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Certification of Standard Reference Material® 114r
Portland Cement Fineness

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Abstract

The SRM 114 series for portland cement fineness has been provided by NIST since 1934 for use in calibrating instruments for measuring fineness characteristics of portland cement and other powdered construction materials. SRM 114r is the latest of this series providing certified values for cement fineness by Blaine apparatus, + 45 μm (No. 325) Sieve, and informational values for particle size distribution by light scattering. ASTM C01.25 Committee on Fineness provided a range of desired materials properties, and the assistance of the Cement and Concrete Reference Lab provided several candidate plants with materials conforming to the Committee request. Approximately 1000 kg of cement was procured, homogenized, and packaged. An inter-laboratory study was initiated to develop the data set used to establish certified values. The certified values represent the consensus means and uncertainties for the Blaine and + 45 μm residue. Informational values on particle size distribution are provided as the mean of the combined wet and dry laser diffraction data.

Key words

Blaine Fineness, Cement, Particle Size Distribution, Specification, SRM.

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1. Introduction

The SRM 114 series for portland cement fineness has been provided by NIST since 1934 for calibrating instruments for measuring fineness characteristics of portland cement and other powdered construction materials [1]. Certified values are provided for cement fineness by ASTM C204 [2] Standard Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus (commonly called the Blaine method), ASTM C430, Standard Test Method for Fineness of Hydraulic Cement by the 45-um (No. 325) Sieve [3], and informational values for particle size distribution by light scattering following AASHTO T 353-14 [4].

The Blaine apparatus measures total surface area relative to the SRM by drawing a fixed quantity of air through a bed of cement having a specific porosity where the pore volume and size is related to the particle size of the cement. The Blaine fineness is used as a material test requirement or an optional requested material test. ASTM C430 measures the material residue greater than 45 μm using a No. 325 mesh sieve (45 μm opening). SRM 114 is used to calibrate the sieve by comparing the amount retained on the lab sieve to the certified value and is also part of a material specification or an optional reported material property. The particle size distribution is a newer measure of cement fineness and is standardized in the AASHTO T 353 test method [4].

2. Characteristic of a SRM Candidate

A poll was conducted with members of ASTM C01.25 on Cement Fineness on the materials characteristics for the new 114 cement. The Committee consensus was for a Blaine fineness around 400 m^2/kg and +45 μm residue between 4 % and 6 %. Reviewing the CCRL proficiency test data (CCRL.US) shows that a small number of cements have +the desired characteristics as modern grinding systems are more effective at producing a finer, more uniform particle size. The larger retention is helpful to calibrate laboratory #325 sieves for analysis of cement and other materials used with cements, such as fly ash. Low retention values make sieve calibration more difficult.

2.1. Materials

Once prospective candidate material sources were identified, cement manufacturers were contacted to confirm current production characteristics (Blaine, +45 μm) and a selection was made to procure about 1100 kg of material. The cement was packed in plastic barrels lined with heavy plastic bags, sealed and shipped to the Cement and Concrete Reference Laboratory in Frederick Maryland for homogenization using their large V-blender, capable of mixing the entire 1100 kg in one run. The homogenized material was re-packaged in the barrels and staff from the Standard Reference Materials Program (SRMP) picked up the barrels for delivery to NIST in Gaithersburg for packaging. Packaging in vials of about 5g each, with 20 vials per unit to remain consistent with past SRM 114 production and material use needs. Vials were stored in boxes of 200 as packaging proceeded.

2.1.1. Density

Cement density (Table 1) was measured using helium pynchnometry [5]. This method was demonstrated to provide density measurements for SRM 114q consistent with the Le Chatelier method used in ASTM C188 [5]. For some of the CCRL cements tested, He

Pyncnometry provided values slightly greater than the Le Chatelier method, likely due to the more complete permeation of the helium into fine fractures and pores within the cement particles. These density values are provided as informational values. Section 13.2 of [3] requires a density difference of no greater than 0.06 g/cm^3

Table 1. Density Measurements for 114r and SRM 46h using helium pynchnometry

Trial		114r	46h
	1	3.1422	3.2118
	2	3.1474	3.1971
	3	3.1435	3.1975
	4	3.1572	
Mean		3.1476	3.2021
σ		0.0068	0.0084

2.2. Inter-Laboratory Study

Participants were recruited through ASTM Committee C01.25, Fineness, and membership of the Portland Cement Association to provide Blaine (ASTM C204) and particle size distribution data (AASHTO T353) from samples of SRM 46h and the candidate SRM 114. This practice of using an inter-laboratory study to establish the data set from which the consensus values provide certified and reference values is consistent with previous editions of the SRM 114 series. The tests and their significance will be briefly described, and references provided which provide greater detail on the procedures and application of the SRM for their calibration procedures.

2.3. Sample Randomization

Once the list of participants and tests were established a randomized assignment of samples, drawn from storage trays was established and recorded. The Participants and Test Methods (Blaine, Laser PSD, +45 μm residue) were matched to the list as the participant list developed. For each test procedure results could be evaluated by lab and by box, representing the packing sequence or by testing sequence to assess for trends.

2.3.1. Blaine Fineness, ASTM C204

The Blaine fineness test used in ASTM C204 is based upon the relationship between the air permeability of a packed bed of cement powder and its fineness and, consequently, its surface area. Cement fineness influences the rheological and strength gain properties and are material specification criteria in ASTM C150 [6] and an optional reporting element in Specifications C595 [7] and C1157 [8]. The current cement fineness SRM 46h served to calibrate the Blaine apparatus so the new SRM 114 measurements would be referenced against a recent measurement of a current fineness SRM. This approach ensured each lab was consistent in their calibrations if the test procedure was properly followed. Section 13.2 of [3] requires a Blaine fineness difference of no greater than 200 m^2/kg.

2.3.2. Particle Size Distribution by Laser Diffraction, AASHTO T353

A particle size distribution measurement provides a more complete measurement of a cement, but the test results are not yet part of any cement specifications. AASHTO T353 involves the suspension of cement particles in either air or isopropanol, measuring the scattering of laser light to calculate a particle size distribution. Each participant was instructed to follow the AASHTO procedure and record their results in a spreadsheet provided with the samples. The spread sheet compiled the laser diffraction data into bins consistent with previous SRM 114 samples. Data from SRM 46h were also collected to see if a lab's test protocol produced results consistent with the SRM values and may serve as a means of identifying outliers.

2.3.3. Percent Retained on #325 Mesh Screen, ASTM C430

The residue of cement on a calibrated #325 (45-µm) sieve is a specification property for cement and supplementary cementitious materials. Since a direct certification of sieve openings is impractical for production-scale work, sieves are calibrated by using a certified reference material, SRM 114. A sieve correction factor is calculated after measuring SRM 114 on the selected sieve and calculation of a correction factor.

Three sieves with nominal openings of 38 µm, 45 µm, and 54 µm were directly calibrated for use in reference material certification. Interpolation was then used to obtain the value at 45 µm. These sieves consist of three cylindrical bodies about 50 mm in diameter with an individually calibrated screen held in the base. The test involves wet-sieving a 1.000 g sample of cement through each of the three sieves and measurement of the mass of the residue on each sieve.

The +45-um data were measured at NIST using a set of three cement wet sieves with calibrated screens following ASTM E11-20 [9] with the certified mean opening value and uncertainty listed in Table 2. The procedure for sieving is provided in Section 2.3.4 below, with results presented graphically in Figures 1 and 2 and teat results provided in Appendix A.

Table 2. Cement Sieve Hole and Wire Diameter Size and Uncertainties following ASTM 11 [2].

No. 400	Diameter	Tolerance	Warp Diameter	Shoot Diameter	Uncertainty
Hole	0.038 mm	0.003 mm	0.03834 mm	0.03812 mm	0.00059 mm
Wire	0.030 mm	0.0045 mm	0.2898 mm	0.02759 mm	0.00059 mm
No. 325	Diameter	Tolerance	Warp Diameter	Shoot Diameter	Uncertainty
Hole	0.045 mm	0.003 mm	0.04496 mm	0.04347 mm	0.00059 mm
Wire	0.032 mm	0.0048 mm	0.03198 mm	0.03153 mm	0.00059 mm

No. 270	Diameter	Tolerance	Warp Diameter	Shoot Diameter	Uncertainty
Hole	0.053 mm	0.004 mm	0.05148 mm	0.05412 mm	0.00059 mm
Wire	0.036 mm	0.0054 mm	0.03650 mm	0.03447 mm	0.00059 mm

2.3.4. Cement Wet Sieving Procedure

Materials & Tools:

1) Cylindrical sieves made of chromium-plated brass
2) Type 304 stainless steel woven-type cloth mounted to the sieve base without distortion or wrinkling
3) ASTM C430-compliant spray nozzle and pressure gauge
4) Low-temperature oven (105 °C) for drying; leather gloves for heat protection when removing sieve from oven
5) Nitrile or latex gloves for hand protection while sieving
6) Safety glasses with side shields for eye protection

Method:
1) Place a 1.000 g sample of the cement in the clean, dry sieve
2) Wet the sample thoroughly with a gentle stream of water
3) Wash for 1.0 min, moving the sieve with a circular motion in a horizontal plane at the rate of one motion per second in the spray. The bottom of the spray nozzle should extend below the top of the sieve frame about 12 mm (0.5 in.)
4) Immediately upon removing the sieve from the spray, rinse once with about 50 cm^3 of distilled water, using caution not to lose any of the residue
5) Displace the water using about 25 ml of ethanol
6) Dry the sieve and residue in an oven or over a hot plate supporting the sieve in such a manner that air may pass freely beneath it
7) Cool the sieve; then carefully brush the residue from the sieve and weigh on an analytical balance capable of reproducing results within 0.0005 g. Alternatively, weigh the sieve and residue after drying, subtract the mass of the sieve to calculate residue mass
8) *Acceptable Cleaning Procedures*— Clean sieve using a stream of water in the opposite direction followed by a rinse using ethanol to displace the water and dry in low-temperature oven.

