were listed. In 1944, the Blaine measurement was added to the certificate of the SRM 114. In 2003, the PSD measured by laser diffraction was added as an information value, i.e., not certified. The PSD was obtained under the sponsorship of ASTM Task Group C01.25.01 [4, 5, 6].

The supply of SRM 114p, which was released in 1994, was depleted in 2004. Therefore, a new batch of SRM 114 needed to be developed. The development process included the selection of a cement, packaging of the cement in small vials, and determination of the values for the ASTM tests reported. In this case, the Blaine, Wagner and 45 μm sieve residue were the tests used. Later the particle size distribution (PSD) will be added.

The values given in this report were obtained through a round-robin inter-laboratory study by volunteer participants from companies participating in the CCRL certification program. The particle size distribution by laser diffraction was also collected in the same round-robin study but will be discussed in a separate report.

The purpose of this report is to provide the description of the development of SRM 114q. It includes a detailed description of the process used to package and certify the cement. Also a brief description of the ASTM tests in the certificate is given. All measurements used for the certifications are provided along with the statistical analysis.

2 Description of methods used

2.1 Blaine ASTM C 204

The Blaine measurement described in ASTM C 204 was adopted by ASTM in 1946. R.L. Blaine published the test in 1943 [7]. The principle of operation is that the permeability of a bed of fine particles is proportional to the fineness of the particles. Therefore, the test is a measurement of the flow rate of air through a bed of cement particles. From the beginning, it was stated that this is a relative test as it depends on the shape of the particles, and the compaction level or porosity of the bed. For this reason, ASTM C 204 section 4.1 states that the calibration of the instrument needs to be done by using a reference material, such as SRM 114 [8].

In brief, the test is carried out by packing the cement to be measured in a cell of known volume and placing it on top of a U-tube manometer that contains a non-hygroscopic liquid of low viscosity and density, e.g., dibutyl phthalate or a light grade of mineral oil. The cell is placed on the U-tube in such a way that a tight seal is created and a vacuum is created under the cement cell so that the liquid in the manometer is higher toward the cell. Then, the air is allowed to flow back only through the cement sample. The time for the liquid in the manometer to descend a set distance is measured. This time is used to calculate the fineness quantified by the surface area S of the cement defined using the following formula:

$$S = \frac{S_s \sqrt{T}}{\sqrt{T_s}} \quad (1)$$

where S_s is the surface area of the reference material, i.e., SRM 114

T_s is the time of flow using the reference material, i.e., SRM 114

T is the time of flow of the material under test

S is the surface area of the material under test

Therefore, the surface area of the material tested can be calculated from the reference material.

The results requested from the participants in the round-robin study to determine the value of SRM 114q were calculated using SRM 114p as the reference material. All participants were requested to measure the SRM 114p material immediately before measuring the SRM 114q material and to report both results.

2.2 Wagner ASTM C 115

The Wagner test method described in ASTM C115 was adopted by ASTM in 1934 after a paper published by L.A. Wagner in 1933 [9]. This test is also referred as the turbidimeter fineness test because it measures the turbidity of a cement suspension in kerosene.

A source of light shines through the cement suspension and is detected by a photoelectric cell. The intensity of the current generated by the cell is recorded. The calibration is done by calculation of a calibration factor, K , which depends on the cell used. To determine this factor a reference material, such as SRM 114, must be used.

Using a reference with known surface area, this test can be used to obtain the surface area of materials similar to the reference, (i.e. Portland cements), by comparing the two relative levels of turbidity. This method could also determine the particle size distribution, but is limited to particles larger than 7.5 μm . Due to this bound on particle size and various other sources of error, it is rarely used for PSD determinations.

The results requested from the participants in the round-robin study for SRM 114q used SRM 114p as the calibrant.

2.3 Sieve Residue ASTM C 430

The principle of this test is to measure the residue or retained amount of cement on a calibrated sieve. The sieve was selected as having a 45 μm opening (No. 325²). Since a direct certification of sieve openings is impractical and expensive for production-scale work, sieves are calibrated by using a reference material, such as SRM 114. A sieve correction factor is calculated by measuring SRM 114 on the selected sieve and correcting the result with the certified value of the SRM 114.

To avoid the need to propagate the SRM 114p uncertainty in the certification of the sieve residue for the SRM 114q material, however, three sieves with nominal openings of 38 μm , 47 μm , and 56 μm were directly calibrated for use in reference material certification. Interpolation was then used to obtain the value at 45 μm .

Although direct sieve calibration was also used for previous generations of this material, The procedure used for the 114q material was different in several respects from the certification of the two preceding lots of SRM 114:

- For SRM 114n sieves with openings of 42 μm to 43.9 μm were used. The average results were plotted against the average sieve opening. The certified value was calculated by extrapolation to 45 μm .
- For SRM 114p sieves with openings of 42 μm to 46 μm were used and all data points (rather than the averages) were plotted against the minimum sieve opening. The certified value was calculated by interpolation to 45 μm .

² Sieve number follow the USA definition given in ASTM E11

3 Materials

3.1 Characteristics of the cement

Based on the properties of past lots of SRM 114, CCRL and NIST identified a plant with a suitable cement for SRM 114q from Lehigh Cement Company³, Union Bridge, Maryland, who donated 1300 kg of the cement for this SRM. The material selected was Type I according to the ASTM C 150 Standard Classification, and had a mass fraction of less than 8 % tricalcium aluminate (C₃A) as defined by ASTM C 150. This requirement was the same as for SRM 114p. Material was collected for shipment to NIST directly from the finish mill process stream into bags.

The approximate chemical composition has been determined by ASTM Standard Test Method C 114-02 to provide additional information on this cement. The analyses of this cement (CCRL Portland Cement Proficiency Sample No. 150) were performed by 70 to 170 laboratories. The composition, which is not certified but is provided for information only, is shown on Table 1.

Calculation of cement compounds from this chemistry, according to ASTM C 150-02, are shown in Table 2, again for information only.

The density of the cement was also measured using a modified ASTM C 188 method. The modification was to use isopropanol (IPA) as the medium instead of kerosene, otherwise a calibrated Le Chatelier flask was used as described in the ASTM test. Two measurements were done: 3.255 g/cm³ and 3.248 g/cm³. This leads to an average of 3.25 g/cm³ ± 0.005 g/cm³.

Table 1: Chemical composition

| | CaO | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | SO ₃ | K ₂ O | TiO ₂ | P ₂ O ₅ | Na ₂ O | MgO | loss on ignition |
|--------------------------|-----|------------------|--------------------------------|--------------------------------|-----------------|------------------|------------------|-------------------------------|-------------------|-----|------------------|
| Percent by mass fraction | 64 | 20.7 | 4.7 | 3.2 | 2.4 | 0.7 | 0.3 | 0.12 | 0.07 | 2.2 | 1.67 |

³ Commercial equipment, instruments, and materials mentioned in this report are identified to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology (NIST), nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 2: Cement compounds according to ASTM C150

| Compound | Percent by mass fraction |
|---|---------------------------------|
| C ₃ S (tricalcium silicate) | 60 |
| C ₂ S (dicalcium silicate) | 14 |
| C ₃ A (tricalcium aluminate) | 7 |
| C ₄ AF (tetracalcium aluminoferrite) | 10 |

3.2 Packaging

Upon its arrival at NIST, the cement was blended in a V-blender (1.7 m³ or 60 ft³) and then transferred to 0.2 m³ (55 gal) drums lined with 0.15 mm (6 mil) polyethylene liners to minimize hydration of the cement in storage prior to preparation and packaging. Over the next two days, the cement from each drum was sealed in foil bags, each containing about 16 kg of cement. The foil bags were stored, and subsequently packaged as described below into vials, in a climate-controlled area.

Each foil bag was packaged into vials and capped and boxed. Each box contained approximately 500 sealed vials and the boxes were sequentially labeled from 1 to 118. Usually about five boxes were filled per day. The more than 60 000 glass vials produced, each containing approximately 5 g of cement, were subsequently sealed into smaller individual foil bags. The vials were randomly selected (see section 4) and shipped to the participating laboratories for measurements. After the analysis of the results was completed the vials were packaged in boxes containing 20 vials each.

3.3 Homogeneity determination

After the material was packaged, it was necessary to determine whether the material in different vials was the same. A special concern was any absorption of moisture during packaging from original delivery to placement in the vials. Therefore, the first test that was performed was loss of ignition (LOI). Cement taken from random vials was submitted to the following procedure:

- The content of each sampled vial was divided among 3 crucibles (about 2 g of cement per crucible) and the mass was recorded
- The crucible was placed in an oven at 105 °C for 12 h, and then allowed to cool down in a desiccator containing a desiccant.
- The mass was recorded, and the crucible was placed in a furnace at 950 °C for 3 h.
- The sample was allowed to cool down in a desiccator and the mass was recorded.
- The LOI was calculated as the water loss during the residence in the furnace per mass of the sample (after the oven drying).

The LOI value is an indication of the amount of water loss during the drying procedure. A high value would be an indication of improper handling of the cement.

The LOI values are shown in Table 3. The Box # identifies a randomly sampled vial from that box. The LOI averages are very small (less than 0.15 %). Therefore, we conclude that there was no significant intake of moisture during packaging.

Nevertheless, further checks were performed during the analysis of the round-robin data by determining whether a box-to-box or vial-to-vial variation was detected (see Section 4).

Table 3: Results from the LOI tests

| Box # | LOI [%] | Average [%] | St. Dev [%] |
|-------|---------|-------------|-------------|
| 9 | 0.224 | 0.11 | 0.10 |
| | 0.045 | | |
| | 0.050 | | |
| 27 | 0.097 | 0.15 | 0.07 |
| | 0.224 | | |
| | 0.137 | | |
| 35 | 0.100 | 0.13 | 0.10 |
| | 0.243 | | |
| | 0.048 | | |
| 47 | 0.047 | 0.08 | 0.09 |
| | 0.179 | | |
| | 0.000 | | |
| 53 | 0.000 | 0.02 | 0.03 |
| | 0.000 | | |
| | 0.046 | | |
| 56 | 0.049 | 0.11 | 0.16 |
| | 0.000 | | |
| | 0.290 | | |
| 72 | 0.048 | 0.11 | 0.05 |
| | 0.135 | | |
| | 0.141 | | |
| 76 | 0.139 | 0.13 | 0.07 |
| | 0.189 | | |
| | 0.049 | | |
| 98 | 0.091 | 0.03 | 0.05 |
| | 0.000 | | |
| | 0.000 | | |
| 118 | 0.089 | 0.08 | 0.03 |
| | 0.046 | | |
| | 0.100 | | |

4 Experiment Design and Data Analysis

4.1 Blaine ASTM C 204

The data for the Blaine test, ASTM C 204, were collected using a nested experiment design with three factors, laboratory, box, and vial. In this design, two randomly selected laboratories who use the Blaine test were each supplied two randomly drawn vials of cement from a randomly assigned box of vials. The laboratory was then asked to measure duplicate samples from each vial. Use of this design allows assessment of lab-to-lab variability, box-to-box heterogeneity of the cement, and vial-to-vial heterogeneity of the cement.

For the analysis of the Blaine data, the first step was to screen the data for non-statistical problems. Based on this screening, results from three labs, 209, 247, and 932, were eliminated from the data set. In each case, the data from these laboratories were omitted from the analysis because Blaine measurements were made on one vial from the box designated for the Blaine measurements and one vial from the box designated for the Wagner measurements. Due to the nature of the experiment design, including these results would have made it more difficult to distinguish between the different potential sources of variation.

Next, a nested, random-effects, analysis of variance (ANOVA) model [10] was fit to the remaining data to check for box-to-box, lab-to-lab, and vial-to-vial variability. Residual plots to verify that the model fit the data showed that two labs each had two extreme outlying measurements. Since there were a large number of labs (66) for this analysis and only two had unusual data, the data from these two labs (123 and 886) was also omitted from the analysis. After refitting the model, residual plots indicate that the model fits the data well. The output from the analysis of variance is shown in Table 4. The low p values (<0.05) corresponding to the F tests for the significance of lab-to-lab and vial-to-vial variability provide strong evidence that there is significant lab-to-lab variation and vial-to-vial heterogeneity. The high p value for the F test for box-to-box variability (0.365) indicates that there is not strong evidence of significant box-to-box heterogeneity in this data.

Table 4: ANOVA Output from the Fit of a Nested, Random-Effects Model with Factors Box, Lab, and Vial to the Blaine Data

| Source | Degrees of Freedom | Sums of Squares | Mean Squares | F Statistic | p value |
|---------------------------|--------------------|-----------------|--------------|-------------|---------|
| Box-to-Box | 37 | 18703.91 | 505.511 | 1.143 | 0.365 |
| Lab-to-Lab (in Box) | 26 | 11494.21 | 442.085 | 29.328 | 0.000 |
| Vial-to-Vial (in Box/Lab) | 64 | 964.72 | 15.074 | 2.960 | 0.000 |
| Residuals | 128 | 651.83 | 5.092 | | |

Because there is significant vial-to-vial heterogeneity, a prediction interval should be used for certification. Based on the ANOVA results above, a summary of the material's properties as measured with the Blaine procedure is given in Table 5.

Table 5: Summary of the Specific Surface Area Results for SRM 114q using the Blaine Procedure

| | | |
|--|--------|--------------------|
| Mean Blaine Result: | 381.81 | m ² /kg |
| Standard Uncertainty of Mean: | 1.31 | m ² /kg |
| Effective Degrees of Freedom: | 26.00 | |
| Standard Uncertainty of Heterogeneity: | 2.23 | m ² /kg |
| Effective Degrees of Freedom: | 26.55 | |
| Standard Uncertainty of 114p | 2.93 | m ² /kg |
| Effective Degrees of Freedom: | 60.00 | |
| Combined Standard Uncertainty: | 3.91 | m ² /kg |
| Effective Degrees of Freedom: | 102.66 | |
| Coverage Factor (95 % coverage): | 1.98 | |
| Expanded Uncertainty: | 7.76 | m ² /kg |

4.2 Wagner ASTM C 115

The experiment design and analysis used for the Wagner [2] data are analogous to the design and analysis used for the Blaine data. As the first step in the analysis, the Wagner data were also screened for non-statistical problems. Based on this screening, data from one lab, 247, were eliminated from the data set because Wagner measurements were made on one vial from the box designated for the Wagner measurements and one vial from the box designated for the Blaine measurements.

Next we fit a nested, random effects, analysis of variance model to the remaining data to check for box-to-box, lab-to-lab, and vial-to-vial variability. Residual plots indicate that the model fits the data well, although there is only one degree of freedom for estimating the lab-to-lab variability. The analysis of variance results are shown in Table 6. The high p values for the tests for significant box-to-box and vial-to-vial variability do not provide strong evidence that the material is heterogeneous as measured by the Wagner method. Even with one degree of freedom, however, significant lab-to-lab variation is evident as indicated by the low p value for the F test for the significance of lab-to-lab variability.

Table 6: ANOVA Output from the Fit of a Nested, Random-Effects Model with Factors Box, Lab, and Vial to the Wagner Data

| Source | Degrees of Freedom | Sums of Squares | Mean Squares | F Statistic | p value |
|---------------------------|--------------------|-----------------|--------------|-------------|---------|
| Box-to-Box | 4 | 250579.600 | 62644.900 | 0.335 | 0.841 |
| Lab-to-Lab (in Box) | 1 | 186966.100 | 186966.100 | 295.696 | 0.000 |
| Vial-to-Vial (in Box/Lab) | 6 | 3793.800 | 632.300 | 0.907 | 0.522 |
| Residuals | 12 | 8367.500 | 697.300 | | |

Since there is no evidence of vial-to-vial or box-to-box variability, it was decided to base the certified value and uncertainty for the Wagner method on the mean and standard deviation of the laboratory means. Based on this decision, a summary of the certification results is given in Table 7. The final uncertainty is large because so few labs are using the Wagner method and could provide results using this method in the inter-laboratory study.