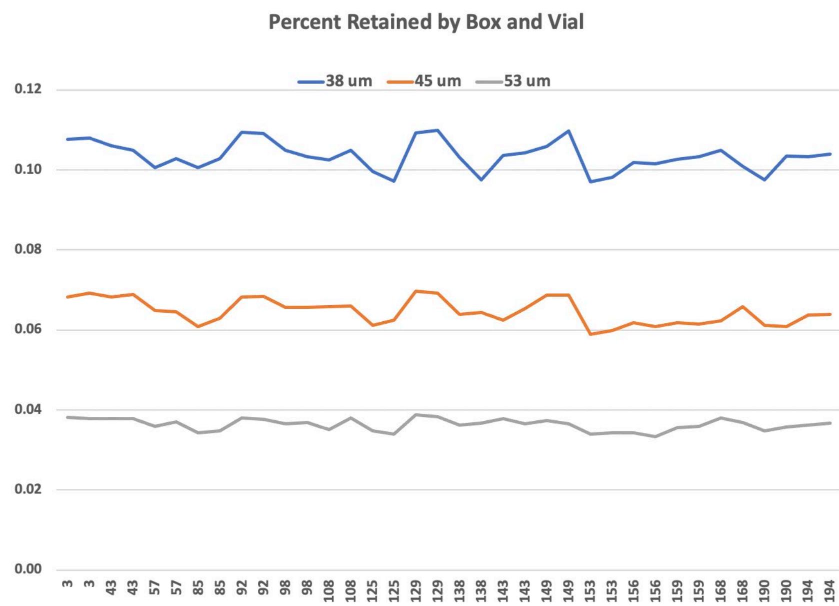

Figure 1. Percent retained on 38 um, 45 um, and 53 um micron sieves do not appear to show any trend based upon sample packing sequence.

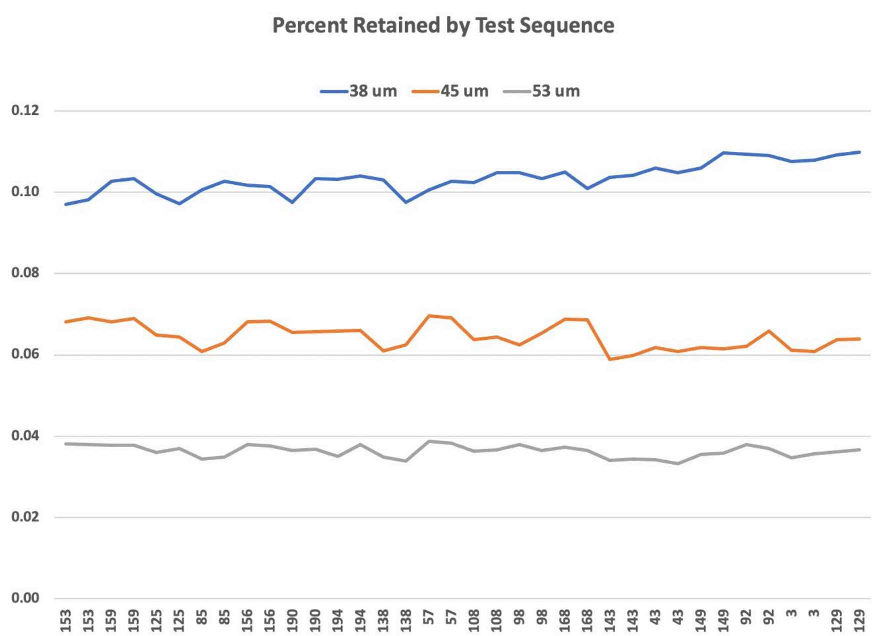

Figure 2. The 38 μm sieve shows an increase in percent retained as testing progressed resulting from trapped particles removed upon. This trend was not found for the 45 μm and 53 μm sieves.

To determine the mass fraction of cement that would be retained by a 45 µm sieve, determinations of sieve residue were obtained using calibrated sieves of three sizes, 38.0 µm, 45.0 µm, and 53.0 µm. The measurement data based on two vials of 36 different boxes is given in Table 3.

Table 3. Sieve residue measurements for the three calibrated sieves

Box	Vial	Residue for 38 µm sieve	Residue for 45 µm sieve	Residue for 53 µm sieve	Sequence position
153	1	9.699	5.89	3.397	1
153	2	9.812	5.979	3.43	2
159	1	10.268	6.178	3.55	3
159	2	10.324	6.142	3.586	4
125	1	9.951	6.106	3.479	5
125	2	9.707	6.236	3.387	6
85	1	10.054	6.077	3.43	7
85	2	10.278	6.289	3.479	8
156	1	10.171	6.177	3.422	9
156	2	10.14	6.08	3.33	10
190	1	9.747	6.106	3.475	11
190	2	10.339	6.077	3.571	12
194	1	10.317	6.369	3.616	13
194	2	10.387	6.392	3.661	14
138	1	10.306	6.38	3.625	15
138	2	9.74	6.437	3.668	16
57	1	10.051	6.481	3.592	17
57	2	10.264	6.447	3.693	18
108	1	10.236	6.578	3.509	19
108	2	10.473	6.595	3.788	20
98	1	10.483	6.555	3.644	21
98	2	10.329	6.565	3.678	22
168	1	10.483	NA	3.795	23
168	2	10.091	6.581	3.69	24
143	1	10.354	NA	3.787	25
143	2	10.417	6.53	3.648	26
43	1	10.581	6.818	3.774	27
43	2	10.486	6.885	3.78	28
149	1	10.582	6.872	3.727	29
149	2	10.97	6.866	3.645	30
92	1	10.926	6.813	3.789	31
92	2	10.897	6.83	3.762	32
3	1	10.764	6.816	3.81	33
3	2	10.789	6.912	3.784	34

129	1	10.911	6.961	3.877	35
129	2	10.98	6.906	3.824	36

The sieve sizes were measured with uncertainty of 0.59 µm. Also, it is known that as a sieve is being used over time, small amount of the residue is retained, thus increasing the residue measurements over time. The data in Table 3 is arranged in time sequence. The following statistical model can capture the effects of sieve size on residue in terms of a quadratic function, also the effects of sequence and the uncertainty in the sieve size.

The measurements are $y38_i$, $y45_i$, and $y53_i$.
$y38_i \sim N(\mu38_i, \sigma38), i = 1, \dots, 36,$
$y45_i \sim N(\mu45_i, \sigma45), i = 1, \dots, 34,$
$y53_i \sim N(\mu53_i, \sigma53), i = 1, \dots, 36.$

The means for the i[th] set of measurements were modeled using a quadratic equation in terms of the sieve size, and the added residue due to sequence, as

$$\mu38_i = \beta_1 + \beta_2\,(\mu.s38 - \overline{\mu.s}) + \beta_3(\mu.s38 - \overline{\mu.s})^2 + \beta_4(i-1), i = 1, \dots, 36,$$
$$\mu45_i = \beta_1 + \beta_2\,(\mu.s45 - \overline{\mu.s}) + \beta_3(\mu.s45 - \overline{\mu.s})^2 + \beta_5(i-1), i = 1, \dots, 34,$$
$$\mu53_i = \beta_1 + \beta_2\,(\mu.s53 - \overline{\mu.s}) + \beta_3(\mu.s53 - \overline{\mu.s})^2 + \beta_6(i-1), i = 1, \dots, 36.$$

The sieve sizes were measured with uncertainty and were modeled as:

$\mu.s38 \sim N(38.0, 0.59^2),$
$\mu.s45 \sim N(45.0, 0.59^2),$
$\mu.s53 \sim N(53.0, 0.59^2),$
$\overline{\mu.s} = \frac{1}{3}(\mu.s38 + \mu.s45 + \mu.s53)$.

This statistical model was fitted using Bayesian MCMC (code is in the Appendix) to obtain parameter estimates of the regression coefficients with uncertainties. Prior distributions were as non-informative as possible, using half Cauchy for the variances $\sigma38^2, \sigma45^2$, and $\sigma53^2$, and uniform distributions for the regression parameters. The results for the parameter estimates, which are given in Table 4, are not sensitive to the prior distribution assumptions.

Table 4. Parameter estimates with uncertainty.

parameter	Posterior mean	Posterior std	Lower 95%	median	Upper 95 %
β_1	5.774	0.341	5.056	5.782	6.433
β_2	-0.443	0.033	-0.519	-0.440	-0.388
β_3	0.017	0.008	0.004	0.017	0.035
β_4	0.028	0.003	0.022	0.028	0.035
β_5	0.031	0.002	0.027	0.031	0.034
β_6	0.012	0.001	0.009	0.012	0.015
$\sigma 38$	0.213	0.026	0.169	0.211	0.272
$\sigma 45$	0.097	0.013	0.076	0.096	0.126
$\sigma 53$	0.080	0.010	0.063	0.079	0.104
$\mu.s38$	38.04	0.57	36.92	38.04	39.14
$\mu.s45$	45.02	0.53	43.94	45.04	45.95
$\mu.s53$	52.94	0.59	51.74	52.94	54.10

The fit of the statistical model was examined using prediction intervals and residuals. The following three plots (Figures 3,4,5) show the measured data values together with the 95% prediction intervals for the three sieves. Except for one or two points the data is included in the prediction intervals.

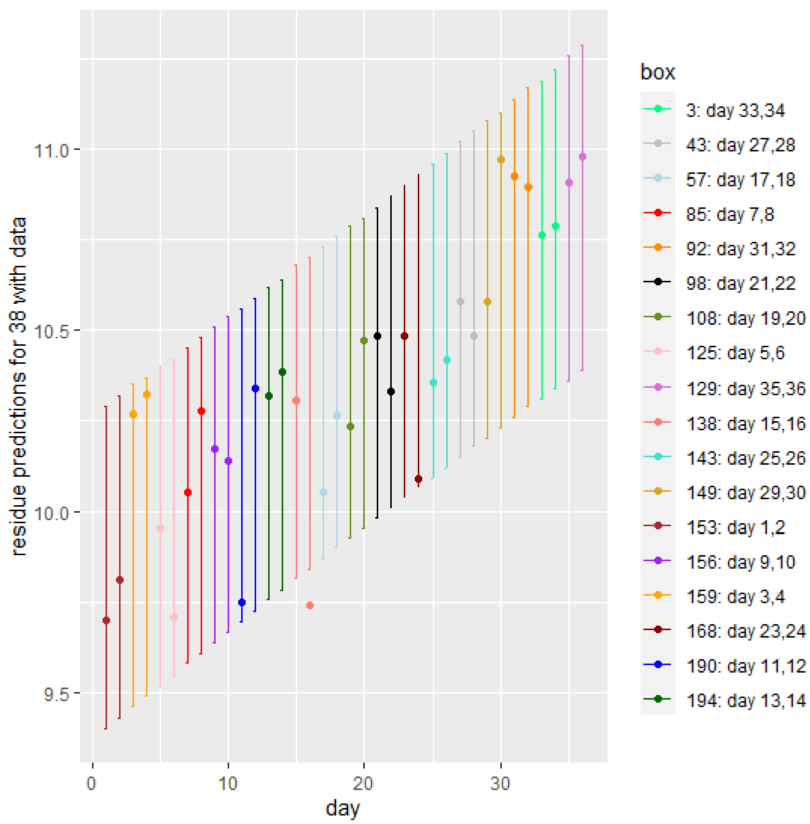

Figure 3. 95% prediction intervals with observed data for the 38 µm sieve.

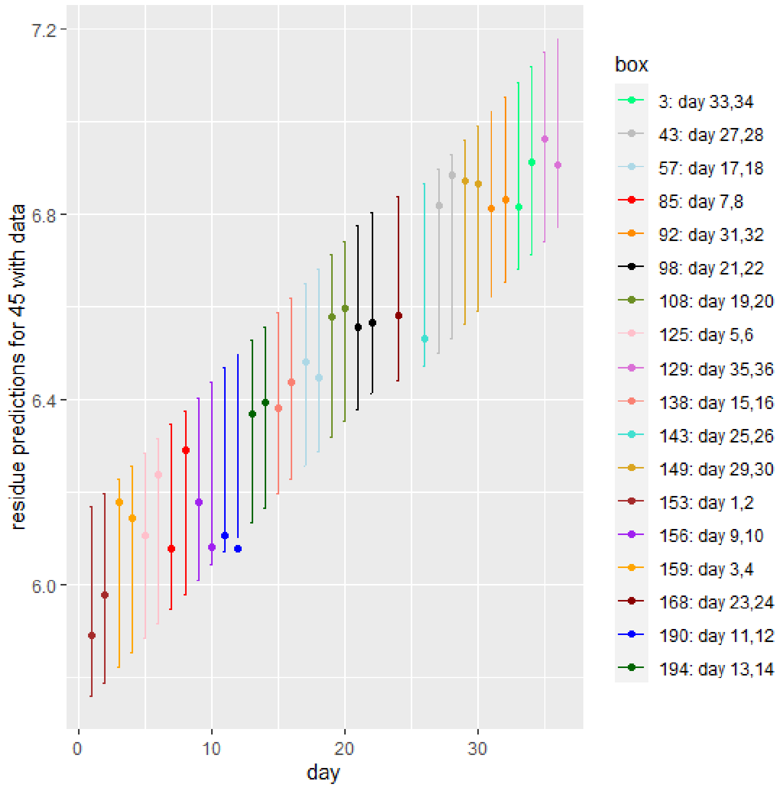

Figure 4. 95% prediction intervals with observed data for the 45 µm sieve.

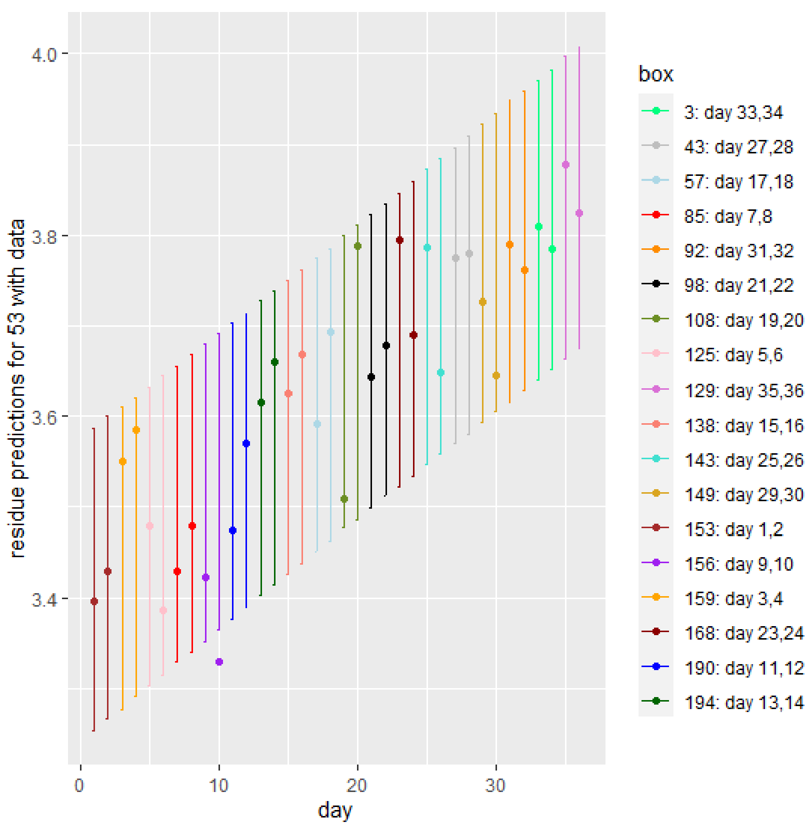

Figure 5. 95% prediction intervals with observed data for the 53 µm sieve.

The residuals from the statistical model with their 95% uncertainty interval are plotted in Figure 6. The residuals appear to be random, not systematic, and the intervals are wider for the smaller sieve sizes as could be anticipated.

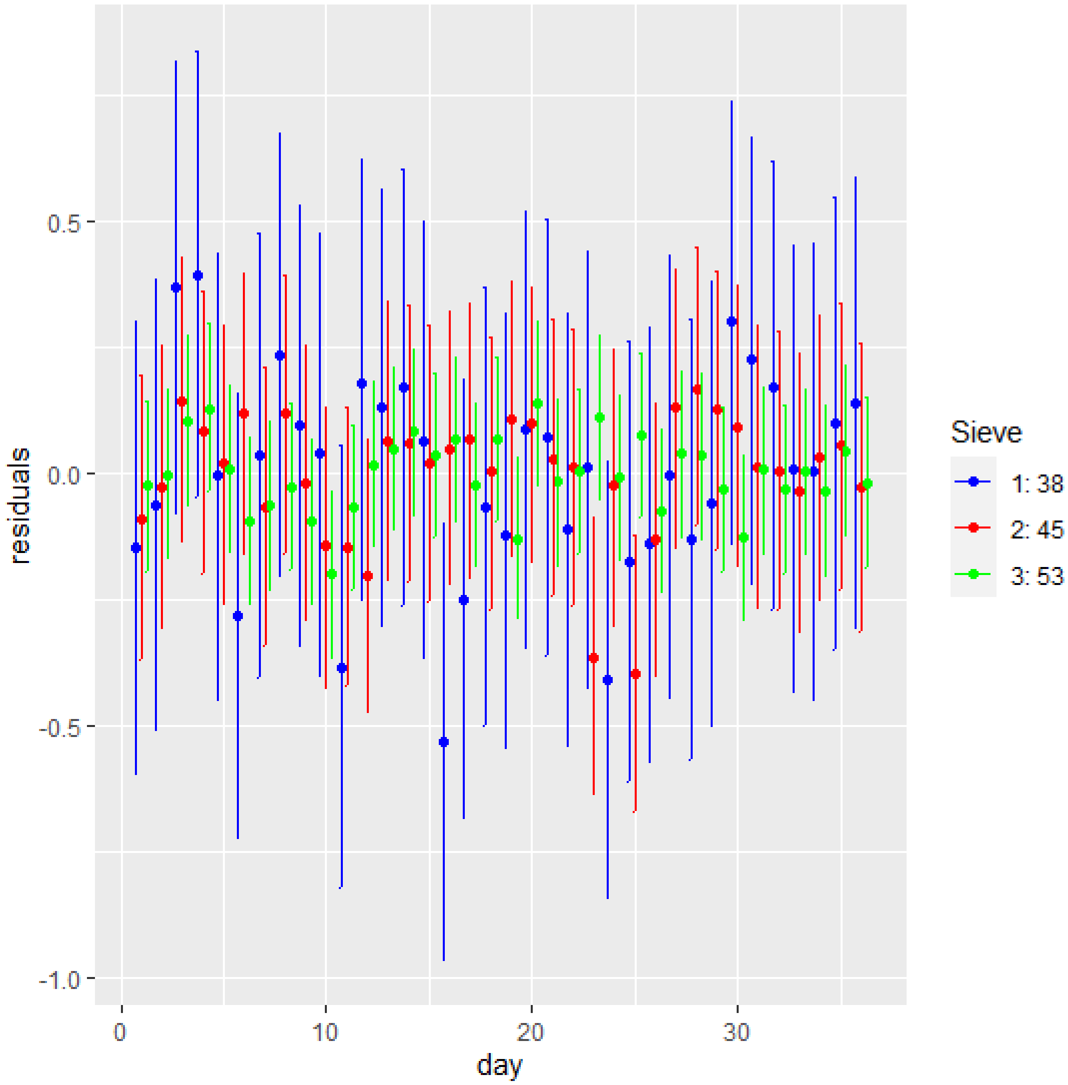

Figure 6. Residuals with 95% uncertainty intervals.