Table 7: Summary of the Specific Surface Area Results for SRM 114q: Wagner Procedure

| | | |
|----------------------------------|--------|--------------------|
| Mean Wagner Result: | 218.33 | m ² /kg |
| Standard Uncertainty of Mean: | 6.04 | m ² /kg |
| Effective Degrees of Freedom: | 5.00 | |
| Standard Uncertainty of 114p: | 2.35 | m ² /kg |
| Effective Degrees of Freedom: | 60.00 | |
| Combined Standard Uncertainty: | 6.48 | m ² /kg |
| Effective Degrees of Freedom: | 6.62 | |
| Coverage Factor (95 % coverage): | 2.44 | |
| Expanded Uncertainty: | 15.86 | m ² /kg |

4.3 Sieve Residue ASTM C 430

A nested design with three factors, day, box and vial was used to collect the sieve residue data. The same design values were used to make measurements on each of the three sieves with one measurement per sieve made from each vial tested. This design allows for the estimation of day-to-day, box-to-box, and vial-to-vial variation. In addition, since the three sieves are each of different sizes spanning the sieve value of interest, 45 μm , the data from this experiment can also be used to fit a model relating sieve residue to the sieve opening size.

For the analysis of the sieve residue data, the data were first plotted to graphically identify potential factor effects and to look for any unusual observations. The plot in Figure 1 identifies two outliers, from measurements on a vial that had been noted to contain a different color of cement than the others. Since these points were obtained from a suspect sample and were very different from all of the other data, they were eliminated from the analysis. Measurements of other samples from this vial, which did not appear to

have unusual values, were still used, however. After removal of the outliers, some evidence of day-to-day variation between the samples is also evident in Figure 2.

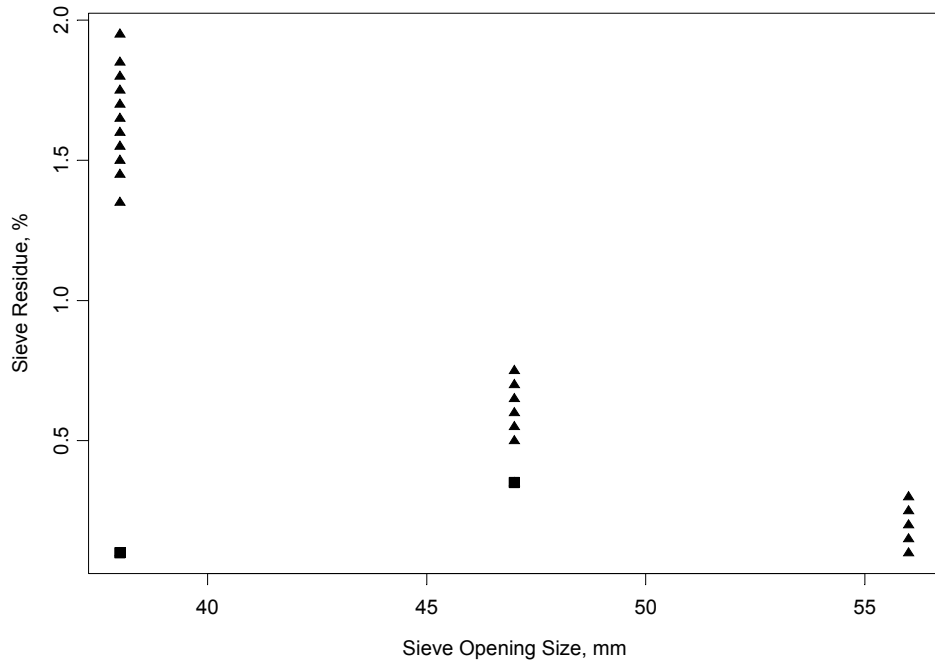


Figure 1: Plot of sieve residue data showing two outliers (square plotting symbols) from a vial that had been noted as containing cement of a different color than the other vials.

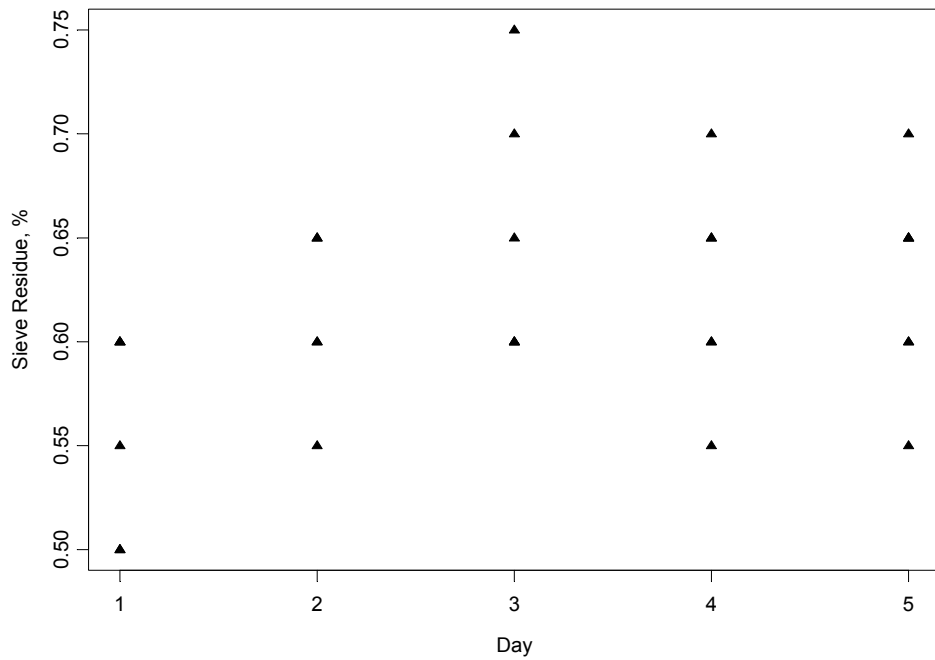


Figure 2: Plot of sieve residue data for the 47 μm sieve (with the outlier omitted) that shows evidence of day-to-day variation in the measurement process.

Next, a nested, random effects analysis of variance model was fit to the data from each sieve to check for day-to-day, box-to-box, and vial-to-vial variability. To maintain the balance of the design for the two sieves for which 1 of the 40 measurements was omitted, the means of the remaining data from the same box as the omitted vial were substituted for the missing data points. Residual plots from each fit verify that the models fit the data from each sieve reasonably well. The ANOVA results, showing the output from the fit of the model to the data from each sieve, are given in Table 8 to Table 10. The relatively high p values in Table 8 ($p > 0.05$) indicate that there is not strong evidence for day-to-day, box-to-box, or vial-to-vial in these data collected using the 38 μm sieve. The low p value for the F test for significant day-to-day variation (0.025) does indicate that there is significant day-to-day variation in the data collected using the 47 μm sieve. There is no evidence of significant variability from the other two factors, however. Finally, the high p values for day-to-day and box-to-box variability for the 56 μm sieve indicate that those factors are not significant, but the low p value for the F test for vial-to-vial variability indicates that there is significant vial-to-vial variation in the measurements made with the 56 μm sieve

Table 8: ANOVA Output from the Fit of a Nested, Random-Effects Model with Factors Box and Vial to the Sieve Residue Data for the 38 μm Sieve

| Source | Degrees of Freedom | Sums of Squares | Mean Squares | F Statistic | p value |
|---------------------------|--------------------|-----------------|--------------|-------------|---------|
| Day-to-Day | 4 | 0.124 | 0.031 | 1.126 | 0.439 |
| Box-to-Box (in Day) | 5 | 0.138 | 0.028 | 2.085 | 0.095 |
| Vial-to-Vial (in Day/Box) | 10 | 0.178 | 0.018 | 1.614 | 0.174 |
| Residuals | 20 | 0.220 | 0.011 | | |

Table 9: ANOVA Output from the Fit of a Nested, Random-Effects Model with Factors Box and Vial to the Sieve Residue Data for the 47 μm Sieve

| Source | Degrees of Freedom | Sums of Squares | Mean Squares | F Statistic | p value |
|---------------------------|--------------------|-----------------|--------------|-------------|---------|
| Day-to-Day | 4 | 0.047 | 0.012 | 7.406 | 0.025 |
| Box-to-Box (in Day) | 5 | 0.008 | 0.002 | 0.722 | 0.612 |
| Vial-to-Vial (in Day/Box) | 10 | 0.017 | 0.002 | 0.679 | 0.731 |
| Residuals | 20 | 0.049 | 0.002 | | |