The estimate of the residue fraction for 45 µm sieve can be estimated as

$$\mu 45 = \beta_1 + \beta_2\,(45 - \overline{\mu.s}) + \beta_3(45 - \overline{\mu.s})^2.$$

This is equal to 5.97 % with standard uncertainty of 0.24 %, and with 95% uncertainty interval of (5.44%, 6.39%).

3. Blaine Test Results

Each lab received a single vial of SRM 46h and two vials of SRM 114r for analysis. They were instructed to run the 46h as a calibration and to provide air flow times; the calculated Blaine surface area was performed at NIST using their air flow times. Using SRM 46h for calibration provided for consistency across labs. For the candidate SRM 114r, each lab was requested to run their analysis in duplicate, with the test result being the mean of the four measurements. This requires the sample be removed and broken up to form a powder. ASTM C 204 cautions against comparing cements that are too different regards density and fineness. As mentioned previously, SRM 46h and candidate 114r are just within the limits for density and fall well within the limits for fineness (+- 200) so these cements may be compared.

3.1. The Data

The Blaine measurements were obtained as described in ASTM C 204 [2]. There were 42 different boxes, each sampled 2 times and each sample measured twice. Table 5 gives the measurements by box and a plot of test results is shown in Figure 7.

Table 5. Blaine measurements in m^2/kg.

Sample Box	**Blaine 114r #1**		**Blaine 114r #2**	
replicate	**1**	**2**	**1**	**2**
8	387.6	385.5	384.8	383.1
15	395.4	386.8	386.8	391.1
17	395.5	395.4	389.2	389.0
21	390.1	391.9	390.3	393.3
22	398.0	396.8	394.2	393.0
26	391.5	390.7	391.5	391.1
35	366.6	367.3	370.1	371.1
36	378.8	381.1	381.1	375.1
40	397.3	397.3	397.3	395.2
44	400.4	411.9	417.5	417.5
49	389.2	390.7	394.3	395.9
58	394.9	390.9	395.4	391.4
61	395.9	392.9	391.5	392.1
70	392.3	392.7	392.7	391.3
73	392.7	397.1	393.5	395.6
78	374.8	363.5	405.2	415.3
87	398.5	399.0	392.4	394.3
88	371.8	374.8	368.8	375.4
90	398.2	395.1	395.1	399.2
101	394.1	389.6	394.5	390.0
107	398.3	395.7	394.6	396.1
110	396.4	398.5	396.3	399.0
115	391.4	392.4	391.2	392.0
122	391.7	393.2	389.4	389.5
124	386.6	386.4	387.5	387.6
131	385.3	385.7	395.7	395.8

145	402.4	404.6	400.1	402.4
148	396.8	396.2	390.9	391.7
152	396.9	396.9	394.9	395.9
157	388.9	390.1	390.6	391.8
160	396.4	394.6	395.6	396.1
166	400.4	398.3	398.3	398.3
171	398.6	396.9	396.8	395.4
173	396.2	397.4	397.6	399.0
174	391.1	390.7	391.0	390.6
175	393.6	396.0	388.9	391.3
176	389.6	389.6	387.3	389.6
177	403.1	398.8	396.4	396.9
178	394.3	394.9	393.6	394.9
196	377.4	377.2	377.7	377.2
200	394.8	396.8	395.8	396.8

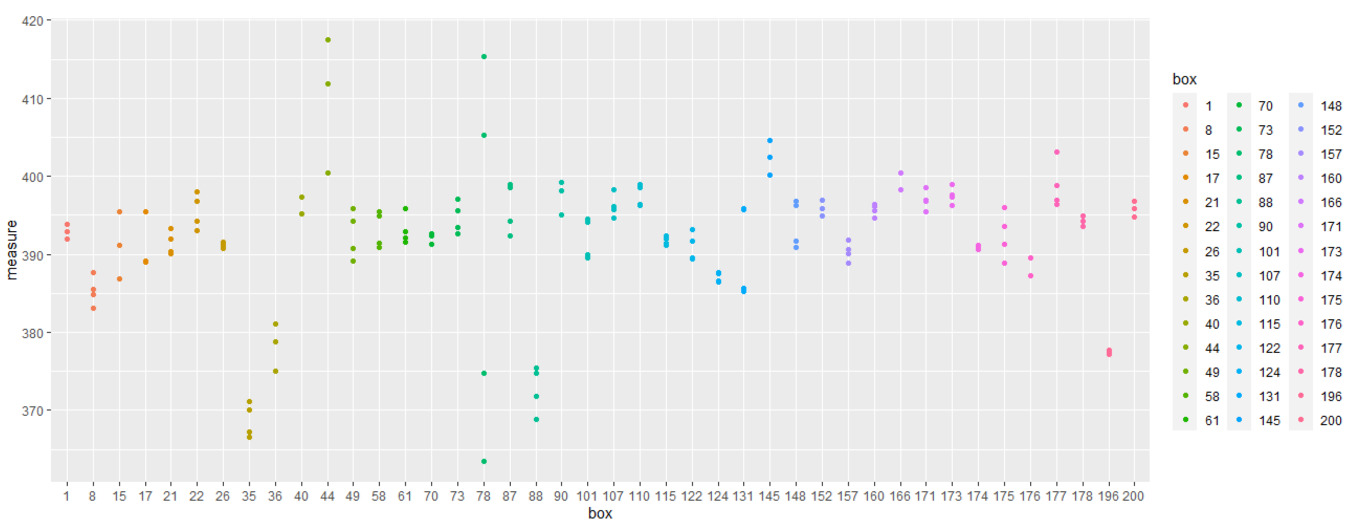

Figure 7. Blaine measurements in m^2/kg by lab.

3.2. Analysis

Figure 7 shows that the data may be somewhat inhomogeneous. A formal test of between box heterogeneity called Cochran's Q is provided as part of the NIST Decision Tree [11]. It indicates heterogeneity with p-value of < 0.001. A further test of symmetry (passed) and of Gaussian distribution (rejected with p-value <0.001), both included in the NIST Decision Tree, indicate that a random effects model which includes between box heterogeneity using the Laplace probability distribution is the best choice to obtain a reference value and its uncertainty. The statistical model is as follows:

$y_{ijk} \sim N(\gamma_i, \sigma_i^2)$, is the measurement where i denotes box ($i = 1, \ldots, 42$), j denotes sample (1, 2), k denotes replicate (1, 2),

$\gamma_i \sim Laplace(\mu, \tau)$, γ_i is the mean value of box i, μ is the consensus value, τ is the scale parameter of the Laplace distribution.

The estimation of the parameters of this statistical model is accomplished using Bayesian MCMC implemented in OpenBUGS [12, 13]. Table 6 gives the estimates of the box means γ_i with their standard uncertainties and 95% uncertainty intervals.

The estimated μ (reference value) is 393.2 m^2/kg with standard uncertainty of 0.9 m^2/kg and 95% uncertainty interval of (391.5, 394.9). It may also be of interest, since there is heterogeneity among the boxes, to include a prediction interval for the value of a randomly selected box. This is (383.3, 403.3).

Figure 8 shows the estimated box means with 95% uncertainty intervals, the consensus (reference) value with its 95% uncertainty interval in green, and the 95% prediction interval in red.

Table 6. Estimated box means with uncertainties for SRM 114r.

number	box	Mean m^2/kg	Standard uncertainty	l95	u95
1	88	373.5	1.6	371.0	377.0
2	70	392.3	0.3	391.7	392.8
3	73	394.7	0.9	392.9	396.4
4	173	397.5	0.5	396.4	398.6
5	148	393.8	1.3	391.2	396.4
6	171	396.9	0.6	395.7	398.0
7	110	397.4	1.1	395.1	399.6
8	122	391.0	0.8	389.4	392.5
9	58	393.1	1.3	390.5	395.7
10	15	390.2	1.7	386.9	393.6
11	200	395.9	1.2	393.5	398.2
12	176	389.0	0.5	388.0	390.0
13	21	391.4	0.8	389.8	393.0
14	124	387.0	0.3	386.4	387.7
15	131	390.8	2.3	386.2	395.4
16	61	393.1	1.0	391.1	395.0
17	107	396.1	1.0	394.1	398.0
18	8	385.4	0.9	383.7	387.1
19	26	391.2	0.4	390.5	391.9
20	177	398.6	1.3	395.8	401.0
21	196	377.4	0.3	376.8	378.0
22	40	396.8	0.5	395.7	397.8
23	90	396.8	1.1	394.6	398.9
24	162	396.1	0.5	395.2	397.1
25	35	369.3	1.2	367.4	371.7
26	160	395.7	0.4	394.9	396.4
27	36	379.5	1.3	377.2	382.3
28	44	407.3	4.3	397.1	414.1
29	166	398.8	0.6	397.6	399.9
30	101	392.1	1.1	389.8	394.3
31	115	391.8	0.2	391.3	392.2
32	174	390.9	0.1	390.6	391.1
33	17	392.3	1.9	388.5	396.1
34	22	395.4	1.1	393.3	397.5
35	145	402.3	0.8	400.6	403.8
36	87	395.9	1.4	393.1	398.5
37	78	391.1	5.6	380.4	402.2

38	157	390.4	0.5	389.3	391.4
39	49	392.5	1.3	389.8	395.1
40	178	394.4	0.3	393.8	395.1
41	175	392.4	1.5	389.4	395.4
42		393.1	0.8	391.6	394.7

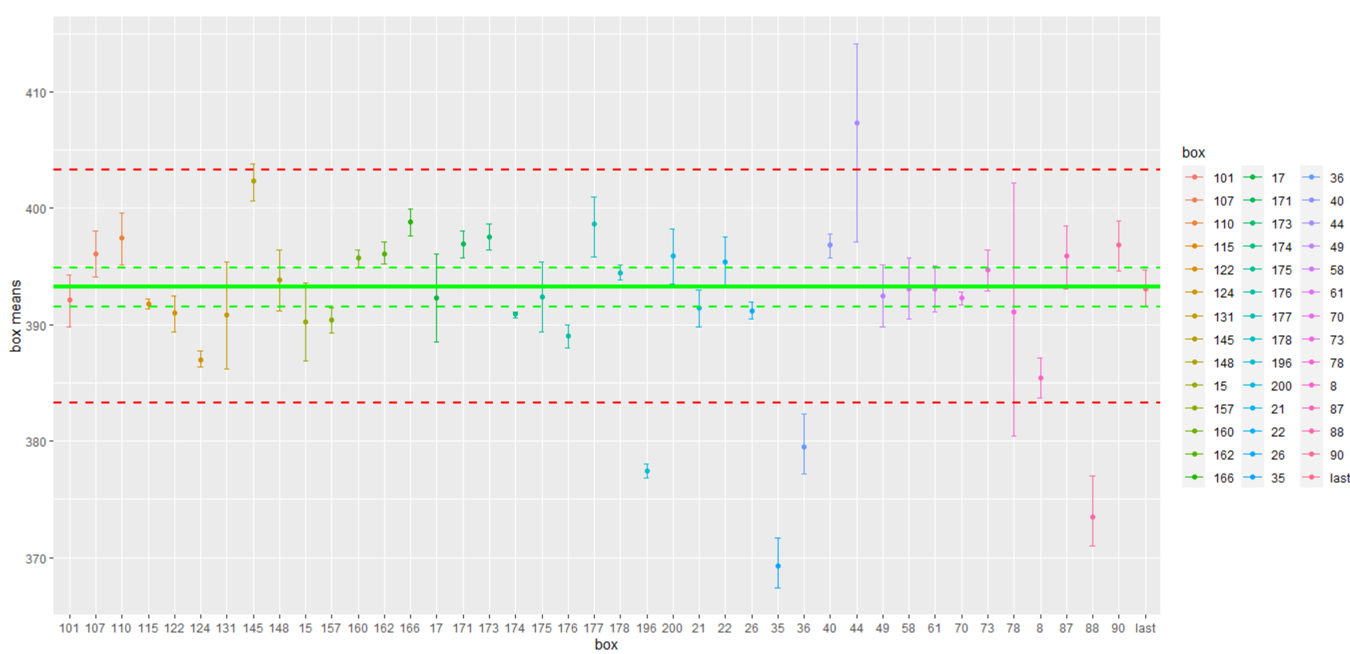

Figure 8. Estimated box means with 95% uncertainty intervals, reference value with 95% uncertainty in green, 95% prediction interval in red.

4. Particle Size Distribution

Informational values are provided for particle size distribution measured by laser diffraction. The values are means of the combined wet and dry PSD measurements as only nine data sets were returned. Two of these were deemed outliers as the fell distinctly below the cluster of results from other participants and represent the wet and dry data contribution from one participant. The black line represents the mean of the measurements, with the values for each size interval provided in Table 7.

4.1. The data

To determine the particle size distribution (PSD), laser diffraction measurements were performed on samples of the material either as dry powder or in liquid suspension. Table 7 gives the cumulative percent by bin size. The data in Table 7 can be represented by a bar graph [Fig. 9] with separate bars for dry and wet method.

Table 7. Cumulative % by bin size in microns for wet and dry measurements

Bin size in microns	Sample 1 wet	Sample 2 wet	Sample 3 wet	Sample 4 wet	Sample 1 dry	Sample 2 dry	Sample 3 dry	Sample 5 dry	Sample 7 dry
1	3.32	7.13	3.68	2.25	4.33	9.78	7.3	6.02	7.13
1.5	4.92	11.41	5.74	4.42	5.83	13.26	12.56	8.94	12.21
2	6.12	15.26	7.71	6.33	7.73	15.94	16.32	11.79	16.03
3	10.18	22.12	12.08	11.06	10.95	20.76	21.76	17.22	21.84
4	13.78	28.02	16.78	16.28	13.38	25.02	25.83	22.11	26.28
6	19.38	37.5	25.96	25.69	17.2	31.12	32.84	30.21	33.56
8	24.65	44.64	34.2	33.18	20.58	34.78	39.03	36.54	39.61
12	34.09	54.88	47.65	44.69	27.64	40.56	49.67	46.36	49.74
16	41.9	62.88	58.01	53.95	34.23	47.32	58.54	54.4	58.19
24	55.2	76.01	72.91	68.74	46.74	62.53	72.63	67.83	71.79
32	66.92	85.19	82.75	79.41	57.25	76.13	82.69	78.18	81.74
48	85.36	94	93.4	91.67	75.36	93.05	94.31	90.72	93.43
64	92.68	97.18	97.72	96.93	85.09	98.87	98.62	96.31	98.1
96	96.16	99.42	99.87	99.67	90.66	100	99.97	99.55	100
128	97.41	99.93	100	99.89	92.48	100	100	100	100

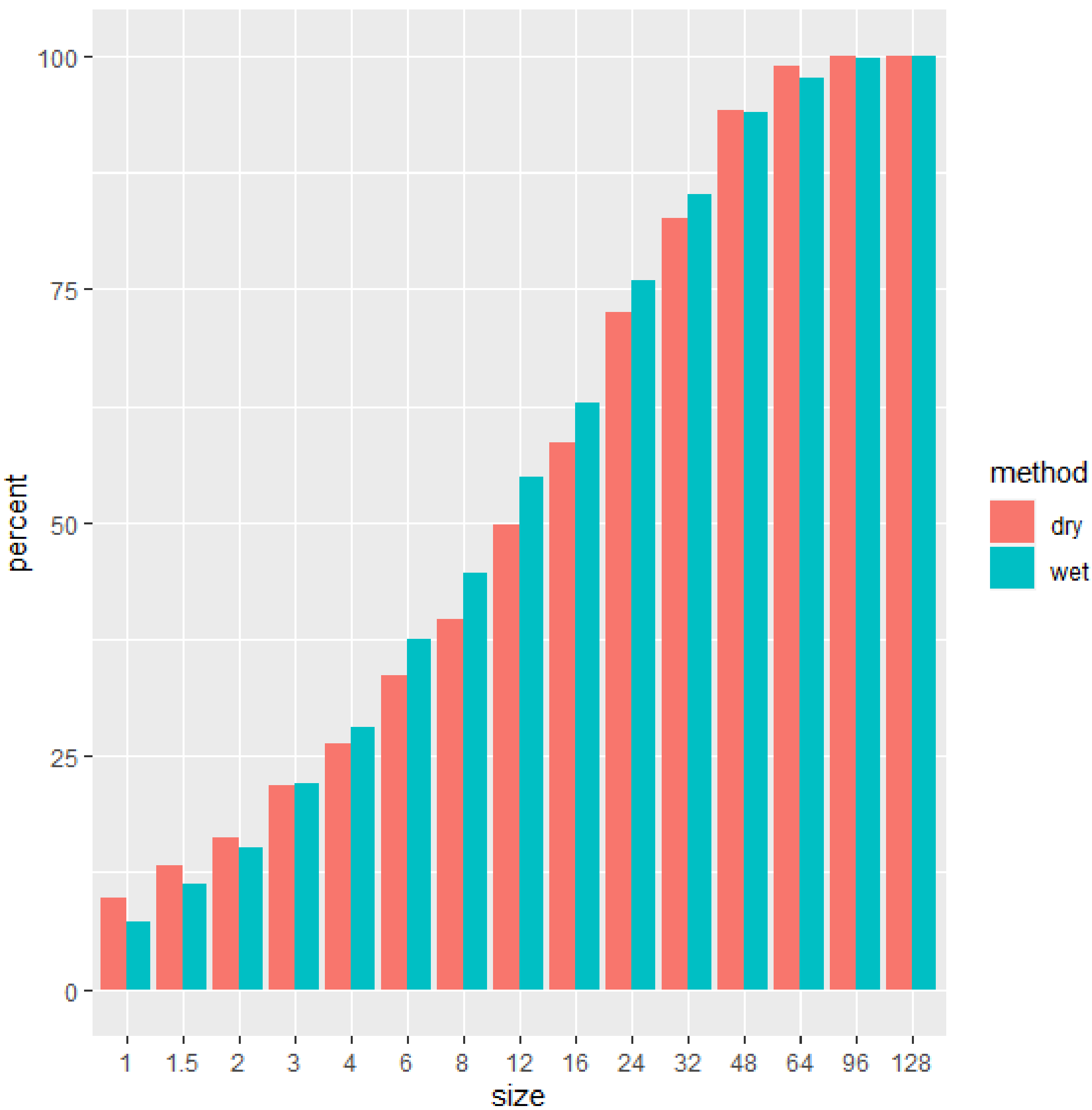

Figure 9. Bar graph of the cumulative frequencies given in Table 7 separated by method.

4.2. Statistical Analysis

Data in Table 7 can be combined using a Bayesian hierarchical model (estimated using Markov Chain Monte Carlo in OpenBUGS [13]) to obtain a consensus cumulative distribution table with values, their standard uncertainty, and 95% uncertainty interval per each bin. The results are given in Table 8. Consensus values with uncertainty for the cumulative particle size distribution are presented in Table 8 and are displayed as a graph in Figure 10.

Table 8. Consensus values with uncertainty for the cumulative particle size distribution. The results in Table 8 are shown in Figure 10.