Table 10: ANOVA Output from the Fit of a Nested, Random-Effects Model with Factors Box and Vial to the Sieve Residue Data for the 56 μm Sieve

| Source | Degrees of Freedom | Sums of Squares | Mean Squares | F Statistic | p value |
|---------------------------|--------------------|-----------------|--------------|-------------|---------|
| Day-to-Day | 4 | 0.010 | 0.003 | 0.965 | 0.500 |
| Box-to-Box (in Day) | 5 | 0.013 | 0.003 | 1.066 | 0.399 |
| Vial-to-Vial (in Day/Box) | 10 | 0.4690 | 0.005 | 3.261 | 0.012 |
| Residuals | 20 | 0.029 | 0.001 | | |

For the certification analysis, a Bayesian model relating the sieve residue of the 114q material from each sieve to the measured sieve size was developed. A Bayesian model was used in order to be able to account for the uncertainty in both the sieve calibrations and the measurements of the reference material made using those sieves. Because of the apparent day-to-day and vial-to-vial variation, and to allow for the fact that those effects were not consistently visible for all sieves, the Bayesian model accounts for all four possible sources of random variation that were included in the design, day-to-day, box-to-box, vial-to-vial, and measurement uncertainty. Although it might not be necessary to treat all of the sieves in the same way, since only one exhibited significant apparent day-to-day variability and one exhibited significant apparent vial-to-vial variability, it was decided to treat them consistently in case those effects (or box-to-box effects) were simply not detected for the other two sieves.

In order to specify inter-relationships among the data, the model specifies the hypothesized relationships between the unknown true values underlying the data (called parameters in statistical jargon), which are to be estimated. In this case, the model specifies a quadratic relationship between the true sieve residues and true sieve sizes. The data gives us the quantitative information needed to estimate the parameter values. Then the model is used to interpolate the result that would be obtained from a sieve with openings of exactly 45 μm .

In order to use the Bayesian model, initial assessments of the values of each parameter in the model must be provided. The initial assessment for each parameter is specified as a probability distribution for the parameter's unknown value. Because these probability distributions are specified independently of the data (i.e. before the data are observed or used), these distributions are commonly called prior distributions. For this analysis, non-informative prior distributions were used. These distributions are essentially very flat and have very large variances so that they will not provide any quantitative information about the values of the parameters as part of the model.

A probability distribution for each measurement, given with respect to the parameters in the model, is also specified. This distribution is called the likelihood of the data. For this analysis, based on typical assumptions for the statistical analysis of metrological data, normal distributions with means specified by the quadratic relationship between residue and sieve size and standard deviations based on sieve size were used for the likelihood. Then, based on the hypothesized relationships between the parameters, the likelihood, and the observed data, the prior distributions for each parameter are updated using Bayes'

Theorem to obtain new distributions for each parameter given the information in the data. Finally, these new distributions, called posterior distributions, are used to obtain uncertainty intervals about each quantity of interest.

The Bayesian model was fit using Markov Chain Monte Carlo simulation [11]. Figure 3 shows a plot comparing the within and between chain variation [12] that indicates that the Markov Chains had converged by the 50 000th iteration of the simulation. Then 10 000 additional iterations were run for each of 4 parallel Markov chains to estimate the parameter values. Based on these results, the posterior probability distribution describing the sieve residue for a 45 μm sieve is shown in Figure 4. A 95 % probability interval for the value of the sieve residue obtained from this posterior distribution is 0.79 % \pm 0.19 %.

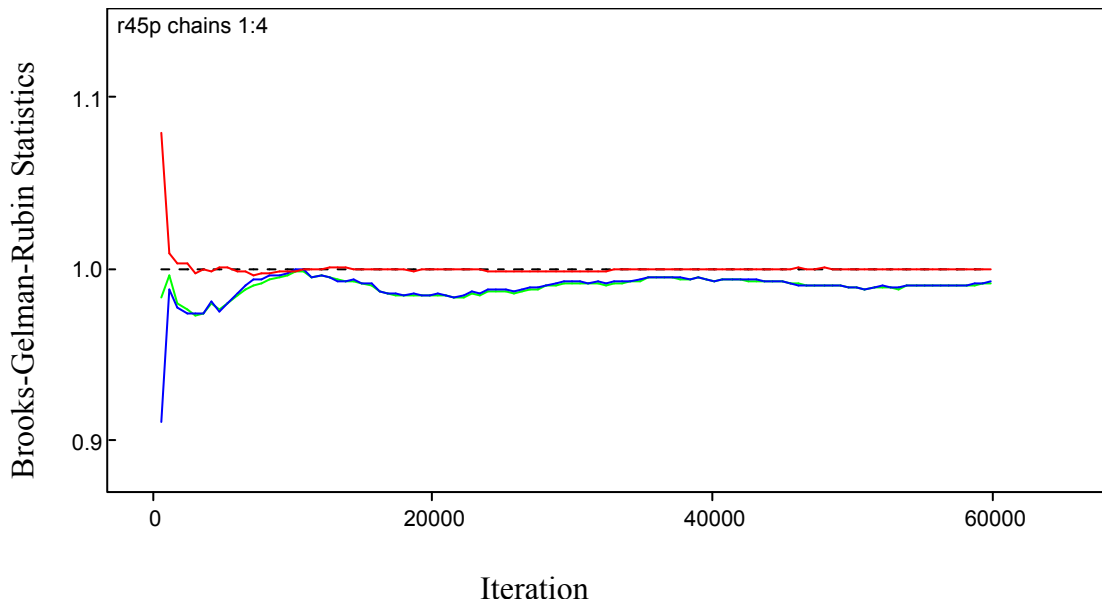


Figure 3: A Brooks-Gelman-Rubin plot [12] showing the ratio (upper trace) of the variation between and within (lower pair of traces) four parallel Markov chains that were simulated for this model. The fact that the ratio approaches 1 and the variation within and between chains levels off by 50 000th iteration indicates convergence.

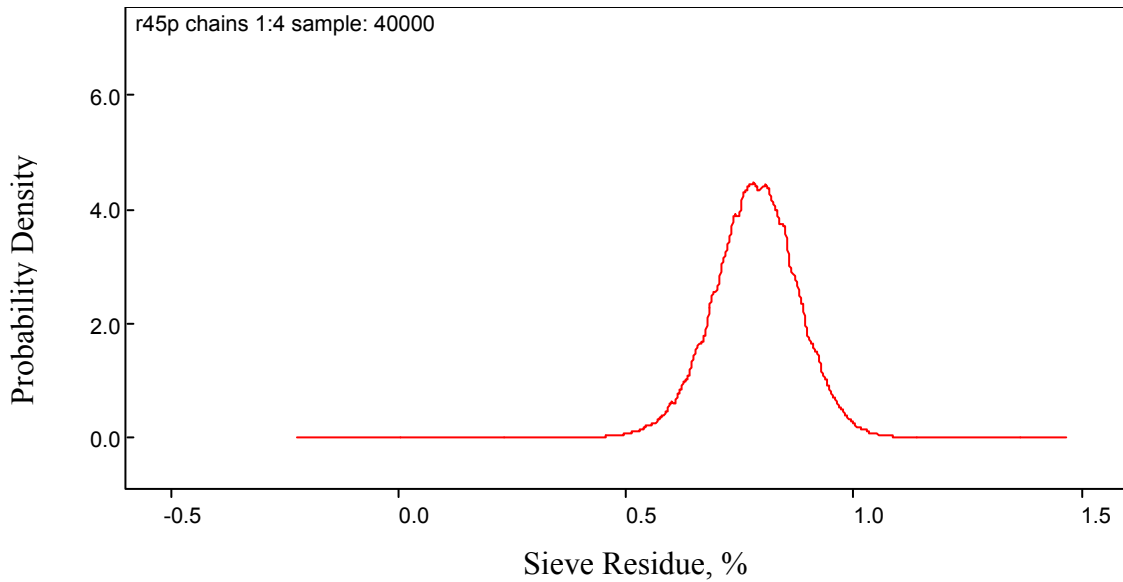


Figure 4: The posterior probability distribution of the sieve residue for a 45 µm sieve.