Bin size in microns	Consensus mean of cumulative %	Standard uncertainty of cumulative %	L95 in %	U95 in %
1	5.97	1.12	3.59	7.92
1.5	9.02	1.27	6.41	11.32
2	11.82	1.32	9.06	14.26
3	16.82	1.37	13.97	19.36
4	21.18	1.43	18.25	23.88
6	28.42	1.66	25.12	31.54
8	34.49	1.83	30.94	38.04
12	44.54	2.10	40.42	48.6
16	52.74	2.16	48.55	56.9
24	66.78	2.18	62.52	70.97
32	77.51	2.24	73.11	81.89
48	91.29	2.74	85.94	96.66
64	96.84	2.87	91.23	100
96	99.54	2.91	93.88	100
128	100	2.92	94.42	100

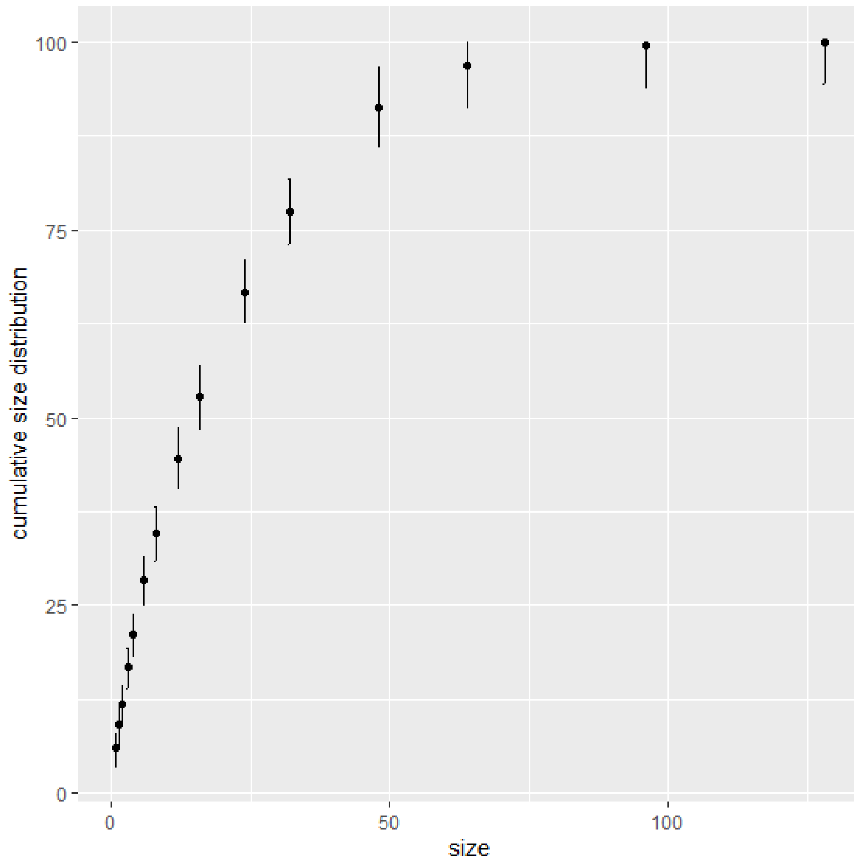

Figure 10. Estimated cumulative particle size distribution with 95% uncertainty bounds.

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Participants

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Appendix A: Wet Sieve Test Results

Sieve Test Results by box and vial showing mass of cement and mass retained on each of three calibrated sieves.

		38 um		45 um		53 um	
Box	Vial	Mass	Mass Retained	Mass	Mass Retained	Mass	Mass Retained
153	1	1.00018	0.09701	1.00044	0.05893	1.00061	0.03399
153	2	1.00062	0.09818	1.00033	0.05981	1.00084	0.03433
159	1	1.00010	0.10269	1.00072	0.06182	1.00089	0.03553
159	2	1.00045	0.10329	1.00084	0.06147	1.00045	0.03588
125	1	1.00095	0.09960	1.00040	0.06108	1.00061	0.03481
125	2	1.00065	0.09713	1.00067	0.06240	1.00083	0.03390
85	1	1.00028	0.10057	1.00033	0.06079	1.00021	0.03431
85	2	1.00015	0.10280	1.00086	0.06294	1.00011	0.03479
156	1	1.00086	0.10180	1.00043	0.06180	1.00052	0.03424
156	2	1.00078	0.10148	1.00041	0.06082	1.00022	0.03331
190	1	1.00090	0.09756	1.00096	0.06112	1.00083	0.03478
190	2	1.00024	0.10341	1.00030	0.06079	1.00090	0.03574
194	1	1.00036	0.10321	1.00045	0.06372	1.00085	0.03619
194	2	1.00085	0.10396	1.00024	0.06394	1.00050	0.03663
138	1	1.00004	0.10306	1.00014	0.06381	1.00094	0.03628
138	2	1.00087	0.09748	1.00025	0.06439	1.00082	0.03671
57	1	1.00027	0.10054	1.00090	0.06487	1.00046	0.03594
57	2	1.00076	0.10272	1.00011	0.06448	1.00022	0.03694
108	1	1.00055	0.10242	1.00040	0.06581	1.00016	0.03510
108	2	1.00093	0.10483	1.00031	0.06597	1.00043	0.03790
98	1	1.00040	0.10487	1.00063	0.06559	1.00066	0.03646
98	2	1.00064	0.10336	1.00080	0.06570	1.00078	0.03681
168	1	1.00088	0.10492	1.00082	0.06221	1.00015	0.03796
168	2	1.00016	0.10093	1.00024	0.06583	1.00032	0.03691
143	1	1.00091	0.10363	1.00088	0.06241	1.00048	0.03789
143	2	1.00038	0.10421	1.00023	0.06532	1.00021	0.03649
43	1	1.00124	0.10594	1.00001	0.06818	1.00015	0.03775
43	2	1.00051	0.10491	1.00080	0.06891	1.00013	0.03780
149	1	1.00083	0.10591	1.00008	0.06873	1.00080	0.03730
149	2	1.00011	0.10971	1.00004	0.06866	1.00006	0.03645

92	1	1.00072	0.10934	1.00015	0.06814	1.00076	0.03792
92	2	1.00128	0.10911	1.00065	0.06834	1.00026	0.03763
3	1	1.00021	0.10766	1.00029	0.06818	1.00005	0.03810
3	2	1.00017	0.10791	1.00063	0.06916	1.00071	0.03787
129	1	1.00061	0.10918	1.00079	0.06966	1.00010	0.03877
129	2	1.00011	0.10981	1.00098	0.06913	1.00067	0.03827

. +45 Sieve results for 38 um, 45um and 53 um sieves

Appendix B. Blaine fineness test results.

Sample Box	**Blaine 114r #1**		**Blaine 114r #2**	
replicate	**1**	**2**	**1**	**2**
8	388	386	385	383
15	395	387	387	391
17	396	395	389	389
21	390	392	390	393
22	398	397	394	393
26	391	391	391	391
35	367	367	370	371
36	379	381	381	375
40	397	397	397	395
44	400	412	418	418
49	389	391	394	396
58	395	391	395	391
61	396	393	392	392
70	392	393	393	391
73	393	397	393	396
78	375	364	405	415
87	399	399	392	394
88	372	375	369	375
90	398	395	395	399
101	394	390	394	390
107	398	396	395	396
110	396	399	396	399
115	391	392	391	392
122	392	393	389	390
124	387	386	387	388
131	385	386	396	396
145	402	405	400	402
148	397	396	391	392
152	397	397	395	396
157	389	390	391	392
160	396	395	396	396
166	400	398	398	398
171	399	397	397	395

173	396	397	398	399
174	391	391	391	391
175	394	396	389	391
176	390	390	387	390
177	403	399	396	397
178	394	395	394	395
196	377	377	378	377
200	395	397	396	397

Appendix C: OpenBUGS code for the sieve residue analysis

```
{
for(i in 1:3)
{
pr.cv.s[i] <- 1/(uc.cv.s[i]*uc.cv.s[i])
mu.s[i]~dnorm(cv.s[i],pr.cv.s[i])
mu.r[i] <- b[1]+b[2]*(mu.s[i]-45.237)+b[3]*(mu.s[i]-45.237)*(mu.s[i]-45.237)

xiNs[i] ~ dnorm(0, 0.0016)I(0.001,)
chSqNs[i] ~ dgamma(0.5,0.5)
tau.wv[i] <- xiNs[i]/sqrt(chSqNs[i])
itau.wv[i]<-1/sqrt(tau.wv[i])
}

b[1]~dunif(0,20)
b[2]~dunif(-5,0)
b[3]~dunif(-1,1)
b[4]~dunif(-1,1)
b[5]~dunif(-1,1)
b[6]~dunif(-1,1)
for(i in 1:36)
{
y38[i]~dt(mu38[i],tau.wv[1],df[1])
y45[i]~dt(mu45[i],tau.wv[2],df[2])
y53[i]~dt(mu53[i],tau.wv[3],df[3])
mu38[i] <- mu.r[1]+b[4]*(i-1)
mu45[i] <- mu.r[2]+b[5]*(i-1)
mu53[i] <- mu.r[3]+b[6]*(i-1)
p.y38[i]~dt(mu38[i],tau.wv[1],df[1])
p.y45[i]~dt(mu45[i],tau.wv[2],df[2])
p.y53[i]~dt(mu53[i],tau.wv[3],df[3])
res[i] <- y38[i]-p.y38[i]
res[i+36] <- y45[i]-p.y45[i]
res[i+72] <- y53[i]-p.y53[i]
pv[i] <- step(res[i])
pv[i+36] <- step(res[i+36])
pv[i+72] <- step(res[i+72])
}
```

```
mr45 <- b[1]+b[2]*(45-45.237)+b[3]*(45-45.237)*(45-45.237)
}
```

Data:

```
list(cv.s=c(38.12,43.47,54.12),uc.cv.s=c(0.59,0.59,0.59),df=c(200,200,200))
```