4.3.1 Cross check of the residue

Because the 114q material has such a different residue than previous generations of this material, it is important to verify that the correct results will be obtained when the certified value is used to calibrate a sieve as described in ASTM C430. The standard implies that the SRM 114q is sieved using a sieve with an unknown opening (although it should nominally be about 45 µm). The result is used with the certified value to calibrate the sieve and correct results of unknown cements.

To verify the performance of the 114q material, we did a double verification as shown in Table 11. The verification consisted of calculating the correction factor using one of the SRMs and testing the other SRM using this correction factor. For example in column 1 (Table 11) SRM 114p is used to calculate the correction factor that is then used to correct a measurement of SRM 114q. If everything is working as expected, the residue found for SRM 114q should match the value found in the certificate, which it does. Similarly, the results obtained for SRM 114p, when 114q is used to calibrate the sieve, do not disagree with the value from the 114p certificate, although the uncertainty in the corrected result for 114p is rather large.

Table 11: Residue Verification Using SRM's 114p and 114q

| Sieve Correction Factor Calculation | SRM 114p | SRM 114q |
|---|---------------------|-----------------------|
| Certified residue on 45 μm sieve [%] | 8.24 ± 0.37 | 0.79 ± 0.19 |
| Residue for 1 g sample [g] | 0.0824 ± 0.0037 | 0.0079 ± 0.0019 |
| Residue on sieve being calibrated [g] | 0.0683 ± 0.0068 | 0.0060 ± 0.0006 |
| Difference [g] | 0.0141 ± 0.0077 | 0.0019 ± 0.0020 |
| Correction factor [%] | 20.6442 ± 13.22 | 31.6667 ± 34.2949 |

| Cement Tested | SRM 114q | SRM 114p |
|---|---------------------|---------------------|
| Residue from sample being tested [g] | 0.0060 ± 0.0006 | 0.0683 ± 0.0068 |
| Corrected residue [%] | 0.7239 ± 0.1074 | 8.9928 ± 2.5090 |
| Certified residue on 45 μm sieve [%] | 0.79 ± 0.19 | 8.24 ± 0.37 |

Note: Uncertainties shown for each value are expanded uncertainties at the 95 % confidence level. A coverage factor of $k = 2$ was used in all cases.

5 Summary of Certified Values

The certified values for the SRM 114q certificate are:

| Measurement and ASTM Method | Result |
|--|--|
| Specific Surface Area C 204-96a (Blaine) | $3818 \text{ cm}^2/\text{g} \pm 78 \text{ cm}^2/\text{g}$ ($381.8 \text{ m}^2/\text{kg} \pm 7.8 \text{ m}^2/\text{kg}$) |
| Specific Surface Area C 115-96a (Wagner) | $2183 \text{ cm}^2/\text{g} \pm 160 \text{ cm}^2/\text{g}$ ($218 \text{ m}^2/\text{kg} \pm 16 \text{ m}^2/\text{kg}$) |
| Sieve Residue C 430-96 (45 μm sieve) | $0.79 \% \pm 0.19 \%$ |

6 Process Improvement

6.1 Packaging

As stated above, the cement received from the manufacturer was sealed in foil bags after being blended. It was the original intention that each foil bag would be open, divided in the vials and any left over would be discarded. This would ensure that the cements left in the foil bags overnight (or longer due to scheduling) would not hydrate in between vial filling. If hydration occurs the cement properties will change. As a further quality assurance, the temperature, relative humidity should be recorded. If a log is kept of the temperature, relative humidity, vials filled, it would result in a better correlation between homogeneity problems and specific vials.

During the filling of this batch of 600 000 vials, the procedure outlined above was followed but no log was kept of the temperature and relative humidity. Therefore, when it was discovered that vials, checked for homogeneity using other methods such as PSD, were found not satisfactory [13], it was not possible to rule out that the packaging conditions were one of the effects.

This situation did not affect the data presented here probably because none of the tests used are sensitive to complete dispersion, as it is the case for PSD measurements.

6.2 Testing

As shown in Table 3, one vial per box was used for the LOI testing. With this design, box-to-box and vial-to-vial differences in the LOI results cannot be differentiated. One minor improvement to the design for LOI testing in the future would be to sample two vials per box so that potential vial-to-vial variability could be separated from box-to-box variability in the LOI results. If necessary, half as many boxes could be sampled in order to keep the total number of measurements the same. Reducing the number of boxes sampled would reduce our ability to identify box-to-box differences, since some of the degrees of freedom would be used to check for vial-to-vial variability, but the added ability to separate vial-to-vial variability from box-to-box variability would give us the potential for additional insight into the process that would offset that loss.

Because of the small sieve residue for this cement, some digitization was evident in the sieve residue data. For future sieve residue assessments on cements with residues in this low range, use of a balance with a finer resolution would be advantageous.

7 Acknowledgments

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9 Appendices

Appendix A: Data received from the round robin for Blaine

| CCRL Lab Code | Raw data as received: Grey [m ² / kg]; white [s] | | | | | | | | Calculated Blaine (SRM 114p & data provided) | | | | | |
|---------------------|---|---------|---------|---------|---------|---------|--------|---------|--|--------|---------|---------|--------|---------|
| | SRM 114p | | Vial #1 | | | Vial #2 | | | Vial #1 | | | Vial #2 | | |
| | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 |
| 6 | 85.1 | | 19 | 89.9 | 89.46 | 19 | 89.01 | 88.35 | 19 | 3878 | 3869 | 19 | 3859 | 3845 |
| 10 | 104 | | 45 | 90.7 | 89.8 | 45 | 88.56 | 89.3 | 45 | 3525 | 3507 | 45 | 3483 | 3497 |
| 11 | 74 | | 89 | 80 | 78 | 89 | 79 | 79 | 89 | 3924 | 3875 | 89 | 3899 | 3899 |
| 18 | 85 | 87.5 | 30 | 71 | 66.5 | 30 | 64.5 | 67.5 | 30 | 3424 | 3314 | 30 | 3264 | 3339 |
| 19 | 118 | | 70 | 129.2 | 129.4 | 70 | 129.6 | 128.8 | 70 | 3949 | 3952 | 70 | 3955 | 3943 |
| 20 | 85.3 | | 114 | 91.6 | 90.01 | 114 | 91.29 | 92.6 | 114 | 3912 | 3877 | 114 | 3905 | 3933 |
| 27 | 152 | 149 | 107 | 157 | 157 | 107 | 158 | 157 | 107 | 3855 | 3855 | 107 | 3867 | 3855 |
| 28 | 3800 | | 81 | 3910 | 3910 | 81 | 3880 | 3900 | 81 | 3910 | 3910 | 81 | 3880 | 3900 |
| 40 | 109.6 | 108.7 | 109 | 121.1 | 119.9 | 109 | 115.5 | 117.9 | 109 | 3975 | 3956 | 109 | 3882 | 3922 |
| 41 | 75.7 | | 9 | 76.9 | 76.7 | 9 | 75.9 | 76 | 9 | 3804 | 3799 | 9 | 3779 | 3781 |
| 43 | 91.4 | | 55 | 95.2 | 95.1 | 55 | 95.7 | 95.8 | 55 | 3852 | 3850 | 55 | 3862 | 3864 |
| 54 | 3772 | 3770 | 89 | 3814 | 3748 | 89 | 3878 | 3882 | 89 | 3814 | 3748 | 89 | 3878 | 3882 |
| 56 | 3820 | 3840 | 50 | 3810 | 3770 | 50 | 3810 | 3790 | 50 | 3810 | 3770 | 50 | 3810 | 3790 |
| 60 | 114 | 114 | 80 | 121 | 120 | 80 | 119 | 120 | 80 | 3888 | 3872 | 80 | 3856 | 3872 |
| 69 | 77.4 | 76.8 | 40 | 78.8 | 79.1 | 40 | 78.6 | 78.2 | 40 | 3815 | 3823 | 40 | 3811 | 3801 |
| 73 | 80 | | 58 | 83 | 82 | 58 | 80 | 81 | 58 | 3844 | 3821 | 58 | 3774 | 3798 |
| 75 | 3810 | 3800 | 77 | 3695 | 3715 | 77 | 3697 | 3708 | 77 | 3695 | 3715 | 77 | 3697 | 3708 |
| 84 | 115.4 | | 120 | 116.555 | 115.7 | 120 | 122.1 | 121.4 | 120 | 3793 | 3779 | 120 | 3881 | 3870 |
| 92 | 68.8 | 66.56 | 118 | 71.35 | 68.0 | 118 | 70.17 | 69.5 | 118 | 3876 | 3784 | 118 | 3844 | 3827 |
| 95 | 77.5 | 77 | 80 | 85.5 | 85 | 80 | 85 | 85.5 | 80 | 3970 | 3959 | 80 | 3959 | 3970 |
| 105 | 85.4 | 85.9 | 11 | 86.7 | 86.9 | 11 | 85.7 | 86 | 11 | 3797 | 3801 | 11 | 3775 | 3782 |
| 121 | 81.4 | | 47 | 83.20 | 84.6 | 47 | 84.3 | 84.1 | 47 | 3816 | 3847 | 47 | 3840 | 3836 |
| 123 | 84 | | 88 | 105 | 80 | 88 | 83 | 83.5 | 88 | 4219 | 3683 | 88 | 3751 | 3763 |

| CCRL Lab Code | Raw data as received: Grey [m ² / kg]; white [s] | | | | | | | | Calculated Blaine (SRM 114p & data provided) | | | | | |
|---------------------|---|---------|---------|--------|---------|---------|--------|---------|--|--------|---------|---------|--------|---------|
| | SRM 114p | | Vial #1 | | | Vial #2 | | | Vial #1 | | | Vial #2 | | |
| | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 |
| 124 | 70 | 70.5 | 13 | 75 | 75.5 | 13 | 75.5 | 75.5 | 13 | 3900 | 3912 | 13 | 3912 | 3912 |
| 125 | 3752 | | 11 | 3861 | 3861 | 11 | 3796 | 3752 | 11 | 3861 | 3861 | 11 | 3796 | 3752 |
| 148 | 83.9 | 82.8 | 75 | 81.0 | 79.3 | 75 | 82.8 | 82.2 | 75 | 3720 | 3680 | 75 | 3761 | 3748 |
| 151 | 89.1 | 86.6 | 116 | 87.3 | 86.0 | 116 | 90.8 | 88.7 | 116 | 3762 | 3734 | 116 | 3837 | 3793 |
| 157 | 93 | | 58 | 95 | 94 | 58 | 94 | 93 | 58 | 3814 | 3794 | 58 | 3794 | 3774 |
| 162 | 3745 | 3756 | 14 | 3796 | 3806 | 14 | 3777 | 3788 | 14 | 3796 | 3806 | 14 | 3777 | 3788 |
| 166 | 81 | 81 | 86 | 90 | 90 | 86 | 90 | 90 | 86 | 3978 | 3978 | 86 | 3978 | 3978 |
| 167 | 3750 | 3730 | 98 | 3800 | 3800 | 98 | 3860 | 3850 | 98 | 3800 | 3800 | 98 | 3860 | 3850 |
| 175 | 106 | 110 | 30 | 110 | 111 | 30 | 109 | 111 | 30 | 3809 | 3827 | 30 | 3792 | 3827 |
| 178 | 77.5 | 77.4 | 3 | 67.4 | 69.5 | 3 | 73.6 | 70.3 | 3 | 3521 | 3575 | 3 | 3679 | 3596 |
| 205 | 90 | 89.8 | 50 | 91.9 | 90.9 | 50 | 90.9 | 91.5 | 50 | 3816 | 3795 | 50 | 3795 | 3807 |
| 209 | 85.3 | 84 | 66 | 90.7 | 89.9 | 113 | 88.4 | 91.0 | 66 | 3907 | 3889 | 113 | 3857 | 3913 |
| 222 | 87.7 | | 28 | 87.8 | 86.5 | 28 | 87.6 | 87.3 | 28 | 3776 | 3748 | 28 | 3772 | 3765 |
| 225 | 105 | 106 | 40 | 110 | 111 | 40 | 109 | 110 | 40 | 3854 | 3871 | 40 | 3836 | 3854 |
| 245 | 80 | | 19 | 73 | 72.5 | 19 | 73 | 74 | 19 | 3605 | 3593 | 19 | 3605 | 3630 |
| 246 | 71.5 | | 21 | 81 | 80.3 | 21 | 79.8 | 79.1 | 21 | 4017 | 4000 | 21 | 3987 | 3970 |
| 247 | 88 | | 43 | 93 | | 109 | 92 | | 43 | 3880 | 0 | 109 | 3859 | |
| 254 | 3765 | 3788 | 97 | 3854 | 3846 | 97 | 3807 | 3815 | 97 | 3854 | 3846 | 97 | 3807 | 3815 |
| 255 | 3730 | | 77 | 3800 | 3720 | 77 | 3800 | 3839 | 77 | 3800 | 3720 | 77 | 3800 | 3839 |
| 284 | 97.0 | 97.5 | 86 | 104.0 | 104.0 | 86 | 104.0 | 103.5 | 86 | 3903 | 3903 | 86 | 3903 | 3893 |
| 303 | 92.9 | 92.2 | 32 | 94.5 | 93.9 | 32 | 95.5 | 92.4 | 32 | 3814 | 3801 | 32 | 3834 | 3771 |
| 309 | 88 | 88 | 116 | 90 | 90 | 116 | 90 | 90 | 116 | 3817 | 3817 | 116 | 3817 | 3817 |
| 354 | 77.7 | 76.5 | 31 | 80.95 | 80.65 | 31 | 79.82 | 78.75 | 31 | 3867 | 3860 | 31 | 3840 | 3814 |
| 360 | 100 | 100 | 3 | 100 | 98 | 3 | 99 | 97 | 3 | 3774 | 3736 | 3 | 3755 | 3717 |
| 413 | Too variable to report | | | | | | | | | | | | | |
| 441 | 3760 | 3760 | 114 | 3740 | 3650 | 114 | 3710 | 3690 | 114 | 3740 | 3650 | 114 | 3710 | 3690 |
| 493 | 83 | 84 | 32 | 87 | 90 | 32 | 90 | 88 | 32 | 3852 | 3918 | 32 | 3918 | 3874 |
| 494 | 97 | 98 | 66 | 100 | 100 | 66 | 98 | 98 | 66 | 3822 | 3822 | 66 | 3784 | 3784 |