Box[]	Vial[]	y38[]	y45[]	y53[]	seq[]
153	1	9.699	5.89	3.397	1
153	2	9.812	5.979	3.43	2
159	1	10.268	6.178	3.55	3
159	2	10.324	6.142	3.586	4
125	1	9.951	6.106	3.479	5
125	2	9.707	6.236	3.387	6
85	1	10.054	6.077	3.43	7
85	2	10.278	6.289	3.479	8
156	1	10.171	6.177	3.422	9
156	2	10.14	6.08	3.33	10
190	1	9.747	6.106	3.475	11
190	2	10.339	6.077	3.571	12
194	1	10.317	6.369	3.616	13
194	2	10.387	6.392	3.661	14
138	1	10.306	6.38	3.625	15
138	2	9.74	6.437	3.668	16
57	1	10.051	6.481	3.592	17
57	2	10.264	6.447	3.693	18
108	1	10.236	6.578	3.509	19
108	2	10.473	6.595	3.788	20
98	1	10.483	6.555	3.644	21
98	2	10.329	6.565	3.678	22
168	1	10.483	6.216	3.795	23
168	2	10.091	6.581	3.69	24
143	1	10.354	6.236	3.787	25
143	2	10.417	6.53	3.648	26
43	1	10.581	6.818	3.774	27
43	2	10.486	6.885	3.78	28
149	1	10.582	6.872	3.727	29
149	2	10.97	6.866	3.645	30
92	1	10.926	6.813	3.789	31
92	2	10.897	6.83	3.762	32
3	1	10.764	6.816	3.81	33
3	2	10.789	6.912	3.784	34
129	1	10.911	6.961	3.877	35
129	2	10.98	6.906	3.824	36

END

Appendix D. Consensus Means and Uncertainty Calculations

SRM 114

Consensus Means Analysis

(Full Sample Case)

Data Summary:
Response Variable: Y
Lab-ID Variable: IX
Number of Observations: 164
Grand Mean: 0.3921951E+03
Grand Standard Deviation: 0.8397705E+01
Total Number of Labs: 41
Minimum Lab Mean: 0.3687750E+03
Maximum Lab Mean: 0.4118250E+03
Minimum Lab SD: 0.2362908E+00
Maximum Lab SD: 0.2452251E+02
Mean of Lab Means: 0.3921951E+03
SD of Lab Means: 0.7464031E+01
SD of Lab Means (wrt to grand mean): 0.7464031E+01
Within Lab (pooled) SD: 0.4580903E+01
Within Lab (pooled) Variance: 0.2098467E+02

Table 1: Summary Statistics by Lab

Lab ID	n(i)	Mean	Standard Variance	Standard Deviation	Deviation of the Mean
1	4	0.3852500E+03	0.3470000E+01	0.1862794E+01	0.9313968E+00
2	4	0.3900250E+03	0.1694917E+02	0.4116937E+01	0.2058468E+01
3	4	0.3922750E+03	0.1344917E+02	0.3667311E+01	0.1833655E+01
4	4	0.3914000E+03	0.2253333E+01	0.1501111E+01	0.7505553E+00
5	4	0.3955000E+03	0.5293333E+01	0.2300725E+01	0.1150362E+01
6	4	0.3912000E+03	0.1466667E+00	0.3829708E+00	0.1914854E+00
7	4	0.3687750E+03	0.4689167E+01	0.2165448E+01	0.1082724E+01
8	4	0.3790250E+03	0.8022500E+01	0.2832402E+01	0.1416201E+01
9	4	0.3967750E+03	0.1102500E+01	0.1050000E+01	0.5250000E+00
10	4	0.4118250E+03	0.6498250E+02	0.8061172E+01	0.4030586E+01
11	4	0.3925250E+03	0.9642500E+01	0.3105238E+01	0.1552619E+01
12	4	0.3931500E+03	0.5416667E+01	0.2327373E+01	0.1163687E+01

13	4	0.3931000E+03	0.3813333E+01	0.1952776E+01	0.9763879E+00
14	4	0.3922500E+03	0.4366667E+00	0.6608076E+00	0.3304038E+00
15	4	0.3947250E+03	0.4002500E+01	0.2000625E+01	0.1000312E+01
16	4	0.3897000E+03	0.6013533E+03	0.2452251E+02	0.1226125E+02
17	4	0.3960500E+03	0.1036333E+02	0.3219213E+01	0.1609607E+01
18	4	0.3727000E+03	0.9240000E+01	0.3039737E+01	0.1519868E+01
19	4	0.3969000E+03	0.4486667E+01	0.2118175E+01	0.1059088E+01
20	4	0.3920500E+03	0.6803333E+01	0.2608320E+01	0.1304160E+01
21	4	0.3961750E+03	0.2409167E+01	0.1552149E+01	0.7760745E+00
22	4	0.3975500E+03	0.1963333E+01	0.1401190E+01	0.7005950E+00
23	4	0.3917500E+03	0.3033333E+00	0.5507571E+00	0.2753785E+00
24	4	0.3909500E+03	0.3376667E+01	0.1837571E+01	0.9187854E+00
25	4	0.3870250E+03	0.3758333E+00	0.6130525E+00	0.3065262E+00
26	4	0.3906250E+03	0.3504917E+02	0.5920234E+01	0.2960117E+01
27	4	0.4023750E+03	0.3375833E+01	0.1837344E+01	0.9186720E+00
28	4	0.3939000E+03	0.9180000E+01	0.3029851E+01	0.1514926E+01
29	4	0.3961500E+03	0.9166667E+00	0.9574271E+00	0.4787136E+00
30	4	0.3903500E+03	0.1443333E+01	0.1201388E+01	0.6006940E+00
31	4	0.3956750E+03	0.6225000E+00	0.7889867E+00	0.3944933E+00
32	4	0.3988250E+03	0.1102500E+01	0.1050000E+01	0.5250000E+00
33	4	0.3969250E+03	0.1715833E+01	0.1309898E+01	0.6549491E+00
34	4	0.3975500E+03	0.1316667E+01	0.1147461E+01	0.5737305E+00
35	4	0.3908500E+03	0.5666667E-01	0.2380476E+00	0.1190238E+00
36	4	0.3924500E+03	0.9283333E+01	0.3046856E+01	0.1523428E+01
37	4	0.3890250E+03	0.1322500E+01	0.1150000E+01	0.5750000E+00
38	4	0.3988000E+03	0.9286667E+01	0.3047403E+01	0.1523702E+01
39	4	0.3944250E+03	0.3825000E+00	0.6184658E+00	0.3092329E+00
40	4	0.3773750E+03	0.5583333E-01	0.2362908E+00	0.1181454E+00
41	4	0.3960500E+03	0.9166667E+00	0.9574271E+00	0.4787136E+00

1. Method: Mandel-Paule

Estimate of (unscaled) Consensus Mean:	0.3921526E+03
Estimate of (scaled) Consensus Mean:	0.5430326E+00
Between Lab Variance (unscaled):	0.5223863E+02
Between Lab SD (unscaled):	0.7227630E+01
Between Lab Variance (scaled):	0.2818678E-01
Standard Deviation of Consensus Mean:	0.1135894E+01
Standard Uncertainty (k = 1):	0.1135894E+01
Expanded Uncertainty (k = 2):	0.2271788E+01
Expanded Uncertainty (k = 1.9599640):	0.2226311E+01
Normal PPF of 0.975:	0.1959964E+01
Lower 95% (normal) Confidence Limit:	0.3899262E+03
Upper 95% (normal) Confidence Limit:	0.3943789E+03

Note: Mandel-Paule Best Usage:
6 or More Labs:

3. Method: Vangel-Rukhin Maximum Likelihood

Estimate of (unscaled) Consensus Mean:	0.3920690E+03
Estimate of (scaled) Consensus Mean:	0.5410923E+00
Between Lab Variance (unscaled):	0.4807536E+02
Between Lab SD (unscaled):	0.6933640E+01
Between Lab Variance (scaled):	0.2594037E-01

Standard Deviation of Consensus Mean:	0.1111312E+01
Standard Uncertainty (k = 1):	0.1111312E+01
Expanded Uncertainty (k = 2):	0.2222624E+01
Expanded Uncertainty (k = 1.9599640):	0.2178131E+01
Normal PPF of 0.975:	0.1959964E+01
Lower 95% (normal) Confidence Limit:	0.3898909E+03
Upper 95% (normal) Confidence Limit:	0.3942472E+03

Note: Vangel-Rukhin Maximum Likelihood
Best Usage: 6 or More Labs

4a. Method: DerSimonian Laird (original variance)

Estimate of Consensus Mean:	0.3921593E+03
Estimate of Variance of Consensus Mean:	0.1518269E+01
Estimate of Between Lab Variance:	0.5966491E+02
Standard Uncertainty (k = 1):	0.1232181E+01
Expanded Uncertainty (k = 2):	0.2464361E+01
Degrees of Freedom:	40
t Percent Point Value:	0.2021075E+01
Lower 95% (t-value) Confidence Limit:	0.3896689E+03
Upper 95% (t-value) Confidence Limit:	0.3946496E+03

Note: DerSimonian-Laird Best Usage:
Any Number of Labs:

4b. Method: DerSimonian Laird - Horn-Horn-Duncan Variance

Estimate of Consensus Mean:	0.3921593E+03
Estimate of Variance of Consensus Mean:	0.1328099E+01
Estimate of Between Lab Variance:	0.5966491E+02
Standard Uncertainty (k = 1):	0.1152432E+01
Expanded Uncertainty (k = 2):	0.2304863E+01
Degrees of Freedom:	40
t Percent Point Value:	0.2021075E+01
Lower 95% (t-value) Confidence Limit:	0.3898301E+03
Upper 95% (t-value) Confidence Limit:	0.3944884E+03

Note: DerSimonian-Laird Best Usage:
Any Number of Labs:

4d. Method: DerSimonian Laird - Bootstrap Variance

Number of Bootstrap Samples	100000
Estimate of Consensus Mean:	0.3921593E+03
Estimate of Variance of Consensus Mean:	0.1522168E+01
Standard Uncertainty (k = 1):	0.1233762E+01
Expanded Uncertainty (k = 2):	0.2467524E+01
Lower 95% (percentile bootstrap) Confidence Limit:	0.3897386E+03
Upper 95% (percentile bootstrap) Confidence Limit:	0.3945844E+03
Lower 95% (symmetric bootstrap) Confidence Limit:	0.3897341E+03
Upper 95% (symmetric bootstrap) Confidence Limit:	0.3945844E+03
K (symmetric bootstrap) Coverage Factor:	0.1965654E+01
Lower 95% (kernel bootstrap) Confidence Limit:	0.3897266E+03
Upper 95% (kernel bootstrap) Confidence Limit:	0.3945951E+03
K (kernel bootstrap) Coverage Factor:	0.1974298E+01

Note: DerSimonian-Laird Best Usage:
Any Number of Labs:

Table 2: 95% Confidence Limits

Method	Consensus Mean	Lower Limit	Upper Limit	Uncertainty (k*SE)
1. Mandel-Paule	0.3921526E+03	0.3899262E+03	0.3943789E+03	0.2226311E+01
3a. Vangel-Rukhin ML	0.3920690E+03	0.3898909E+03	0.3942472E+03	0.2178131E+01
4a. DerSimonian-Laird (original)	0.3921593E+03	0.3896689E+03	0.3946496E+03	0.2490330E+01
4b. DerSimonian-Laird (H-H-D)	0.3921593E+03	0.3898301E+03	0.3944884E+03	0.2329151E+01
4d. DerSimonian-Laird (perc. bootstrap)	0.3921593E+03	0.3897386E+03	0.3945844E+03	0.2425148E+01
4d. DerSimonian-Laird (symm. bootstrap)	0.3921593E+03	0.3897341E+03	0.3945844E+03	0.2425148E+01
4d. DerSimonian-Laird (kern bootstrap)	0.3921593E+03	0.3897266E+03	0.3945951E+03	0.2435813E+01

Table 3: Standard Uncertainties (k = 1)

Method	Standard Consensus Mean	Relative Uncertainty (k = 1)	Standard Uncertainty (%)
1. Mandel-Paule	0.3921526E+03	0.1135894E+01	0.2896562E+00
3a. Vangel-Rukhin ML	0.3920690E+03	0.1111312E+01	0.2834480E+00
4a. DerSimonian-Laird (original)	0.3921593E+03	0.1232181E+01	0.3142042E+00
4b. DerSimonian-Laird (H-H-D)	0.3921593E+03	0.1152432E+01	0.2938683E+00
4d. DerSimonian-Laird (bootstrap)	0.3921593E+03	0.1233762E+01	0.3146073E+00

Table 4: Expanded Uncertainties (k = 2)

Method	Consensus Mean	Expanded Uncertainty (k = 2)	Relative Expanded Uncertainty (%)
1. Mandel-Paule	0.3921526E+03	0.2271788E+01	0.5793123E+00
3a. Vangel-Rukhin ML	0.3920690E+03	0.2222624E+01	0.5668960E+00
4a. DerSimonian-Laird (original)	0.3921593E+03	0.2464361E+01	0.6284083E+00
4b. DerSimonian-Laird (H-H-D)	0.3921593E+03	0.2304863E+01	0.5877366E+00
4d. DerSimonian-Laird (bootstrap)	0.3921593E+03	0.2467524E+01	0.6292147E+00

Appendix E. Dataplot code for consensus means

```
. Name:     blaine.dp
. Purpose:  Run a consensus means analysis for blaine.dat
.
. Step 0:   Define the output devices
.
device 2 close
let string fplot = blaine.ps
set ipl1na ^fplot
call checksys.dp
.
. Step 1:   Read the data
.
skip 25
read blaine.dat x y1 y2 y3 y4
skip 0
.
let x = sortc x y1 y2 y3 y4
let nlab = size x
loop for k = 1 1 nlab
   let aval = x(k)
   let string s^k = ^aval
end of loop
.
let ix = code x
let ix = combine ix ix ix ix
let y = combine y1 y2 y3 y4
.
. Step 2:  Initializations for consensus means analysis
.
set write decimals -7
set modified mandel paule off
set vangel rukhin on
set vangel rukhin bootstrap off
set dersimonian laird minmax off
set dersimonian laird bootstrap on
set schiller eberhardt off
set mean of means off
set grand mean off
set graybill deal off
set generalized confidence interval off
set fairweather off
set bayesian consensus procedure off
set bob off
bootstrap samples 100000
set random number generator fibonacci congruential
```

```
seed 66783
.
let string sx1 = 1
let string sx2 = 2
let string sx3 = 3
let string sx4 = 4
let string sx5 = 5
let nmeth = 5
.
let string srmid = SRM 114
date
.
. Step 3:  Set plot control features
.
frame corner coordinates 15 20 90 90
line solid all
character blank all
character fill on
. character hw 1 0.75
character hw 0.5 0.375
character circle
line      blank
.
case asis
title asis
tic mark label case asis
title offset 2
.
.  Step 3b: Generate strings for lab data
.
loop for k = 1 1 nlab
    let icnt = nmeth + k
    . let ival = mod(k,2)
    . if ival = 1
       let string sx^icnt = ^s^k
    . else
    .    let string sx^icnt = sp()cr()^s^k
    . end of if
end of loop
let ntot = nmeth + nlab
.
xlimits 1 ntot
major xtic mark number ntot
minor xtic mark number 0
tic mark offset units data
xtic mark offset 0.5   0.5
```

```
x1tic mark label size 1.7
x1tic mark label direction vertical
title case asis
title offset 2
.
.  iflagm  = 1   => 95% Confidence Interval
.          = 2   => Two Standard Errors Confidence Interval
.          = 3   => One Standard Errors Confidence Interval
.
let iflagm = 2
. set consensus mean plot sorted on
if iflagm = 1
   set consensus mean plot error confidence intervals
else if iflagm = 2
   set consensus mean plot error two standard errors
else if iflagm = 3
   set consensus mean plot error one standard errors
end of if
.
feedback off
capture screen on
capture cons_114.out
capture suspend
.
capture resume
print "
"
print "SRM: ^srmid"
print "Date: ^currdate"
print " "
print " "
.
loop for l = 1 1 ntot
    let string sxnew^l = ^sx^l
end of loop
.
title ^srmid
x1tic mark label format group label
let igx = group label sxnew1 to sxnew^ntot
x1tic mark label content igx
.
consensus mean plot y ix
.
capture suspend
.
line dotted
```

```
let xcoor = nmeth + 0.5
drawdsds  xcoor 20  xcoor 90
justification center
moveds 2.5 8
if iflagm = 1
   text Consensus Method: 95% Confidence Limits
else if iflagm = 2
   text Consensus Method: Two Standard Errors
else if iflagm = 3
   text Consensus Method: One Standard Error
end of if
moveds 25 8
text Method/Laboratorycr()meansp()+/-2*sd/sqrt(n)
line blank
just right
move 98 4
height 1.5
text Date: ^currdate
.
height 1.4
justification left
move 91 88
text Method:
move 91 86
text 1: MP
move 91 84
text 2: VR
move 91 82
text 3: DSL
move 93 80
text    Original
move 91 78
text 4: DSL
move 93 76
text HHD
move 91 74
text 5: DSL
move 93 72
text Bootstrap
.
end of capture
feedback on
.
. Step 4: Device close
.
device 2 close
```

```
system ps2pdf ^fplot
.
quit
```

Appendix F Blaine Fineness Test Results formatted for Dataplot

```
Name:    blaine.dat
Source:  Paul Stutzman

Description:   Column 1:  Lab ID
               Column 2:  Vial 114r #1 Replicate 1
               Column 3:  Vial 114r #1 Replicate 2
               Column 4:  Vial 114r #2 Replicate 1
               Column 5:  Vial 114r #2 Replicate 2

X     Y1    Y2    Y3    Y4
--------------------------
8  387.6 385.5 384.8 383.1
15 395.4 386.8 386.8 391.1
17 395.5 395.4 389.2 389.0
21 390.1 391.9 390.3 393.3
22 398.0 396.8 394.2 393.0
26 391.5 390.7 391.5 391.1
35 366.6 367.3 370.1 371.1
36 378.8 381.1 381.1 375.1
40 397.3 397.3 397.3 395.2
44 400.4 411.9 417.5 417.5
49 389.2 390.7 394.3 395.9
58 394.9 390.9 395.4 391.4
61 395.9 392.9 391.5 392.1
70 392.3 392.7 392.7 391.3
73 392.7 397.1 393.5 395.6
78 374.8 363.5 405.2 415.3
87 398.5 399.0 392.4 394.3
88 371.8 374.8 368.8 375.4
90 398.2 395.1 395.1 399.2
101 394.1 389.6 394.5 390.0
107 398.3 395.7 394.6 396.1
110 396.4 398.5 396.3 399.0
115 391.4 392.4 391.2 392.0
122 391.7 393.2 389.4 389.5
124 386.6 386.4 387.5 387.6
131 385.3 385.7 395.7 395.8
145 402.4 404.6 400.1 402.4
148 396.8 396.2 390.9 391.7
```

152	396.9	396.9	394.9	395.9
157	388.9	390.1	390.6	391.8
160	396.4	394.6	395.6	396.1
166	400.4	398.3	398.3	398.3
171	398.6	396.9	396.8	395.4
173	396.2	397.4	397.6	399.0
174	391.1	390.7	391.0	390.6
175	393.6	396.0	388.9	391.3
176	389.6	389.6	387.3	389.6
177	403.1	398.8	396.4	396.9
178	394.3	394.9	393.6	394.9
196	377.4	377.2	377.7	377.2
200	394.8	396.8	395.8	396.8