| CCRL Lab Code | Raw data as received: Grey [m ² / kg]; white [s] | | | | | | | | Calculated Blaine (SRM 114p & data provided) | | | | | |
|---------------------|---|---------|---------|--------|---------|---------|--------|---------|--|--------|---------|---------|--------|---------|
| | SRM 114p | | Vial #1 | | | Vial #2 | | | Vial #1 | | | Vial #2 | | |
| | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 | Box # | Meas#1 | Meas.#2 |
| 551 | 3751 | 3769 | 112 | 3892 | 3887 | 112 | 3926 | 3930 | 112 | 3892 | 3887 | 112 | 3926 | 3930 |
| 557 | 100 | 100 | 18 | 101 | 100 | 18 | 100 | 101 | 18 | 3793 | 3774 | 18 | 3774 | 3793 |
| 605 | 3816 | | 28 | 3879 | 3879 | 28 | 3859 | 3816 | 28 | 3879 | 3879 | 28 | 3859 | 3816 |
| 690 | 3774 | | 88 | 3801 | 3801 | 88 | 3828 | 3828 | 88 | 3801 | 3801 | 88 | 3828 | 3828 |
| 736 | 99.6 | 97.8 | 71 | 108.2 | 105.3 | 71 | 107.6 | 106.5 | 71 | 3951 | 3898 | 71 | 3939 | 3920 |
| 886 | 3705 | 3735 | 119 | 3832 | 3784 | 119 | 3624 | 3851 | 119 | 3832 | 3784 | 119 | 3624 | 3851 |
| 932 | 115.4 | | 19 | 121.4 | 122.9 | 75 | 123.2 | 123.8 | 19 | 3871 | 3895 | 75 | 3899 | 3909 |
| 1042 | 111.6 | 111.1 | 21 | 114.4 | 114.3 | 21 | 110.9 | 112.6 | 21 | 3824 | 3823 | 21 | 3766 | 3795 |
| 1054 | 83 | 85 | 14 | 93 | 93 | 14 | 90 | 90 | 14 | 3971 | 3971 | 14 | 3907 | 3907 |
| 1251 | 3765 | 3757 | 107 | 3891 | 3874 | 107 | 3883 | 3883 | 107 | 3891 | 3874 | 107 | 3883 | 3883 |
| 1715 | 72.9 | 72.3 | 13 | 74.8 | 75.1 | 13 | 75.6 | 75.5 | 13 | 3829 | 3837 | 13 | 3850 | 3848 |
| 1773 | 101.1 | 101.4 | 47 | 91.3 | 91.6 | 47 | 90.4 | 90.7 | 47 | 3584 | 3590 | 47 | 3566 | 3572 |
| 1819 | 82.59 | 83.00 | 9 | 86.4 | 87.1 | 9 | 86.1 | 86.5 | 9 | 3855 | 3870 | 9 | 3850 | 3858 |
| 1916 | 88.9 | 88.7 | 31 | 94.1 | 95.2 | 31 | 95.6 | 94.2 | 31 | 3884 | 3908 | 31 | 3916 | 3887 |
| 1940 | 3716 | 3753 | 18 | 3870 | 3901 | 18 | 3825 | 3922 | 18 | 3870 | 3901 | 18 | 3825 | 3922 |
| 2116 | 47.5 | 48.0 | 118 | 50.5 | 50.0 | 118 | 50.5 | 49.5 | 118 | 3881 | 3862 | 118 | 3881 | 3843 |
| 2190 | 85.3 | 86.0 | 111 | 86.3 | 85.8 | 111 | 87.5 | 86.3 | 111 | 3789 | 3778 | 111 | 3815 | 3788 |
| 2191 | 75.4 | 74.3 | 111 | 73.6 | 77.4 | 111 | 79.7 | 81.4 | 111 | 3744 | 3837 | 111 | 3896 | 3937 |
| NA | 65.2 | 64.8 | 70 | 66.7 | 67.0 | 70 | 66.5 | 66.5 | 70 | 3824 | 3832 | 70 | 3816 | 3818 |

Appendix B: Data received from the round robin for Wagner

| CCRL code | 114p | | Box A | | | Box B | | |
|--------------|----------|----------|-------|----------|----------|-------|----------|----------|
| | Meas. #1 | Meas. #2 | Box # | Meas. #1 | Meas. #2 | Box # | Meas. #1 | Meas. #2 |
| 73 | 2049 | | 9 | 1984 | 1986 | 9 | 1997 | 2040 |
| 125 | 2130 | | 9 | 2330 | 2290 | 9 | 2310 | 2300 |
| 205 | 2070 | | 119 | 2380 | 2380 | 119 | 2380 | 2350 |
| 247 | | | 43 | 2280 | 2240 | 109 | 2260 | 2280 |
| 254 | 2060 | 2085 | 43 | 2046 | 2020 | 43 | 2117 | 2043 |
| 494 | 2088 | 2078 | 66 | 2113 | 2126 | 66 | 2135 | 2079 |
| 551 | 2045 | 2067 | 113 | 2227 | 2262 | 113 | 2272 | 2232 |

Appendix C: Data obtained for Residue

| Day | Box | Vial | Residue | | |
|-----|-----|------|----------|----------|----------|
| | | | Sieve 1 | Sieve 2 | Sieve 3 |
| 1 | 118 | 1 | 0.0010 g | 0.0035 g | 0.0015 g |
| 1 | 35 | 1 | 0.0150 g | 0.0060 g | 0.0030 g |
| 1 | 118 | 1 | 0.0160 g | 0.0050 g | 0.0010 g |
| 1 | 35 | 2 | 0.0160 g | 0.0050 g | 0.0020 g |
| 1 | 35 | 2 | 0.0155 g | 0.0060 g | 0.0010 g |
| 1 | 35 | 1 | 0.0150 g | 0.0055 g | 0.0030 g |
| 1 | 118 | 2 | 0.0170 g | 0.0050 g | 0.0025 g |
| 1 | 118 | 2 | 0.0150 g | 0.0060 g | 0.0025 g |
| 2 | 47 | 2 | 0.0160 g | 0.0060 g | 0.0025 g |
| 2 | 27 | 1 | 0.0180 g | 0.0065 g | 0.0010 g |
| 2 | 27 | 1 | 0.0180 g | 0.0060 g | 0.0020 g |
| 2 | 47 | 2 | 0.0160 g | 0.0060 g | 0.0025 g |
| 2 | 27 | 2 | 0.0185 g | 0.0060 g | 0.0020 g |
| 2 | 47 | 1 | 0.0175 g | 0.0060 g | 0.0030 g |
| 2 | 27 | 2 | 0.0160 g | 0.0055 g | 0.0025 g |
| 2 | 47 | 1 | 0.0170 g | 0.0065 g | 0.0020 g |
| 3 | 53 | 2 | 0.0150 g | 0.0060 g | 0.0020 g |
| 3 | 98 | 1 | 0.0165 g | 0.0065 g | 0.0025 g |
| 3 | 53 | 2 | 0.0160 g | 0.0065 g | 0.0025 g |
| 3 | 98 | 2 | 0.0170 g | 0.0075 g | 0.0025 g |
| 3 | 98 | 2 | 0.0155 g | 0.0060 g | 0.0020 g |
| 3 | 98 | 1 | 0.0150 g | 0.0060 g | 0.0025 g |
| 3 | 53 | 1 | 0.0170 g | 0.0070 g | 0.0025 g |
| 3 | 53 | 1 | 0.0150 g | 0.0060 g | 0.0015 g |
| 4 | 76 | 2 | 0.0135 g | 0.0055 g | 0.0025 g |
| 4 | 72 | 2 | 0.0165 g | 0.0060 g | 0.0025 g |
| 4 | 76 | 1 | 0.0170 g | 0.0065 g | 0.0025 g |
| 4 | 76 | 2 | 0.0150 g | 0.0065 g | 0.0025 g |
| 4 | 76 | 1 | 0.0135 g | 0.0060 g | 0.0020 g |
| 4 | 72 | 1 | 0.0195 g | 0.0070 g | 0.0025 g |
| 4 | 72 | 1 | 0.0175 g | 0.0065 g | 0.0025 g |
| 4 | 72 | 2 | 0.0145 g | 0.0065 g | 0.0025 g |
| 5 | 56 | 2 | 0.0160 g | 0.0055 g | 0.0025 g |
| 5 | 56 | 2 | 0.0160 g | 0.0060 g | 0.0020 g |
| 5 | 9 | 1 | 0.0180 g | 0.0065 g | 0.0030 g |
| 5 | 56 | 1 | 0.0170 g | 0.0070 g | 0.0025 g |
| 5 | 9 | 2 | 0.0155 g | 0.0065 g | 0.0025 g |
| 5 | 56 | 1 | 0.0165 g | 0.0060 g | 0.0025 g |
| 5 | 9 | 1 | 0.0180 g | 0.0065 g | 0.0025 g |
| 5 | 9 | 2 | 0.0165 g | 0.0065 g | 0.0025 g |

Note: Box 118 vial 1 - slightly different color than that of box 118 vial 2 and both vials 1 and 2 from box 35

10 Certificate of SRM 114q

Below the certificate of SRM 114q is provided for information only. As, the certificate will be updated later in 2005 to add the particle size distribution of the cement [13], the official version is the available online at: https://srmors.nist.gov/certificates/view_cert2gif.cfm?certificate=114q

Or by selecting “Standard Reference Materials” from the website www.NIST.GOV and typing in 114q



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 114q

Portland Cement Fineness Standard

This Standard Reference Material (SRM) is intended for use in calibrating fineness testing equipment according to ASTM Standard Methods. The SRM unit consists of 20 glass vials with plastic caps containing powdered cement (each vial is contained in a sealed foil bag). Each vial contains approximately 5 g of cement.

Certified Values and Uncertainties: A NIST certified value is a value for which NIST has the highest confidence in its accuracy and that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for specific surface area and sieve residue are given in Table 1. The certified values for the surface area are the mean of results from analyses performed by cooperating laboratories. The certified value for sieve residue was calculated from a quadratic fit of NIST data using three sieves having openings ranging from 38 μm to 56 μm .

The expanded uncertainties of the certified values for specific surface area were calculated according to the NIST uncertainty policy described in the NIST Technical Note 1297 [1], and are at the 95 % confidence level. The uncertainties include measurement variability within and between laboratories. The surface area uncertainties also include material variability and the uncertainty of the surface area values for the superseded SRM 114p *Portland Cement Fineness Standard*, which was used as the calibrant for this material. The expanded uncertainty for the sieve residue was computed using a Bayesian analysis and is also at the 95 % probability level. The expanded uncertainty accounts for the variability of random measurement effects, sieve calibrations, and material inhomogeneity.

Table 1. Certified Values

| Measurand | ASTM Method | Certified Value and Expanded Uncertainty |
|--|--------------------------|--|
| Specific Surface Area (Blaine) | C 204-96a ^(a) | 3818 cm ² /g \pm 78 cm ² /g (381.8 m ² /kg \pm 7.8 m ² /kg) |
| Specific Surface Area (Wagner) | C 115-96a ^(b) | 2183 cm ² /g \pm 160 cm ² /g (218 m ² /kg \pm 16 m ² /kg) |
| Sieve Residue (45 μm residue) | C 430-96 ^(c) | 0.79 % \pm 0.19 % |

^(a) Standard Test Method for Fineness of Portland Cement by Air Permeability Apparatus [Blaine].

^(b) Standard Test Method for Fineness of Portland Cement by the Turbidimeter [Wagner].

^(c) Standard Test Method for Fineness of Hydraulic Cement by the 45 μm (No. 325) Sieve.

Expiration of Certification: The certification of SRM 114q is valid, within the measurement uncertainties specified, until **31 December 2016**, provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is contaminated or otherwise modified.

Maintenance of Certification: NIST will monitor representative samples from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration date, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

James St. Pierre, Chief
Materials and Construction Research Division

Gaithersburg, MD 20899
Certificate Issue Date: 24 March 2005

Robert L. Matters, Jr., Chief
Measurement Services Division

See Certificate Revision History on Last Page

The preparation of the material and the coordination of the technical measurements leading to certification were performed by C. Ferraris of the NIST Materials and Construction Research Division.

Statistical consultation on measurement design and analysis of the certification data was performed by W.F. Guthrie and A.I. Avilés of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

Stability and Use: This material is considered to be extremely hygroscopic. Based on measurements in 1993 of several earlier renewals of SRM 114, the properties certified are stable as long as the foil bag remains sealed. The specific surface area of cement changes on exposure to the moisture in air. Therefore, this cement should be used immediately after opening the outer foil bag.

Allow the sealed foil bag to equilibrate to testing temperature before opening. To open the pouch, cut off the end with scissors. Fluff the cement in accordance with ASTM Standard C204, Section 3.4, allow the cement to settle for 2 minutes, and then perform the measurement.

Material Selection and Packaging: The desired properties were determined to be generally the same as those selected for the previous issues of SRM 114; however, in order to better represent current cements, the material selected for SRM 114q consists of a finer particle size distribution than previously issued. The Cement and Concrete Reference Laboratory (CCRL) and NIST identified a plant with suitable cement: Lehigh Cement Company (Union Bridge, Maryland)⁴ donated 1300 kg of appropriate cement for this SRM. The material selected was Type I according to the ASTM C 150 Standard Classification and had a mass fraction of less than 8 % tricalcium aluminate (C₃A). The material was collected for shipment to NIST directly from the finish mill process stream into bags. Upon arrival at NIST, the cement was blended in a V-blender (1.68 m³) and then transferred to 208 L (55 gallon) drums lined with 0.015 cm (6 mil) polyethylene liners to minimize hydration of the cement in storage prior to preparation and packaging. Over the next two days, the cement from each drum was sealed in foil bags, each containing about 16 kg of cement. The foil bags were stored in a climate-controlled area. The contents of each bag were subsequently packaged into vials. The vials were then capped and packaged in boxes of about 500 vials per box. The boxes were sequentially labeled from 1 to 118. About 5 boxes were filled per day. Nearly 59 000 glass vials, each containing approximately 5 g of cement, were produced. Each vial was then individually sealed in a foil bag. Vials were selected from the lot by stratified random sampling [2] for both homogeneity and certification analyses. Selected vials were shipped to the participating laboratories for measurements. The remaining vials were packaged into SRM unit boxes of 20 vials each.

Homogeneity Assessment and Certification Analyses: Homogeneity testing of the material was performed on 48 random-selected samples. Measurements of the loss of ignition (LoI) showed no reversible moisture take-up by the cement during packaging. The data received from the round-robin participants were also checked for laboratory-to-laboratory (or day-to-day in the case of sieve residue) variability, box-to-box variability, and vial-to-vial variability. No significant box-to-box or vial-to-vial variability was detected except for the Wagner or sieve residue tests, and therefore it was determined that the samples were homogeneous for the ASTM measurements. Significant vial-to-vial variability was observed using the Blaine test and the certified values reflect this source of uncertainty.

Certification analyses for specific surface areas using ASTM Standard Test Methods C 115-96a and C 204-00 were performed on two samples at each of the participating laboratories. SRM 114p *Portland Cement Fineness Standard* was used for calibration. Raw data were submitted by each laboratory to NIST for tabulation and calculation of surface areas, which for the Blaine test, assumed a density of 3.15 g/cm³. The density was measured twice at NIST: the results were 3.255 g/cm³ and 3.248 g/cm³.

Certification analyses according to ASTM Standard Test Method C 430-96 for the 45 μm sieve residue were performed at NIST on 40 samples from 20 vials of cement.

Laboratories performing certification analyses are members of the CCRL (<http://www.ccril.us>) proficiency program. The full list is provided in the report describing the details of the certification process [2].

⁴Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Information Values: NIST information values are considered to be of interest to the SRM user, but are not certified because insufficient information is available to assess adequately the uncertainty associated with the values or only a limited number of analyses were performed. Information values for SRM 114q are given in Tables 2 and 3. Table 2 provides the approximate chemical composition determined by ASTM Standard Test Method C114-02. The analysis of this cement (CCRL Portland Cement Proficiency Sample No. 150) was performed by 70 to 170 laboratories; the number of participating laboratories depends on the value measured.

Table 2. Information Values for Chemical Composition

| Compound | Mass Fraction (%) | Compound | Mass Fraction (%) |
|--------------------------------|-------------------|-------------------------------|-------------------|
| CaO | 64.0 | K ₂ O | 0.70 |
| SiO ₂ | 20.7 | TiO ₂ | 0.30 |
| Al ₂ O ₃ | 4.7 | P ₂ O ₅ | 0.12 |
| Fe ₂ O ₃ | 3.2 | Na ₂ O | 0.07 |
| SO ₃ | 2.4 | MgO | 2.2 |
| Loss on Ignition | 1.67 | | |

Table 3 provides the calculation of cement compounds according to ASTM C 150-02.

Table 3. Information Values for Cement Compounds (Calculation from Table 2)

| Compound | Mass Fraction (%) |
|--|-------------------|
| C ₃ S (tricalcium silicate) | 60 |
| C ₂ S (dicalcium silicate) | 14 |
| C ₃ A (tricalcium aluminate) | 7 |
| C ₄ A _F (tetracalcium alumino-ferrite) | 10 |

REFERENCES

- [1] Taylor, B.N.; Rugatt, C.F.; NIST Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results* (1993).
- [2] Ferraris, C.F.; Abilés A.J.; Guthrie M.; Haupt, R.; *Certification of SRM 114q, Phase I, NIST SP260-161* (2005).

Certificate Revision History: 24 March 2005 (This technical revision corrects the certified values and expanded uncertainties for the measurand); 23 March 2005 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.

